

Heinz Martin

Polymers, Patents, Profits

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Polymers, Patents, Profits

A Classic Case Study for Patent Infighting

Karl Ziegler, the Team, 1953 – 1998



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75 Years
Studien- und Verwertungs-GmbH (1925–1955)
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Trustee for the Max-Planck-Institute for Coal Research

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Foreword

In recent times, an ever increasing number of success stories have emerged which focus on the patent and licensing activities of US universities, who, for example in 1997, filed a total of approximately 6000 patent applications, obtained more than 2400 issued patents, and made about 611 million US dollars in profit from roughly 7000 license agreements. This situation has now also been replicated in Germany, with these achievements placing non-industrial and particularly university-conducted research efforts at the center of research politics and public interest as a potential source for innovation. The German Research Society (DFG), the University Presidents Conference, and the Federal Ministry for Education and Research (BMBF) have all reevaluated their position on the issue of patent protection for the results obtained from publicly-funded research. Their previously rather guarded view of patents as an avenue for knowledge and technology transfer has yielded to an understanding that in order to make any research results commercially profitable, it would be essential, as a rule, to have the research results protected by either a regular or a utility design patent.

In order to enable universities to build up an infrastructure of patent rights and to commercially exploit their research results, plans are being considered to revoke the so-called professor privilege of Section 42 of the Employee Invention Law and have it superseded by a regulation which guarantees universities a share in any inventions made by a professor during the time of his employment. At the same time, any university personnel are to be guaranteed a 30% participation in the exploitation of their research results. Should these plans become a reality, then the technology transfer arms of universities would join the ranks of the patent department handling the German research of the Fraunhofer Gesellschaft, Garching Innovation GmbH of the Max-Planck-Gesellschaft zur Förderung der Wissenschaften, e.V. (Max-Planck Society for Promotion of the Sciences, r.A.), and Studiengesellschaft Kohle GmbH, who, for many years, have successfully taken care of the protection and exploitation of research results.

The ups and downs that may challenge any future exploiter of university-derived inventions are descriptively illustrated in Dr. Heinz Martin's book. Dr. Martin, a co-inventor, together with Professor Karl Ziegler, Dr. H. Breil and Dr. E. Holzkamp, of the Ziegler polyolefin catalysts, invented in 1953/1954 and was honored with the Nobel Prize in 1963. Since 1970 Martin has been Co-Director of the Studien- und

Verwertungs GmbH which was established in 1925 (known as Studiengesellschaft Kohle mbH since 1955), and in this book he recounts a story that is unique in many ways.

- It is unique in that the technology established by Professor Ziegler in the 1950s dominated the worldwide production of polypropylene and polyethylene, the materials on which the entire plastics industry is based, for a period of more than 40 years.
- Unique because Professor Ziegler was able to obtain worldwide patent protection for the basic inventions of his research team despite a difficult starting position.
- Unique because, due to the significance of his inventions and through his extraordinary negotiating skills, Professor Ziegler was able to enter into option and license agreements with such industry giants as Farbwerke Hoechst, Hercules Powder, Gulf Oil, Dow Chemical, Union Carbide or, for instance, ESSO, Du Pont, Mitsui Chemical and others, and this he managed to achieve at a very early stage in the development of the inventions, practically within 20 months of inception, that is, even before he obtained patents for his inventions in either Germany, Japan, the United States, or many other countries. In 1954 these agreements earned him and the Max-Planck-Institute for Coal Research a total of almost 19 million Deutsch Marks (compared to the Institute's then annual budget of 1.2 million Deutsch Marks) and, thus, established the financial basis for his subsequent activities centering around the protection and defense of his patent rights.
- Unique because practically all of his license partners, as well as a large number of others, later moved heaven and earth to prevent the granting of his patents, or to limit their scope, and/or to challenge their validity, and Professor Ziegler, always firmly supported by Dr. Martin's energetic assistance, never hesitated to engage in legal disputes with these industry giants and to persevere to the end.
- Unique also because after Professor Ziegler's death, Studiengesellschaft Kohle and Dr. Martin, together with their US and German attorneys, were able to successfully exhaust the special remedies available under US patent law, but only as a result of their extraordinary perseverance up to the end. The situation was only resolved after 45 years, when in 1999 a final settlement of 1.65 million US dollars was agreed with the Formosa Plastics Corp. of Texas. Among other things, they were able to compel Japanese automobile manufacturers to pay royalties for the period from 1988 through 1995(!), because their automobiles which were imported into the United States contained parts made of polypropylene which was produced in Japan with Ziegler-catalysts.

- Finally, the story is also unique because the most recent changes in US patent law now mandate a patent term of 20 years beginning on the date on which the application is filed, as compared to the earlier 17-year term running from the day of final patent issuance, which would thus make it virtually impossible for the history of the Ziegler-catalysts to be repeated.

Even if Dr. Martin ultimately fails to give the reader a complete account of the achievement, he nevertheless illustrates, arguably the most successful worldwide commercial exploitation of all time of inventions based on non-industrial research.

After all, he does reveal that the Max-Planck Institute for Coal Research in Mülheim was sustained for more than 40 years on the proceeds of the exploitation of its patent rights dating back to 1953/1954. This information would certainly enable any math wizard to gain a pretty clear picture of the total license fees collected.

In writing this book, the author certainly satisfies in a very special manner, an apparently decade-old desire to help clarify the issue of the not entirely undisputed and at least partially obscure scientific priority of the achievements attained by Professor Ziegler and his Mülheim team.

One of the author's main concerns was to shed light on the relationship between Professor Ziegler's team and Professor Giulio Natta and his team in Italy. Natta had been awarded the Nobel Prize jointly with Professor Ziegler, and while Dr. Martin in no way challenges Natta and his team's scientific achievements, he traces the roots of these achievements back to the research results which Natta had previously accessed at Mülheim on the Ruhr. The author has also observed that the battle for the acknowledgment of priorities was most often conducted on a less than highly moral or ethical level, and that the scientific community, for various reasons, ignored a specification in its reference to the origin of the Ziegler-catalysts, (see Section (p. 30). Therefore, in his final analysis, Dr. Martin has endeavored to establish the scientific truth by using the innumerable decisions handed down, particularly those made by the US courts – which not only determined patent priority but also scientific priority, thus acknowledging the dominant position which Mülheim's research occupied in this field.

This book makes suspenseful reading, sometimes almost reminiscent of a crime story. It allows insight not only into the developments within the complex field of polymer chemistry and the equally complex arena of patent jurisdiction, which, in the United States particularly, is marked by anomalies, but also highlights in particular the downright phenomenal negotiating skills demonstrated by Professor Ziegler and Dr. Martin himself, although the author and his activities always remain discreetly in the background. This is clearly illustrated in the following single excerpt: "In May of 1967" – that is, 10 years after the controversy with Du Pont had started – a "settlement agreement between Du Pont and Ziegler" was finally negotiated, under the terms of which Du Pont paid 2 million dollars as a settlement sum for its polyethylene license in the United States. In addition, patents that Du Pont had issued in the meantime from various interference proceedings, among others for the production of isoprenyl aluminum and its utilization in the polymeri-

zation of ethylene, were assigned to Ziegler on a worldwide basis. Thus, Ziegler had made up for the cost of the concessions he had made in connection with the settlement with Du Pont concerning the “Lachar-Pease Polyethylene product patent rights”. A significantly sizeable profit was, however, realized due to the fact that Du Pont had neglected to include Canada, even though the major portion of its polyethylene production had been established there in the meantime. Ziegler once again collected the same amount in settlement for the Canadian production.

Within a very short time the transfer of the Du Pont patent rights in the isoprenyl aluminum field would prove to be a propitious arrangement for the Max-Planck-Institute for Coal Research. Farbwerke Hoechst in Frankfurt had decided to use this product for the manufacture of its ethylene polymerization catalysts, being unaware, however, that it was the Max-Planck-Institute which now owned these patents in Germany as well (see p. 123). The effect of this transfer of rights from Du Pont to Studiengesellschaft was that, as Martin notes elsewhere, “it could be urged that Hoechst was obligated to pay royalties for the longer running Du Pont patent rights which Ziegler had acquired in Germany” (see p. 157), and that, in the early 1980s, Hoechst had to pay Studiengesellschaft/MPI royalties supposedly in the eight figure range. Imagine, a German research organization prevailing against Hoechst on the basis of patent rights acquired from Du Pont! This was certainly an unprecedented achievement.

Dr. Martin’s book should be required reading for anyone involved in the protection and utilization of results obtained from publicly-funded research, and in fact anybody who would like to learn what enforcement and utilization of patent rights mean in actual practice, or in any event, what they can mean. Where else could one find more abundant material to illustrate this point. This book offers eloquent testimony of how difficult and costly it is to convert even those research results that have been awarded the Nobel Prize into commercial success stories. If Professor Ziegler had not been able, within a very short period of time, to collect millions from option and license agreements based on inventions that, at the time, had not even matured into patents, money that later on assured him a solid basis for the defense and enforcement of his rights, in other words allowed him to engage first-rate patent attorneys and general legal counsel, it might very well have happened that the Max-Planck-Institute would have either made no profit at all or derived only a modest income from these epoch-making inventions.

Martin was not only a co-inventor, but because of the battles he waged for decades against any potential infringer, he also contributed materially to the commercial success of the exploitation of the Ziegler-catalysts.

The author takes us on a historical journey through the events surrounding the development and commercial exploitation of this technology, events which up to now had barely penetrated public awareness in Germany but which decisively shaped the second part of the 20th century, whilst at the same time offering invaluable object lessons. If anything, this treatise should enlighten both universities and other non-commercial research organizations as well as the Ministries for Research and Education about the fact that successful commercialization of research findings requires peak performances not only by researchers, but also by those who are re-

sponsible for the patenting and commercial exploitation of such research results. But even the latter are helpless if they do not have enough “cash” to vigorously pursue the enforcement and defense of the patented inventions. If these factors are not properly appreciated, any hopes carried by the proposed revoking of the university professor privilege may prove illusionary and may even act as a boomerang. Exceptional individuals of the caliber of Professor Karl Ziegler and Dr. Heinz Martin, who are capable not only of conducting top quality research, but also of commercially exploiting their research results in a highly successful manner, are extremely rare even today. They deserve our praise, appreciation and highest esteem.

The law makers, universities and other non-commercial research institutes, when considering future revisions not only to the law but also to institutional infrastructure and its funding, should not rely on the ready availability of such exceptional individuals.

Munich, June 2001

Joseph Straus

Introduction

In terms of the industrial development brought forth by our society, the last 80 years have been dubbed, among other things, the age of plastic. This period witnessed important advancements with contributions not only from industry, but also from individual research groups and/or researchers themselves. Synthetic materials such as plastics (German: Kunststoffe), as the term implies, are products made mostly from chemical raw materials that are refined to give them properties which render them useful in replacing or enhancing natural materials.

In the 1930s, production plants were built for the manufacture of, for example, polyvinylchloride (plates, rods, tubes, hard foils), polyvinylether (e.g. adhesives), acrylic and metacrylic acid polymers (plexiglas), polystyrene (styropor), and polyvinyl acetate (e.g. chewing gum), to mention only a few of the most significant polymer products marketed at that time. The rubber industry developed “Buna”, a polybutadiene, and “Buna S”, a butadiene/styrene copolymer, as well as butyl rubber made of polyisobutylene. Other familiar products included polyamides, such as nylon and perlon, and polyester, such as celluloid or trevira fibers.

In the mid-1930s, it became possible for the first time to produce polyethylene from ethylene gas by binding ethylene molecules together in a chain (high pressure process: temperatures of 200 ° Celsius and pressures of up to 1500 atmospheres, catalyst: traces of oxygen). Depending on the chain length, this method yielded liquid, waxy or solid, thermoplastic products, that is, products which could be molded into shapes at elevated temperatures. The characteristics exhibited by solid polyethylene were new and, in many respects, different from the properties of the aforementioned plastics. Thus, its significantly enhanced molding properties, coupled with inherent chemical resistance, elasticity, and electrical insulating properties at broad temperature ranges, made polyethylene superior to the foregoing products. Films made from this material began to increasingly dominate the packaging industry. For 20 years, this technology evolved unencumbered by competition.

In 1953/54, at the Max-Planck-Institute for Coal Research in Mülheim on the Ruhr, Karl Ziegler and co-workers discovered a significantly improved process for the production of polyethylene with a high crystalline content – a linear structure with practically no branching of the polymer chain, and, therefore, suitable for a comparatively much broader scope of applications than high pressure polyethylene. The crucial factor was the discovery of a catalyst which caused the individual ethy-

lene molecules to bind together under very simple conditions and with unaccustomed efficiency. But the spectacular results did not end there. A whole new class of Ziegler-catalysts had now become available – catalysts, which were also capable of transforming the next higher homologs of ethylene, such as propene, butene, etc., into entirely new, solid, plastic-like, partially crystalline products and, in a comparatively rather simple manner, polymerizing the respective dienes, such as butadiene and isoprene, into elastomer materials. Thermoplastic and rubber-like properties can also be found in materials obtained by the same method from the co-polymerization of different olefins. But that was not all. The catalysts furthermore proved to be the key to guaranteeing that, with each repetition of the process, the desired polymer chains would exhibit a standardized broad range of characteristics. The versatility in the application of the Ziegler-catalysts led to a multiplicity of new products.

The scientific magnitude of this discovery is undisputed. This fact and the ensuing worldwide, industrial-scale production of the multifarious new plastics was acknowledged by the presentation of the Nobel Prize for chemistry to Karl Ziegler in 1963.

To this day, it is the solid, plastic-like polypropylene which over the years has been the most marketable product springing from the research efforts of the Max-Planck-Institute in Mülheim. Almost 50 % of the proceeds from the licensing transactions conducted during the period 1953–2000 came from the production of polypropylene, co-polymers and terpolymers (polyethylene approx. 24%).

Polypropylene, therefore, has been given preferential status in the account and analysis of the historical developments presented here.

It was almost 50 years ago that polypropylene was synthesized at the Max-Planck-Institute in Mülheim on the Ruhr – a colorless, solid, plastic polymer, which, at temperatures of above 140 °C could be molded into foils and plates for example, and which is made from propene by polymerization with a catalyst composed of alkyl aluminum compounds and titanium halides. Even though polypropylene had been previously produced elsewhere, whenever it exhibited the same characteristics, its production, was not independent, owed a debt to the discoveries made at the Max-Planck-Institute for Coal Research. The conflicts arising from this situation were ever present throughout the entire history of this evolution.

Much has been written about the research into, improvement of the properties, and industrial-scale production of this material. Each year, more than 25 million tons of polypropylene are produced and sold worldwide. Given the importance of the material to today's industrial society, one could hardly expect to find a more explosive "new product" story anywhere.

Not only did I participate in the discovery of the new material, but for more than 40 years I was instrumental in shaping ongoing events and provided guidance throughout many pivotal proceedings in both a scientific and legal capacity, initially as Karl Ziegler's assistant, and ultimately as the General Manager of Studiengesellschaft Kohle mbH, acting as Trustee for the Max-Planck-Institute.

Although the inventors and/or Karl Ziegler owned a monopoly right for long periods of time, they had to overcome countless obstacles in order to obtain maximum commercial benefits for the Max-Planck-Institute.

As I recount the mostly unfamiliar historical events, I refer to a large number of documents, all of which I have listed in an earnest effort to be as thorough as possible. While a substantial portion of these papers are available to the public, some of the evidentiary material is subject to confidentiality. To the extent that the latter documents are essential to the understanding of events, their pivotal points have been excerpted without violating confidentiality.

The story of polypropylene has been impacted by many institutions and personalities, and in many instances some significant developments occurred independently from one another at the same point in time. However, the continuity of the story, including some interesting cross-connections, and the chronology of events that can be demonstrated on the basis of these documents remain its most important aspects.

As the history of an invention is retold by a third party, it will hardly remain free from a personal slant. Surely, a comprehensive portrayal would, therefore, require not only self presentation, supported by pertinent documentary evidence, but also critical opposing views, and finally the balancing judgment of an arbiter.

The present account is aimed at chemists, in particular research chemists, attorneys, particularly patent attorneys, managers of chemical companies, the exploitation manager, and also any reader who is interested in natural sciences.

The actual story began on 13 July 1954.

Mülheim, December 2006

H. Martin

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December, 2006

H. Martin

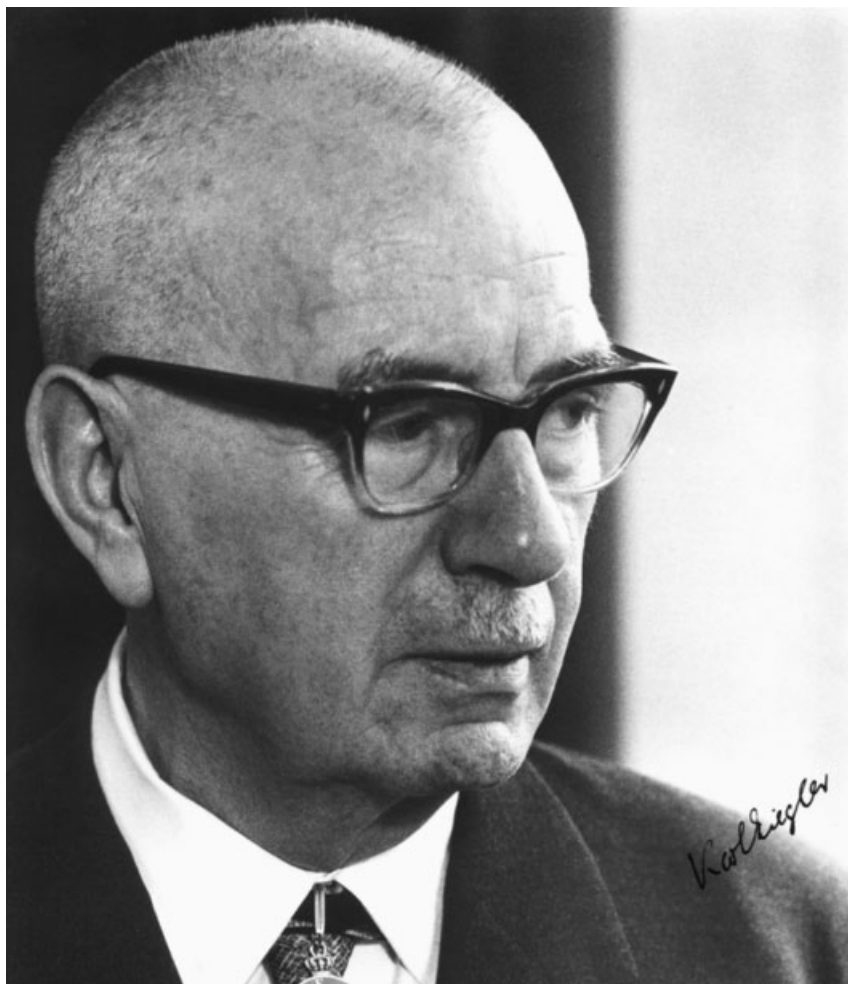


Fig. 1.1 Professor Karl Ziegler

1

The Invention

1.1

First Observations 1950/1953

As dramatic and exciting as the history of the development of the Ziegler catalysts may be on its own, nevertheless revisiting the events that took place around the world in the same area of research is no less fascinating. It was particularly in the United States, where, in different parts of the country, within the span of a few years, simultaneous, but independent, efforts were underway to investigate transition metal compounds as catalyst components in connection with the conversion of olefins.

Numerous litigations during the last 30 years provided an opportunity for a number of individuals, among others, those in research organizations, to disclose and provide supporting evidence for the developments taking place within their own institutes as well as to establish legal positions concerning the events that took place between 1950 and 1955. Through countless cross-examinations conducted during these litigations, the facts of the case, the historical chronology of the events, and the functions performed by individual research personalities were disclosed to the public.

In 1958, the US Patent Office declared a so-called “interference”¹⁾ [1] between Du Pont, Standard Oil of Indiana, Phillips Petroleum, Hercules Powder and Montecatini. The issue involved was a product claim directed to the new and up to that time unknown polypropylene. The product claim to the new “solid, crystalline polypropylene” material which the Office proposed to all parties involved, read as follows:

*“Normally solid polypropylene consisting essentially of recurring
[2] polypropylene units, having a substantial crystalline polypropylene content.”*

Neither the parties nor the US Patent Office had considered including Karl Ziegler’s patent rights in this proceeding. The claim proposed by the Examiner did not

1) “Interference” is a procedure carried out by US-Patent law according to which the Board of Patent Appeals in the US Patent Office

determines the priority of two or more timely overlapping inventions of identical or similar claim content.

indicate whether or not high molecular “thermoplastic” polypropylene was included. But more on this later.

US law provides that three criteria must be met when determining prior inventorship (priority), criteria which are also internationally respected:

- (1) Production of a composition of matter satisfying the limitation of the Count.
- (2) Recognition of the composition of matter; and
- (3) Recognition of a specific practical utility for the composition.

Product yields of barely a few milligrams were not sufficient to satisfy these requirements, but not only could US inventors assert the earliest filing date of a patent application as evidence of priority, but even before that, they also could establish an earlier priority based on testimony-supported documentation (such as laboratory journal entries).

In 1971 [1], the US Patent Office found, in connection with this interference, that Montecatini was entitled to the earliest product claim directed to “polypropylene”. The losing parties appealed this decision to the competent district court. In 1980, i.e. more than 25 years after the discoveries had been made, the court [3] determined that it was neither Standard Oil nor Du Pont nor Montecatini, but Phillips Petroleum who was entitled to the benefit of priority of invention [4]. The sensational aspect of this decision will be discussed later. For now suffice it to say that the court found that Montecatini had committed fraud on the Patent Office where Phillips Petroleum was concerned and had, therefore, forfeited any right to an award of priority (see p. 31; Chapter 3, footnote 33; p. 115; p. 159; Chapter 5, footnote 1).

1.1.1

Standard Oil of Indiana

In 1950, at the “Exploratory Research Division” at Whiting, Indiana, Dr. Bernhard Evering and his research team were exploring the effects of catalysts on alkylation reactions with ethylene. A. Zletz, Carmody and E. F. Peters were the team members who worked together on this project during that time. They obtained substantially solid polymers as by-products.

The catalysts in question consisted of “reduced” molybdenum oxide on aluminum oxide carriers. One of the earliest entries in Zletz’s laboratory journal (dated July 18, 1950) also suggests that this catalyst could be used for the polymerization of propylene [5]. The reference to the polymerization of olefins, including propylene, in Mr. Zletz’s journal was initialed by J.C. Stauffer (“Senior Patent Adviser”) on July 21, 1950. Presumably this was intended to establish a date of conception and/or priority. Although Zletz described such polymerization experiments in his journal entries dated August 11, 1950 and August 31, 1950, these experiments never became the subject matter of a patent application.

During his experiment on September 29, 1950, Carmody isolated a propylene polymer [6]. It was fabricated into a thin, “somewhat flexible, not brittle” film. The product was also subjected to viscosity measurements and infrared spectra [7]. A later de-

termination of crystallinity showed 38%, according to Heinen [8], and 27–30% according to Luongo [9]

That same day, Carmody conducted a further propylene polymerization experiment [10], and later on, so did Peters on April 30, 1953 (P-1) and July 17, 1953 (P-9), using similar catalysts (cobalt molybdate) [11]. When repeating this experiment in 1965, these products were characterized as 1,2-polypropylene having 77–95% crystallinity. No yields were specified.

Patent applications filed in 1951 [12, 13], naming A. Zletz as the inventor, may serve as an indication of how, in general, chemists at Standard Oil of Indiana were dealing with transition metal catalysts. In 1952, E. Field and M. Feller [14, 15], who were attached to the same research laboratories, described, from a large number of experiments using ethylene, two examples which used propylene alone (see US Patents ‘647, Example 21, and ‘453, Example 7). Whether it was a clever patent attorney who while working on the patent applications, suggested including these Examples – the 80 mg yield and/or complete lack of data on yield are less than convincing – or whether the experiment did in fact lead to the described results, will forever be shrouded in mystery. Much of the attention of numerous chemists, expert witnesses, and legal counsel was centered on these two examples during subsequent litigations. At no time did Standard Oil of Indiana either assert or designate these patent applications as priority applications [16].

Propylene polymerization experiments conducted in 1953 were included in a patent application by Zletz and Carmody (US SN 223,642), but this application was abandoned on May 20, 1954 in favor of US Application SN 462,480, filed October 15, 1954, which however, no longer contained these experiments. One can only surmise the reasons for such action: the yields [6, 10] (0.7 and/or 3.83 g) were too small, the product could not be reproduced, and had not been recognized as polypropylene. They contained a high methylene/methyl ratio (the court tolerated 1.1, but not 1.5 and higher – theoretically the ratio should be “1.0”), which led to the conclusion that these could not be polymers with a regular 1,2-propylene chain configuration. It was eventually learned that up to the year 1958, it had been impossible to determine the presence of crystalline polypropylene from infrared data alone. A retroactive interpretation of infrared spectra was, however, legally inadmissible.

Although the 1953 X-ray images of the products appeared to indicate crystalline fractions, quantitative figures were not available. In the final analysis, there was the possibility of an amorphous ethylene/propylene co-polymer content.

The documented experiments carried out by Standard Oil of Indiana in September of 1950 and between April and the end of July, 1953, were not accepted as proof of priority, “since neither the making of the product had been adequately described, nor had the product been recognized or a utility therefore been given,” as the judge later noted. It was not until approximately June 15, 1954, that Zletz realized that molybdenum oxide on aluminum oxide carriers was suitable as a catalyst for the polymerization of olefins. Dr. W. Bailey, an expert witness engaged by one party, told the court that disproportionation of propylene on molybdenum catalysts in butene and ethylene is known, so that, since ethylene polymerizes more rapidly than propylene, ethylene/propylene co-polymers must necessarily be present in the product.

Judge Wright ruled that Standard Oil was to be granted a priority date of October 15, 1954, the date that the Zletz Application Serial No. 462,480 was filed in the US Patent Office.

Due to the unsatisfactory results obtained when using the molybdenum catalysts for propylene polymerization, Standard Oil of Indiana had neither developed its own commercial production, nor offered licenses, conceivably because of the unfavorable priority situation. It was not until the late 1960s and early 1970s that Standard Oil started producing polypropylene, this time, however, using Ziegler catalysts²⁾ (organoaluminum compounds and titanium halides). In 1972/1973, Amoco Chemical Corporation, a wholly-owned subsidiary of Standard Oil of Indiana, obtained a license for this production from Ziegler.

1.1.2

Phillips Petroleum Company

Phillips Petroleum Company, a crude oil processing company like Standard Oil of Indiana, maintained a process development section for catalytic processes in Bartlesville, Oklahoma, USA, which was a subdivision of an exploratory station for hydrocarben conversion. Dr. W.C. Lanning was the head of this operational section, while J.P. Hogan was a group leader, and R.L. Banks, G. Nowlin and E. Francis worked as research chemists. At that time, Hogan's group was focusing specifically on the conversion of gaseous olefins to liquid oligomers. They were exploring transition metal oxides, such as nickel oxide on a silica – aluminum oxide carrier, in terms of their effectiveness as catalysts.

In June 1951, the team worked with a mixture of chromium- and nickel oxide using the same carrier material. Ultimately, it was the type of catalyst which uses chromium oxide alone on the carrier material in the form of a reactor bed which, on October 9, 1951, for the first time caused the polymerization of propylene into, *inter alia*, semi-solid polypropylene [17]. E. Francis isolated 40–50 g of a polymer which G. Nowlin later fractionated, and one fraction of which he characterized as polypropylene gel, insoluble in both chloroform and benzene.

In November of 1951, when repeating the experiment under identical conditions, Hogan, Banks and Francis [18] recovered 70.5 g of a sticky polymer containing fractions which, again, were insoluble in boiling methylisobutyl ketone and n-pentane. Some of the products thus satisfied the claim that they were solid and moreover, that an infrared spectrum would show recurring polymer units (head – tail polymerization or 1,2 addition). This discovery, albeit fragmentary, was probably made between February and June of 1952. Crystallinity would be verified later by way of an infrared spectrum and X-ray analysis. In December of 1952, one of the products originally recovered in April of that year (PO-116) was determined to have a melting point of as high as 130 °C. The fact that the product was insoluble in boiling methylethylketone

2) G. Natta, for the first time, had defined the catalysts used by him and formed from aluminum alkyls and titanium halides as Ziegler

catalysts (Lit. 165 and 166, first patent applications 1954).

led to the conclusion that it contained crystalline fractions. The measurement results obtained from an analysis of the polypropylene products recovered during the second half of 1952 [19] were set forth in a patent application filed January 27, 1953 [4]. But Phillips' assertion that they had demonstrated the utility of the polypropylene products as solid thermoplastics lacked evidentiary support, even though the usefulness of the products as wax additives had been established. The text of the patent application contained neither the word "crystalline" nor an indication that the products were useful as wax additives. Nevertheless, it was found that Phillips had documented both criteria. The mere supposition that the new material might be useful for the same purposes as polyethylene did not, however, constitute sufficient evidence. Furthermore, the melting point measurements obtained did not guarantee that the products would remain stable when subjected to molding temperatures (molded plastic). There was no indication that the products were useful as solid thermoplastics. As Judge Wright stated in 1980:

"The Court accordingly finds that Phillips failed to prove that its scientists knew enough about their product to conclude that it was useful as a solid plastic [20]."

The ruling with regard to this defect would prove to be of great significance later on. To indicate the presence of crystalline fractions in the polypropylene product, the Phillips application mentioned the lack of solubility of the polymers in pentane at room temperature, a melting point range of the solid residue between 240 and 300 °F (115–149 °C), a density range between 0.90 and 0.95, an "intrinsic" viscosity between 0.2 and 1.0, and a molecular weight range of between 5000 and 20,000, including specific fractions between 200 and 50,000. According to the specification, the sticky polymer, having a molecular weight between 500 and 5000, could be separated into semi-solid, pentane-soluble fractions and solid, pentane-insoluble fractions. Examples were given only with respect to the semi-solid and/or sticky products.

Later on, that is, many years after the filing of the 1953 application, a number of experts repeated the examples presented in that application³⁾. In 1980, the court [21]

3) In 1955 C. Capucci of Montedison, found that solid polypropylene can be produced as described. But the method of work-up differed from the disclosure of the Phillips application. Nevertheless an 'intrinsic' viscosity (I.V.) of 1.02 was measured on a sample of the product, i.e. at the upper limit of the disclosed range. Yet, this sample was not a raw product, but an insoluble fraction [22].

In 1956, H.S. Eleuterio of Du Pont, repeated examples out of the parallel Belgian Phillips patent 530,617 (equivalent to the Phillips US application 1953). He extracted the raw polymer from the catalyst, evaporated the solvent and isolated a solid product, 56 % crystallinity. In 1962 G. Trada of the Edison Company, repeated the Phillips experiments. He

extracted the product with boiling xylene and after evaporating the xylene extracted the residue with pentane. The viscosity of the residual polypropylene was 1.31 and the product was 57% crystalline. After extraction with boiling heptane the viscosity of the residue was raised to 2.0 and the crystallinity remained unchanged.

Following the Phillips disclosure for the work up D. Witt of Phillips, isolated a material having a melting range of 125–130 °C and a viscosity (I.V.) of 0.73–0.95. In 1964 reproducing the procedure J.A. DeLap of Phillips, was not able to show different data.

Results from reproduction experiments were described by G. Longi of Montedison, during 1965 [23].

found that these products were not covered by Phillips' disclosure. Furthermore, it was not verified whether these products had been subjected to infrared or X-ray analysis. Longi proved that significantly higher-melting, crystalline products (melting point up to 167 °C, I.V. 1,37, crystallinity 71.5%) could be isolated. The same court further found that while the 1953 Phillips application did contain a description of the method for producing crystalline polypropylene, it failed to disclose all of the ranges of crystalline polypropylene and, therefore, was not entitled to claim the entire range. The application indicated that the described products had a molecular weight of approximately 5000–20,000. Whether products having a molecular weight of up to 50,000 were included in this range must remain doubtful. The only definite disclosure made by Phillips in its 1953 application was that a solid, at least partially crystalline material had been produced.

1.1.3

Du Pont

1.1.3.1 From the Beginning up to the Patent Application

According to statements made by Du Pont, it was in late January and February of 1954 when a group of chemists at the "Petrochemical Department" started a systematic search for suitable polymerization catalysts for the co-polymerization of norbornene and ethylene. At that time, the fundamental discovery which formed the basis for the development of the Ziegler catalysts for the polymerization of ethylene into solid, plastic-like polyethylene at the Max-Planck-Institute for Coal Research at Mülheim was 2–3 months old. Patent applications [24] directed thereto had been filed in the German Patent Office, and Montecatini, Farbwerke Hoechst, and Ruhrchemie AG [25] had been advised by Ziegler, through confidential communications, of the composition of the best-performing catalysts known at that time.

Whether and to what extent the Du Pont research group led by W.F. Gresham (Research Director), Anderson, Robinson, Merckling and Truett had knowledge of the work being carried out at the Max-Planck-Institute at Mülheim, could not be conclusively be established.

However, a memorandum [26] from the Du Pont Patent Department, dated August 4, 1954, mentioned that Gresham knew that Ziegler had developed a new polyethylene product, and that he had an idea about the composition of the Ziegler catalyst. In August of 1954, Du Pont filed patent applications in the US Patent Office disclosing and claiming the production of high molecular weight, solid polyethylene [27, 28] as well as polypropylene [29].

The initial experiments conducted at the Du Pont laboratory were based on the use of catalysts according to Max Fischer [30]⁴⁾. Upon repeating the Fischer experi-

4) Max Fischer (BASF, Ludwigshafen) varied the efficiency of aluminum trichloride as a Friedel-Crafts-catalyst for the production of lubricating oils from ethylene, by addition of titanium tetrachloride and a small amount of aluminum powder. In addition to viscous oils solid ethylene polymers were also formed. It was known that aluminum powder can reduce the titanium in titanium tetrachloride.

That was the basis on which the Du Pont team developed the working hypothesis that the formation of solid high MW polymers required that the titanium in the catalyst must be in a lower valence and later, that the valence must be below 3. Titanium dichloride by itself polymerizes ethylene into solid high MW polymers as shown, but titanium trichloride is ineffective.

ments using ethylene, the only products initially recovered were oils, and Truett, one of the researchers, characterized the Fischer reaction as “capricious” [31], because he was unable to determine the external conditions that would account for the formation of either nothing but oils, or actual solid polymers, as were subsequently found. Meanwhile the research group had expanded to more than a dozen chemists. A working hypothesis had been established according to which it was a transition metal halide alone, in reduced form [32], that would have to be responsible for the catalytic activity. The reducing agents employed were metals (aluminum or magnesium), but also – it was April, 1954 by now – Grignard-compounds [33]. No conclusion was reached as to the usefulness of aluminum alkyls or alkyl aluminum chlorides.

At that time, Ziegler had already entered into a number of option and/or license agreements [34], and there were test facilities from which polyethylene samples were distributed to interested parties and the curious. It was not difficult to analyze such samples for ash residues and to conclude that they contained at least aluminum and titanium. It was not until the early summer of 1954 that Gresham initiated the use of organoaluminum compounds. The suggestion had been made by a chemist by the name of Hyson [35], who, even though he had not been previously involved with this project, was still knowledgeable and experienced in working with Ziegler’s aluminum alkyls. At the end of July, 1954, Hyson conducted experiments with ethylene, during which equimolecular amounts of aluminum trimethyl and titanium tetrachloride under extreme pressures (up to 1000 atmospheres) and temperatures (100–200 °C) were found to be effective as catalyst components. Later on, however, Hyson switched from pure aluminum alkyls to lithium aluminum tetraalkyls, since he considered the short-chained aluminum alkyls in particular, to be too dangerous to use due to their inflammability when exposed to air. Hyson also suggested that propylene as well as olefins in general could be used for polymerization [36].

Subsequent developments are described in the specifications of the patent applications filed between August 16 and 19, 1954 which claimed the production of catalysts for the polymerization of ethylene [27, 28]; the August 19, 1954 application claimed, *inter alia*, the production of polypropylene. [29]. The former suggested Grignard-compounds, lithium aluminum tetraalkyls and aluminum trimethyl as reducing agents for titanium tetrachloride, while the latter contained examples showing the use of lithium aluminum tetraalkyls or Grignard-compounds as the reducing component. None of these applications names Hyson as an inventor. The use of phenyl magnesium bromide and titanium tetrachloride as a catalyst mixture is described in an initial experiment of May 21, 1954, all further experiments are dated in August, a detail of vital significance for DuPont from the standpoint of patent rights.

1.3.1.2 Only a Scant Experimental “Polypropylene”

During the period from April through August of 1954, Stamatoff and Baxter conducted a series of experiments at DuPont using different catalysts for both ethylene and propylene. A large number of these experiments yielded either no polymer at all or liquid polymers (oils) only. In some cases, where minute quantities of solid poly-

mer were formed, the products were never analyzed. On May 21, 1954, Baxter [37] had converted propylene with the aid of a mixture of Grignard-compounds and titanium tetrachloride, but the yield, 0.5 g of a powder, was not conclusive. Even though a film was formed from this product, which was characterized as “tough and elastic” – infrared spectra indicated absorption at 8.69 μm (internal methyl group) – no quantities were specified for this methyl content, nor was crystallinity mentioned – thus, the analysis provided no conclusive results.

In 1980, within the context of the above-described litigation between Standard Oil of Indiana, Phillips Petroleum Co., Du Pont and Montecatini, Judge C.M. Wright [38] affirmed a 1971 decision of the Board of Patent Interferences [39] (the board of appeals for interference proceedings) which held that the earliest priority established by Du Pont with respect to the production of solid, crystalline polypropylene was August 19, 1954 [29]. Du Pont’s assertion that they were entitled to the earlier priority of the May experiment was rejected by the judge, since the experimenters had not only failed to recognize, in a legal sense, the polypropylene product as such, but had also neglected to show any utility, as required by the rules. The judge stated: “It is clear that the infrared spectrum of the product of the May experiment, fabricated by Mr. Beck (Du Pont), showed no indication that the product had any polypropylene crystallinity whatsoever.”

In August of 1954, Baxter obtained a larger yield of polypropylene when using a catalyst combination of titanium tetrachloride and lithium aluminum tetrahexyl. An X-ray analysis of these products now showed a crystalline content of 15–10%, but even these values were not submitted to the Du Pont Patent Department in August (see p. 223).

No later than 1955, it had become obvious to Du Pont that its own position, in particular with respect to polypropylene was poor as compared to all of its competitors. But it took the individuals involved another 25 years before they would become aware of this (see above). Any yields and insights obtained from these experiments were too slight for Du Pont to expect an award of priority. Du Pont has never seriously considered the commercial production of polypropylene.

1.2

Max-Planck-Institute, Mülheim on the Ruhr

1.2.1

K. Ziegler, H. Breil, E. Holzkamp and H. Martin

While the three above-mentioned companies and their research teams working in the polymer field had obtained the described results independently from one another, the development at the Italian chemical company Montecatini is inseparably entwined with the developments at the Max-Planck-Institute for Coal Research. In other words, the initial impulse for all of the activities taking place at Montecatini with respect to “polyolefins” was provided by the Institute at Mülheim on the Ruhr, as will be documented hereinafter (see p. 29).

Giulio Natta, Director of the Instituto Di Chimica Industriale Del Politecnico in Milan, a research institute subsidized by Montecatini within the framework of a contractual association⁵⁾, was training a number of young chemists for Montecatini among others. Early on, he had pointed out to Montecatini that the research carried out at the Max-Planck-Institute for Coal Research at Mülheim might be of potential importance for Montecatini. According to his own statements, Natta had closely followed Ziegler's publications and lectures since at least 1952. At that time, Ziegler and his co-workers were exploring the so-called growth reaction⁶⁾ of ethylene on aluminum triethyl and the selective dimerization of higher alpha-olefins on organoaluminum compounds. Obviously of particular interest was the dimerization of propylene to 2-methylpentene-1 as well as the dimerization of butene to 2-ethylhexene-1. In January of 1953, Montecatini and Ziegler entered into an "agreement" [34], granting Montecatini an exclusive license for Italy under certain patent rights [40] concerning

"the conversion of olefins with the use of organometallic catalysts."

The license also included any

"ancillary processes, as well as all other future processes which would be developed in the field of olefin conversion with the use of organometallic compounds as catalysts."

The definition of the subject matter under contract was so comprehensive that, 1 year later, it prompted a debate as to whether the "Ziegler catalysts" (organometallic mixed catalysts) discovered during the second half of 1953 would fall under this definition [41].

The agreement furthermore provided that any subsequent inventions made by Montecatini in the contract field should be offered to Ziegler, and that Montecatini would retain the right to grant licenses in other countries for any of its own inventions falling under the above definition [41]. The down payment of 600,000 Deutsch Marks was attractive to Ziegler, especially because the existing "know-how" for conversion to industrial-scale production was by no means complete. Only a small experimental pilot plant was available.

Ziegler's Italian patent rights at that time covered, *inter alia*, the "polymerization of ethylene" [40]. But it should be mentioned here that while the products in question were, at best, wax-like in character, they consisted primarily of hydrocarbon oils (see "Growth reaction", this chapter, footnote 6). The aluminum trialkyl catalysts used were free from transition metal compounds.

In the Spring of 1953, in line with the agreement, Montecatini sent two chemists, Paolo Chini and Roberto Magri, as well as an engineer, Giovanni Crespi, to Mülheim

5) G. Natta assigned patent rights to Montecatini. The chemists whom he trained were partly employees of Montecatini.

6) Growth reaction: the stepwise addition of ethylene molecules to aluminum triethyl leads to the formation of a longer hydrocar-

bon chain i.e. the chain grows. Contrary to this result a replacement reaction was defined if the chain was removed from the aluminum. This could occur after one, two or several steps of growth, according to specific conditions.

with the aim of familiarizing themselves with the licensed subject matter and to learn the process production and method of use of aluminum alkyls. The three guests arrived at Mülheim to start their visit on February 24, 1953 [42] at a point in time when a number of interesting observations were being made at the Max-Planck-Institute for Coal Research during work with aluminum alkyls.

While investigating specific growth reactions on ethylene, E. Holzkamp, a doctoral student with K. Ziegler, had noticed an important development which deviated from the usual test results: instead of a chain-building reaction, he observed a displacement reaction, which for the most part produced butene, the dimer of ethylene, a portion of which, was again dimerized. The search for the cause of this reaction, as is generally known pointed to traces of nickel metal from the chromium – nickel – steel of the autoclave acting as a co-catalyst. By adding finely dispersed nickel, the addition reaction could be curtailed. However, the exploration of the use of other transition metals and their effect was considered too ambitious an undertaking for a doctoral thesis.

A young undergraduate, H. Breil, was then assigned to the task of preparing suitable transition metal compounds and, in collaboration with E. Holzkamp, investigating their action as so-called displacement catalysts.

Although the Italian guests worked inside the Max-Planck-Institute, they were being introduced to the organic chemistry of aluminum in a different area of the building, away from the Holzkamp/Breil laboratory. At that time, the Institute was also accommodating another guest, Dr. A. Glasebrook of the US-based Hercules Powder Co. Following an invitation by Ziegler, Dr. Glasebrook had been assigned by his company to become acquainted with organoaluminum chemistry in order to see whether its use on an industrial scale would be of sufficient interest, given his company's field of interests. Among other places, Dr. Glasbrook also worked at the Holzkamp laboratory. Every day, all of the guests, the three Italian gentlemen and A. Glasebrook, had lunch together at a nearby restaurant and exchanged information.

1.2.2

Experiments between May and December, 1953

In May of 1953, E. Holzkamp conducted two back-to-back experiments, in both cases using a combination of chromium acetylacetonate and aluminum triethyl as the catalyst mixture, the intent being of course, to test the effect of the chromium compound in place of nickel, as a displacement co-catalyst on ethylene [43, 44]. In the first instance, he recovered "approximately 30 g of a mostly higher molecular weight product, about 30% of which was non-melting," while the second experiment yielded a "residue of 16 g of a non-melting product." The only variation in the two experiments was the degree of ethylene pressure being applied.

Around the same time, and taking into account the nickel co-catalysis reaction previously observed, H. Martin, a postdoctoral assistant with Ziegler, was testing the theory that, if the reaction vessel being used was completely free of any potential traces of nickel, the growth reaction of ethylene on aluminum triethyl should proceed wholly unencumbered, all the way to yielding true polyethylene. This re-

quired a simultaneous, step-by-step reduction in the amount of aluminum triethyl employed, so as to allow the chains forming on the aluminum to develop with a high excess of ethylene [45]. To that end, an anodized aluminum vessel was fitted into the pressure reactor, into which aluminum triethyl and ethylene were introduced at a 1 : 280 ratio, a temperature of 55 °C and a pressure of 150 bars, and brought to reaction in hexane as the reaction medium. The reaction continued for 20 h, yielding a high fraction of solid paraffin products [46].

In a letter dated the end of July 1953 [47], K. Ziegler reported to Montecatini (R. Orsoni) on the status of the developments, stating *inter alia*:

“For approximately the past 2 to 3 months, the entire realm of polymerization problems has, again, been intensively addressed by Dr. Martin and others. I believe that here, too, we are on the threshold of new developments and insights.”

In September, 1953, the research endeavors yielded products with melting points between 80 and 100 °C [48]. The molecular weights of these products, however, failed to rise above 10,000 [49]. Parallel thereto, there was an increase in the olefin content of the products, which indicated that the displacement reaction was still taking place even under these conditions⁷⁾.

During the process of systematically exploring any available transition metal compounds, Breil also used titanium tetrachloride together with aluminum triethyl as a catalyst mixture under ethylene pressure. He made the following entry in his laboratory journal [50]: “Yield 3.5 g of butene ... additionally, a high polymer residue, black (titanium), rubber-like mass.” It was noteworthy that despite a lowering of the exterior heating, the reaction mixture heated up by itself within a few minutes and maintained this elevated temperature level for several hours. Upon review, it can merely be said that apparently, a repetition of the experiment had neither been planned nor was it executed at the time, possibly because the results had been unsatisfactory and did not comply with the aim of titanium displacement catalysis.

This assessment of the situation was later confirmed by Karl Ziegler during a deposition conducted in 1967 [52] in connection with a patent litigation involving Ziegler, Du Pont, and Natta, and/or in 1969 [53] between Montecatini, Dart Industries Inc., Chevron Chemical Co. and Enjay Chemical Co., Humble Oil and Refining Co. and Avisun Corporation regarding the subject of polypropylene. The person asking the question, Mr. Irons, US patent attorney for Montecatini, was actually inquiring as to whether an experiment using titanium tetrachloride had been conducted prior to August 1954 (he presumably meant in connection with polypropylene). Ziegler corrected him by saying that, in his opinion, Irons obviously meant 1953, and denied that any experiment had been conducted prior to August 1953 during which the formation of polyethylene had been observed. Mr. Irons con-

7) Almost 40 years later H. Martin and co-worker [51] found in connection with other activities that pure aluminum triethyl and also other aluminum trialkyls converts ethy-

lene into high molecular weight solid polyethylene if the temperature is lowered to room temperature. The reaction is then very slow, but the displacement reaction is suppressed.



Fig. 1.2 Erhard Holzkamp, Heinz Breil and Heinz Martin

firmed this view [54] saying that by the end of July 1953, “you had not yet made a high molecular weight solid polyethylene.”

After the August 1953 vacation period, the repetition of an experiment using chromium acetylacetonate together with aluminum triethyl and ethylene [55] was being reported. Breil recovered small amounts of “soft paraffins”, and when using molybdenum acetylacetonate, found “paraffins of medium consistency plus a minor quantity of butene” [56]. At the beginning of October, the influence of vanadium acetylacetonate was being investigated, whereby some polymers (paraffins) were recovered along with butene [57–59]. Manganese acetylacetonate catalyzes the formation of butene [60], as does platinum acetylacetonate [61].

On October 26, 1953 [62], Breil used zirconium acetylacetonate together with aluminum triethyl and ethylene. Of the converted ethylene 90% now consisted of solid products: 38 g of “hard paraffin, polyethylene”. The triumph was complete. The product was pressed into an initial sheet (100–300 atmospheres, 130–150 °C Fp). On November 13, 1953 [63], Breil repeated this experiment, this time however, in the presence of hexane as the reaction medium. While, from a later viewpoint, this proved to be trivial, at the time it represented a significant variation of the process. The surface availability of the heterogeneous catalyst was being enhanced. Up to that time, reactions involving organoaluminum compounds had been carried out practically free of solvents. The yield of solid, finely powdered polyethylene, was tripled and stressed bands were tested for tensile strength (30 kg/mm²). An infrared spectrum failed to indicate the presence of methyl groups. Three days later [64], propylene was used for the first time in place of ethylene with the same catalyst. “No solid, but only liquid products, dimerisate” were obtained. Thus, the concept of polymerizing propylene in place of ethylene existed on paper, but could not be reproduced in practice [65].

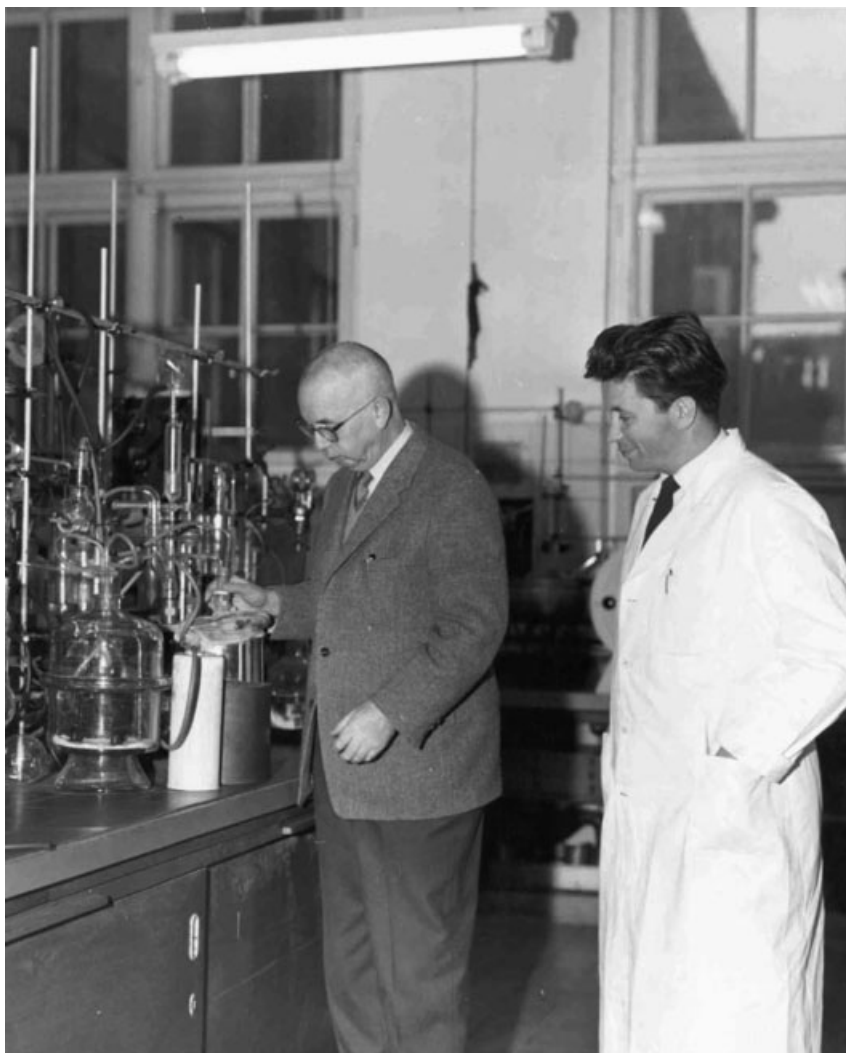


Fig. 1.3 Karl Ziegler and Heinz Martin at work in the laboratory

During the months of May through December, 1953, the three Italian visitors, and/or one of them⁸⁾, maintained written contact with their, and/or his, supervisors, Orsoni and Ballabio, both engineers at Montecatini in Milan. The Max-Planck-Institute and Ziegler had no knowledge of these reports, but they were uncovered during the course of subsequent litigations. While the letters written between May and July,

⁸⁾ In August G. Natta had suggested, that after the summer vacation, only one chemist, R. Magri, should return to Mülheim [82].

1953 [66-80] dealt with the dimerization of olefins, as covered by the agreement, including dimerization with added colloidal nickel, as well as the production of the starting materials (06/17/1953, page 3 “Ziegler Processes” [72]), Magri, in a letter dated 11/15/1953 [81], reported to Milan that he had learned from certain co-workers of Karl Ziegler – no names were mentioned – that during the course of investigating the influence of different “metals” on the reaction of aluminum triethyl and ethylene, a “high polymer of the polythene type had been obtained.”

He had afterwards seen the product for himself and described its properties as “true and genuine polythene.” He also conveyed some further information he had received at the Institute, according to which one of the polyethylene samples had purportedly resulted from an experiment conducted with aluminum triethyl in the presence of traces of zirconium at 100 bar and 100 °C.

On November 16, 1953 [83], K. Ziegler filed, in the German Patent Office in Munich, a patent application, which he himself had drafted, entitled “Process of the Polymerization of Ethylene” and was granted a priority date of November 17, 1953. The application claimed “a process for the production of a high molecular weight polyethylene useful as a plastic”, using “mixtures of aluminum trialkyls with salts of the metals titanium, zirconium, hafnium, vanadium, niobium, tantalum, chromium, molybdenum and tungsten as polymerization catalysts” and specifying “ethylene at pressures of more than 10 atmospheres and temperatures above 50 °C as process characteristics.” The claim was limited to the production of polyethylene. At the time, no practical results had as yet been obtained with the use of propylene and higher olefins towards the formation of solid polymers.

On November 27, 1953 [84], encouraged by the activity of the zirconium acetylacetonate in connection with ethylene polymerization, Breil experimented with a mixture of aluminum triethyl and titanium tetrachloride in the presence of hexane as the reaction medium. The polymerization reaction proceeded at a rate of 90% conversion of the starting ethylene. As compared to zirconium acetylacetonate, this experiment, conducted at only slightly elevated temperatures and an ethylene pressure of 55 atmospheres, was highly exothermic in nature, and the resulting product was black.

Around the same time, H. Martin sought to produce larger amounts of polyethylene by using the zirconium acetylacetonate/aluminum triethyl mixture [85] as well as the titanium tetrachloride and aluminum triethyl combination [86]. Not only did the result, 200 and/or 900 g of polyethylene, confirm past findings, but it also clearly demonstrated that, for the first time, the catalysts were so highly active that polymerization of ethylene at room temperature and normal pressure now appeared possible. After a few days, ethylene polymerization in a three-necked flask and later in a 5-l jar [87] was being undertaken as a standard experiment. The number of guests being taken by surprise by these amazingly simple procedures increased rapidly, and the news spread quickly and uncontrollably out of the Institute. Any disclosure regarding the composition of the catalysts was, however, scrupulously avoided. On December 12, 1953, Ziegler filed an addition application [88] in the German Patent Office, which, again, he had drafted himself. This time, the claims were directed to a method using an ethylene pressure of 1 atmosphere and lower temperatures above –20 °C. The Patent Office accorded the application a filing date of December 15, 1953.

Further results then followed in quick succession. Martin was successful in polymerizing ethylene with ethyl aluminum chlorides, particularly diethyl aluminum chloride, but also ethyl aluminum sesquichloride and ethyl aluminum dichloride in place of aluminum triethyl as a component of the catalyst, thus broadening the range of available catalysts [89–94]. This was a significant achievement, since dialkyl aluminum chloride compounds alone were not suitable for the growth reactions⁹⁾ with ethylene [95]. At the same time, Breil gained his first insight into the reaction products resulting from a conversion of titanium tetrachloride with aluminum triethyl, i.e. the catalyst components [96–98].

In mid-December 1953, Breil attempted to repeat the propylene polymerization process [99]. In preparing the catalyst mixture, he replaced aluminum triethyl with aluminum trioctyl and zirconium acetylacetonate with titanium tetrachloride. Of all the experiments from the early days in the historical development of the Ziegler catalysts, it was this one that was to be minutely scrutinized, commented on and criticized by experts, chemists and lawyers for the next 25 years. This experiment elicited the entire spectrum of judgments and opinions, some in written form, from “fall out” to “polypropylene must have been present”.

There was no doubt that the product was characterized as “polyethylene, sticky” and furthermore that propylene was initially pressed onto the catalyst mixture. The absence of a reaction, recognizable by a fall in pressure and/or rise in temperature, caused Breil, after “blowing off” a portion of the original propylene, to test the efficiency of the catalyst by adding more pressurized ethylene. Even then, “there was only a minor pressure drop after shaking.” A weight balance of recovered “polyethylene” on the one hand and the amount of ethylene and propylene introduced on the other should have led to the conclusion that propylene had been polymerized to a solid product, which must have been present either as a mixture of pure polypropylene and pure polyethylene or as a co-polymer [100]. At the time, however, no one sought further clarification of the result. The intention was rather to fully and completely devote all available time to the development of the catalysts and their effect on ethylene. Fourteen years later (in 1967), H. Martin repeated the first part of this experiment – catalyst mixture and propylene [101]. With spontaneous heating, the experiment produced more than 90% solid, high molecular weight polypropylene.

1.2.2.1 Montecatini Points to the Contractual Rights and Obligations

Inspired by Mr. Magri's reports and his visit in late-November 1953 [102], Mr. Orsoni [103] inquired whether the production of “high molecular weight ethylene polymers” would be covered by the scope of the January 1953 contract, based on the state of the discovery and crucial finding. As bait and in order to attain the information on the catalysts quickly, he mentioned the possibility of pushing for the development of the “polymerization of ethylene ... on a major industrial scale in Italy”.

Shortly before Christmas 1953, R. Magri, the last remaining guest from Italy, approached Ziegler and asked for a definite declaration regarding the nature of the new catalysts and to have them incorporated into the existing contract with Monte-

9) See p. 9 + 10

catini. Ziegler put him off, but handed him the two patent applications [83, 88] that he had meanwhile filed, with a request to pass them on to Mr. Orsoni. In his letter of January 6, 1954 [104] to Mr. Orsoni – receipt of which was acknowledged by Orsoni in his response dated January 25, 1954 [105] – Ziegler intimated that “an extensive field ... had been opened up.” In the same letter, Orsoni invited Ziegler to visit him in Milan. For the first time, it appeared that Ziegler himself must have had scruples and doubts regarding his own conduct, because in the last paragraph, he suggested:

“I trust that you and I are in complete agreement that any further development of this group of new catalysts should remain, for the time being, exclusively in our hands.”

and

“Surely you will appreciate that it is very important to us that we ourselves initially exploit our new discoveries for maximum benefit. Actually, my request to you that the completely undisturbed progression of this work be left exclusively to us for the time being, should be self-evident within the framework of a partnership such as it exists between you and us.”

Would Ziegler have been in breach of the agreement, if he had delayed passing on the most recent results for 6 months? Secure ownership of the new discoveries and the inherent unique chance of being in possession of a guaranteed commercially viable product and working with this monopoly was certainly the pivotal motivation for seeking out a strong industrial partner even at this early stage.

1.2.3

Experiments from December 1953 through April 1954

Not only did Martin’s December experiments aim to produce the optimum activation of ethylene with the aid of ethyl aluminum chloride compounds and titanium tetrachloride at normal pressure and low temperatures, leading to greater diversity within the catalysis field, but this class of catalysts also became extremely important particularly in the commercial production of polypropylene that was to follow later. The diethylaluminum chloride/titanium tetrachloride catalyst system was then used to verify the formation of ethylene/propylene co-polymers [106], to successfully test the polymerization medium – higher molecular weight saturated hydrocarbons [107] – and the co-polymerization of ethylene with dienes [108] and, for the first time, to convert butadiene¹⁰⁾ into a polymer with the aid of the aluminum trialkyl/titanium tetrachloride catalyst combination, even though the latter results were less favorable than those obtained with ethylene [109].

The same polymerization activity was observed when the chlorine in the diethylaluminum chloride was replaced with alkoxy groups [110, 111], such as methoxyl, and the compound employed in combination with titanium tetrachloride. Catalysts

¹⁰⁾ See p. 74

formed on this basis would rise to prominence as so-called “high speed” catalysts 30 years later when they were used in the polymerization of propylene (see p. 226).

During the reaction of diethylaluminum chloride and titanium tetrachloride, a fine, brown-colored powder deposits in the reaction vessel, which, on its own, is virtually inactive with respect to ethylene, but in combination with fresh diethylaluminum chloride constitutes an extremely effective catalyst combination useful for converting ethylene to solid high molecular weight polymers [112, 113]. This powder was determined to be titanium trichloride¹¹⁾, to which poorly adsorbed aluminum compound was still attached. A further reduction of the titanium does not take place under these conditions. This finding, of course, begged the question whether the purple, commercially available variety of titanium trichloride was also effective. Indeed, in combination with diethyl aluminum chloride [114] as well as aluminum triethyl [115], it was capable of converting ethylene to solid high molecular weight polymers, albeit at a slower rate than had been previously observed.

A similar result was obtained when titanium trichloride was replaced with titanium dichloride; thus, aluminum triethyl or diethyl aluminum chloride in combination with titanium dichloride led to the formation of active polymerization catalysts [119]. Violet titanium trichloride and black titanium dichloride were highly crystalline starting products.

At that time, Breil discovered that the product consisting of titanium tetrachloride and aluminum triethyl would have to be a partially hydrocarbon-soluble, black-colored, low-valence titanium halide compound. The composition led him to conclude that the product could not be a uniform compound, but that the titanium, in large part, was present in divalent form. The insoluble moiety alone was effective as a catalyst for the polymerization of ethylene [120], which, as already mentioned, was not true for titanium trichloride (see also Chapter 5, footnote 45).

Breil was furthermore able to show that effective catalyst mixtures could also be obtained, on the one hand, by replacing the aluminum trialkyls with metal alkyls such as magnesium dimethyl [121], Grignard compounds [122] as well as zinc diethyl [123], sodium phenyl [124] and lithium butyl [125] and also, as was to be expected, sodium aluminum tetramethyl and sodium aluminum tetraethyl [126], but also, unexpectedly, lithium hydride [127] and lithium aluminum tetrahydride [128]. On the other hand, titanium tetrachloride could be replaced with compounds of transition metals from subgroups IV, V and VI of the Periodic Table, particularly the halides, in addition to nickel chloride [129], iron chloride [130, 131], and manganese chloride [132]; the latter three, however, were effective only if combined with diethylaluminum chloride, (reduction not to titanium metal) but did not work in combination with pure metal alkyls (displacement catalyst in the dimerization of ethylene). Tungsten hexachloride [133], molybdenum pentachloride [134] and chromium trichloride [135], on the other hand, could be activated only when combined with alu-

11) Amorphous (β)-titanium trichloride is an unstable modification or a mixture of various modifications. The “brown” powder described was indentified by X-ray analysis as partly crystalline [116]. The powder as well as β -titanium trichloride is transformed into the

stable violet, pure crystalline α -titanium trichloride by longer tempering. Only about 4 mole% of the brown titanium trichloride [117] (measured surface 240 m²/g) is available for adsorption with diethyl aluminum chloride [118].

minum trialkyls for the conversion of ethylene to high molecular weight polymers. However, only part of these experiments were included as examples in subsequent patent applications, a disadvantage which would hamper further patent claim delineation.

1.2.4

Letter from Ziegler to his Patent Attorney von Kreisler

On January 7, 1954, Ziegler asked Dr. A. von Kreisler [95] to represent him as his patent attorney before the German Patent Office and sent him copies of the patent applications, drafted by himself, which had been filed in November and December of 1953. When explaining the situation, he pointed out that “the recent newly discovered catalytic processes no longer have any immediate connection to our earlier reaction processes.” By this statement he meant that the previously studied catalytic effects were limited to “genuine aluminum trialkyls”, whereas the finding that alkylaluminum chlorides with titanium tetrachloride were also useful as effective catalysts was a “complete novelty.” From this he concluded that the “real catalysts were ... most likely organometallic compounds of the added heavy metals with unusually low valencies.” This was a newly gained insight into polymer chemistry. Accordingly, he asked von Kreisler to prepare a further patent application directed to the use of alkyl aluminum halides and those of the general formula R_2AlX .

In this context, he mentioned later on in the same letter a prior patent application by BASF, Ludwigshafen [136] (actually, it was a patent), the disclosure of which led him to arrive at a serious conclusion:

“The object of this application is undoubtedly a forerunner of our new processes. Except the inventors failed to recognize what they had in hand. For the polymerization of ethylene, they used a mixture of aluminum chloride, metallic aluminum, titanium tetrachloride, and, besides oily products (see p. 2, line 85), obtained perhaps a 40% yield of a polyethylene material which had characteristics similar to our own products. The inventors added the aluminum powder to bind hydrochloric acid (p. 1, line 20, and p. 2, lines 1–2). Actually, however, a combination of aluminum metal, aluminum chloride and ethylene at higher temperatures regularly leads to the formation of genuine organoaluminum compounds; this result has even found its way into the literature. The true mechanism of the process according to the BASF patent, thus, consists in the primary formation of a small amount of ethyl aluminum sesquichloride from aluminum chloride, aluminum metal + ethylene (the reaction does not completely tally, and a whole line of dehydrogenation by-products are also formed), and these genuine organoaluminum compounds, in combination with the titanium tetrachloride, then lead to the solid polyethylene, the same as we have obtained at the Institute. The inventors, of course, failed to see this connection.”

In the course of subsequent adversary proceedings, especially in the United States, this letter was disclosed to opposing counsel and, thus, moved into the public domain. As was to be expected, the opponents used Ziegler's letter as an admission that the Ziegler catalysts were covered by this BASF patent and, therefore, were no longer patentable.

As late as mid-1955, when assessing the same BASF patent in his doctoral thesis [137], Breil wrote that the "white powder" as described in the patent, "obviously, is a polyethylene of the same type as my polyethylenes." And regarding the catalysts he went on to say:

"It seems obvious to me that in experiments of this type, a catalyst of the type of my catalysts would be formed. The inventor of the patent failed, however, to recognize the essential nature of his process. The polymerization catalyst is formed either by Al, AlCl₃ and ethylene combining to primarily form organoaluminum compounds (see C. Hall, A.W. Nash, J. Inst. Petrol, Technol. 23, 679 1937 and 24, 471 1938), which react with titanium tetrachloride, or the metallic aluminum directly reduces the titanium tetrachloride."

Neither of these statements was supported by any experimental data. It was not until 1958, and continuing intermittently through 1992, in between long intervals, that experiments were conducted at the Max-Planck-Institute at Mülheim under scientifically exact standards, to test the mode of action of the catalyst described by Max Fischer in the BASF patent. Not only were the above-described first impressions found in need of extensive revision, but, based on these experiments, it was also subsequently possible to identify the intermediate compounds and end-products of the Max-Fischer-catalyst [138–140]. But more on this later.

Back to mid-January 1954: von Kreisler filed the next two patent applications in the German Patent Office, the first of which was directed to the use of, among other compounds, alkyl aluminum halides in combination with transition metal compounds [141], and in the second application the aluminum compounds were replaced by magnesium alkyls [142].

1.3

Montecatini, Milano/G. Natta and Co-workers

1.3.1

The First Experiments with Ziegler catalysts

Based on the disclosure of the first two patent applications which had been divulged to Montecatini/Natta between late December and early January 1954, as well as three additional Ziegler patent applications [143], Ziegler's work was being discussed by Natta and his co-workers during January and February [144]. Magri translated the first two patent applications together with Chini and Crespi. Copies of these docu-

ments were made available to Dr. Varda, the Head of Montecatini's Patent Department, and engineer Orsoni [145], who was in charge of the Technical Development Section. On February 9, 1954, Chini began to repeat certain of the experiments described by Ziegler, starting with Example 2 in combination with Example 4 from Ziegler's second patent application [88, 146, 147]. He polymerized ethylene with a catalyst mixture of aluminum triethyl and titanium tetrachloride at atmospheric pressure, resulting in solid, crystalline, high molecular weight polymers. He was struck by the great ease of the polymerization process. While the results of these reproduced experiments had been discussed at the time with both Natta and Orsoni, no such discussions took place with Ziegler or his co-workers.

At that time, P. Pino and G. Mazzanti also worked under Natta. Pino supervised and conceived parts of the experimentation program, and Chini, Mazzanti, Longi, Angelini and Giachetti were Pino's co-workers [148].

On March 8 and 9, 1954, while passing through on his way to Southern Italy, where he planned to spend his vacation, Ziegler accepted an invitation from Montecatini to stop in Milan, and he met Natta and Orsoni. At the hosts' request, the discussion was recorded [149] and signed in the form of a contractual arrangement entitled "Cooperation between Professor Ziegler and Montecatini", that is, the content was legally acknowledged. The Memorandum, dated March 9, 1954, regulated the division of labor regarding future developments as between Ziegler's Institute, on the one hand, and Natta/Montecatini on the other.

Ziegler:

"b) Exploration of new mixed catalysts for the polymerization of olefins (particularly ethylene) based on the principles of production of the mixed catalysts described in the prior Ziegler polyethylene applications."

Natta:

"a) Production of branched-chain polymers resulting from the action of aluminum alkyl contacts ... on substances containing interchangeable active groups."

"b) Kinetic study of the chain elongation reaction of ethylene ... and"

"c) X-ray analysis of the structures of the polyethylene materials obtained from various different production methods."

Further assignments were to be carried out at Montecatini's Ferrara plant:

"d) Production of high molecular weight polyethylene on a technical scale, ..."

"f) Production of alpha-butene ..."

"g) Technological running comparison of the end polymers."

The last paragraph of the Memorandum explicitly provided for Ziegler that

"any investigations involving the new catalysts and regarding the manipulation of the polyethylene molecular weight should initially be handled exclusively by him and his organization."

Ziegler additionally told his contract partners of the successful co-polymerization of ethylene and propylene [150].

Two days after the meeting, on March 11, 1954, Natta instructed his co-worker, Chini, to use propylene while otherwise using the exact same parameters as employed in previous catalyst preparation. The propylene was added to the Ziegler catalyst initially at room temperature and atmospheric pressure [151], and later at elevated pressure. Chini recovered 3.5 g of solid polypropylene. The internal designation, according to a statement by Corradini, was a “Ziegler–Chini” product [152]¹²⁾. The following day, a shaped piece of the polypropylene thus obtained was stretched up to 500%, and an X-ray image revealed the typical characteristics of a crystalline polymer [153]. The raw product was then fractionated by solvent extraction and the insoluble fraction presumed to be crystalline. Pino instructed Mazzanti to work together with Chini on the experiments [154]. On March 15, Mazzanti and Chini together made a second attempt [155] at polymerizing propylene and performed a third test on March 24, 1954 [156], again based on Ziegler’s first patent application. In the latter experiment, propylene pressure was applied from the very outset. The details of this test were recorded in Chini’s laboratory journal, but not in that of Mazzanti. Further experiments were conducted on March 26, April 5, and May 26, 1954 [157, 158].

As early as March, Natta compiled all the data thus far available on propylene polymerization as well as the findings previously obtained regarding ethylene polymerization and forwarded them to the Patent Department at Montecatini [159].

The sequence of events which shaped the early history is described here in such detail, because it was the very results obtained from Chini’s initial experiments which formed the basis for Natta/Montecatini’s assertion that they were the first to “independently” produce high molecular weight polypropylene. All of the involved individuals at Milan were well aware that they had deliberately breached the agreement made with Ziegler and that Montecatini [160] “invade(d) a sphere of polymerization with our tests which Ziegler had explicitly reserved for himself when giving us advance notice of the discovery of his co-catalyst.” and that Ziegler

“might one day request the restitution of the invention on the strength of illegal derivation.”

and furthermore, that the possibility of polymerizing propylene would not have existed without the detailed information supplied by Ziegler [161].

Chini:

“I expressed in my opinion, which is an opinion that Professor Ziegler should have been so mentioned in the patent.”

“I believed that if we did not have the information about the polyethylene preparation we would not have been able to prepare the polypropylene.”

12) Corradini had prepared the X-ray images.

1.3.2

Natta, Orsoni and DeVarda's Visit to Ziegler at Mülheim on May 19, 1954

The preparation, execution and results obtained from the experimental program implemented by Natta and his co-workers at Milan—as is evident from the sequence of events – were neither disclosed to either Ziegler or his co-workers nor discussed with them. Up to the end of July/beginning of August, 1954, Ziegler had no inkling of what had meanwhile occurred there. Even when Natta, Orsoni and DeVarda came to Mülheim on May 19, 1954 [162] to visit Ziegler [162], nothing was said to Ziegler on that subject. This was with full intent on Montecatini's part.¹³⁾ As had become apparent during an earlier internal discussion at Montecatini, the individuals involved wanted to avoid triggering a confrontation with Ziegler based on a patent application, filed solely in Montecatini's name and directed to the production of polypropylene, without knowing whether Ziegler had already previously polymerized propylene. They decided to find out during this visit to Ziegler [163]:

"... it would have been quite a complicated matter if by filing our patent we would have found that Ziegler had made ahead of us the polypropylene -the invention of polypropylen. Now, these doubts were clarified in May 19th (1954) when we went to Mülheim and Ziegler volunteered the information not only that he had tried to polymerize propylene, but that he had not succeeded, and therefore he thought it would not work. I mean the catalyst he had employed or had used would not."

Further a notice by DeVarda [162]:

*"He [Ziegler] did say "it does not go. It does not run. It does not succeed." ...
... words he [Ziegler] used and I remember them absolutely exactly "Polypropylene does not work."*

According to his recollection [164], Ziegler's response to a casual question as to whether he had also polymerized propylene was:

"No, not up to now, but we have successfully co-polymerized ethylene and already successfully co-polymerized ethylene and propylene."

13) In a later deposition (for this reason the court had moved to Milan in 1969) G. Natta was asked why he did not inform Ziegler about his experimental results: "I did not tell him because I had to take patents first."

In March 1970 G. Natta was deposed again as a witness. He presumed to declare that his question was whether or not somebody should try to polymerize propylene, Ziegler's reaction was: "Propylene could not polymer-

ized." "If I have not been able to succeed, nobody would succeed." [147]

The Nobel-price laudatio for G. Natta does not mention Natta as the inventor of polypropylene, but indicates that Natta found that certain types of Ziegler catalysts led to stereoregular macromolecules, a diplomatic détourné of the conflict between Ziegler and Natta.

The visitors from Italy believed that Ziegler's attitude justified their conclusion that to file a patent application quickly claiming the polymerization of propylene with the use of a Ziegler catalyst was entirely within the bounds of legal propriety [162]. This Montecatini application [165] was accepted by the Italian Patent Office in Rome on June 8, 1954. It was directed not only to the method of production [166] – the polymerization of propylene using Ziegler catalysts: TiCl_4 and AlEt_3 – but also to the high molecular weight polypropylene products, the structure of which was determined, *inter alia*, by X-ray structural analysis. G. Natta was named as the sole inventor. The first four examples of this patent application corresponded to the above-described experiments of March 15 and 26, April, and May 4 and 26, 1954. The first Chini experiment was not included in the application, nor was Chini later named as a co-inventor [161].

On July 27, 1954, a second application [167] was filed, claiming enhancement of the regular structure of the solid, crystalline polypropylene due to the presence of asymmetrical carbon atoms, and similar polymers from olefins with four or more carbon atoms. The catalysts disclosed in the specification were, again, Ziegler catalysts. Initially, no inventors were indicated, but the applicant subsequently named G. Natta, P. Pino and G. Mazzanti. The propylene polymers, as described in both of these applications, were of the same kind [168], and even the product obtained from Chini's first experiment was encompassed by the claim of the first application [169].

As it turned out later (see p. 118), Montecatini's inconsistency in naming the inventors, on the one hand, and its assertion, on the other hand, that the polypropylene products according to both applications were similar and also corresponded to the product obtained from Chini's first experiment, would prove disastrous from a standpoint of patent law. The issue of what Pino and Mazzanti had contributed to the invention was never fully resolved.

It should be reiterated here that neither had Ziegler been informed nor his consent been requested in regard to the filing of these applications. Ziegler first learned of the fact that Natta's team had polymerized propylene from Orsoni's letter [170] of July 30, 1954, which stated:

"During our meeting on May 20 at Mülheim, you declared that propylene and styrene did not lend themselves to the formation of superpolymers. This assessment took us somewhat by surprise, since even then we were convinced that we had been successful in producing at least the superpolymer of propylene."

A few days later Natta also contacted Ziegler [171], just after the latter had sent to Milan on July 21, 1954 [172] both an initial sample of the high molecular weight, solid polypropylene produced at Mülheim (see p. 30 "July 1954, Polypropylene") and a pressed sheet made of polypropylene. In his accompanying letter, Ziegler intimated that a systematic testing program focusing on the polymerization of alpha-olefins, styrene, methyl styrene, butadiene as well as the halogenated ethylenes and possible mixed polymerizations had been set up.

Through X-ray analysis, Natta observed that these polypropylene products manifested a 45% crystalline content:

"We had already obtained similar products last March."

Natta further reported that other polyolefins, such as poly-alpha-butene and polystyrene, exhibited similar structures as the polypropylene.

Later on, Montecatini's US patent attorney, Harry Toulmin, writes [173]:

"An issue that I have long feared might be raised in connection with the question of what Professor Natta contributed over Dr. Ziegler and therefore whether Professor Natta was a genuine inventor has now been precipitated by the attached editorial."

"Therefore, Natta, using the exact catalyst of Ziegler produced polypropylene in his early work. It was not until later that he began to be selective in his selection of the catalyst."

"Dr. Orsoni, in one of his communications, indicated that he thought, as we understood him, that you could avoid this situation because of the selection by Natta of a special catalyst, but unfortunately, in the early invention, which was fundamental, Professor Natta used the exact catalyst of Ziegler, and it was not until later that Natta began his selectivity."

1.4

K. Ziegler and Co-workers

1.4.1

H. Martin: Experiments May–July 6, 1954; H. Breil: Masters Thesis

The main focus which gave direction to the series of experiments conducted at Mülheim between May and mid-July 1954 was established by the fact that upon determining the molecular weight of the polyethylenes produced, very high values (several hundred thousands up to several millions) had been ascertained. Processing on conventional extruders was not possible. The solution to the problem presented itself in the finding that the molar ratio of the components employed in the preparation of the catalyst appeared to be crucial in determining the chain length of the polyethylene produced. This finding was confirmed and re-confirmed through a large number of experiments, whereby products with molecular weights between 20,000 and about 1 million could be easily synthesized [174]. Relatively short-chained products were more easily moldable than products with molecular weights in the several hundred thousands range. Highly crystalline polyethylene products with molecular weights between 50,000 and 80,000 could then be processed in standardized machines by established methods and formed into fibers, injection molding and sheet products.

By varying the catalyst composition, not only could the chain length be controlled, but the activity (conversion of gram ethylene/gram catalyst) was also affected. With a decrease in the amount of organoaluminum compound per titanium component used, conversion increased dramatically.

All in all, this fruitful period not only opened up new possibilities for the practical application of low pressure polyethylene, which could now be produced with extreme ease, but for the first time it also became apparent that the catalysts, depending on the method used for their production, showed great selectivity in their effect.

On June 28, 1954, H. Breil presented his Masters Thesis [175] "On Organometallic Mixed Catalysts" to Bonn University. It contained a description of his previous experiments, provided a summary of the effectiveness of the catalysts being explored, and supplied indications which helped clarify the reaction mechanism of the new polymerization catalysts:

"4. We were able to establish that the polymerization of ethylene to produce high molecular weight 'polyethylene' involves an entirely new reaction mechanism, and that the organoaluminum compounds represent merely auxiliary components useful in the production of the catalyst."

"8. It is now conceivable that the new polymerization catalysts contain low-valent metal compounds of the heavy metals, the valencies of which are, in part, saturated by organic radicals."

1.4.2

H. Martin, July 1954: Polypropylene

During the second week in July, Martin co-polymerized [176] ethylene and propylene by contacting the monomers with an aluminum trialkyl/titanium tetrachloride catalyst in hydrogenated diesel oil, thus reducing to one-sixth the amount of aluminum in relation to the amount of titanium, as compared to the January 7, 1954 [106] experiment. On July 13, 1954 [177], the turbulent polymerization that occurred upon introducing ethylene into the propylene-saturated reaction mixture, persuaded him to use propylene alone at a low pressure of between 6 and 12 bars under otherwise identical conditions. The reaction product obtained was a thick, black sludge. Of solid polypropylene 160 g was recovered. It was obvious that the raw product contained low molecular weight fractions. At a temperature of 135 °C, it was at least possible to draw filaments from the opaque molten mass.

The components of the catalyst were similar to those employed by Breil in December of 1953 [99]. Prior to the actual polymerization, the components reacted with each other for 2 h at room temperature in a solution and/or suspension medium of hydrogenated diesel oil. The amount of organoaluminum compound relative to the titanium tetrachloride was reduced to one-fifth and the propylene subsequently pressed on had a high degree of purity. (Contact with organoaluminum compound had rendered the propylene free from traces of oxygen and moisture). The polymerization reaction then took place in the presence of additional hydrocarbon diluting agents. Whether the relatively minor differences outlined with regard to the reaction procedure were responsible for the clearly positive results now obtained could not be determined with certainty.

When the experiment was repeated using aluminum triethyl as the aluminum component, the polypropylene yield was more than doubled [178], and with the use of diethylaluminum chloride almost tripled [179]. In the same manner, n-butene [180] and styrene [181] were converted to solid polymers.

The new findings constituted the basis of a further patent application [182], which was filed in the German Patent Office on August 3, 1954. Since the German Patent Law, at the time, did not provide for product protection, the new polypropylene was described in the specification only, without being included in the claims, on the one hand in terms of its characteristics:

“colorless, solid, plastic polypropylene, capable of being formed into transparent sheets and plates at temperatures above 140°C”

and, on the other hand, in terms of the “fingerprint” of the infrared spectrum. The spectrum left no doubt that the product had a high crystalline content. Accordingly, the claims of this patent application were directed to a process for the polymerization and co-polymerization of olefins.

1.5

1952–1954 Polypropylene (Review)

1.5.1

Appraising the Historical Course of the Invention from a Year-2000 Vantage Point

The second half of the 20th century witnessed an incredible stimulation not only in polymer chemistry, but in particular in the chemistry of heterogeneous and homogeneous catalysis as a result of the inventions of Ziegler and his co-workers (Ziegler catalysts and their application). The hallmark of the enormously vast number of publications, biographies, and issued patents generated during that period is infinite their variety.

On December 10, 1963, His Majesty, the King of Sweden presented Karl Ziegler with the Nobel Prize for Chemistry [183], which he shared with Giulio Natta. In his Presentation Speech, Professor A. Fredga [184], Member of the Nobel Committee for Chemistry of The Royal Swedish Academy of Sciences, states, *inter alia*:

“However, Professor Ziegler has found entirely new methods of polymerization..... The combination of aluminum compounds with other metallic compounds gives Ziegler catalysts. These can be used to control polymerizations and to obtain molecular chains of the required length. However, many systematic experiments – and indeed some accidental findings - were necessary to reach this stage. Ziegler catalysts, now widely used, have simplified and rationalized polymerization processes, and have given us new and better synthetic materials. Your excellent work on organometallic compounds has unexpectedly led to new polymerization reactions and thus paved the way for new and highly useful industrial processes.”

The added remark “and indeed some accidental findings” calls for an explanation. During the past 40 years, the media in particular, have classified the invention of the Ziegler catalysts as a so-called “chance invention”. This characterization was to convey to the layman that by no means did the researchers work towards the invention in a goal-oriented manner, but rather that they had accidentally stumbled across the results obtained while exploring a side-road. It was, however, easy to see that the goal of adding ethylene to the Al-C bond with any desired chain length had existed from the start, and since this was continually borne in mind by the researchers, a solution had been pursued in a methodical manner. Moreover, finding a solution was possible only for researchers who were dealing with the production and properties of metal alkyls. These two prerequisites imposed such narrow limitations on both the number of individuals and the location that the word “chance” could only have meaning if within these groupings, the interaction between metal alkyls and transition metal compounds was discovered. Admittedly, the point in time when this was made possible was propitious, since observations had been documented worldwide, even though coming from a different direction and were rather weak in their effect. After all, the discoveries made by Ziegler and his co-workers in this connection occurred within only a few short months and/or days as similar or identical results became available at Du Pont.

Professor Natta’s contribution was acknowledged by Mr. Fredga, when he stated, *inter alia*:

“The individual molecules strung together to form polymers are often so built that the resulting chain exhibits small side groups or side-chains at certain points, generally one at every other carbon atom. But the picture is more complicated, since these side groups can be oriented either to the left or to the right. When their orientations are randomly distributed, the chain has a spatially irregular configuration. However, Professor Natta has found that certain types of Ziegler catalysts lead to stereoregular macromolecules, i.e. macromolecules with a spatially uniform structure. In such chains, all the side groups point to the right or to the left, these chains being called isotactic. How is this achieved when the microstructure of the catalyst is probably highly irregular? The secret is that the molecular environment of the metal atom, at which new units are stuck on to the chain as mentioned before, is so shaped that it permits only a definite orientation of the side groups.

Isotactic polymers show very interesting characteristics. Thus, while ordinary hydrocarbon chains are zigzag-shaped, isotactic chains form helices with the side groups pointing outwards. Such polymers give rise to novel synthetic products such as fabrics which are light and strong at the same time, and ropes which float on the water, to mention only two examples.

Nature synthesizes many stereoregular polymers, for example cellulose and rubber. This ability has so far been thought to be a

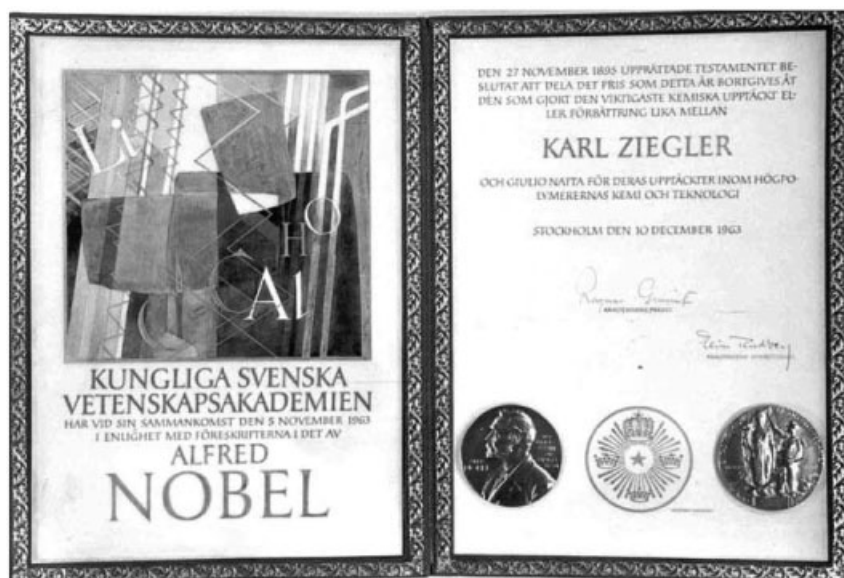


Fig. 1.4 The King of Sweden, Gustav VI. Adolf, presenting the Nobel Prize for Chemistry to Karl Ziegler on December 10, 1963; the Certificate is shown below

monopoly of Nature operating with biocatalysts known as enzymes. But now Professor Natta has broken this monopoly. Towards the end of his life, Alfred Nobel was thinking of the manufacture of artificial rubber. Since then, many rubber-like materials have been produced, but only the use of Ziegler catalysts enables us to synthesize a substance that is identical with natural rubber. You have succeeded in preparing by a new method macromolecules having a spatially regular structure.'

The last three paragraphs will be discussed in greater detail at a later point. Such attributions as are made are only in part backed up by the historical events surrounding the invention. For now, suffice it to say that the initial polypropylene polymers produced by Natta on the one hand, and Martin on the other, were structurally practically identical. The infrared spectrum showed that both products were crystalline in nature. But it was Natta who had specifically recognized that the structure of these polymers was of regular character. Later on, through the development of crystalline TiCl_3 preparations, he and others were able to gradually increase the crystalline content of the polymers, a further indication of the highly selective mode of action of certain Ziegler catalysts (see p. 25).

Any appraisal and evaluation of an invention must take into account whatever patents, priorities and infringement actions are associated with it, because – as has been shown – verifiable facts, relevant documents and legal arguments in support of factual findings are often more illuminating in terms of the course and historical analysis of such inventions than are the occasional opinions espoused by some scientists.

Thirty years after the invention, Judge C.M. Wright of the US District Court at Wilmington, Delaware, after having inspected reams of evidentiary material, paid tribute to the invention itself [185]:

"Through application of Ziegler's discovery (catalyst), Natta at Montecatini, Martin at the Max-Planck-Institute, and subsequently, many others were able to produce crystalline polypropylene on a commercial scale."

Two years later, on the issue of priority by Ziegler and his co-workers with respect to the process for the production of polypropylene, the US Court of Appeals for the Federal Circuit in Washington, D.C., held that [186]:

"It was Ziegler and his named coinventors who invented those catalysts and told Natta about them. It is here immaterial who was the first to use those catalysts to polymerize polypropylene."

The Judges wanted to make it clear, that the infringer had literally used a patented Ziegler catalyst to polymerize propylene. Who was the first to use the catalyst for the polymerization of propylene was immaterial in this context. This, however, presupposed that both parties had demonstrated that propylene could be polymerized. More on this later.



Fig. 1.5 Giulio Natta

Aside from Natta himself (in his initial patent applications), both Professor Fredga, Stockholm, and the quoted judges in their decisions spoke of Ziegler catalysts, particularly in connection with the production of polypropylene and/or polyolefins, rather than referring to them as Ziegler–Natta catalysts, as was erroneously done later by a number of writers [187]. The scientific world had ignored the accurate terminology, and had done so for various reasons which will be discussed at an appropriate point.

The battle for priority rights is waged more often than not on a less than highly moral or ethical level, especially where international scientific information is communicated on a confidential basis, and is not treated as such. Depending on the commercial significance of the invention, a controversy may ensue, as it did in the present case, that would last over a whole generation.

The fact that Natta shed light on the crystalline structure and chain configuration of stereoregular polypropylene products with the aid of X-ray crystallography did not change the properties of the polymers, but it was definitely a scientifically important, and brilliant finding¹⁴⁾. The subsequent illustration of the regular construction

14) Prior to this time C. Schildknecht, had for the first time, reported on the stereoisomer structures in crystalline polyvinylisobutylether [188]. Highlighting this publication P.J. Flory discussed the possibility of crystallinity

because of the stereoregularity in polymers [189]. In 1953 J.D. Watson and F.H. Crick presented the double helix structure of DNA [190].

of the molecules with the aid of a helix structure was certainly of gradual importance in terms of the utility of the materials. The dependent invention of polypropylene at Milan and the independent invention at Mülheim can be explained only with the understanding that the teams at both locales started out with knowledge of the Ziegler catalysts. Giulio Natta and his co-workers had received prior information on the catalysts from Ziegler and his co-workers. Natta had reported his findings to Ziegler after the results on polypropylene had become available at the Max-Planck-Institute.

Martin had the benefit of two experiences, first his participation in the development of the Ziegler catalysts, and second the negative results obtained by Karl Ziegler and his co-workers in connection with their initial attempts to recover solid polypropylene. In the final analysis, any discussion about the fact that these negative results caused Ziegler to believe that high molecular weight polypropylene could not be produced in this manner would be irrelevant.

Neither statement made outside of court nor anything gleaned from actual court testimony proved helpful in arriving at a decision. Only the presentation of verifiable facts was sufficient to determine the issue of priority. Montecatini itself was well aware of this situation. G. Natta could not be acknowledged as an inventor of polypropylene without “derivation” being mentioned. It is this factual situation that the answer to the question of who invented polypropylene must ultimately be reduced to.

A memorandum [191] prepared in 1989 by attorney Sprung, knowledgeable with respect to the legal and particularly the patent position¹⁵⁾ involved, finally summarizes the situation. This memorandum was the basis for a number of important, profitable ramifications for Studiengesellschaft and the Max-Planck-Institute. Part of this has already previously been addressed.

Aside from the patent law aspect, which may be illustrated, through numerous oppositions and heated court battles, by way of this intermediate conclusion, the economic side of the picture also deserves consideration. Up to this day, polypropylene is produced on an industrial scale exclusively with the use of Ziegler catalysts. Even though, as time has passed, the catalysts have undergone some improvements and, based on the events described above appear under different names, nevertheless, the nature of the processes involved has remained the same: the combining of a transition metal component, particularly a titanium chloride-containing component, with an alkyl aluminum-containing species is as essential now as it was then.

Eight years earlier, in 1980/81, the controversy centering on the question of who was the first to actually make the “polypropylene” product irrespective of the method of its production, had been resolved by the highest patent appeal court in the United

15) Dr. Martin, in his experiments done in July of 1954, produced such solid, flexible polypropylene, and this experiment appears to represent the first independent invention of the solid, flexible polypropylene. From a scientific and practical standpoint, while Natta et al. may have run an experiment in Italy and

obtained the solid, flexible polypropylene shortly prior to Dr Martin's experiment, the Natta work was not independent, as it was based on and depended on information obtained from a team working at the Max-Planck-Institute under Professor Ziegler, of which Dr. Martin was a member.

States. Ziegler/Martin were not involved in these proceedings¹⁶⁾. Each of the litigants – Standard Oil of Indiana, Du Pont, Montecatini and Phillips Petroleum – had an opportunity to document its story, its argumentation, and its contribution towards the genesis of the invention, and thus its claim of priority.

All relevant and, in the court's view, crucial documents were taken into consideration [3]. These have also been addressed and/or cross-referenced in the outline of the early history. The outcome is being anticipated here to assist the reader at this juncture to properly integrate and appreciate any relevant connections and adversary proceedings involved.

The court accepted the evidence as showing that, among the litigating parties, it was Phillips Petroleum and/or the inventors J.P. Hogan and R.L. Banks, who were entitled to the benefit of the January 27, 1953 priority for solid, crystalline polypropylene and, thus, that they had recognized the new substance ahead of any of the other parties involved. However, not only did this particular polypropylene have other characteristics which rendered it unsuitable for commercial exploitation, but it was also produced with catalysts not suitable for use from a technical standpoint. The court expressly stated that Phillips' documentary evidence did not mention use as a thermoplastic material, but that only the application as a wax was described in certain relevant documents. The material¹⁷⁾ was brittle, but partially crystalline.

While W.N. Baxter, at Du Pont, had produced polypropylene with characteristics comparable to the products according to Hogan and Banks prior to August 19, 1954, i.e. prior to Ziegler/Martin, they had not recognized the product as such and had also failed to show utility. These same findings were applied to A. Zletz, of Standard Oil of Indiana, with a date of October 15, 1954. Natta and his co-workers, at Montecatini/ Montedison, were denied priority (see p. 2), i.e. Natta's claim to the invention of "polypropylene" was rejected. The priority date of Ziegler's patent application was August 3, 1954, prior to Baxter and Zletz, but clearly after Hogan and Banks.

16) The conflict had started at an early stage in 1957, at a point in time when Ziegler was not ready to engage in substantial financial efforts. The priority patent application for polypropylene filed by Ziegler contained a product claim covering solid polypropylene. At least during the late 1980s a decision was made regarding this issue (see Section 5.9)

17) Memo A. Sprung, Lit 191: The product produced by Hogan and Banks was a brittle material, having no real commercial value or interest. Hogan and Banks' earlier work entitled Phillips to a broad patent claim covering solid polypropylene with a substantial crystalline portion.

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- 2 In the structure of the polymer chain uniform repeating propylene-sequences (head – tail polymerization or 1,2-addition).
- 3 494 Fed. Supplement, p. 370–461, 1981: Civil Action No. 4319, District Court of Delaware – Decision Jan. 11, 1980 Standard Oil of Indiana, Phillips Petroleum Co., E.I. Du Pont de Nemour & Co. vs. Montecatini S. p.A. et al, confirmed by the Court of Appeal, 3rd Circuit, 1981.
- 4 Phillips Petroleum Company, USA, US P 4,376,851 (SN 558,530, Jan. 11, 1956, Continuation-in-part of SN 333,576, Jan. 27, 1953, abandoned, and SN 476,306, Dec. 20, 1954, abandoned), J.P. Hogan, R.L. Banks, priority Jan. 27, 1953.
- 5 494 Fed. Supplement, p. 398, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980, Standard Oil of Indiana, Phillips Petroleum Co., E.I. Du Pont de Nemour & Co. vs. Montecatini S. p.A. et al, confirmed by the Court of Appeal, 3rd Circuit, 1981. Molybdenum oxide was reduced with hydrogen, 850 °F (455 °C) at 400 psi pressure, adding xylene the polymerization was tried (propylene pressure 420 psi (ca. 30 atm), temperature 302 °F (160 °C).
- 6 494 Fed. Supplement, p. 398, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980. Exp. Nr. EP-34 with the same catalyst von A. Zletz, 0.7 g solid polymers, 0.1 g of that insoluble in xylene, from Carmody described as ‘rubbery, non-tacky’.
- 7 494 Fed. Supplement, p. 400, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980. IR- (micron 8.63, 9.07, 10.05, 10.29, 11.12, 11.85, 12.35) (Affidavit G. Natta of Sep.13, 1956, unknown to the parties, IR of crystalline polypropylene: micron 10.02, 10.28, 11.89, as well as strong bands at 9.05, 11.12, 12.39).
- 8 W. Heinen, 1959, *J. Polymer Sci.*, 38, 545 ratio of 1171 cm⁻¹ to 846 cm⁻¹. (micron 8,84/11,82).
- 9 J.P. Luongo, 1960, *J. Appl. Pol. Sci.* III/9, 302, Fig. 1 ratio of 974 cm⁻¹ to 995 cm⁻¹. (micron 10.26/10.05).
- 10 494 Fed. Supplement, p. 398, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980. EP-35, with the same catalyst, yield 3.83 g.
- 11 494 Fed. Supplement, p. 399, 1981: Civil Action No. 4319, District Court of Delaware - Decision Jan. 11, 1980. Reduction at 805 °F (429 °C), pressure 200 and 500 psi, polymerization at propylene pressure of 160 psi, room temperature, 27 days (experiment P-1) and 3000–600 psi, temperature 205–260 °F (91–127 °C) in xylene (experiment P-9); yield 7 g resp. 4.6 g in xylene-insoluble products. In product exp. P-1 and P-9 crystallinity, IR absorption (Apr. 30, 1953) 10.03 and 11.85 resp. 10.03 and 10.27 (Luongo).
- 12 Standard Oil of Indiana, USA, US P 2,692,257 (SN 223,641), A. Zletz, priority Apr. 28, 1951 – “Ethylene Polymerization”.
- 13 Standard Oil of Indiana, USA, US P 2,780,617 (SN 288,501), A. Zletz, priority Apr. 28, 1951 – Ethylene polymerization with conditioned molybdenum catalyst.
- 14 Standard Oil of Indiana, USA, US P 2,691,647, (SN 324,610), E. Field and M. Feller, priority Dec. 06, 1952 – “Conversion of ethylene and/or propylene to solid polymers ...”.
- 15 Standard Oil of Indiana, USA, US P 2,731,453 (SN 324,608), E. Field and M. Feller, priority Dec. 06, 1952 – “Conversion of ethylene and propylene to solid polymers ...”.

- 16 Repetition of experiments von Field and Feller:
1. **Declaration Martin, Nov. 19, 1987**
Field and Feller, US P 2,691,647, Ex. 21.
Cobaltmolybdate US P 2,393,288 (p. 2, right col., line 3–22), no solid polypropylene.
2. **Declaration Martin, Dec. 22, 1988**
Field and Feller US P 2,731,453, Ex. 7 (col. 13, line 71–75, – col. 14, line 1–7): (see chapter V, [284]).
1. Catalyst US P 2,393,288, A.C. Byrns (p. 2, left col., line 62–75 and right col., line 1–2 and 25–59) formed from cobaltnitrate and ammoniummolybdate, 9 weight-% MoO₃.
2. Catalyst US P 2,320,147, E.I. Layng (p. 2, left col., line 54–73) MoO₃ on Al₂O₃, 10 weight-% MoO₃.
According to description Ex. 7, US P 2,731,453 no solid polymer in the first case and 16 mg of a rubbery solid polymer was isolated. CH₂/CH₃ > 4:1, IR-Spectrum: no crystallinity, atactic polymers, melting range 119–130 °C.
3. **Declaration Martin, March 01, 1989**
US P 2,731,453 and 2,691,647 Field et al.
This declaration was related to the decision in Civil Action 4319, District Court of Delaware. There the two patent rights were not considered because Standard Oil of Indiana did not use the filing dates for a claim of priority.
Also G. Natta tried to repeat Field and Feller. In a declaration, dated Sep. 13, 1956, Natta confirmed no solid propylene only hydrocarbon oils were formed repeating Ex. 21, using five different catalysts. Changing the preparation of the catalyst, not described in the 647 patent Natta received 0.055 g of a waxy polymer product, melting point 135 °C, molecular weight 13,000. The product was totally soluble in boiling heptane, CH₂/CH₃ = 2:1.0.
17 494 Fed. Supplement, p. 411, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980.
E. Francis: Exp. Nr. 4721–16 (Okt. 9, 1951) 5.5 h at 88 °C and nearly 40 bar pressure, isopentane as solvent.
18 494 Fed. Supplement, p. 411, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980.
Hogan, Banks and Francis: Exp. 4721–26, Nov. 19, 1951, silica-aluminum oxide-carrier, containing 4 weight-% chromium oxide, was heated and activated at 390 °C. Activation means that by treating the catalyst granules with dry air 38 h, 82 °C, 35 bar.
19 494 Fed. Supplement, p. 412, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980.
Exp. 4958–13, -15, -18; Feb. 1952, 15 h at 90–100 °C, 30 bar propylene pressure. The product PO-133, melting point 113 °C, IR-Absorption at 10.03 and 10.27 or 11.85 micron.
From the fact that the material was insoluble in pentane and the possibility to conduct measurements of viscosity i.e. the solubility in any solvent it was concluded that the product was not cross-linked but crystalline.
20 494 Fed. Supplement, p. 418, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980.
21 494 Fed. Supplement, p. 429 ff, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980.
22 After treating the raw product with toluene or benzene the concentrated solution was placed in a cool mixture of acetone and methanol. The precipitated product was filtered and stepwise extracted with hot acetone, ether and boiling heptane. The residue was analyzed as described above.
23 494 Fed. Supplement, p. 429, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980.
Catalyst on carrier (87.3% silicon oxide, 12.4% aluminum). The carrier was impregnated with chromium nitrate, activated at 480–500 °C with dry air. It contained 2.77% chromium, polymerizations: in heptane at 88–90 °C, pressure 20–32 bar, 2 h. Polypropylene-product was sepa-

- rated from the catalyst by xylene at 120 or 130 °C and precipitated with acetone/methanol. The product was insoluble in heptane I.V. 5.70, melting point 167 °C.
- 24 Karl Ziegler, German P 973 626, K. Ziegler, H. Breil, E. Holzkamp, H. Martin, priority Nov.17, 1953 – Process for the production of high molecular polyethylenes.
 - 25 Ziegler to Orsoni, Montecatini, Jan. 06, 1954 - Farbwerke Höchst AG, Horn, Scherer: Notice (Memo) visit at MPI Nov. 25, 1953 – Ruhrchemie AG: Program for discussion with Prof. Ziegler March 02, 1954.
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 - 28 E.I. Du Pont, de Nemours and Co, USA, US P 3,541,074 (SN 450 244), Anderson et al, priority Aug.16.1954 – Olefin polymerization catalysts comprising divalent titanium and process for polymerization of ethylene therewith.
 - 29 E.I. Du Pont, de Nemours and Co, USA, US P 4,371,680 (SN 451 064), W.N. Baxter, N.G. Merckling, I.M. Robinson, G.S. Stamatoff, priority Aug.19, 1954 – Polymer Composition.
 - 30 Badische Anilin and Soda-Fabrik, German P 874 215, M. Fischer, priority Dec.18, 1943 – Process for the production of solid polymers from ethylene or ethylene-rich gases.
 - 31 US District Court of Delaware, Civil Action 3952, Studiengesellschaft Kohle mbH vs. Dart Industries, Plaintiff's Post-Trial Brief, Mid 1982, p. 38.
 - 32 549 Fed. Supplement, p. 734: Studiengesellschaft Kohle mbH vs. Dart, C.A. 3952 – Decision Okt. 05, 1982.
 - Lab journal Anderson, Du Pont, Feb. 11.1954.
 - 33 549 Fed. Supplement, p. 734: Studiengesellschaft Kohle mbH vs. Dart, C.A. 3952 - Du Pont April 1954 'Grignard-compounds' Dart Decision Okt. 5, 1982.
 - 34 Agreements up to Mid 1954: Montecatini Jan. 21,1953, Hoechst AG Nov. 1952, Ruhrchemie Dec. 23, 1953, (draft).
 - 35 Lab journal Hyson, Du Pont, July 29–31, 1954 – Organoaluminum-compounds and polymerization of ethylene.
 - 36 Lab journal Hyson, Du Pont, Aug.11, 1954, Suggestion also to polymerize propylene.
 - 37 494 Fed. Supplement, p. 390–397, 1981: Civil Action No. 4319, District Court of Delaware, Decision Jan. 11, 1980. Du Pont Ex. HPL 4427–86, identical with 4460–41; 6 g TiCl₄, 40 ml PhenylMgBr, 200 ml cyclohexane, filtered and dried. Polymerization with 3 g of this catalysts, 100 ml cyclohexane, 50 g propylene, 30 °C. 500 mg polypropylene were isolated.
 - 38 494 Fed. Supplement, p. 370 ff, 1981: Civil Action No. 4319, District Court of Delaware, Standard Oil of Indiana, Phillips Petroleum Co., Du Pont and Montedison- Decision Jan. 11, 1980.
 - 39 Patent Interference Nr.89,634, Nov. 29, 1971.
 - 40 K. Ziegler et al, Italian Patent Applications Nr.6850 and 6851 June 21, 1951.
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 - 42 Correspondence Ziegler/Montecatini Jan. 27–Feb. 20, 1953.
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- 80 Magri, Chini, Crespi to Orsoni, July 19, 1953.
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- 82 Natta to Orsoni, July 09, 1953.
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- 86 Lab journal H. Martin, Exp. 2, Dec. 02, 1953.
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- 92 Lab journal H. Martin, Exp. 14.
- 93 Lab journal H. Martin, Exp. 15.
- 94 Lab journal H. Martin, Exp. 16, Jan. 04, 1954.
- 95 Ziegler to von Kreisler, Jan. 07, 1954.
- 96 Lab journal H. Breil, Exp. 41, Nov. 30, 1953 (p. 309–310).
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- 98 Lab journal H. Breil, Exp. 45, Dec. 08, 1953 (p. 318–323).
- 99 Lab journal H. Breil, Exp. A 75, Dec. 16, 1953 (p. 329–330): 200 cm³ iron autoclave, 50 cm³ aluminum alkyl (average chain length C₈), 1.5 cm³ TiCl₄, 68 g propene, 'At room temperature (T 20 °C, p 20 atm) during shaking no reaction and no

- self heating'. Then, the temperature from outside increased to 45 °C, next day up to 95 °C. Thereby the pressure increase to 65 atm. 'No reaction' can be determined, thereafter 31 g propene released and without opening the pressure vessel (25 °C, 14 atm residual pressure) 38 g ethylene pressed into the vessel. The autoclave now contained besides 38 g ethylene 37 g residual propene. After 4 days the autoclave was opened, from which 5 g residual propene were isolated. The entrée in the lab journal '86.5% of the original C₃H₆ has reacted, 100% of the C₂H₄'. The solid product was washed and dried: '51 g PE'.
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 - 141 Karl Ziegler, Z 3941 IVb/39c (priority Jan. 19, 1954, published July 18, 1957, issued as German P 1 012 460, Aug. 05, 1960) inventor: K. Ziegler, H. Breil, E. Holzkamp, H. Martin.

- 142 Karl Ziegler, Z 3942 IVb/39 c (priority Jan. 19, 1954, published Sep. 19, 1957, issued as German P 1 016 022, Dec. 28, 1960) inventor: K. Ziegler, H. Breil, E. Holzkamp, H. Martin.
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- 144 Deposition Chini, March 1970, p. 1193.
- 145 US-Interference 99478 Ziegler/Natta, Ziegler's "Main Brief", p. 14.
- 146 Deposition Chini, March 1970, p. 1196.
- 147 Consolidated Civil Action No. 3343, March 09, 1970, Deposition of Natta, pp. 85, 86.
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- 149 Agreement Mar. 09, 1954.
- 150 US-Interference 99478 Ziegler/Natta, Ziegler's Reply Brief, p. 25, (Document ZX 137, identified as A 0059 - A 0061) Remarks of Montecatini besides the memo, dated Mar. 9, 1954, discussion: ZX 130 and ZX 116 (M 4517): Here it is stated, that after the visit of Ziegler, other olefins such as propylene and sterene were then tried.
- 151 Deposition Chini 1969, p. 10 ff, and 1970, p. 1221–1232, Chini lab journal p. 42: 8.4 g TiCl_4 , 20.4 g AlEt_3 , mol ratio 1 Ti : 4 Al, in petrol ether, propylene at first normal pressure – then up to 12 atm, 17–75 °C, 1.25 h, yield: 3.5 g solid polymeres, thereafter extracted with ether.
- 152 Deposition Chini 1970, p. 1230.
- 153 Consolidated Civil Action No. 3343, March 09, 1970, Deposition of Natta, p. 80.
- 154 Consolidated Civil Action No. 3343, March 09, 1970, Deposition of Mazzanti, pp. 1475, 1478.
- 155 Consolidated Civil Action No. 3343, March 25–26, 1970, Deposition of Mazzanti, pp. 1631–1632, 1634–1635 and 1639–1641.
- 156 Consolidated Civil Action No. 3343, March 16–18, 1970, Deposition of Pino, p. 631.
- 157 Consolidated Civil Action No. 3343, March 25–26, 1970, Deposition of Mazzanti, pp. 1649, 1650.
- 158 Zeugenaussage Chini March 24, 1970, p. 1272–1294
2. Exp.: catalyst $\text{TiCl}_4 + \text{AlEt}_3$ 1 : 10, room temperature, and pressure. The product was extracted: 7% with acetone, 40% diethylether, 28% heptane and 25% heptane-insoluble.
3. Exp.: 50–60 °C and pressure, yield 193 g.
- 159 US-Interference 99478 Ziegler/Natta, Ziegler's "Main Brief", p. 17.
- 160 US-Interference No. 99,478 - Ziegler's "Reply Brief", ZX 166, p. 29 Montecatini – internal memo.
- 161 US-Interference No. 96,101, Deposition Chini 1969, p. 72 and 171, 1970, p. 1233.
- 162 US-Interference 99,478, Brief "Final Hearing", Natta et al, p. 31 (ZX 164, p. 615, ZX 167, p. 755–756).
- 163 US-Interference 99,478, Brief "Final Hearing", Natta et al, p. 32 (ZX 166).
- 164 Deposition Ziegler, Duisburg, Dec. 09, 1969, p. 140.
- 165 Italian Patent Application 24.227/54, Montecatini, filed June 08, 1954, issued as No. 535 712, Nov. 17, 1955, inventor: G. Natta.
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- 167 Italian Patent Application 25.109/54, Montecatini, filed July 27, 1954, issued as No. 537 425, Dec. 28, 1955.
- 168 Consolidated Civil Action No. 3343, March 9, 1970, Deposition of Natta, p. 264.
- 169 Consolidated Civil Action No. 3343, March 25–26, 1970, Deposition of Mazzanti, p. 1658–1659.
- 170 Orsoni to Ziegler, July 30, 1954.
- 171 Natta to Ziegler, Aug. 04, 1954.
- 172 Ziegler to Orsoni, July 21, 1954.
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Catalyst: $\text{TiCl}_4 + \text{Et}_2\text{AlCl}$: Exp.-Nr. 55, 72, 78–80, 85, 86.

- Catalyst: other metal compounds + AlR₃: Exp.-Nr. 52, 56, 57, 63.
- 175 Diplomathesis H. Breil University Bonn, June 28, 1954, p. 24.
 - 176 Lab journal H. Martin, Exp. 87, July 09, 1954.
 - 177 Lab journal H. Martin, Exp. 88, July 13, 1954.
 - 178 Lab journal H. Martin, Exp. 93, July 21, 1954.
 - 179 Lab journal H. Martin, Exp. 101, July 30, 1954.
 - 180 Lab journal H. Martin, Exp. 94, July 23, 1954.
 - 181 Lab journal H. Martin, Exp. 97, July 27, 1954.
 - 182 Karl Ziegler, Z 4348 39b 4 (priority Aug. 03, 1954, published Dec. 28, 1967, issued as German P 1 257 430, Dec. 17, 1973) inventor: K. Ziegler, H. Breil, H. Martin, E. Holzkamp.
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2

The Chemical Industry Connection

The commercial world learned of the nature and direction of the chemistry Ziegler and his co-workers were working on through lectures, publications and private conversations which Karl Ziegler held and/or conducted throughout 1952. Interest in Ziegler-chemistry sprang up almost simultaneously in Germany, England, Italy and the United States when Ziegler announced that he had successfully completed the production of polyethylene – at the time this meant the production of waxy materials from ethylene (the so-called “growth reaction”) – the selective dimerization of ethylene, propylene and butene – leading to the production of p-xylene/terephthalic acid/synthetic fiber – as well as improvements in the synthesis of organoaluminum compounds.

His first connection to Italy has been discussed in the foregoing chapter. The term “polyethylene” always had an exciting effect in those days, because ICI in England, Du Pont in the United States, and BASF in Germany were sharing a monopoly for thermoplastic polyethylene and were running an economically interesting operation, without interference by any independent competitive production or product. Even though the “polyethylene” – such as that produced by Ziegler in 1952 – did not constitute a competitive product, nonetheless, a mixture of hope and trust in Ziegler-chemistry prompted some companies, even in those early days, to enter into binding contracts with Ziegler to make sure they had “a foot in the door”. Montecatini had done this with its preliminary January 1953 agreement, and others attempted to follow suit.

As much as Ziegler was interested in pursuing the commercial exploitation of the chemistry he had developed up to that point after almost 10 years of research at the Institute, industry had an traditional policy of securing its access to a whole area of research as early and as cheaply as possible, and, on an exclusive basis of course, if possible. Promises were made to Ziegler that one or other of his products would be produced on an industrial scale, but experience showed that in the end it would always be one or other improved product that would be more interesting for future development.

When the Institute was founded, a binding promise was made to its first Director, Franz Fischer, as early as 1912, “that any inventions made at the Institute by him or his co-workers would be filed as patent applications, paid for by the Institute, both at home and abroad, maintained, and commercially exploited.” This commitment was

broadened and solidified in 1925 through foundation of the Studien- und Verwertungsgesellschaft¹⁾ [1]. Emil Kirdorf as representative of the Rhine-Westphalian Coal Syndicate and August Thyssen, a “factory owner at Castle Landsberg near Mintard”, were the founding members, and thus partners, both having an equal share in the company (capital contribution 15,000 Reichsmark each). The founding partners had made a commitment as trustees to transfer part of their company shares to the other members of the Coal Syndicate. In the course of time, up to 61 companies, domiciled mostly in the Rhine–Ruhr area, acquired company shares [2].

The company assumed the prior-established obligations of the Kaiser-Wilhelm-Institute regarding the protection of new technologies by securing patent rights and exploitation of these patent rights at home and abroad. The position of the Manager of the company was always to be held by the then Director of the Institute. Distribution of income from commercial exploitation was established as follows: 30% to the Institute, 40% to the Director of the Institute and/or other inventors for inventions leading to patents that were to be assigned to the company, and the remaining 30% to the partners proportionate to their share holdings.

On review, it is particularly remarkable how meticulously Karl Ziegler laid the groundwork during the period 1952–1954, for the commercial exploitation of the new chemistry he had launched. He did this by paying close attention to the details of language structure of any agreements he entered into, whereby, with each new case, he immediately applied what he had learned in each negotiation to discussions with the next interested parties.

The immediately obvious challenge here was to balance the requirements of any prospective licensees in such a way that, while maintaining a healthy competitive structure, no licensee would receive disproportionately more favorable license terms than any other. This was certainly more easily accomplished when selling a monopoly than in cases where competing processes were already available in the marketplace. The number of interested parties eventually grew so large, on a global scale, that it was safe to speak of a monopoly creating situation.

A further problem arose from the fact that there was no body of experience to be relied on in connection with the drafting and negotiating of license agreements. Consequently, Ziegler had to deal with the written contract offers of any interested parties, whereby one or other point of agreement reached with certain licensees would necessarily contain within it the seed of subsequent disputes, because the interested parties of different “couleur” would always be conscious of the element of “corriger la fortune” when facing an inexperienced “professor”.

From one case to the next, the option and license agreements developed with such speed and intensity that it was often impossible to maintain the necessary distance from the events, a distance which would have demanded the reassessment of one or other request.

1) See p. 63.

2.1

Farbwerke Hoechst

In February of 1952, Karl Ziegler paid a visit to Dr. Otto Horn, of Farbwerke Hoechst, a former assistant research scientist under Karl Ziegler's predecessor, Privy Councilor Professor Franz Fischer, of the Kaiser-Wilhelm-Institute for Coal Research at Mülheim, during which they discussed, *inter alia*, the chemistry of organoaluminum compounds [3], a field which Ziegler was exploring at the time.

Three months later, Karl Ziegler gave a lecture at Mülheim entitled "Novel Catalytic Conversions of Olefins" [4]. During the lecture, he mentioned "that we are not yet able to produce genuine plastic-like polythene." Very soon thereafter, Ziegler received a draft option agreement from Hoechst at Frankfurt, which led to an agreement being signed in late 1952 [5]. The agreement centered on four German patent applications, the subject matter of which was disclosed to Farbwerke Hoechst, coupled with an option to obtain a non-exclusive production license for Germany in conjunction with the right to also sell abroad any products manufactured according to the licensed process. As a consideration, Farbwerke Hoechst paid DM 50,000. The option term was limited to 1 year. The individuals at Hoechst who had negotiated the agreement with Ziegler were the Director of Research, Dr. Sieglitz, and Dr. Eishold, Head of the Patent Department. During the option year, the parties were engaged in a lively exchange of know-how related to the contract field.

It became apparent that three particular areas would be of special interest to Farbwerke:

1. The selective dimerization of butene to octene and recovery of p-xylene through aromatization. However, the Ziegler version appeared too expensive.
2. The synthesis of C14–C20-olefins from ethylene and production of detergents therefrom through oxidation. The commercial possibilities could not yet be assessed.
3. Although high polymer ethylene is interesting, a process for its production using aluminum trialkyls as catalysts is not yet available. In any event, Hoechst did make efforts to clarify the patent situation.

The consensus at Hoechst was that it would not be possible, nor was it intended, to arrive at a final evaluation during the remainder of the option period. It was therefore recommended that an extension of the option with Karl Ziegler should be negotiated [6]. Hoechst was reluctant to sign a license agreement. The situation changed when, in the Fall of 1953, polymerization catalysts for the production of thermoplastic polymers were discovered. One of the first ramifications was that the option agreement was informally extended through January 15, 1954 [7].

Prior to the extension, the Institute had received a visit from Drs. Horn and Scherer in November of 1953, during which time the guests observed the production of a sheet formed between two heated aluminum plates: a thermoplastic polyethylene produced with the aid of aluminum trialkyls and co-catalysts at tempera-

tures up to 100 °C and pressures between 50 and 100 atmospheres [8]. As early as December, Horn and Ziegler exchanged data regarding the tensile strength of the initial sheets produced at Mülheim [9]. In January of 1954, Drs. Horn, Scherer and Sennewald accepted Ziegler's invitation to watch an experiment at Mülheim resulting in the polymerization of ethylene under normal pressure [10]. The experimental procedure left a lasting impression.

Two days later, the parties agreed that, for an additional payment of 100,000 Deutsch Marks, the existing option would be extended initially for another year [11]. Hoechst was now anxious to gain insight into the details of the new polymerization process, which were disclosed in a memorandum of January 28, 1954 [12]: Catalyst production using diethylaluminum monochloride and titanium tetrachloride and polymerization at normal pressure and temperatures between 20 and 70 °C [13].

Six months later [14], Ziegler reported to Hoechst (Dr. Sieglitz) on the progress that had been made with respect to the polymerization technology:

1. Pre-adjustment of the molecular weight of the polyethylenes produced between 24,000 and 50,000, the commercially desirable range.
2. Polypropylene as a novel polymer²⁾.
3. Synthesis of butene-(1) from ethylene based on aluminum trialkyl/titanium tetrabutylester as the catalyst mixture.

A license agreement was finally entered into in August of 1954 [15]. The license covered patent rights for which applications relating to the contract field had been filed in the Patent Office up to that point in time i.e. conversion of olefins using organometal catalysts, production of such catalysts as well as patent rights obtained by Ziegler during the term of the contract. It was a non-exclusive production and sales license for Germany which included the right to sell the products abroad except for Italy (where exclusive rights had previously been granted). In consideration, Hoechst paid a sum of 600,000 Deutsch Marks to Studien- und Verwertungsgesellschaft as the legal contractual partner and representative of the Max-Planck-Institute, whose Managing Director, under the by-laws, was Professor Karl Ziegler. The agreement provided for running royalties on a scale of 4% of the net sales price for the first 1200 tons and 3% for any sales in excess of 1200 tons. Disputes were subject to arbitration. Half of the amount paid upon signing of the agreement was creditable as an advance-payment against future royalties.

2.2

Petrochemicals Limited

Sir Robert Robinson (a 1947 Nobel Prize winner), a manager with Petrochemicals Limited, a young British company, had apparently closely followed Karl Ziegler's publications of 1952 and earlier. He suggested that his Director of Research at Manchester, Dr. E.T. Borrows, contacted Ziegler in order to discuss commercializa-

2) In spring 1954 experiments to the polymerization of propylene were conducted by Hoechst as later reported, but Dr. Rehm

(Hoechst) neither gave Karl Ziegler notice of this nor did he characterize the product.

tion of the results thus far generated by Ziegler-chemistry. As early as September 1952, the parties signed a letter agreement [16], outlining the principles of a cooperative venture. Pursuant to the undertaking, Ziegler would act as a consultant to Petrochemicals Ltd for a period of 5 years, during which time he would make information on his chemistry available for an annual fee of 50,000 Deutsch Marks. The arrangement further provided for exclusive rights in Great Britain and Northern Ireland and required that Petrochemicals Ltd bear all patent costs in connection with British applications to be filed corresponding to seven German home applications, and additionally pay a royalty of 5% of the “product value” with the consultant fee being offset. The subject area was defined as Ziegler’s entire field of research, and the contract was to expire after a total of 400,000 Deutsch Marks had been paid, but the option for an extension would be included.

Both parties were interested in replacing this letter agreement with a formal license arrangement. But this did not immediately come about. In addition to an exchange of scientific/technical information and discussions particularly with respect to propylene and ethylene dimerization, the production of waxes by means of the so-called “growth reaction”, and the production of terephthalic acid and phthalic acid, the parties also exchanged drafts for a more formal contractual arrangement, however the events that occurred at Mülheim in 1953 [17] initially delayed the conclusion of a definitive license agreement. Even though E.T. Borrows believed that the scope of the 1952 agreement also encompassed the most recent findings, Sir Robert Robinson and Karl Ziegler signed a contract [18] which, as of March 1954, also included the British equivalent of the first German patent applications for the production of polyethylene with catalysts composed of aluminum trialkyls and transition metal halides dating back to November–December 1953. This agreement re-affirmed the exclusive license for Great Britain and Northern Ireland.

From an early stage, it had been Ziegler’s desire that the exclusivity should apply only to the production, but not the sale of products, i.e. that other European licensees should remain free to enter the British market as long as Petrochemicals Ltd enjoyed reciprocal rights.

Dr. Borrows was informed of the ongoing technochemical developments at the Max-Planck-Institute for Coal Research, such as the mastering and control of the molecular weight of polyethylene and the successful polymerization of propylene [19]. While the relevant correspondence does not indicate that Petrochemicals Ltd had already, at the time, carried out any propylene polymerization, it does allude to the production of copolymers of ethylene and propylene [20], dated however, after the successful experiments conducted by H. Martin in January of 1954.

Since Petrochemicals Ltd was a complete novice in the field of plastics production and processing, Sir Robert Robinson suggested that the company persuade ICI to enter into a cooperative venture [21]. In October of 1954, Karl Ziegler advised E.T. Borrows [22] of all relevant German patent applications, the list now also included patent applications filed in 1954, and reminded him of the need to file corresponding British applications. During that same year [23], Borrows reported to Ziegler on first contacts made with ICI, with the aim of establishing a cooperative venture in the licensed field, and sent him an “announcement” [24], detailing Petrochemical

Ltd's offer of small quantities of 2-methylpentene-1 as a new product generated from the Ziegler-chemistry – a “good will” promotion.

Based on numerous license agreements concluded with companies in other foreign countries, Ziegler felt that it had become necessary to upgrade the March 1954 agreement. This was finally accomplished in February of 1955. The revised version was signed under the title of “Supplemental” [25]. The minimum annual royalty was increased to 75,000 Deutsch Marks and the total paid-up amount to 600,000 Deutsch Marks; the running royalty was to be paid on a scale of 4% for up to 1200 tons, 3% for up to 12,000 tons, and 2% of the net sales price for amounts in excess of 12,000 tons. If any sub-licenses were to be granted, 50% of Petrochemicals Ltd's proceeds would go to Ziegler. The exclusive rights granted to Petrochemicals Ltd in the 1954 agreement for the British Commonwealth were rescinded, that is, Petrochemicals Limited retained the right to a non-exclusive license regarding the Commonwealth countries [26].

2.3

Steinkohlenbergbauverein (Hard Coal Mining Society)/Bergwerksverband (Mine Association)/Ruhr Area Coal Industry

In 1952, Ziegler-chemistry, as understood through lectures and written publications, appeared sufficiently promising to Farbwerke Hoechst in Germany, Petrochemicals Limited in England, and Montecatini in Italy to prompt these companies to enter into option agreements with Ziegler so that each would secure rights for itself at an early stage. However, at this point it was not positively clear to these prospective licensees whether any, and/or which, products and/or processes encompassed by the Ziegler-chemistry should be considered for commercial exploitation. It was company policy to secure access to any of the most promising advances in chemistry that had been developed in Germany.

Such a position could not be expected from the mining-related industry, even though the Ruhr Area coal industry (Ruhrkohle) as represented by Aktiengesellschaft Rheinisch-Westfälisches Kohlensyndikat at Essen as well as the City of Mülheim on Ruhr and the Kaiser-Wilhelm-Society for the Advancement of the Sciences (now Max-Planck-Society) were among the foundation carriers [27] of the Max-Planck-Institute for Coal Research, and despite the fact that the by-laws still required that a “scientific advisory board” promote communication between the Institute and industry. After all, the mining-related industry included well-known chemical companies such as Deutsche Erdöl AG, Hibernia AG, Rheinpreußen AG, Ruhrchemie AG, Gelsenberg Benzin AG und Ruhröl GmbH.

However, Karl Ziegler's research efforts were not immediately connected to coal, and the mining industry likewise failed to recognize any direct links with the scientific discoveries generated at the Institute. It was only the sensation created by the synthesis of polyethylene with the aid of the “Ziegler catalysts” that finally prompted eight gentlemen³⁾ from coal mining companies and “companies closely associated

3) v. Blumencron, Wesseling; Busch, Gelsenberg; Dietzel, Mannesmann; Grimme, Rheinpreußen; Kleingrothaus, GHH; Krüger,

Harpen; Paul, Ruhrchemie; Reerink, Steinkohlenbergbau-Verein.

with them” to meet in mid-December of 1953 [28] at the Max-Planck-Institute to discuss the latest findings with Ziegler; this was arranged by Dr. Broche of Rheinisch-Westfälischer Bergbau.

The talks ended with Ziegler’s promise not to engage in commercialization negotiations with any third parties until the end of January 1954, except for a continuation of his discussions with Hoechst. As early as February 1954, the parties exchanged drafts for a license agreement.

Dr. Heinrich Tramm, Chairman of the Board of Ruhrchemie at Oberhausen-Holten and successor to Professor Dr. F. Martin, learned of the latest developments in ethylene polymerization, possibly through contact on the occasion of a lecture event held at the Max-Planck-Institute for Coal Research. He pressured Ziegler into entering an option agreement with Ruhrchemie at the earliest possible opportunity [29]. It was not only that Dr. Tramm had recognized the commercial significance of the process invented at the Institute, but he also had access to the potential capacity of the company over which he presided to make rapid technical advancements including building a pilot plant.

After a round of discussions with mining industry delegates at which Mr. Tramm was represented by his Technical Director, Mr. Paul, Ziegler regretfully had to curtail Dr. Tramm’s zeal and postpone signing the proposed option agreement [30].

Steinkohlenbergbau-Verein as the representative of the German coal mining industry expected to acquire for itself the preferably full-scale, and as far as possible exclusive, use of the Ziegler-chemistry, while Ziegler wanted to make sure that these aspirations were kept in check and that he would retain for himself any attainable freedom.

Thus, the license [31] granted to Steinkohlenbergbauverein, dated March 3, 1954, was semi-exclusive, since the existing option agreement with Farbwerke Hoechst was to remain unaffected by the new agreements. The subject area – conversion of olefins with the aid of organometal compounds and their production – was narrowly defined and limited to methods and processes “in which the coal mining and any related industries have an interest.” “Steinkohlenbergbauverein has no intention, as a principal licensee, to be involved in the exploitation of the contracted inventions where such exploitation falls outside of the mining industry’s area of interest.” The license however, failed to define the “interests of the mining industry” and, as events progressed, these interests became subject to different interpretations.

Geographically, the semi-exclusive rights under the license were restricted to the Federal Republic of Germany, with the license having an initial running time of 3 years, whereby this period was additionally limited to each individual process. After the end of 3 years, it became clear that an industrial-scale production was not in the offing. The exclusivity was changed to a simple license. Ziegler’s intentions were unmistakable.

Since it was impossible at the time to predict the added value to be gained from the individual processes or products, figures on running royalties could not be established. The down-payment of 300,000 Deutsch Marks required from Ruhrkohle was larger than comparable option fees paid by others, but the subject area of the contract was also more comprehensive. The agreement furthermore provided for the right to grant sub-licenses. At the time, Ziegler referred to it as a friendship price.

“The German coal mining industry and related industries (should) have an opportunity, according to circumstances, to make the best possible use of the inventions of Professor Dr. Ziegler and his co-workers covered by the agreement.” (Ziegler’s cover letter accompanying the agreement [32]). Two-thirds of the down-payment were considered to be an advance payment creditable against future royalties.

Two further cover letters [33], expressed Ziegler’s intention that, in view of his existing contractual arrangement with an older licensee, Petrochemicals Limited, the sale of licensed products between Great Britain and the Federal Republic of Germany was to remain unrestricted so long as there was reciprocity, and that this principle should be adhered to with respect to any subsequent foreign licenses. Ziegler additionally included a loose most-favored-licensee clause for Steinkohlenbergbau relative to any license requests made by prospective foreign licensees.

In March of 1954, Ruhrchemie and the Institute entered into an initial arrangement for the mutual exchange of know-how regarding the technical feasibility of polyethylene production in a pilot plant [34]. Within a few weeks a small plant was completed in which polyethylene was produced using a diethylaluminum chloride/titanium tetrachloride catalyst with the products exhibiting molecular weights greater than several hundred thousand.

It was not until May of 1955, however, that an option [35] was formally granted to Ruhrchemie through Bergwerksverband, the legal successor of Steinkohlenbergwerks-Verein, which, at that time, signed and took responsibility for Ruhrchemie.

2.4

The “Run” of Prospective US Licensees

2.4.1

Hercules Powder Company

Hercules Powder Company of Wilmington, Delaware, maintained an office in Den Haag as liaison for its European connections. Mr. Riemersma, the Manager of this office, contacted Karl Ziegler in May of 1952. He referred to a lecture Ziegler had given in Frankfurt, called on Ziegler at Mülheim together with Dr. R. Wiggam, “Manager of Development”, and asked for an option agreement, which, however, was only concluded and signed by both parties in September in the form of a letter agreement [36].

The subject matter of the agreement was defined as the production of organoaluminum compounds and their use as catalysts for the production of higher olefins and aromatic hydrocarbons, such as p-xylene, derived therefrom. Geographically, the agreement was to cover the United States and Canada, and the option guaranteed Hercules exclusive rights, and specifically an exclusive option,

“to study and evaluate the Ziegler processes for a period of nine (9) months....”.

The price was \$10,000 at the then prevailing conversion rate (4.20 Deutsch Marks/1 US \$), comparable to the option payments made by other prospective licensees. In

the event that it was not possible to conclude negotiation of the terms for an exclusive license within the required time period, then Hercules would in any event, receive a non-exclusive license for the production of 2-ethylhexene-1, the butene-1 dimer. The running royalty would be fixed at 4% of the net sales price of the product, payable on quantities up to 100 million pounds. If Hercules failed to exercise the license by January 31, 1957 (start-up of an industrial plant), the license would be cancelled.

In March of 1953 [37], Wiggam inquired as to Karl Ziegler’s ideas regarding a further contract, particularly his ongoing evaluation of the Ziegler chemistry, the prevailing patent situation, and finally his ideas on an exclusive license for the United States and Canada. One month later, during Wiggam and Riemersma’s visit to the Institute, Hercules submitted a rather voluminous draft license, in response to which Ziegler merely promised [38] to extend the option agreement which had in the meantime been renewed, up to the end of 1953 and to render his opinion on the draft license by July 1953.

In the Spring of 1953, Hercules sent one of its chemists, Dr. Arthur Glasebrook, to Mülheim so that he could gain an “on-site” impression of the practical aspects of the chemistry under consideration.

Ziegler’s commentary on the Hercules draft license was initially talked over internally with patent attorney Dr. von Kreisler at Cologne, but not with Ziegler’s US attorneys, and was discussed with Messrs. Wiggam, Rutteman and Glasebrook at the end of July 1953, without leading to an actual revision and/or redrafting of the existing contract. A noticeable uneasiness developed on Ziegler’s part based on Hercules’ desire to negotiate an exclusive license covering the full range of the Ziegler chemistry, instead of restricting the license – as originally optioned – to the dimerization of butene and subsequent aromatization to produce p-xylene. A problem was created by the fact that the license was even to include future developments. In a second draft [39] presented in October of 1953, Hercules offered to limit the license to a “Technical Field” and to define any developments outside of this area as “Extended Technical Field”, the latter to be addressed separately. As advance payment, Hercules offered a total sum of \$40,000.

In mid-November, Rutteman sent a reminder regarding a commentary on the most recent draft license and learned from Ziegler of the spectacular new developments in the production of high molecular weight ethylene polymers. A letter from Ziegler [40] apprised Hercules of the details. As a consequence, Ziegler expected Hercules to appreciate his position that “at this time, he could not sign” a new agreement, but confirmed that he was willing to be bound by the earlier option agreement. At the end of 1953[41], he forwarded the first polyethylene samples in the form of various sheets, describing their characteristics thus: Production of a polymer powder at normal pressure, room temperature, separated with purification, and molded into sheets, determining the tear strength ($20\text{--}25\text{ kg/mm}^2$) of extended ribbons. This type of promotion was sure to have its effect. In February 1954 [42], Ziegler submitted his commentary on the latest license draft of October, 1953 and, referring to the most favored licensee terms granted to prospective German licensees, now expected a substantially higher option fee of \$50,000 for certain portions of the contract field. Furthermore he offered in any event, to separate the agree-

ment into the previous “Technical Field”, polyethylene plastics, and the “Extended Technical Field”. The period for acceptance of an exclusive license was to expire at the end of 1954.

It had become increasingly obvious by now that any delay in signing an agreement would not result in any more favorable terms for the licensee than had already been granted, particularly to an earlier German licensee.

The license agreements with Petrochemicals Ltd. in England and Steinkohlenbergbau-Verein in Germany had meanwhile been signed, and in May of 1954, Hercules submitted a further written analysis of the license agreement being negotiated, when Steinkohlenbergbau-Verein, after reviewing the Hercules license drafts, warned Ziegler explicitly not to grant exclusive licenses [43].

Alluding to the role Bergbau played in supporting the Max-Planck-Institute, Ziegler [44] reminded Hercules of his moral obligation to accede to Bergbau’s wishes and offered non-exclusive licenses. At this point in time, at the end of June 1954, he was still in a position to confirm that “no license agreement whatsoever has been concluded with any US company.”

Developments progressed at a heated pace. At that time, visitors streamed through the Max-Planck-Institute as if through a revolving door. Negotiating committees of numerous prospective US licensees were staying concurrently either at the Hotel Petersberg at Königswinter near Bonn, or at the Hotel Breidenbacher Hof at Düsseldorf, eyeing one another skeptically. Hercules realized that time was of the essence. In July, Messrs. Wiggam and Rutteman called on Ziegler at Mülheim, discussed the new situation, and negotiated two new agreements, the “Technical Field Contract” and the “Polyolefin Contract” [45]. Both contracts were signed on September 24, 1954. The license also encompassed polypropylene and, thus, polymers other than polyethylene; additionally, it covered processes for their production as well as products containing polyolefins. Further inclusions were the future patent rights which had been filed in the United States and Canada prior to January 1, 1960. As consideration for the 1-year option to obtain a license, Hercules made a down-payment of \$50,000, and when they exercised the option (September 12, 1955) paid an additional \$300,000, whereby this amount could be applied as an advance payment creditable against future running royalties. The license also included worldwide export rights with the exception of Italy, Germany and Great Britain due to the exclusive licenses granted in these countries, in addition to a “most favored licensee clause” which would upgrade Hercules in the event of the subsequent issue of US licenses containing more favourable terms than those presently accepted (see Chapter 5, footnote 71, and p. 234). The running royalty for production was scaled as follows, 4% of the net sales price for amounts up to 1200 tons per year, 3% for sales in excess of 1200 tons per year up to a limit of 10,000 tons per year, and for sales over and above that, 2%.

The provision that payment of running royalties was contingent upon the issuance of a patent was not in Ziegler’s favor. Any royalties due were initially paid into an escrow reserve account. Up to that time, there were only German patent applications. Ziegler furthermore conceded that Hercules’ payment obligation was limited to 15 years beginning with the start-up of commercial production and that

the license would be converted to a paid-up license once 2 million dollars had been accounted to Ziegler and paid. This provision applied to patent rights that were owned by Ziegler within the first 5 years following the signing of the agreement and was further limited to a production capacity of 10,000 tons annually. For any production in excess of 10,000 tons, a reduced royalty of between 1 and 2% was to be re-negotiated.

Also significant was a provision regarding “Dominating Patents”, that is, patent rights owned by third parties which partially or wholly dominated the Ziegler process. The agreement did not include a definition of this term. Ordinarily, what is meant are older patent rights which the parties had not been aware of when they signed the contract, but which claimed an earlier priority date, i.e. which had been filed prior to November 1953. Hercules, however, subsequently interpreted this paragraph to mean that whenever the process could not be freely carried out without infringing a third-party patent, a license would have to be obtained for such dominating patent rights. In that case, Hercules planned to deduct any payments made to third parties from the royalties due Ziegler.

Further difficulties arose from the provision concerning infringement of the Ziegler patent rights by unlicensed third parties. Under this clause, Ziegler was committed to prosecute and stop any infringement without guidelines by which to determine when infringement had in fact taken place and who was to produce the evidence. If Ziegler failed to take action, Hercules had the right to stop paying running royalties. The same applied in the event of Hercules being sued by any third parties for infringing extraneous patent rights while carrying out the Ziegler process.

Jurisdiction for the settlement of any disputes arising under the agreement was vested in the District Court at Wilmington, Delaware. In fact, a large number of suits were later brought to this court, and not only against Hercules, which prompted one of Du Pont’s chemists to quip that Du Pont had been the winner in all of the litigations fought at Wilmington. Du Pont owned the only suitable hotel in town, where both or all of the parties involved in suits were obliged to stay for several weeks’ duration each time they had to appear in court.

The second agreement of the same date, September 24, 1954, the “Technical Field Contract” covered five subject areas: the production of organometal compounds, utilization of these compounds for the dimerization of olefins as well as the polymerization of olefins, use of these compounds in further conversion procedures

“to produce compounds containing a functional group other than a double bond.”

What was meant here was the production, for example, of alcohols, used as intermediaries for insecticides, herbicides, fungicides, and utilization in the production of specific olefins suitable for aromatization with further processing. The definition was couched in very broad terms and, obviously, could not be narrowed after these many years of negotiation. The license was non-exclusive as far as the last four subject areas were concerned. But in terms of the sale of aluminum trialkyls in the United States, it was exclusive. The basis for future litigation had, thus, been laid.

The agreement provided for a running royalty of 5% of the net sales price for capacities of up to 2 million pounds per year of product sold and a reduced royalty for production above this capacity. Finally, Hercules was granted permission to reduce its running royalties by 50% once a total sum of 1 million dollars had been paid. Any provisions regarding dominating patents and the course of conduct of each party in the event of third-party infringement were parallel to the first agreement.

After the contracts were signed, Hercules' representatives received a packet of written information and disclosures regarding knowhow disclosures (approx. 80 pages concerning the first agreement and 120 pages relating to the second).

One point of interest is Karl Ziegler's statement [46] made in connection with these two concurrently dated agreements, according to which he personally acted as a party to the contract, rather than as the Director of the Max-Planck-Institute for Coal Research or Manager of the Studien- und Verwertungs-GmbH. This was because it had been agreed internally between the Max-Planck Society, Max-Planck-Institute for Coal Research and Karl Ziegler, that Ziegler would act as trustee for the Institute [47].

2.4.2

Gulf Oil, Koppers, Dow, Union Carbide and Monsanto

The behavior pattern of large oil companies was markedly different from that customary for the chemical industry proper. By nature, these companies were ever on the lookout for new methods of refining petroleum-derived products and were willing to make major investments rapidly whenever a promising prospect appeared on the market. Gulf Oil Corporation was one of the companies manifesting such a typical behavior pattern. It was through Ruhrchemie, who had acquired petroleum processing knowhow from Gulf Oil, that contact with Karl Ziegler was first established. In May of 1954[48], "The Gulf Companies" approached Ziegler through their legal department in Pittsburgh. In the name of Goodrich Gulf Chemical Inc., B.F. Goodrich Company, and Gulf Oil Corporation, W.I. Burt, Chairman of the Technical Committee, not only confirmed a verbal agreement, according to which Ziegler was willing to make samples of the new polyethylene available to Gulf, but already acknowledged safe receipt of such samples. According to Burt the three companies undertook not to conduct any chemical analyses whatsoever on these specimens in order to ascertain the probable composition of the catalyst, and not to file any patent applications whatsoever on the basis of any tests conducted by Gulf employees, which, on the one hand and in hindsight, was a naive promise but, on the other hand, heralded the beginning of a long-standing, loyal cooperation between the parties.

The participants at the negotiations and business partners interacted with one another in an almost exaggeratedly proper manner. Only 2 months later, on July 27, 1954 the parties agreed on the language for an option and license agreement in the event of the tests conducted at Gulf on the specimens would lead to a positive assessment of the technical and economic utility of the product. At that point, Karl Ziegler provided access to his as yet unpublished German patent applications, announced

the granting of an option, and stipulated that Gulf must decide within 45 days whether or not it was going to take a license. Gulf immediately paid \$50,000 for the option; all technical information was to be kept confidential.

There were two aspects of interest. The prospective US licensees requested that, in addition to the United States and Canada, Mexico also be included in the licensed territory. Moreover, the agreement and all prior drafts were directed to “polyolefins”, despite the fact that at the time the drafts were prepared polypropylene and higher olefins were not yet in existence and, were only 2 weeks old on July 27, 1954, at which time they had neither been filed for a patent, nor had any details concerning them been disclosed to Gulf’s representatives. The term “polyolefins” may possibly have been adopted to cover the production of higher olefins from ethylene using the so-called growth reaction. The “Definitions” clause of the license agreement did not, however, reflect this interpretation, but did address the production of co-polymers, which had already been synthesized in January of 1954.

As early as September 2, 1954 [49], the President of Goodrich Chemical Inc. and B.F. Goodrich Corporation, Mr. W.S. Richardson, and the Vice-President of Gulf-Oil Corporation, Mr. W.L. Naylor, jointly signed the statement regarding exercise of the option and the license agreement. This statement became effective on September 15, 1954 when Karl Ziegler co-signed the document. The wording of the license agreement, thus covered polypropylene, but did not extend the licensed territory to include Mexico.

The license grant could only be non-exclusive and was contingent on payment of an additional \$250,000, \$150,000 of which was creditable against royalties subsequently due from production. Determining the running royalties proved to be complicated, because the licensee’s US representatives could not be definite as to whether or not US taxes should be withheld, and if so, how much. The parties agreed on percentage-based royalties, based on net sales values, to which tax at a rate of 0–30% would be added. If no taxes were due, the royalty rate would be fixed at 3.6%.

Koppers Company Inc., with headquarters in Pittsburgh, also moved more quickly than Hercules. As early as July of 1954, its European representative Mr. C.F. Winans offered Ziegler an option agreement [50] for polyethylene, which was executed by both parties on July 22, 1954. It covered the first five German applications filed in 1953 and early 1954. The agreement was effective for 1 year, with an option fee of \$50,000. In the event that Koppers took a license under the corresponding issued US patents, the down-payment would be \$350,000, with the running royalties calculated on the basis of the previously introduced descending scale of 4, 3 and, 2% of the net sales price. The remaining conditions were similar to the terms of the Hercules agreement, except that in this case, the licensed territory was limited to the United States. In May of 1955 [51], before the option period had expired, Koppers announced its intention to exercise the option, and thus became a very early licensee for the production of polyethylene. The required down-payment was made by the end of May. By June 1955, Koppers had already launched its new polyethylene product under the name of “Super Dylan” [52], listing its properties and utilities.

Actually, the number of US companies operating in this way under a Ziegler license should definitely have been sufficient to satisfy any market requirements. But the reverberation from the new developments at Mülheim was so immense that other companies tried to initiate a dialogue with Ziegler. Nevertheless, he put off Dow Chemical until November, even though Dow, through Mr. Hirschkind, “Technical Advisor to the President of the Dow Chemical Company” had approached Ziegler as early as July of 1954 [53],

“because our entire time was taken up by negotiations with future (particularly European) licensees to whom we are already contractually bound” [54].

Thus pressed for time, Mr. Hirschkind signed an option agreement [55], and on November 22, 1954 [56], the Executive Vice President of Dow Chemical Co. signed a license agreement for the production and sale of olefin polymers. Dow paid \$50,000 as a down-payment and, upon expiration of the option period on January 7, 1955, another \$350,000 in the form of a down-payment, \$200,000 of which was creditable against future royalties, whilst at the same time accepting the previously introduced sliding scale for running royalties. However, both parties were apparently not completely satisfied with the terms of the agreement, with Ziegler being the more dissatisfied party.

The great haste with which the draft agreement had been discussed by Dow and negotiated by Ziegler alone, was obviously the reason for the resulting lack of precision. Ziegler was soon to notice this himself: the contract did not contain a confidentiality clause, and the royalty obligation covered only the filed US applications which corresponded to the first three but not the later German applications. The latter point meant that Dow’s royalty obligation would cease if the US applications in question failed to mature into patents [57]. Ziegler was able to eliminate only one of the problems, i.e. in the event that his US and Canadian applications did not lead to patents, Dow would be obligated to pay royalties for any export to countries where Ziegler had patent rights [58]. All further requests for modification however, were ignored by Dow.

At the same time, Ziegler’s patent attorney, Dr. Andreas von Kreisler of Cologne, negotiated the terms of a license agreement with Union Carbide and Carbon Corporation (UCC) of South Charleston, West Virginia. The company maintained a European branch office in Geneva and a technical laboratory in Brussels, both designed for the purpose of effective public relations with UCC’s European business prospects. Between April [59] and November, 1954, UCC representatives attempted to initiate contact with Ziegler, but since they were unable to convince him of the sincerity of their approach, it eventually fell to von Kreisler as the middleman, to clear the way for discussions between his client UCC and Ziegler [60].

Ziegler harbored doubts as to whether it would be prudent, on top of the previously concluded agreements and the running negotiations with Dow, to accept a further licensee for the United States. On 10/28/1954 [61], Ziegler outlined his practical concerns in a confidential letter to von Kreisler and resolved to wait and see. This hesitation was rooted in the fact that, in the meantime, chemical literature had

been published which might prove potentially harmful to his patent position. Von Kreisler dispelled Ziegler’s misgivings, and on November 24, 1954 [62], an agreement was signed which, in consideration of a \$50,000 payment, granted UCC the right to an option for the United States and Canada, to exercise the option within 4 weeks, and to obtain a license under the terms defined in the same agreement: \$350,00 down-payment, with 50% creditable against future royalties, or \$450,000 down-payment, with the full amount creditable against future royalties. The option fee too, was to be fully creditable. Royalties were determined according to the familiar scale – 4, 3, and 2% of the net sales price – export was allowed without further payment, except to countries where exclusive licenses had already been granted, i.e. Germany, Great Britain and Italy. The license was limited to non-exclusive rights for the production and sale of polyethylene and co-polymers containing at least 50% ethylene.

UCC hesitated to effectively declare the exercise of its option during the option period. Word had spread in the United States that Phillips Petroleum had developed its own “low pressure process” for the polymerization of ethylene, presumably using “compounds of Group IV of the Periodic System of Elements, particularly, chlorides” as catalysts. This information was only partially accurate. UCC made an arrangement with Phillips according to which both companies apparently agreed to a mutual exchange of licenses [63]. Before Christmas 1954 [64], UCC decided to pay the larger of the two proposed down-payments and thus exercise the Ziegler option. The license agreement, *inter alia*, included the so-called “15-year clause” which provides that the licensee’s royalty obligation is limited to 15 years, starting with the first commercial sale. In a supplemental agreement dated November 1955 [65], this clause was revised to specify that the royalty period became effective on the date the first commercial plant went on stream. This royalty period was again to play a role in a different context.

Early in 1956, UCC decided to broaden the contract in two different directions: first, toward the production and sale of general polyolefins [66] for a payment of an additional \$200,000 (fully creditable against future running royalties) within 30 days, and second, toward the remaining processes encompassed by Ziegler chemistry, summarized and defined as “Restricted Field” [67], except for the sale of organoaluminum compounds, this latter restriction took into account the exclusive sales rights that had been granted to Hercules Powder Company. The fee for the “Restricted Field” agreement was \$480,000.

It was customary for US chemical companies, among others, to send representatives to Europe in the Spring and Fall of each year to personally obtain an overview of any interesting developments in chemistry that may have occurred, and where and to what extent such developments were being put into actual practice. Besides Union Carbide, Monsanto Chemical Company, with its headquarters in St. Louis, Missouri, also maintained a “Technical Representative” European branch office in Geneva. As early as March/April 1954 [68], such a routine meeting took place between Monsanto’s then representative, E.B. Seaton, and Dr. Koch of the Max-Planck-Institute, with whom he was acquainted. This procedure was repeated again in September, but this time with Karl Ziegler as the second participant, in the form of a detailed discussion of the catalytic polymerization of ethylene.

A letter [69] written by the General Manager of Monsanto Chemicals Ltd in London, J.W. Barrett, highlighted Monsanto's genuine interest. Nevertheless, Ziegler treated Monsanto only as an "interested party", which was understandable in view of the fact that a considerable number of companies had already entered into negotiations with him. Ziegler summarized the substance of the conversations that took place in late November/early December 1954 in a letter [70] to his patent attorney, von Kreisler.

One event now followed another in rapid succession. Ziegler's initial reserve notwithstanding, I.R. Wilson, Vice President, and R.K. Mueller, another Vice President and General Manager, Plastics Division, of Monsanto Chemicals Corporation, signed a draft option and license agreement even before Christmas of 1954, at a point in time, when in fact, Monsanto was building a high-pressure polyethylene production plant under the terms of an ICI license [71]. The final agreement was signed by Karl Ziegler in January of 1955 [72]: an option to obtain a non-exclusive license for the production and sale of polyethylene for a \$75,000 option fee, payable upon execution, with an option period of 4 weeks, and, upon exercise of the option, a \$325,000 down-payment for the license, \$200,000 of which was creditable against future running royalties, so that, as also specified in the other contracts, only half of the annual running royalties could be credited against the \$200,000. The running royalties were payable according to the same scale as set forth in prior agreements. As a "perk" Ziegler offered to ensure that the total down-payment, in other words the entire \$400,000, would be creditable against future royalties if Monsanto paid an additional \$200,000 prior to the start-up of commercial production. Monsanto made this payment in May of 1963, practically at the same time as its industrial plant went on stream.

To start with, a team of Monsanto's experts [73] visited the Max-Planck-Institute as early as January 1955: Dr. E.W. Gluesenkamp, Mr. Eli Perry and Dr. Richards, who together with Monsanto's European Representative, D.W. Weddell, handed over a check for the option fee and – as had occurred in all previous cases – were introduced by Martin to the subject matter of the licensed field. Subsequent correspondence [74] reflects the intensity with which Monsanto prepared for its commitment, which in February 1955, speeded up its exercise of the option and payment of the license fee. Monsanto's interests were by no means limited to the United States and Canada, since the company was engaged in worldwide activities involving polymer technology, but for now, Ziegler left this territorial restriction unchanged.

2.4.3

Esso, the Straggler

Belatedly and "startled out of its repose", Esso AG, Hamburg, contacted the Institute to convey the desire of its parent company, Standard Oil Development Company (S.O.D.) to be allowed to call on Ziegler in late November/early December 1954 [75]. A group of Esso delegates, made up of Vice Presidents W.C. Asbury and C. Morrell as well as P. Smith, paid a visit to the Institute at Mülheim on the Ruhr. Further preliminary negotiations were conducted by von Kreisler in Cologne. The parties en-

tered into a dialog which also covered subject areas beyond polyethylene. In January [76], in parallel with the Hercules agreements, Karl Ziegler drafted the outline for a “Technical Field” for von Kreisler covering these areas, whereby the specific terms, again provided for a division between polyethylene as a thermoplastic on the one hand, and “the remainder” excluding polyethylene, on the other. In this connection, he offered a license for the production of organoaluminum compounds for internal use, but not for sale, because of the exclusive rights he had already granted Hercules. Additionally he offered a license for the synthesis of alcohols.

The aim, however, that Mr. Asbury (S. O.D.) had in mind for these negotiations, as it soon turned out, was to obtain a polypropylene license at the lowest possible cost. Neither von Kreisler nor Ziegler had realized this at the beginning of the negotiations. Even though a license offer for polyethylene was on the table [77], the discussions very quickly led to an arrangement in the “Restricted Field” area, which now included polypropylene, higher polyolefins, and co-polymers.

At this juncture, at the latest, it should have been obvious to all participants that the proposed division into a polyethylene license agreement, on the one hand, and the remaining subject areas summarized in the “Technical Field” contract on the other, was no longer useful. At that time however, it was polyethylene which in Ziegler’s estimation, was obviously the product with the greatest potential for exploitation.

On January 26, 1955 [78], W.C. Asbury, as Vice President of S. O.D., later Esso Research and Engineering Company, with headquarters in Elizabeth, New Jersey, and Karl Ziegler put their signatures to an agreement, which, as an Appendix, incorporated the aforementioned correspondence regarding a “Restricted Field”, but did not include a polyethylene license. The contract package became effective on February 7, 1955. The option fee was \$175,000. This sum was relatively large, because the disclosure of the method for the production of polypropylene necessarily had to include disclosure of how to produce polyethylene, in other words, the amount included the option fee attending the “polyethylene” offer. Upon expiration of the 12-month option period, the agreement called for another \$425,000 as a fully creditable down-payment, in other words, the initial cost of the license was \$600,000. The running royalty was to be a flat 2% of the net sales price for all products, with an option to re-negotiate the royalty rate if a product price was realized that was considerably higher or lower than had been anticipated. The “15-year” clause familiar from the earlier agreements was replaced by an obligation to pay running royalties for a period of 17 years, starting at the earliest issue date of a patent covering the licensed production. As it later became apparent, the contract included one disastrous provision. Under this clause, S. O.D. had the right to withhold payment if they were sued for infringement, and furthermore, any costs incurred in defending the suit would be fully creditable against running royalties if the plaintiff was successful, and creditable at a 50% rate should S. O.D. be successful in having the complaint dismissed, concessions which, from today’s point of view, were unnecessary (see p. 169 and 178).

During negotiation of the terms of the license agreement which had been concluded in the meantime, the “polyethylene license” question had remained open.

Ziegler's offer of a polyethylene license – production and sale – had been on the table since early January 1955. On March 7, 1955, von Kreisler, on Ziegler's behalf, extended the option period for polyethylene until March 21, 1955 [79]. Two days prior to expiration, Mr. Asbury and US attorney Whelen [80] traveled to Cologne, where they met Karl Ziegler and Andreas von Kreisler, and *inter alia*, drew attention to two of Esso's own US patents dating back to 1938 and 1943, which claimed ownership of a catalyst made of aluminum trichloride/titanium alcoxide compounds for polymerization reactions taking place below 0 °C and/or of a process for the polymerization of olefins using methyl aluminum chloride compounds at room temperature. Additionally, Esso announced its plans for incorporating experiments conducted in 1942 on the polymerization of isobutene using catalysts consisting of ethylaluminum halides and/or aluminum triethyl and titanium tetrachloride, into a new application to be filed in the US Patent Office. The subject matter aroused great interest in view of the fact that 10–15 years earlier, Esso's researchers had come very close to approximating the Ziegler catalysts. At that time, isobutene had evidently shown greater promise than ethylene and propylene.

The revelation was apparently calculated to bring down the cost of the license. But an infringement opinion ordered shortly thereafter with respect to the Esso patent rights concluded that these rights did not present any threat to the validity of Ziegler's patents. Any major influence on Ziegler's position that Esso may have hoped for failed to materialize. The US negotiators walked away with merely a promise that the option period of both agreements would be extended until February 7, 1956. Finally, a memorandum was signed wherein Esso vouched that among other things, it would not assert either the two aforementioned US patents or the planned patent application against any Ziegler licensee.

The terms governing option periods and related payments were very confusing at that point in time. Therefore, for purposes of clarification, on June 23, and August 3, the parties executed [81] an "Agreement on Status ...", wherein Esso, again, undertook not to bring a suit against any Ziegler licensee based on the aforementioned Esso patent rights and/or newly proposed US patent application. The option period for the polyethylene field expired on the same date as the option term for the "Restricted Field" area, i.e. February 7, 1956. With respect to the advance payments, both paid and to be paid, the agreement made it clear that the \$175,000, remitted upon execution of the first option, and the \$1.025 million, to be paid no later than the expiration of the option period (February 7, 1956) in the event that both options (Technical Field and Polyethylene) were to be exercised, or \$625,000 in the event that only one option was to be exercised, would both be creditable against future running royalties (4, 3, and 2 % scale).

Even this arrangement was not final. In early January 1956 [82], S. O.D. pressed for an extension of the option period and, thus, its payment obligation. Von Kreisler, on behalf of Karl Ziegler, responded [83] by reaffirming that the option for polyethylene was to be exercised by February 7, 1956, with the concurrent payment of \$625,000, and agreeing to an extension of the option period for the "Restricted Field" until August 1, 1956, at which time a payment of \$400,000 was expected. On February 2, 1956, S. O.D., through Esso AG, Hamburg, paid 2,631,526 Deutsch Marks, the equivalent of \$625,000 (at a 1 : 4.21 conversion rate) [84].

Details on S. O.D.’s exercise of the “Restricted Field” option on August 3, 1956 [85] and additional \$400,000 payment to Ziegler will be described at a later point, since in order to fully appreciate the situation, the reader must be acquainted with certain facts which will be presented here in connection with the continuing developments at Montecatini.

2.4.4

Du Pont

Initial contact between Ziegler and Du Pont was established through Du Pont’s London representative, G.S. Garstin, and very hesitantly at that, in fact, not until the summer of 1954 [86], that is, at a time when four other companies [87] had already signed an option and/or a license agreement with Ziegler for the production of polyethylene. The parties tried to read each other so as to learn, on the one hand, what Ziegler’s catalysts were composed of, and on the other hand, how far Du Pont’s own findings and developments in the same area had progressed by that time.⁴⁾ At the end of November, the first discussion between the parties took place in Düsseldorf [88].

The emerging formulation of a contract was difficult under the circumstances, since Ziegler was not in a position to grant Du Pont any more favorable terms than he had granted to those licensees who had previously signed an agreement; but he also had to ensure that Du Pont would not hamper other producers on the strength of its own patent rights. The February/March 1955 contract [89] reflected the parties’ mutual distrust. Initially it became apparent that Du Pont, by its own evaluation, could only assert an effective patent position with respect to the production of polyethylene and catalysts, but not for polyolefins in general. On the other hand, Du Pont was willing to pay \$50,000 for immediate access to Ziegler’s patent applications. The agreement, therefore, included an option, while the license offer called only for running royalties, but not, as in other cases, for additional down-payments. As compensation in the event that Du Pont declined the license offer, Du Pont offered Ziegler’s licensees a license under its own dominating patents to the extent that they existed.

Beyond the free choice of either accepting or declining the license offer, Du Pont was not willing to disclose the contents of its own patent applications. The poker game played by Messrs. Habicht and McAlevy of Du Pont had paid off. Nevertheless, Ziegler initially felt relieved.

W.F. Gresham (Research Director at Du Pont), called on Ziegler in late March of 1955. He too, like Garstin, had received copies of Ziegler’s 1953 and 1954 priority applications.

Only 1 week later, in April of 1955, the General Manager of Du Pont’s Polychemicals Department, accepted the license offer and forwarded a license agreement [90] for the United States and Canada, signed by Du Pont, which was countersigned by Ziegler shortly afterwards. The contract spelled out rules for the contingency that

4) See Chapter 1, Section 1.1.3. Du Pont believed that it had a negotiation position on the basis of its patent applications of August 1954.

Du Pont would subsequently establish its own dominating patent position in the area of the licensed subject matter which, in principle, corresponded to the terms set forth in the preliminary agreement. As early as June 1955, Du Pont publicly announced that a pilot plant for the production of a new type of polyethylene, called Alathon, produced according to the licensed patents of Karl Ziegler, would go on stream in the Fall. These events were followed, 1 year later, by a second license agreement with Karl Ziegler [91] on the production of co-polymers of ethylene with other olefins, whereby the ethylene content of the co-polymers was to be at least 50 mol%.

This limitation had become necessary in light of the developing relationship between Ziegler and Montecatini. Execution of the license agreement for the United States and Canada was based on a down-payment of only \$200,000, while the provision for running royalties called for the customary percentages.

This was the first contract which provided for the contingency of Ziegler applications becoming involved in interference⁵⁾ proceedings with Du Pont patent rights. It was agreed that in that case, running royalties would be paid into an escrow account which was to be closed in the event of a dispute with the monies being distributed either entirely to Ziegler if he prevailed, or 50/50 if Du Pont was successful. Additionally, both the Du Pont/Ziegler agreements contained the previously mentioned “15-year” clause.

2.5

Mitsui Chemical, the First Japanese Licensee

Mitsui's elderly President Ishida had visited the Max-Planck-Institute in late 1954. The news regarding the innovative Ziegler catalysts had not bypassed Japan, and specifically, had not gone unnoticed by the numerous Japanese industry representatives in Europe. Mr. Ishida sat down on a laboratory stool at the Institute and watched as a normal-pressure ethylene polymerization experiment was being conducted before him. As the suspended polyethylene powder formed during the process started to emerge, he jumped up and searched the equipment for the mysterious source of the polymer powder. He rushed to Ziegler to ask, in a downright demanding manner, for a license – with exclusive rights for Japan, of course.

The prevailing consensus of opinion in those days regarding the attitude of Far-East contract partners toward agreements, was that the stiffest possible financial terms should be imposed in order to ensure protection against the risk of potential disputes arising, if for no other reason than as a result of communication difficulties due to the language barrier – this was a misconception. In fact, it was with extraordinary loyalty and letter-perfect precision that the Japanese licensees fulfilled any contracts they had entered into. As early as December 21, 1954 [92], the parties exchanged draft agreements regarding an exclusive option for a license for the production of polyethylene.

5) “Interference” see Chapter 1, footnote 1.

Ziegler actualized his ideas based on the above-mentioned beliefs by making the following stipulations to the agreement: a limited option period of 3 months; limited export – essentially restricted to Asia, Africa, Australia – this was to circumvent the risk of the Japanese flooding the market in countries where there were already existing Ziegler licensees; \$150,000 for the option [93] to obtain an exclusive license, and an additional down-payment of \$1,050,000 upon exercise of the option; and further patented Mitsui improvements were to be free of charge to Ziegler in Germany. On December 31, 1954 Ziegler requested that the Japanese signed the document no later than January 7, 1955 in order to make this option agreement legal and binding.

By early January 1955 [94], everything was in place. There was, however, no way of estimating the time it would take before Mitsui Chemical could obtain approval of the agreement together with payment authorization from the Japanese government agency “MITI”. Never before had MITI approved amounts of this magnitude for a license. Communication with Japan was difficult. The exchange of documents was mostly handled by Japanese couriers. In February of 1955, the Japanese became acquainted with the contents of the first three German patent applications [95]. In March, three chemists [96], M. Suzuki, T. Suzuki and K. Yamamoto, arrived at the Institute and studied the process on the basis of the additional information provided to them.

In late June/early July, both parties signed the license agreement [97]: an exclusive license for the production of polyethylene, including the production of the polymerization catalysts required. As was requested in existing European and US licenses, a running royalties clause according to the customary 4, 3, and 2%-scale was stipulated.

In support of the application filed for approval with the Japanese government, Ziegler furnished promotional material [98], such as a brochure from the US company Koppers, an announcement by the US company Hercules Powder to budget 10 million dollars for the construction of an industrial polyethylene plant, and a publication by Union-Carbide disclosing that they had started operation of a pilot plant for the production of Ziegler polyethylene. Additionally he provided some samples of the new polyethylene with different molecular weights. Nevertheless, the MITI government agency was very non-committal. ICI had licensed the competitive process (high-pressure polyethylene) in Japan to Sumitomo⁶). MITI demanded revision of the contract [99] with respect to Ziegler’s offer, according to which the agreement was to be changed to now provide for a 15-year effective term with an option for prolongation. Ziegler accepted [100], and in early November 1955, government approval was granted [101].

6) Further competitors in the field of polyethylene were Mitsubishi Petrochemical, a licensee of BASF, Furukawa Chemical, (“Stafilen”) a

licensee of Standard Oil of Indiana and Showa Denko, (“Sholex”) a licensee of Phillips Petroleum.

2.6

Summary

Table 2.1 summarizes the achievements of 1954 to 1955. Within a period of approximately 20 months, the income from the United States alone was more than 4 million dollars, which, at the prevailing exchange rate, amounted to almost 17 million Deutsch Marks. Payments made by the licensees were contractually non-refundable. The Institute's then annual budget was 1.2 million Deutsch Marks.

At that time, there were in existence only seven German patent applications, none of which had been examined yet by the German Patent Office, and the "demonstration lesson" of a polymerization experiment in a glass vessel using an active catalyst, as well as the utility of the polymer powder produced.

The option periods granted became progressively shorter in each new instance and the down-payment higher, and, except in the case of Esso, the option periods were not extended – a unique situation. The financial cushion appeared ample enough to allow for any future problems to be tackled without pressure. For 40 years,

Table 2.1 US Licence Agreements 1954 – 1955

Company	Contract Object	Teritory Countries	Option Agreement	Option Period days	Option Payment \$	Option Exercise	License Agreement	License Payment \$
Goodrich	Polyolefines	USA	07,27,54	45	50,000	09,03,54	09,15,54	250,000
Gulf	Co-polymers	Canada						
Koppers	Polyethylene Co-polymers	USA	07,22,54	360	50,000	05,03,55	07,22,54	350,000
Hercules Powder	a) "Ziegler- Processes"	USA Canada	Letter Agreement 09,11,/ 19,52	270 360	10,000 50,000			
	b) Polyole- fines		09,24, 54			09,12,55	09,24,54	300,000
Dow Chemical	Polyolefines Co-polymers	USA Canada	11,22, 54	30	50,000	01,07,55	11,22,54	350,000
Union Carbide	Polyethylene Co-polymers Polyolefines	USA Canada	11,23, 54	30	50,000	12,23,54	11,23,54 01,21,56	450,000 200,000
Monsanto	Polyethylene	USA Canada	01,10, 55	30	75,000	02,07,55 05,10,63	01,10,55	325,000 200,000
E.I. Du Pont	Polyethylene	USA	02, 03, 18,03,55	30	50,000	04,07,55	04,03,55	0
Esso	Polyethylene "Restricted Field"	USA Canada	02,07,55	360 510	0 175,000	02,07,56 08,01,56	02,07,56 02,07,55	625,000 400,000

* Including polyolefines.

longer than a whole generation, the Max-Planck-Institute for Coal Research profited from the ongoing exploitation of the above-described basic patent rights. The prospective US licensees had pursued their licensing activities in a much more focused manner that had their European counterparts in the meanwhile. Companies based on broad hydrocarbon chemistry tried to obtain “last minute” licenses. Such an occurrence was then known as “Hunting License”.

In Europe in 1954, Montecatini in Italy, Petrochemicals Ltd in England, Farbwerke Hoechst and Steinkohlenbergbauverein in Germany had each entered into a license agreement. The total income from these contracts, up to the end of 1954, was 1.7 million Deutsch Marks. The exclusive licenses and, therefore, non-existent competition impeded the rapid growth of the licensing business.

2.7

Back to the Ruhr: Ruhrcoal and Bergwerksverband

As the representative of Ruhrcoal and its affiliated industry, it behoved Steinkohlenbergbauverein (Coal Mining Society) to implement the agreement entered into with Ziegler in March 1954 (see page 47). It was, however, not until August 1954 [102] that Chairman, retired General Manager and retired Mine Assessor, A. Wimmelmann, in a letter to the members of the Board of Directors of the Society, convened a meeting for September 14, 1954 for the purposes of adopting the final Articles of Association of the newly to be established “Syndicate for Olefin Chemistry”. The interested mining companies had already been in agreement regarding the necessity for forming such a syndicate [103]. The by-laws adopted at the meeting provided, *inter alia*, for a standing committee [104] which, at the time, consisted of Messrs. Broche (Ruhröl), Chairman, Söhngen (Rheinstahl) Deputy Chairman, Wimmelmann (Steinkohlenbergbauverein), Braune (Mannesmann), Busch (GBAG), Curtius (Rheinpreußen), Rindtorff (Hibernia) and Tramm (Ruhrchemie). The object of the syndicate [105] was to establish an association of interested parties among the Steinkohlenbergbauverein member companies for purposes of exploiting the Studien- und Verwertungsgesellschaft agreement ⁷⁾.

At the end of October 1954, [106] the Manager of the Syndicate for Olefin Chemistry, Dr. Heinz Reintges, informed Ziegler that Deutsche Erdoel-AG, through Steinkohlenwerk Graf Bismarck as a member of the Steinkohlenbergbauverein, as well as Ruhrchemie, Hibernia, Gelsenkirchener Bergwerks AG (GBAG) and Mannesmann, had gained approval for building pilot plants for the polymerization of ethylene. The number of interested parties and approval for the operation of pilot plants was extended on February 4, 1955 [107] to also include Arenberg-Bergbau-Gesellschaft, Essen, Rheinpreußen AG für Bergbau und Chemie, Homberg, Steinkohlenbergwerk Hannover-Hanibal AG, Bochum, and Krupp Kohlechemie GmbH, Wanne-Eickel.

7) See page 41 and 42.

At approximately the same time, the Syndicate for Olefin Chemistry (AfO) urged that the March 1954 license be extended to include all European countries, except Germany, Great Britain and Italy. For the first time, the Bergwerksverband (Mining Association) claimed the right to charge a percentage surcharge, up to 25%, as a service fee for its efforts each time a sub-license was granted. Ziegler wanted to curtail this practice by pointing out that “according to its own, repeatedly stated explanation, the sole purpose of Steinkohlenbergbauverein acting as an intermediary in connection with the granting of licenses was to exercise reasonable control over licenses and manufacture”. And further: “No mention had ever been made that perhaps a considerable portion of the royalties collected should remain in the hands of Steinkohlenbergbauverein and/or its successor.” [108]

Bergwerksverband GmbH (BMV) as the successor to Steinkohlenbergbauverein subsequently enforced the latter’s interests, albeit in a limited form. In hindsight, BWV’s attitude on this issue would have to have been accepted, particularly because –as will be described later – Studiengesellschaft Kohle GmbH’s⁸⁾ share-holders were excluded from sharing in the profit from the license fees. After all, the influx of annual investments on the part of Bergwerksverband into the Institute would have to generate a certain financial backflow. The fact that the BWV-affiliated companies merely received a preferred option could not possibly be sufficient, particularly in view of the fact that in such cases additional, investments were initially necessary in order to realize adequate profits from any potential production.

(BWV’s contractually assured share of the royalty income received from any BWV-procured licenses was up to 25% of the running royalties. In actual fact, the percentage rate was between 7.5 and 25%).

As the situation progressed, it became evident that BWV actually generated a surplus between income and expenditure for patent rights and their defense through the Max-Planck-Institute for Coal Research and Studiengesellschaft Kohle mbH – Bergwerksverband had taken on that contractual obligation which was approximately 25% based on BWV/AFO’s total income [109].

The “Europe” agreement signed on December 20, 1954 [110] included a provision calling for compensation of Bergwerksverband GmbH. In a cover letter accompanying the agreement, “Studien- und Verwertungsgesellschaft [111]” stated that a prerequisite for this compensation was that Steinkohlenbergbauverein would continue its financial support of the Institute provided that they were apprised of any changes in monetary value, and that otherwise the planned compensation would be adjusted to the new circumstances. This, however, never happened.

Steinkohlenbergbauverein had assigned all rights and obligations of the first 1954 Agreement to Bergwerksverband GmbH (BMV). One year later this transfer was officially confirmed [112].

The “Europe” agreement proper defined the subject matter covered by the contract as polymerization of olefins and the production of catalysts required for the

8) Successor of Studien- und Verwertungsgesellschaft mbH (see p. 41).

process. Sub-licenses granted by BWV presupposed that any parties acquiring either licenses or options were willing to make appropriate down-payments either upon execution of the agreement or at the start-up of production. In a further letter accompanying this agreement [113], BWV undertook to guarantee that by January 10, 1957, the total amount of down-payments rendered by third parties to Ziegler/MPI would reach 1.5 million Deutsch Marks. Royalties were to be fixed between 4 and 5%. BWV was liable for any expenses incurred in connection with the patent rights in question, including litigation costs.

While no payment was due from Bergwerksverband upon execution of the agreement, the latter was merely authorized to grant options and licenses on behalf of Studien- und Verwertungsgesellschaft.

A further agreement, entered into by and between Studien- und Verwertungsgesellschaft mbH and Bergwerksverband GmbH in December of 1955, expanded the licensed territory to include Central and South American countries as well as Asia and Africa, with all other terms and conditions remaining the same [114].

The list of countries did not mention Australia, New Zealand or the Union of South Africa. Developments during the next few years required the agreement to be amended in that respect. In 1960, these three countries were included by way of a supplemental agreement [115].

Now Bergwerksverband GmbH requested that the first March 1954 agreement too, be upgraded to spell out, in writing, terms for a “remuneration of BWV”. The contract was replaced by a new one, which, although signed on December 15, 1955 [116], was effective as of the date of the first agreement of March 1954. All other terms and conditions of this contract, had, on the one hand, been adopted from the first agreement and, on the other hand, conformed to the “Europe Agreement”. In other words, instead of the originally intended exclusive license, BWV merely received authorization to grant options and licenses [117] in the name of Studien- und Verwertungsgesellschaft. This also included BWV’s general option to acquire any continuing patent rights owned by Studien- und Verwertungsgesellschaft, and, bearing any costs incurred in connection with the latter, the right to grant licenses thereunder in the name of Studien- und Verwertungsgesellschaft. The period for declaring its exercise of this option was set to expire after 1 year.

In accord with Studien- und Verwertungsgesellschaft’s Board of Directors and the Administrative Board of the Max-Planck-Institute for Coal Research, Karl Ziegler endeavored to make sure that even the very first profits derived from the patent rights would remain with the Institute. In 1955, with the active support of Retired Mining Assessor Hermann Kellermann, who, after Dr. Springorum, had reassumed his position as Chairman of the Board, attempts to obtain the partners’ consent for an appropriate amendment of Studien- und Verwertungsgesellschaft’s Articles of Association were successful.

In accordance with the new by-laws, the company’s name had now been changed to Studiengesellschaft Kohle mbH, and its sole and immediate purpose, as stipulated, was the advancement of the aims of the Max-Planck-Institute for Coal Research, with the company acting as a Trustee for the Institute [118]. The profit would not be distributed to the partners. As compensation to the partners, the Director of

the Institute contractually undertook [105, 106], by way of a preferred option, to allow Bergwerksverband first and unrestricted access to any inventions originating from the Institute. The structure of this contract which secured the financial foundation of the Institute while at the same time protecting the interests of the companies in partnership with and having an interest in Studiengesellschaft, was certainly progressive and worked in a positive manner for all parties involved.

At the Special Partners Meeting of December 22, 1955, the partners passed Studiengesellschaft Kohle mbH's new by-laws which, to a large extent, would allow the company to enjoy the advantages of a non-profit organization.

As stated by Karl Ziegler, in 1955 [119]:

"The Finance Minister of the Land of Northrhine-Westfalia, in his letter of March 5th of this year, has endorsed these by-laws and, in that connection, noted that the granting of licenses, by the Institute or Studiengesellschaft as a Trustee for the Institute, under patents growing out of the research work done at the Institute, does not constitute a commercial business on the part of the Institute. This statement, which we had aimed for and which has now been expressed, is of crucial importance to us, because it means that any license income received by the Institute would be one-hundred percent tax-free. The Finance Ministry, on the other hand, failed to also accept Studiengesellschaft Kohle as an all-around, generally non-profit organization, because, as a Trustee for the Institute, it would fulfill the non-profit purpose, i.e., advancement of the sciences, not in its own right, but, as a trustee, do so only in an indirect manner. No tax or other disadvantages flow from this, or at least not to any significant extent, so that I can state that we have accomplished the intended goal."

The granting of licenses through BWV in Germany, Europe and countries outside of Europe got off to a slow start. By the end of March/April 1955, both Deutsche Erdöl AG [120] (DEA) and Mannesmann [121] obtained a license under identical terms, for the production of polyethylene and co-polymers, each license incurring a \$100,000 down-payment. The contracts defined the licensed territory as the Federal Republic of Germany, a non-exclusive grant, of course, calling for running royalties of 3 % up to 600 tons, 2.25 % for any additional 6000 tons, and 1.5 % for production in excess thereof, as well as a minimum royalty payment beginning in 1958. The intent was to operate a pilot plant as a joint venture with Mannesmann AG of Düsseldorf and Farbwerke Hoechst of Frankfurt and, in a commercial plant, to produce 6000 tons annually starting in April of 1957, and 12,000 tons annually beginning in October 1957. This established the capacity limit up to that amount.

In May 1955, Rheinpreussen [122] and Ruhrchemie AG [123], who were each paying 50,000 Deutsch Marks, were granted an option to obtain a license (polyethylene and co-polymers), which, in 1959, [124] and/or 1957 [125] led to the signing of the license agreements. It was noteworthy that the agreement with Ruhrchemie AG was made retroactively effective as of January 1, 1954.

Table 2.2 License Agreements for polyethylene and co-polymers in the Federal Republic of Germany granted through Bergwerksverband GmbH since 1954

Company	Option Agreement	Option Payment DM	License Agreement	License Down-payment DM
DEA	–	–	03,30,04,29,55	100,000
Mannesmann Kokerei AG	–	–	03,29,55	100,000
Rheinpreussen	05,09,55	50,000	08,31,11,21,59	100,000
Ruhrchemie	05,26,55	50,000	05,03,57	100,000
Gelsenkirchner Bergwerks-AG	01,11,56	100,000	03,10,65	100,000
Chemische Werke Hüls AG	–	–	05,18,05,23,55	100,000
Hibernia AG	–	–	09,23,55	100,000

In 1956, Gelsenkirchner Bergwerks-AG had obtained an option for a license to produce polyethylene, but did not sign the license agreement until 1965 with the intent of transferring it, within that same year, to Chemische Werke Hüls, of Marl [126]. “Hüls” already owned a license, dated 1955, for the production and sale of polyethylene [127], for a down-payment of 100,000 Deutsch Marks, and an additional contract, dated 1957, for the production and sale of polyolefins [128] (polypropylene and polybutene), including co-polymers, for an additional down-payment of 75,000 Deutsch Marks.

In the Fall of 1955, BWV and the mining company Hibernia AG entered into a license agreement [129] incorporating practically the same terms as those granted to the previous licensees. In the Spring of 1958, for a payment of 50,000 Deutsch Marks, the agreement was broadened to include polypropylene [130].

As may be gleaned from Table 2.2, the BWV member companies had each received unusually favorable up-front payment terms for obtaining a license. The total amount paid by seven companies in option fees and license down-payments was 900,000 Deutsch Marks. Only three of the above-named licensees – Ruhrchemie AG, Chemische Werke Hüls AG and Hibernia AG – developed the process to a state ready for marketing and went on stream with polyolefin production, but did so after Farbwerke Hoechst had already entered the market in 1955.

2.8

August through December 1954: Montecatini steps up its own Developments

G. Natta, in his above-mentioned letter to Karl Ziegler (see page 23 [171]) not only indicated that by way of X-ray analysis he had discovered an approximately 45% crystalline content in the polypropylene sample which Ziegler had sent to him in July of 1954, and that he had already claimed to have obtained similar products back in

March, but, along with further crystallographic data on the polypropylene, he also enclosed a sample of highly crystalline polypropylene, which had “recently” been produced. This product was apparently the result of an optimal extraction technique, which the Milan researchers regarded as significant.

Nowhere did this letter refer to the arrangement agreed upon in March of that year between Orsoni, on one hand, and Ziegler, on the other, regarding the division of future work in that field. But, to make matters worse, Natta announced that he was intensifying his research⁹⁾ in this field:

“We are convinced that the field is very interesting and requires an enormous amount of work in order to gain an overview. I hope to be able to meet with you in the near future, so that we may discuss these problems in detail.”

Despite Ziegler’s indication that he would pay a visit to Orsoni in Milan after returning from his August vacation, it is not certain whether this visit ever took place; even if it did, it was a very brief visit which had no significance. The period between September and December of 1954 was taken up with licensing negotiations at Mülheim, involving prospective US licensees, which hardly allowed Ziegler time to conduct negotiations with Montecatini, a situation which helped him to distance himself from Montecatini’s and Natta’s disappointing conduct. But then, in late December of that year, Orsoni, DeVarda and Natta did, after all, travel to Mülheim. The effect of this visit was that the guests insisted that, during their previous visit in May, Ziegler had denied the possibility that propylene could be polymerized, while, on the other hand, Natta downplayed propylene polymerization as nothing special, since plastic-like polypropylene was already known. This unsubstantiated statement formed the basis on which Natta promoted the continuing developments which he himself had achieved during the preceding months, as a “great invention” [131].

The matter under consideration was an attempt to selectively alter the crystalline content of the polypropylene products produced. The method met with success when the effect of the solid catalyst components, on one hand, and that of the dispersed and soluble components, on the other hand, on the polymerization reaction was traced by subjecting the resulting products to X-ray crystallographic analysis. It became apparent that the titanium trichloride and/or titanium trichloride/titanium dichloride mixture precipitated during preparation of the catalyst and in combination with alkyl aluminum compounds, caused the formation of markedly higher-crystalline polypropylene than did the soluble or finely dispersed catalyst components.

While, in the patent applications filed in June and July of 1954, Natta referred to the catalysts employed by him as “Ziegler catalysts”, he considered the catalyst preparations which had now been physically modified to be something new.

The crystalline content of the solid titanium trichloride component was now being enhanced by tempering or by modification of the production methods. The

9) During the next 5 years Natta and co-workers published 170 papers. During this time the

Natta-Institute had 100 co-workers in this field.

Italian patent applications [132], [133] filed in December of 1954 gave the impression that these were new inventions.¹⁰⁾

The Laureation Address during the award of the Nobel Prize to G. Natta (Chapter 1, page 27) revealed the belief, even at that time – in 1963 – that “certain types of Ziegler catalysts” might be capable of solving this problem.

What became of the first applications on this subject matter and how their contents was being judged will be discussed at a later point. For now, suffice it to say that in 1973 (see page 174) and/or 1982/84 (see page 201/202), some of the highest patent appeal courts [134] in the United States had found that such catalysts, consisting of crystalline titanium trichloride combined with alkyl aluminum compounds, were Ziegler catalysts, and

“come under the definition of a pioneer patent covering a function never before performed”,

and/or

“Regardless of whether the TiCl_3 is produced by reducing TiCl_4 with aluminum powder, with DEAC (diethylaluminum-chloride), ... the TiCl_3 produced is still a salt of titanium and chlorine ... TiCl_3 molecule, described as $\beta\text{-TiCl}_3$, $\alpha\text{-TiCl}_3$, $\chi\text{-TiCl}_3$ and $\delta\text{-TiCl}_3$... are all titanium salts.”

There could be no recognition of an independent invention under the patent laws.

2.9

The first Ziegler/Montecatini Pool-Agreements

On February 9, 1955, in a letter [131] to his patent attorney von Kreisler, Ziegler summarized the criteria which formed the basis of the position he would adopt during the course of further negotiations. In recounting the events, Ziegler went right back to the second half of 1953 and pointed out that Montecatini had been advised about the new polyethylene developments at a very early stage, even though, when interpreting the first agreement (in January of 1953), Ziegler had been doubtful as to whether the new catalysts would even fall under this agreement. Up to the time of the meeting on March 8, 1954 in Milan, G. Natta, according to his own statements, had merely carried out kinetic studies on the aluminum trialkyl – ethylene reaction and published his findings. Even then, Ziegler was puzzled at such practice, since after all it was not customary anywhere in the world, for any party who had received confidential advance notice of developments in a given scientific field, to actively seek publication. Ziegler then discussed the minutes taken during the March 8 and

10) The applications (U73 and U73 a) were combined in foreign countries. In Germany the first (U73) was issued as DEPS 1 302 122 on September 13, 1979, 7 years after the legal life

time of 18 years. This patent right was heavily opposed by objecting parties during the prosecution phase.

9, 1954 visit and complained that, with the help of these minutes, Professor Natta had obtained Ziegler's approval for conducting investigations.

For purposes of improving the Ziegler catalysts so that they could be employed for either higher- or lower-crystalline polypropylene production, Ziegler urged von Kreisler to check the texts of any Montecatini patent applications thus far unknown at Mülheim, for any truly patentable elements.

In early March 1955, von Kreisler traveled to Milan with the aims of negotiating a resolution to the obviously conflicting views held by the parties regarding the history of polypropylene invention and the ensuing consequences, and establishing criteria for future handling of the mutual contractual relationship. On March 13, 1955, "binding guidelines [135] for revision of the January 21, 1953 agreement between Montecatini and Ziegler" [136] were established and executed.

Of the seven "Montecatini" patent applications which had meanwhile been filed in Italy, five, including the "Higher Polyolefins" patent application Ziegler had filed in Germany in August of 1954, were to become the subject of a special arrangement (pool). In this context, Montecatini demanded that the first two of its applications (June and July, 1954, Chapter 1, references [165, 167]), together with the aforementioned Ziegler application (Chapter 1, reference [182]), be filed in combined form, jointly by Montecatini and Ziegler in countries outside of Italy and Germany, and that the proceeds from the joint exploitation of any resulting patents be split 50/50 between them.¹¹⁾

It had, thus, been worthwhile for Natta/Montecatini to ignore the March 8, 1954 agreement and polymerize propylene with Ziegler catalysts, and to file a patent application covering this method. Montecatini's stringent demand was to be sweetened by the fact that three additional Montecatini applications would also be included in the pool under the same conditions. These involved the polymerization of propylene using a catalyst combination of iron compounds and organoaluminum compounds [137] and – as discussed above – two further applications covering methods for controlling the crystalline content of polypropylene by electively using solid, particularly crystalline transition metal compounds, especially low valent titanium halides and/or soluble, dispersed transition metal compounds. The applications had been filed by Montecatini shortly before the trip to Mülheim, i.e. in December 1954.

There remains to be mentioned the fact that the guidelines stipulated further stated that any Italian patent rights now covered by the Pool Agreement would be the exclusive property of Montecatini, while Ziegler would be the exclusive owner of any patent rights in Germany. The same rule applied to the exploitation of the patent rights.

11) With regard to the USA, Montecatini additionally requested that the patent applications mentioned should kept separate and that the prosecution should be guided by Montecatini's US patent attorneys. Whether it was an act aimed at controlling the prosecution of the

first Ziegler-polyolefin application or, whether for reasons connected to the anti-trust law, the basis on which these decisions were made by Montecatini was not known at this time. It later became apparent that both reasons played a role.

By incorporating a number of exceptions, Montecatini shifted the outcome further in its favor. Thus, on a worldwide basis, they claimed any methods of utilizing the products described by them, to the extent that they were not disclosed in any of the Ziegler applications and were not produced with transition metal compounds of Groups IV to VI as catalyst components. They also insisted on making the final decision with respect to any licensing terms granted to third parties, with Montecatini accepting only one obligation, i.e. to grant licenses to US companies who had previously signed license agreements with Ziegler: Goodrich, Dow, Union Carbide and Esso.

In the co-polymer field, Ziegler managed to reserve for himself the right to the exploitation of those patent rights claiming the production of co-polymers with an ethylene content of more than 50%. The subject matter of the remaining two patent applications [138], [139] will not be discussed here. They were significant only to the extent that they increased the number of Montecatini applications vis-à-vis the single polypropylene application in Ziegler's name and, thus, produced a psychological effect.

It will become apparent later on that Montecatini repeatedly used such leverage as a tool to reduce Ziegler's share.

This was evidently all that could be achieved without filing a complaint and referring the matter to arbitration. Montecatini had at least two "feet in the door."

The prospective agreement was also intended to incorporate the provisions of the January 21, 1953 contract, that is, the terms of an exclusive license for Italy, and thus establish a legal foundation for the future. In this context, the fact had to be taken into account that Bergwerksverband GmbH had meanwhile obtained the right to grant licenses under the Ziegler patent rights in Germany, so that the parties had to make sure that Bergwerksverband GmbH approved of the terms and conditions being negotiated for Germany. Under the new contract, no additional payments were required for polyolefins.

On August 27, 1955 [140], the negotiated agreement was signed¹²⁾ in Basel by Dr. von Kreisler, acting under Ziegler's general power of attorney, and Engineer Giustiniani on behalf of Montecatini. In a cover letter [141] dated August 9, 1955, Bergwerksverband had declared its consent and, on September 21, 1955, in modified form [142], had confirmed to Montecatini that Bergwerksverband had granted no exclusive licenses in the contract territory, taking note of the fact that any inventions which fell within the established parameters of the subject matter under contract would remain the exclusive property of Montecatini, but that Ziegler would be granted an option as preferred buyer of the licensing rights for Germany. This referred particularly to textile fibers and elastomers. The intention was to avoid any conflict between Ziegler/Bergwerksverband, on one hand, and Ziegler/Montecatini, on the other.

12) See letter from von Kreisler to Montecatini [144] confirming the changes made by Giustiniani to the contents of the agreement.

During the final phase of the negotiations, Montecatini requested a declaration from Ziegler

“that his (my) agreement with Montecatini of January 21, 1953 in no way restricted either research or the filing of patent applications on the part of Montecatini ...”.

On August 5, 1955 [143], Ziegler formally complied with this request. The request was obviously intended to solicit Ziegler’s confirmation that the March 8, 1954 agreement would not block either Montecatini’s own research efforts or their freedom to file patent applications. The reason for such a declaration was evidently to give Montecatini a free hand to continue its past practices, in accordance with which Montecatini had even rendered financial compensation for the right to conduct research in the field of the subject matter under contract, to publish findings, and to file patent applications in its own name.

In return, Montecatini granted Ziegler

“the right, as preferred buyer, to potentially secure licenses for Germany under the patent rights it obtains for inventions that it was to make independently in the subject matter field under contract. In all other countries, the right to grant licenses under such patent rights is reserved for Montecatini. Montecatini, however, in the event that Ziegler grants any licenses under these patent rights to other licensees, will not make unreasonable demands and will also consider any wishes Ziegler may have. Joint inventions of Montecatini and Ziegler will be jointly filed as patent applications and jointly exploited, whereby in the case of licensing in third countries both parties will have the right of co-determination.”

In terms of time however, the March 1954 agreement came after the aforementioned 1953 contract and contained a clear accord for the division of research projects between the parties.

The protocol used by Multinational concerns and individuals for handling confidential information differs significantly. Undertakings, stated either verbally or presented in a form other than a formal contract, may now and then be disregarded.

After the conflict with Montecatini had been settled temporarily, Ziegler realized that the contractual arrangement which had been entered into between the two parties did not include a guarantee for his US licensees to produce and sell polyolefins without infringing any of Montecatini’s patent rights. After all, a number of US license agreements, particularly those entered into during the period 1954/55, did contain a warranty that the licensee owned a license to produce, for example, polypropylene. Furthermore he realized that there would be an ongoing and ever-increasing risk of his US licensees being substantially blocked by Montecatini’s patent applications.

As early as January 24, 1956, Ziegler and Montecatini signed a second agreement [145] (Pool 2), which provided that now all patent rights owned by either contract partner up to January 1, 1960 would be included in the agreed upon Pool.

Montecatini insisted that in future, its attorneys and not von Kreisler should prosecute the patent rights. Ziegler relinquished a portion of his projected income, the allocation formula was modified from 50/50 to 70% Montecatini/30% Ziegler. In return, Montecatini undertook to grant licenses in the United States and Canada to Ziegler licensees already-under-contract, i.e. Goodrich Gulf Chemicals Inc., Dow Chemical Company, Union Carbide and Carbon Corporation, Hercules Powder Company, Inc., and Esso Research and Engineering Company, such licenses being confined however, to the commitments Ziegler had made to the companies in question. Furthermore, Montecatini waived its share of any payments rendered to Ziegler by such US licensees.

US licensee Hercules Powder Co. had already approached Montecatini in order to explore the possibility of obtaining a license. Future developments were to show that it was not possible for the unlicensed competitors in the United States, on one hand, and Montecatini, on the other, to settle their differences without the intervention of the courts.

2.10 Polydiene

2.10.1

Karl Ziegler and H. Martin/Max-Planck-Institute for Coal Research; S. E. Horne/Goodrich Gulf Chemicals Inc.; Giulio Natta and Co-Workers/Montecatini; D.R. Smith and R.P. Zelinski/Phillips Petroleum Co.

The sequence of events leading up to the conversion of conjugated dienes such as isoprene and butadiene, into high molecular weight polymers using Ziegler catalysts was not only dramatic but also suspenseful. And especially because many of the details actually passed unnoticed, the outcome does properly belong here. The selective effect of the Ziegler catalysts had first been recognized at the Mülheim Institute in mid-1954 (see page 25).

A further example demonstrating the highly selective effect of the Ziegler catalysts which initially attracted less attention in Europe than did the developments in Milan, was provided by personnel at B.F. Goodrich and the Gulf Oil Company in Akron, Ohio. Goodrich's Research Director, Dr. Waldo Semon, advised Karl Ziegler late in 1954, approximately 2 months after conclusion of the license agreement under the Ziegler polyolefin patent rights, that co-workers had polymerized isoprene using Ziegler catalysts, to obtain polyisoprene which, due to its high *cis*-1,4 content, exhibited the properties of natural rubber (page 29). He further stated that his assistant, S. E. Horne, had repeated the steps for polymerizing ethylene which he had learned from Martin after the Goodrich/Ziegler option agreement had been signed, and had confirmed the results. During the course of his further work, he attempted to enhance the heat resistance characteristics of the polyethylene by adding small amounts of isoprene. The aim was to cross-link the polymer using the tech-

nology which had already been established in the production of butyl rubber. The product, when subjected to infrared analysis, proved to be a mixture of polyethylene and polyisoprene. After removal of the polyethylene, the polyisoprene was shown to have the structural characteristics of natural rubber.

In comparing the test parameters with those employed at the Max-Planck-Institute in January of 1954 (Chapter 1 reference [109]), it is noteworthy that Horne had chosen isoprene, while Martin had used butadiene, and that Horne had employed the same aluminum alkyl (aluminum trioctyl) but in a considerably smaller mol ratio to the titanium tetrachloride, i.e. 1:1, with the same absolute amount of titanium tetrachloride.¹³⁾ The external conditions (inert hydrocarbon solvent, temperature and reaction time) were practically identical. The yields – Horne, 55 g; Martin, 38 g – were of the same order of magnitude. There were, however, three critical features which would account for the difference in product evaluation: For one thing, the highly sophisticated method of analysis of polydienes particularly infrared analysis, was a standard resource for a major cautchouc and rubber processing company and was supported by ample experience, but which the Max-Planck-Institute in Mülheim did not have access to in that form. Further, there was no guarantee that the monomers would be available or of sufficient purity at Mülheim. Since Horne at Brecksville (Akron, Ohio) was unencumbered by these disadvantages, use of the Ziegler method led him to describe, in an unbiased manner. And finally, Ziegler had not been very impressed with his own polybutadiene product as compared to polyethylene, because, from his own experience in the 1930s he was familiar with the effectiveness of butadiene polymerization, for example, with metal sodium [146] or lithium alkyls, and now viewed as his priority the significant results obtained with the easy polymerization of ethylene. It is interesting to note that Horne of course, conducted further tests, and that his following experiments failed, that is, no polymerization took place. To speculate what would have happened if some of the first experiments had failed, hardly seems appropriate but to make use of good fortune is perfectly legitimate¹⁴⁾.

In the United States at the beginning of World War II, the “Big Four” rubber producers – B.F. Goodrich Co., Firestone, Goodyear and US Rubber – competed for a share of the market. Research and development were subsidized by the US government. Despite the information exchange between the US companies, there was, however, little hope that a method would be found for the economical synthesis of natural rubber.

The information channel which B.F. Goodrich and Gulf Oil Co. had opened up through Ruhrchemie in Germany in the early 1950s, led to Ziegler’s new catalysts.

13) At the time of experiments in January 1954 the effect of the cited mol ratio was not known. Then an excess of organo aluminum compounds was used in order to remove possible impurities. At the time of the experiments carried out by Horne, Ziegler and Martin had informed Goodrich of the selective effects of various mol ratios of aluminum compounds to titanium compounds [147].

14) As was later found, mixtures of AlEt_3 and TiJ_4 do not polymerize isoprene, AlEt_3 and TiF_4 do not polymerize butadiene, LiAlH_4 and TiJ_4 mixtures produce 75–95 % trans-1,4-polybutadiene and also trans-1,4-polyisoprene and TiCl_4 used instead of TiJ_4 leads to over 80 % 1,2-polybutadiene [148].

The cis-1,4-polyisoprenes which Horne had synthesized with the use of Ziegler catalysts were celebrated in the United States as a major scientific breakthrough. An announcement in the newspapers came on December 3, 1954, 1 day after the priority date of the basic patent [149]. The owner of the patent was Goodrich Gulf Chemicals Inc. of Pittsburgh, Pennsylvania. The press release did not mention either the inventor, Horne, or Karl Ziegler, but Horne received the famous “One Dollar” in consideration for having made the invention, a customary practice in the United States for recognizing an inventor¹⁵). As a memento, the dollar bill was adorned with the signatures of members of the board.

Together with Goodrich, the other major rubber manufacturers were all committed to one another through a pool agreement which called for a continuous exchange of information between the parties. It is not certain whether a reciprocal, royalty-free license was also intended for all participants in the pool. In any event, the pool partners urged Goodrich to honor its commitment towards them. Goodrich initially resolved the situation by indicating that its agreement with Ziegler prohibited any dissemination of technical information. Goodrich stuck to this arrangement beyond the Fall of 1955, so that any scientific conferences were limited to presentations by Firestone chemists, who, with much applause from the audience, revealed their alkali metal catalysts and their properties with respect to the polymerization of dienes, and Goodyear who disclosed test results obtained with the use of then known Ziegler catalysts, well aware of Goodrich’s priority rights in that field. After years of court litigation, Goodrich acquiesced to the demand to offer sub-licenses to the other pool partners¹⁶).

In actual fact, prior to the Horne experiments Firestone had successfully synthesized high-cis-content polydiene using a different method, but had not consistently pursued the results, since the company apparently had a greater interest in co-polymers (butadiene/styrene).

In any event, Dr. F. Foster, one of Dr. F. Stavelly’s associates, inspired by Ziegler’s early work with metal sodium, had attempted to apply lithium to butadiene. The result, polymers with approximately 35% cis content, was initially not encouraging, but became intriguing when lithium was combined with isoprene. There were no patent applications or publications directed to any optimizations thus far achieved. It was not until August 1955 that Firestone went public with “cis-1,4-polyisoprene”. By that time, the cis-1,4-content had been increased to 94%, not sufficient however, when compared to the 98% present in natural rubber. Improved results were obtained with the use of lithium alkyls in the form of solutions.

Goodyear tried on one hand, to improve their expertise through a mutual cooperation agreement with Firestone, and, on the other hand, in 1960 entered into a license

15) In addition to the “one dollar” an inventor at Esso received a ballpoint pen inscribed with “Esso Inventor”.

16) In 1960 a settlement between the US Government and Goodrich-Gulf finally ensured that the conditions of Goodrich’s license would have to satisfy other interested parties adequately.

agreement with Ziegler [150] covering the selective production of dimers¹⁷⁾ from propylene [151], with the aim of achieving independence from the oil companies in connection with its diene supply. From the total of 860,000 tons of dimer propylene produced during the remaining effective period of the relevant patent, between 1962 and 1971, the Max-Planck-Institute for Coal Research, through Studiengesellschaft Kohle mbH, received approximately 1.9 million dollars in royalties. It was in this way that Ziegler profited from the development of the polyisoprene market.

About 4 months after Goodrich's first priority application in the United States [149], two further patent applications [152], [153] based on accumulated test results were filed. The expanded coverage incorporated two findings: when the aluminum/titanium ratio was reduced to 0.7–0.33 Al : 1 Ti, the structure of the polyisoprene changed to a practically exclusive trans-1,4-configuration. The same was true for butadiene. Even though the claim of the first patent application [149] covered only polyisoprene, for the first time the specification included two examples, according to which, in the presence of a ratio of 1 Al : 1 Ti and/or 1.5 Al : 1 Ti catalyst, butadiene was converted into a mixture of cis- and trans-1,4-polybutadiene. The second patent right additionally included the use of further heavy metal compounds, such as vanadium, zirconium, chromium, tungsten and iron in the form of their chlorides or acetyl acetonates, together with compounds of the formula R_2AlCl instead of R_3Al . Up to that time there were no recipes for the production of pure cis-1,4-polybutadiene, but these were being sought by a number of teams worldwide through intensive research efforts.

The gap was subsequently closed in two ways. First, Horne and Carlson discovered that the aluminum trialkyl/titanium tetrachloride catalyst (2.5 : 1), when heated to 50–100 °C prior to its introduction into the reaction, increased the rate of cis-configuration in the polymer [154], a physically modified process variation. A more elegant solution could be arrived at by using a soluble cobalt catalyst consisting of anhydrous cobalt chloride and either an organoaluminum compound [155] alone (for example, *i*-Bu₂AlCl) or a mixture of alkylaluminum dichloride and dialkylaluminum chloride [156]. More than 95 % of the polymer consisted of 1,4-cis units. The fact that cobalt would prove to be the heavy metal of choice could not have been foreseen.

However the impact of this discovery was not fully realized until D.R. Smith and R.P. Zelinski, members of the research team at Bartlesville, Oklahoma and employees of Phillips Petroleum Company, were named as inventors of a patent application dated October 17, 1955 [157] entitled "Rubbery Polymer of 1,3-Butadiene having a high Cis 1,4-Addition". The scientists had found that by contacting butadiene with a catalyst formed from titanium tetraiodine in place of titanium tetrachloride, together with aluminum triisobutyl (aluminum trialkyls), a practically gel-free polybutadiene¹⁸⁾ product could be obtained. The priority situation was resolved only gradually and was actually not fully appreciated until the late 1960s.

17) The dimer of propene, 2-methyl-pentene-1, was isomerized according to the so called "Scientific-Design" process in Beaumont and thereafter the product, methyl-pentene-2 pyrolysed into isoprene and methane.

18) The addition of iodine or iodine-containing compounds suppresses the formation of gel during the polymerization process and the soluble polymer contains 90% cis units [158].

The results achieved by Messrs. Smith and Zelinski did not of course, constitute an isolated discovery. Rather, everything pointed to the fact that the research conducted at Phillips Petroleum had been extended from the polymerization of ethylene and propylene across the board to the polymerization of dienes (see Chapter 1, Section 1.1.2).

Thus, where two different but equally reliable methods for the production of polybutadiene containing a high percentage of *cis* configuration were available, the party which had obtained a product patent on this hitherto unknown, high-percentage *cis* 1,4-polybutadiene should be able to claim ultimate success. In a long drawn out interference procedure, followed by an infringement action between Phillips and Goodrich, it was ultimately determined that Phillips Petroleum was the first to make, and was entitled to claim a product having a greater than 85% *cis* 1,4 content, irrespective of the method of its production.

It is said that the judge was persuaded after watching a demonstration whereby a rubber ball made of such synthesized caoutchouc exhibited enhanced elasticity properties as compared to polybutadiene with a lower *cis* 1,4-content. The ball simply bounced higher when both balls were dropped from the same height.

Natta and his co-workers also joined in the race for “polydiene” priority. Their contribution to diene polymerization was based on their finding that propylene, in the presence of solid catalysts, preferably composed of titanium trichloride and organoaluminum compounds, could be converted to polymers with a higher crystalline content. On March 12, 1955, Montecatini filed a patent application in Italy [159], describing and claiming the production of polymers from conjugated diolefins to 1,4-polymers, incorporating the experience of using solid catalysts composed of titanium trichloride or vanadium trichloride with aluminum triethyl or zinc diethyl. However, the polymers predominantly exhibited the *trans* 1,4-configuration.

At practically the same time, patent protection was being sought for the mixed polymerization¹⁹⁾ of α - and diolefins using the same catalysts [160]. Polymerization of conjugated dienes leading to the formation of predominantly 1,2-polydienes is the subject matter of yet another application filed by Montecatini, claiming an Italian priority date of July 15, 1955 [161]. It was soluble heavy metal compounds, particularly titanium alkoxides, in combination with organoaluminum compounds which exhibited such selective polymerization activity.

It was not until 1 year later, in mid-1956, that Montecatini sought patent protection for the production of butadiene polymers with predominantly *cis* 1,4-configuration, synthesized with a catalyst mixture composed of titanium tetrachloride and organoaluminum compounds using a mol ratio of Al:Ti which had now been preadjusted to 1–2.5:1, as had been discovered previously by the research team at Goodrich Gulf. This, however, requires subsequent fractionation of the polymerization product [162] with acetone, methylethylketone and ether. The ether extract contains (infrared absorption 13.6 μ) a product of which up to 85% was in the *cis* 1,4-configuration.

19) The mixture of ethylene and conjugated diolefins (vinylcyclohexene) was tested in a copolymerization experiment by Martin in early

1954 without filing for patent protection (see Chapter 1, Section 1.2.3).

Information regarding further developments at Goodrich Gulf was very sparse, so that Ziegler did not follow up on the matter until the occasion arose during personal visits between the parties in 1958. Mr. Crockett of Goodrich Gulf closed the information gap with a letter to Ziegler [163] in June of 1958. Shortly thereafter, the two parties started to explore the substance and finalization of a contractual arrangement between them regarding the exploitation of patent rights, and on August 6, 1958, an agreement [164] was entered into involving the polymerization of diolefins with the use of Ziegler catalysts to form rubbery diolefin polymers, as claimed by Goodrich Gulf patent rights. The amendments of 1960 upgraded the agreement in two ways, first, by broadening the definition of the Ziegler catalysts to include compounds of Group VIII metals, particularly cobalt, as catalyst components, and second, by expanding the polymerization of isoprene²⁰⁾ to the polymerization of dienes in general. Ziegler subsequently received an exclusive right to grant licenses under Goodrich Gulf's German patents. While the exclusive right itself was royalty-free, Ziegler, in return, granted Goodrich Gulf a non-exclusive license under his US and Canadian patents for processes falling within the remit of the re-negotiated contract, and furthermore allowed Goodrich Gulf the exclusive right to grant non-exclusive licenses under both parties' US and Canadian patents. Ziegler initially received as royalty, a contractual guarantee of 0.35% of the net sales price derived from both Goodrich Gulf's own production as well as the production of Goodrich Gulf sub-licensees. Export was royalty-free, except for Germany, Italy and Great Britain. During negotiation of the license terms, particularly pertaining to the revised 1960 agreement, Ziegler, while vacationing in Sils Maria, wrote to his patent attorney von Kreisler [165] stating that the proposed fixed values of 0.35% and/or 0.45% were "visually unattractive", and that he would prefer to negotiate a distribution key between Goodrich and himself regarding their joint income. It was agreed [166] that with regard to its own production, Goodrich would pay Ziegler 0.35% of the profits generated from sales of polyisoprene and 0.5% from sales of polybutadiene, and that Ziegler's share of the proceeds from any sub-licenses would be 35% in cases where Goodrich had not provided any expertise and/or 28% where they had provided any technical assistance.

The granting of a license, through Ziegler, in Germany required that the licensee make a down-payment of at least US \$ 100,000, while further down-payments were expected for any technical facilities which went on stream, the amount depending on the capacity of the facility.

The contractual safeguarding of his rights vis-à-vis Goodrich in 1958 also prompted Ziegler to attempt to resolve the situation with Montecatini through negotiation of an agreement, with the aim of regulating the use of Ziegler catalysts when combined with discoveries made by the Natta School and/or Montecatini in the field of diene polymerization. The third Pool Agreement [167] was signed in mid-1958 by Montecatini and Ziegler as the contract parties. Parallel to the first two agreements

20) At this time Goodrich Gulf had safely secured the priority for polyisoprene before all other competitors. For polydienes in general the sit-

uation was uncertain, particularly for cis-1,4-polybutadiene.

pertaining to the polymerization and co-polymerization of α -olefins, the new contract provided guidelines for granting licenses in the field of diolefin polymerization – particularly the polymerization of isopropene and/or butadiene – whereby the rights of ownership and control were retained by Ziegler in Germany and Montecatini in Italy, while Montecatini reserved exclusively for itself all rights of exploitation, also in Ziegler's name, in the remainder of the licensed territory. The ratio of costs-to-earnings shared by the parties was 80% Montecatini and 20% Ziegler. Montecatini's tendencies and intentions to establish itself as a premier licensor, also on behalf of Ziegler, became increasingly evident.

It was interesting to note in hindsight, that Ziegler, whose rights in the diene polymerization field were actually limited to patents covering only his own catalysts, but not covering the use of these catalysts for the production of polydienes, nevertheless sought to generate his own income from the developments which were unfolding at both Goodrich Gulf and Montecatini. In this he was considerably more successful in his dealings with Goodrich Gulf than with Montecatini, and also – as history revealed – the proceeds that were actually realized from worldwide production had been made possible only through the Goodrich Gulf connection. Even though the Natta School demonstrated in brilliant publications, that they had scientifically worked out the structural characteristics of the new polydienes, their sponsor, the Montecatini Società Generale, did not grant a single license in this field. A summary of the prevailing priorities is given below.

Catalysts consisting of either titanium halides or cobalt compounds in combination with organoaluminum compounds are Ziegler catalysts and are claimed in Ziegler patents. Priority rights for the specific use of these catalysts for the polymerization of, particularly, diene hydrocarbons, isopropene and butadiene are owned by Goodrich Gulf (cobalt catalysts) and/or Phillips Petroleum (titanium iodide catalysts), insofar as the selective polymerization to cis-1,4 polymers was concerned. Montecatini was left empty-handed. Product protection for polybutadiene with a high cis 1,4 content was won by Phillips after a long drawn out court battle against Goodrich²¹⁾.

The results are also interesting from the point of view of patent rights. At the time, German patent law did not provide for straightforward product protection as practiced in the United States. In the US, Goodrich Gulf recognized that the Ziegler catalysts, in their capacity as “new substances”, were highly likely to enjoy product protection *per se*, that is, independent of the method of their production, and that it would be desirable to gain access to them by way of a license. In Europe, Ziegler lacked process claims for the conversion of diene hydrocarbons and, since there was no product protection, he would have come away empty-handed, had he not traded the sale of the catalyst product protection in the US for protection of the Goodrich Gulf process in Germany.

21) (a) See Section 1.5.1, laudation to G. Natta on receipt of the Nobel Prize. (b) See Sec-

tion 5.1.1, (Chapter 5, reference [26]), Ziegler's fruitless attack on Phillips Petroleum.

Still the Ziegler/Goodrich Gulf patent rights package was not complete since – as mentioned earlier – the conflict of interest between Phillips Petroleum and Goodrich Gulf regarding the product protection of cis-1,4-polybutadiene had yet to be resolved. Phillips Petroleum settled the patent infringement action it had brought against Goodrich Gulf when, in 1968, Goodrich Gulf agreed to pay a sum of \$ 1.4 million dollars [168]. Yet Goodrich Gulf was in a favorable position to seek such a solution, since the terms of the Goodrich Gulf/Ziegler agreement called for 50% of such payments to Phillips to be creditable against future royalties due to Ziegler, as Ziegler had to concede that Phillips' product patent dominated any cis-1,4-polybutadiene production ("dominating patent" clause).

Had there been a timely exchange of information between the parties, a trilateral arrangement between Ziegler, Goodrich Gulf and Phillips Petroleum might have provided a more satisfying solution, limited, as it were, to the history of polybutadiene and polyisoprene as discussed in this book. Moreover, a cooperative effort between Ziegler and Phillips promoting the overall development of the Ziegler catalysts and their use in the production of polyolefins would have provided an ideal combination for optimum market development. At that point in time, the self-possessed nature of a man like Karl Ziegler would have been incompatible with the business concepts of a Midwest US oil conglomerate, effectively preventing any joint market policies, if such an idea had ever been discussed between the parties. Thus, the controversy with Phillips Petroleum was predestined.

Phillips owned the patent rights and knowhow necessary for the commercial production of linear, high-crystalline polyethylene, which very soon became available on the market under the trade name "Marlex". While the chromium oxide catalysts used for this production were not covered by any Ziegler patents, the process in terms of parameters and product obtained was at least equivalent. Moreover, as previously indicated²²⁾, Phillips had obtained product protection for crystalline polypropylene as a new material, establishing itself in a brilliantly competitive position vis-à-vis Ziegler.

Later on, it became apparent that although, on the one hand, this situation led to a settlement between Goodrich Gulf and Phillips Petroleum, on the other hand, Ziegler attempted, both alone and together with Goodrich Gulf, to challenge Phillips Petroleum's patent rights in the field of polybutadiene in court, failing to succeed in the United States, but prevailing in Europe.

The first fruitful outgrowth from the contractual arrangements with Goodrich Gulf was a license granted by Ziegler to Chemical Werke Hüls in early 1960. This was the only Ziegler license granted in Germany in the polydiene field. Between 1964 and 1977, roughly 280,000 tons of polydiene were produced at Hüls. Studiengesellschaft Kohle mbH made approximately 6.4 million Deutsch Marks in royalties from this agreement based on a royalty rate of 2.25–1.125% of the (net) sales price.

22) See Chapter 1, Section 1.1.2, Judge Wright explicitly pointed out, that Phillips Petroleum did not mention thermoplastic polypropylene. The product claim was not limited to waxes

however. Both waxes and thermo plastic polypropylene had a substantially crystalline content.

At this point, Dutch-based Shell Internationale Research Maatschappij N.V., another influential owner of patent applications relating to the use of cobalt catalysts for the polymerization of butadiene containing a high percentage of the *cis*-1,4-configuration, entered the arena. Shell was afraid it lacked competitive priority rights and sought a contractual arrangement initially with Goodrich [169], which was finalized in early 1961. As part of this agreement, Shell received a license for France with limited-term exclusivity regarding Goodrich's patent rights for so-called "cobalt"-1,4-butadiene rubber as well as "non-cobalt"-1,4-butadiene rubber. If, within 4 years, Shell went on stream with a plant having an annual capacity of 15,000 tons, it would be entitled, under the agreement, to an extension of its exclusive rights. The differences as compared to equivalent Goodrich patent rights were relatively minor. In the claimed polymerization processes, the recipe included chlorine-rich alkyl aluminum compounds or additional small amounts of a third component. Shell's earliest documented priority date was November 6, 1957, i.e. 6 months after Goodrich's earliest date. All further applications were filed in 1958 and 1959 [170]. Through his agreement with Goodrich, Ziegler gained access to Shell's patent rights in Germany.

In 1961–62, prompted by Goodrich, an attempt was made to integrate the interests of all parties concerned – with the exception of Phillips Petroleum, i.e. Goodrich, Shell, Montecatini and Ziegler – into a single contractual arrangement, an undertaking which, with hindsight, proved to be not only very complicated but also nearly impossible to implement in practice. Without having knowledge of the ultimate priorities, that is faced with an ambiguous situation in that the patent priority rights of the participants had yet to be finally determined, the parties met at the historic Bürgenstock Hotel above Lucern to discuss the distribution of the anticipated future shared income. The impression gained from reading the Minutes of the Meeting is that the negotiations were conducted almost in the manner of a poker game. In October 1962, a worldwide agreement was signed [171], which not only called for the parties granting one another reciprocal licenses under their patent rights, but also spelled out the distribution arrangements for royalty income paid by third parties from different groups of countries. From a total royalties of 3% of the (net) sales price of *cis*-butadiene rubber, Ziegler received 10%, Shell between 10 and 20%, Montecatini between 10 and 50%, and Goodrich the remaining share in each case. The negotiators for both Montecatini and Shell are to be complimented for each having achieved this bargaining result based on a relatively modest patent position.

According to Montecatini's report to Ziegler [172], no income had been posted under the four-party agreement on "High *Cis*-Polybutadiene Rubber" until mid-1969, i.e. there had been zero production in this context.

The realization that a large volume of the commercially employed catalysts for the polymerization of dienes had been Ziegler catalysts, did not gain public recognition until 1967 and 1971 when Karl Ziegler was awarded the "International Synthetic Rubber Medal of "Rubber and Plastic Age" and the "Carl-Dietrich-Harris-Plaque of the Deutsche Kautschukgesellschaft".

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3

Patent Rights Worldwide, Patent Applications: Prosecution, Oppositions, Priority Rights

The scientific sensation caused by the mode of action of the new catalysts developed by Ziegler and his co-workers was overwhelming and broadly acknowledged. For the catalysts to be considered for industrial utilization, issued patents were an “absolute must”. No one would have signed any option or license agreements, had the licensee’s royalty obligation not been offset by Ziegler’s undertaking to guarantee the availability of issued patents. The advance royalty payments mentioned earlier were “roulette stakes” ventured by the interested parties, even though most of the parties involved were very careful to scrutinize any known patent literature before making a payment. There was no available data regarding the quality of any commercial process, except for the spectacular impression created by a polymerization experiment carried out in a set of glass beakers on the laboratory table.

In late 1953 and during the first half of 1954, a frantic search had begun – including, of course, at the Max-Planck-Institute for Coal Research – for possible references to the use of Ziegler-catalysts, and even the catalysts themselves by combing the available literature for earlier indications, or possibly even overlapping findings. Patent applications filed too late are, after all, only of minor value.

While speculating on the mode of action of the new catalysts, H. Breil’s diploma thesis [1] mentioned low-valent metal compounds of the so-called transition metals, whose valencies, according to Breil, are partially saturated by organic radicals, thus containing genuine metal – carbon bonds. Compounds of tetravalent titanium – not low-valent titanium compounds – containing a titanium – carbon bond, had been described by D.F. Hermann and W.K. Nelson [2] in connection with the reaction of phenylmagnesiumbromide with titanium tetraesters, although their effect on olefins had not been examined¹⁾. Complex compounds of aromatic substances and chromium were first mentioned by F. Hein [3].

H. Breil then wrote a dissertation [4] which provided a detailed discussion of the “pertinent literature on ethylene polymerization.” Breil initially cited BASF German Patent 874 215 [5] and concluded that, in accordance with the process described by the inventor, Max Fischer, in addition to large quantities of heavy oils, “a light, al-

1) It was speculated that unstable low valence phenyl titanium compounds were formed; their instability was confirmed by the

polymerization of styrene which was facilitated by the phenyl radicals which had dissociated from the titanium compounds.

most white powder which obviously was a polyethylene of the type of my own polyethylene products” was also formed. H. Breil’s explanation followed²⁾. It should be reiterated here that, up to that time, nobody had repeated any experiments, nor had anybody specifically re-worked the only Example disclosed in the BASF patent.

Repetition of the Example as described by Max Fischer failed to yield any solid product.³⁾ Highly polymeric, solid products formed at an increasing rate only when more aluminum powder was added. These polymers had, however, a highly branched structure and, by comparison, exhibited completely different melting behavior [6]⁴⁾.

In discussions of the established literature considerably more space was devoted to older patents owned by Du Pont [7, 8]. The first one of these described the effect of typical hydrogenation catalysts, such as nickel in particular, in combination with alkali metal alkyls, on ethylene, while the second patent centered on liquid ethylene being subjected to the action of heavy metals or metal salts combined with peroxides. The term “non-ionized salts” as used by Ziegler and his co-workers was in sharp contrast to the subject matter of the Du Pont patents, wherein ionized solvents, such as water and alcohol, were selected⁵⁾. The Du Pont scientists had after all, come close to the new Ziegler catalysts, even though they had made the approach using a completely different aspect of polymerization technology.

They had concentrated on the polymerization of ethylene using radicals, whereby the patentable characteristic was to be the use of liquid ethylene, in other words, the process was an improved, so-called radical, polymerization method. The aim was obviously to avoid the extremely high pressure inherent in the state of the art, technically significant ICI process for the production of “high-pressure” polyethylene. Utilization of aluminum alkyls, as employed by Ziegler and his co-workers as the

2) Quoting from H. Breil once more (see Chapter 1, p. 19): “It seems obvious to me that in experiments of this type, a catalyst of the type of my catalysts would be formed. The inventor of the patent failed, however, to recognize the essential nature of his process. The polymerization catalyst is formed either by Al, AlCl₃ and ethylene to primarily form organoaluminum compounds (see C. Hall, A.W. Nash [9, 10], *J. Inst. Petrol, Technol.* 23, 679 [1937] and 24, 471 [1938]), which react with titanium tetrachloride, or the metallic aluminum directly reduces the titanium tetrachloride.”

3) At the same time a research team at Du Pont also failed to produce solid polymers. This result was not published at the time (see Chapter 1, p. 7).

4) In unpublished experiments by P. Borner and H. Martin, aluminum powder from various

sources and ethylene of varying purity were used in an experimental program in 1957. No solid polymerization products could be isolated when the Example in the BASF patent was repeated. If the amount of aluminum was doubled 20% of the reaction product formed contained a mixture of wax and low melting polyethylenes having a relative low molecular weight while the greater percentage of the product was composed of oils. If the amount of aluminum was tripled a large portion of the product finally consisted of solid polymers having melting points around 100 °C.

5) Except in experiment 23, where a transition metal and not a metal compound was used: lithium butyl as the metal alkyl, but in this case nickel metal on diatomite earth was used. Alcohol and water destroy Ziegler catalysts.

preferred co-catalysts, had not been described by Du Pont. It would therefore be no more than pure speculation to suggest that the Du Pont patent was referring to a combination of aluminum alkyl compounds and transition metal compounds. In fact, this combination had not even been considered by the Du Pont inventors⁶⁾. Breil concluded that the publications were hardly germane to the catalysts which he had described. In addition to the older scientific articles, it became apparent that there was other material that dealt with the identical subject matter. Other early publications were uncovered during the course of various subsequent patent prosecution procedures.

3.1

The Package of the First Six German Patent Applications

The German Patent Office had been slow to take any action after Ziegler had filed his first patent application (Chapter 1, reference [24]), and it was a full year before they issued an Office Action. In this Action, the Examiner maintained that the above-cited British Du Pont patent [8] disclosed catalysts composed of metal alkyls in combination with multivalent metals which, according to some Examples, were used in the form of their salts. The Examiner further combined this element of a previous Du Pont patent with an older Ziegler patent [11], which claimed the polymerization of ethylene with aluminum alkyls alone. He argued that a combination of the two cited references would render the new polyethylene synthesis obvious, and the latter would therefore, no longer be patentable – thus raising serious argument against Ziegler.

An interview with the Examiner was therefore requested and during the course of this interview several points were clarified. First, the earlier Ziegler patent had not disclosed the use of transition metal compounds and had described the resulting products as liquid or, at best, waxy polymerization products of ethylene, and second, the Du Pont patent failed to mention any aluminum alkyls and although there was very limited mention of transition metals those that were specified belonged to Groups VIII and IB of the Periodic Table. In addition, where transition metals identical to those specified by Ziegler were used, either peroxides or methanol were also present in the reaction mixtures thus creating conditions under which the new catalysts would not be able to exist.

The Examiner was persuaded that the new development could be clearly differentiated from the state of the art and was, therefore, patentable. Publication of the patent application, as required by the rules, took place in late 1956. Two months later, BASF filed an opposition to prevent patent issuance. In the Argument section, the author of the Opposition combined the previously mentioned earlier Ziegler

6) In any case, the polymerization described by Du Pont was limited to ethylene and the polymerization of propylene to form solid

high molecular thermoplastic products is not possible using this method.

patent [11] – high molecular weight polyethylene produced with aluminum alkyls alone – with the subject matter of the BASF/Max Fischer patent [5], which allegedly disclosed the polymerization of ethylene to form, at least partially, high molecular weight polyethylene. The fact that the latter did not mention the use of aluminum alkyls required an explanation. Breil had, unwittingly of course, provided the basis for the opponents' argument by way of his dissertation which had meanwhile been published, in which he referred to the publications of Hall and Nash.⁷⁾ Page 687 of the cited Hall and Nash publication [8] does, in fact, contain a sentence to the effect that, under the conditions described there, aluminum trichloride, aluminum and ethylene – the components which Max Fischer had added to the titanium tetrachloride – might also possibly produce traces of aluminum triethyl:

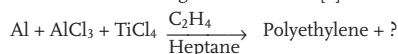
“The original crude fraction is a mixture of aluminum ethyl dichloride and aluminum diethyl chloride, with a possible trace of aluminum triethyl.”

The authors did not offer any experimental proof of the presence of aluminum triethyl, nor would it have been credible, since any aluminum triethyl, no matter how small the amount, would have reacted with excess aluminum trichloride to form ethylaluminum dichloride (Chapter 1, reference [138]).

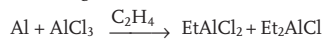
Farbenfabriken Bayer joined the opposition proceedings relying on the same arguments. There was no chemically sound, factual support for BASF and Bayer's motivation in filing the oppositions. The parameters in terms of proportions of the reaction ingredients employed in the production of the Ziegler catalysts, on the one hand, and those used for the titanium tetrachloride/aluminum powder/aluminum trichloride catalyst described by Max Fischer as well as the synthesis of organoaluminum compounds according to Hall and Nash, on the other hand, were so completely different that they defied combination. The authors of the oppositions were well aware of this. Nevertheless, the implication of Breil's conclusions in terms of patent protection was all too obvious⁸⁾.

7) See footnote 2.

8) To understand the chemical facts, once more: if under the conditions of the ethylene polymerization according to Max Fischer [5]



including the results of Hall and Nash



first ethyl aluminumsesquichloride was formed, which together with titanium tetrachloride, formed a Ziegler catalyst which would be responsible for the production of solid, high molecular polyethylene, the catalyst now described as a Ziegler catalyst would not be new and therefore not patentable, in the view of the opposing parties. Experimental proof thereof was not delivered by BASF or Bayer. For the opposing parties such proof was not necessary, as it was considered that H. Breil had already provided the proof. By comparing the different conditions in detail the compelling evidence leads to the conclu-

sion that the different amounts of aluminum powder and aluminum chloride used by Max Fischer, and then by Hall and Nash were of paramount importance. In Fischer's experiment only a very small amount of an aluminum compound would have been formed giving a chlorine/aluminum ratio ≥ 2 while with a relatively large excess of aluminum chloride would also be present. Fischer used one-tenth of the aluminum powder used by Hall and Nash and described this addition as favorable. The aluminum was chosen out of a series of hydrogen chloride-binding metals. Iron or zinc should have been equivalent to aluminum powder. Actually, not a trace of an organo aluminum compound could be isolated from the mixture according to Max Fischer and iron and zinc could not be used for this purpose at all.

At this stage it should be pointed out that the so-called “Max Fischer catalyst” cannot polymerize propylene into solid polymers.

The practice of opposing the issuance of a patent, even in the absence of conclusive, chemically sound arguments, was and is today widespread and suggests itself to anyone attempting to buy entrance into a license agreement cheaply by causing a delay in patent issuance.

Ziegler retaliated. He filed a complaint to have the Max Fischer patent declared invalid, which was partially successful⁹⁾. The purpose of the invalidity action was accomplished, as will be described below in further detail.

In retrospect, it was easy to understand Max Fischer's and also BASF's frustration at having come so close, during the final war years, to making the discovery which Ziegler was to achieve 10 years later. Presumably, Max Fischer had been unaware of work being done with metal alkyls and, working from a completely different direction, had considered the polyethylene he had obtained as an undesirable "by-product". The Hall and Nash publications were unknown to him. The object of the Max Fischer experiments was to improve the yield and quality of the polymerization of ethylene to form lubricants, whereas Ziegler and his co-workers were looking to examine the interaction of ethylene and higher olefins with metal alkyls. Subsequent experiments regarding Fischer/Hall and Nash, which appeared to be urgently needed at that time, demonstrated that there was no connection between the subject matter of the Fischer patent and the Hall and Nash publications. With hindsight the combination suggested by BASF completely lacked experimental support.

In early 1958, the German Patent Office dismissed the oppositions and granted the patent [12]¹⁰⁾. The purpose of blocking issuance of the patent prompted BASF to appeal the Patent Office's decision, without, however, presenting new arguments. The Patent Office did not prosecute the appeal, because in mid-1958, the parties reconciled their differences contractually [13], in that Ziegler recognized the validity of the limited Max Fischer patent and BASF withdrew its oppositions to Ziegler's patent applications. On the same day, the parties entered into a license agreement [14] regarding the production of polyolefins and co-polymers. BASF made no down-payment in the form of an advance license fee, but otherwise paid the usual sliding scale of royalties based on the net sales price, in other words, it did not receive any more favorable terms regarding the sale of the licensed products.

In retrospect, Ziegler had, at that time, vastly overestimated the significance of the subject matter of the Max Fischer patent and its effect as a prior art publication, not least because of a lack of experience in the patent field and a presumption on his part that the subject matter was close to the discoveries made at Mülheim. In a large number of patent prosecution procedures and subsequent infringement actions,

9) The action lasted from 1958 to 1959. Thereafter the claims were limited to the compulsory presence of aluminum powder, but not totally rejected because the Court was not in a position to prove that the single example of the original patent – as claimed by Ziegler – did not result in solid polymers. Arbitrary experiments were not part of a Court order.

10) The Patent Office: there was no proof of the formation of aluminumtrialkyls according to

Hall and Nash; Fischer used a solvent, Hall and Nash did not. The ratio of aluminum to aluminumchloride was 1 : 10 in Fischer's experiment (compared to Hall and Nash). Fischer describes zinc and iron as alternatives to aluminum powder, neither metal was demonstrated to work in the reaction described, according to Hall and Nash. Finally, the polymer products substantially differ in the degree of branching in the polymer chain.

the BASF patent constituted part of the argument presented by Ziegler's opponents, but none of the courts of law had attributed as much significance to the subject matter of the Fischer patent as Ziegler himself had done. In this context, a discussion of the important deposition testimony will follow at a later point.

Subsequently filed patent applications, initially treated as so-called additional applications, were directed to further discoveries made during the developmental stage of the invention.

These discoveries included, for example, use of the catalysts at room temperature and normal ethylene pressure [15]; broadening the range of catalyst components to include uranium compounds [16] and/or aluminum compounds reduced to containing only one or two alkyl radicals, such as, diethylaluminum chloride or diethylaluminum alkoxyl [17]; and finally the utilization of organometal compounds of magnesium and zinc [18]. The delayed issuance of patents maturing from the additional applications (1960) was, of course, due to the oppositions which BASF had filed against the first Ziegler application. It was 7 years after Ziegler's first five German patent applications that the patents were actually issued. Ziegler felt immense satisfaction at the result he had achieved in Germany. But he suspected that this development was merely the start of an obstacle race.

Chronologically, the last patent application in the series was filed on August 3, 1954 [19], and it was this application that expanded the range of olefins that could be polymerized from ethylene to higher olefins to include for example, propylene, butene, etc. Patent issuance was difficult, since, along with the contractual arrangement with Montecatini set forth in the aforementioned Pool Agreements, patent litigation involving Natta's Italian priority application and its corresponding foreign applications was a foregone conclusion. In order to avoid conflicts, the Montecatini Pool Agreement called for any applications in foreign countries, except Germany and the United States, corresponding to the initial Italian applications directed to the same subject matter as Ziegler's August 3, 1954 application, to be filed jointly with Ziegler. The aim was to obtain strong basic patents.

In Germany, the applications claiming the Italian priority dates of June 8 and July 27, 1954 [20] were filed in July of 1955 by Montecatini and Ziegler as contractual joint owners. The applications named G. Natta alone and/or G. Natta, P. Pino and G. Mazzanti as joint inventors, but failed to name Ziegler and his co-workers. Prosecution of the application before the German Patent Office was drawn out for 5 years, and afterwards the application was hampered by massive opposition proceedings extending through 1966 and, was finally effectively rejected by the German Patent Office [21]¹¹⁾.

11) During the opposition procedure the opposing parties (six companies: Staatsmijnen, Limburg, Netherlands; Rhone Poulenc, Paris, France; Hercules Powder, USA; Solvay & Cie, Brüssel, Belgium; Dynamit Nobel AG, Köln, DE; Eastman Kodak, USA) argued that not all of the compounds of group IV–VI metals together with alkyl aluminum compounds would polymerize ethylene as claimed. The Appeal Senat in the German Patent Office: specifically active compounds as the claimed oxy-halides and acetylacetonates of metals to which the claimed metal com-

pounds should be limited besides halides of these metals are not part of the description. Furthermore, the single halides could not be found in the original files (except for titanium tetrachloride). The language of the claim under consideration could not be found in the original file and the claim could not be accepted because of this deficit. With regard to heavy metal compounds, apart from titanium tetrachloride only chromium acetylacetonate was given as an example. Other metal compounds are missing.

Ziegler's priority application of August 3, 1954 was prosecuted in Germany simultaneously with the Natta cases and now assumed fundamental significance as the basis for any license agreements in the polypropylene field. The German licensees pushed for expedited patent issuance and the protection gained thereby for the production of polypropylene, polybutene, and the like. In view of the aforementioned Montecatini/Ziegler application, Ziegler's original priority application was initially "put on hold", that is, the German Patent Office prosecution was suspended. It was resumed in 1967, after the final rejection of the Montecatini/Ziegler case, and, in late 1967, the application was published as a so-called German Disclosure Application (*Auslegeschrift*) [22].

Disregarding the negative decision handed down in connection with the Montecatini/Ziegler application, it was Ziegler's intention to now insist on the broadest possible patent claim for his own application, which, predictably, provoked opposition from the chemical industry, particularly on the part of those companies which had not been granted licenses. Thirteen years after the application was first filed, in 1967, Glanzstoff AG of Wuppertal, and Avisun Corporation of Philadelphia, USA, attempted to further obstruct patent issuance through oppositions.

While Glanzstoff AG's immediate motivation was not clear, Avisun, a subsidiary of Standard Oil of Indiana, with headquarters in Chicago, was operating under an all too obvious agenda. The latter was exporting polyolefin products worldwide and was not willing to tolerate any obstacles curtailing the expansion of its export business. The object of the opposition was limitation of Ziegler's coverage to exclude the use of a catalyst composed of titanium trichloride, ethyl aluminum dichloride, and a third component¹²⁾, silicon tetraethoxyl, since allegedly, this mixture did not fall under Ziegler's claims, yet, according to Avisun's contention, its catalytic action produced the same yield and quality of the desired polyolefin products. The same argumentation was advanced by Standard Oil challenging the Montecatini/Ziegler polyolefin patent rights throughout Europe, a foretaste of the disputes to follow in the United States. It was at this point in time at the very latest, that it had become obvious that Standard Oil was producing polypropylene without a license. This will be discussed in a later chapter (see Chapter 5, p. 180).

Apart from the previously presented arguments, the opposing parties again alleged that a large number of the conceivable combinations falling within the broad range of catalyst mixtures claimed by Ziegler and co-workers simply did not work. Although this contention was not completely refuted, its crucial points were nonetheless, subsequently disproved experimentally. Over time however, the opposing parties' briefs emphasized ever more adamantly the charge that the original patent application disclosed only a small fraction of the numerous possible catalyst mix-

12) Experiments by H. Martin confirmed that silicon compounds and aluminum compounds could exchange ligands. Following Avisun's protocol the reactants were mixed at 60 °C before titanium tetrachloride was added. By chlorine/ethoxy exchange an aluminum compound $\text{ClAl}(\text{OEt})\text{Et}$ was formed which in

combination with titanium halide showed high catalytic activity. The three-component catalyst, claimed by Avisun was changed into a two-component catalyst before the polymerization of the olefin began, as claimed in the Ziegler patents. As expected Avisun did not comment on this result.

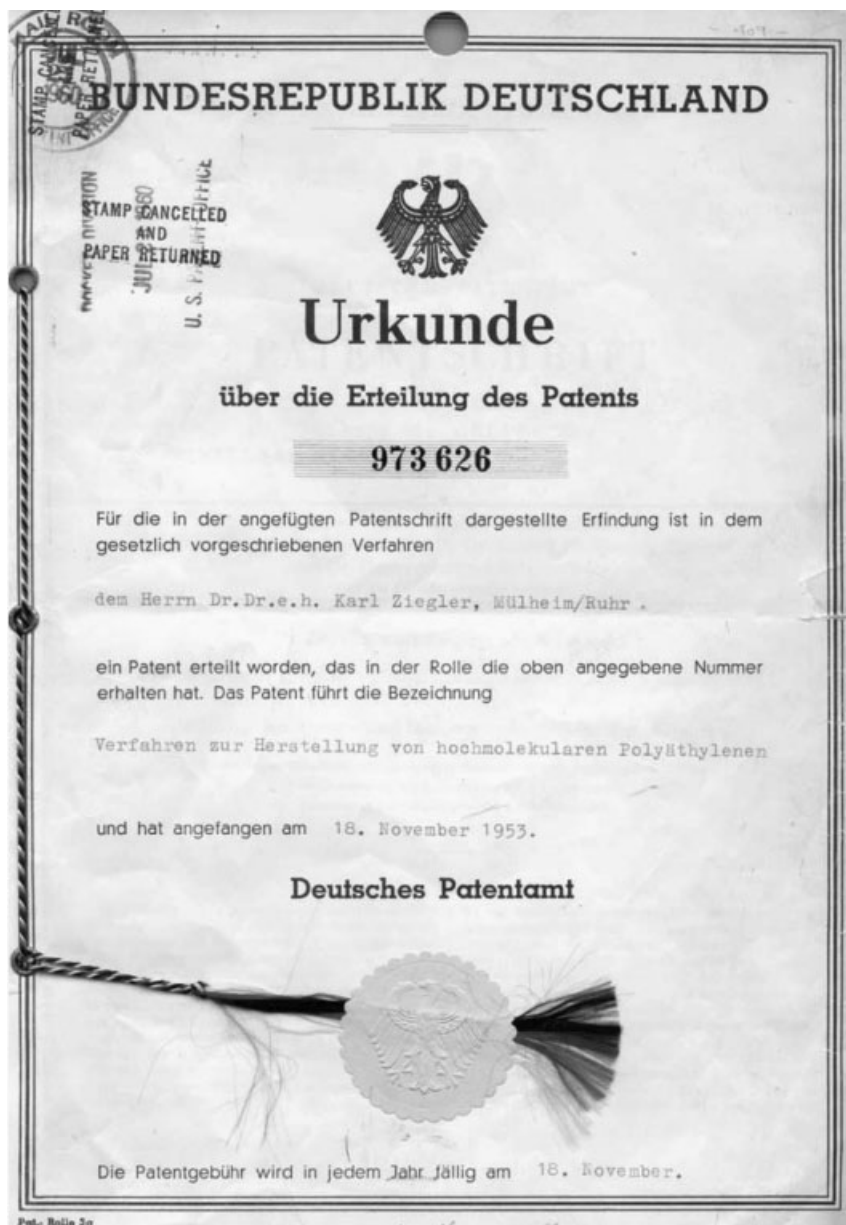


Fig. 3.1 German Patent No. 973 626: (A) cover page;

BUNDESREPUBLIK DEUTSCHLAND



AUSGEGEBEN AM
14. APRIL 1960

DEUTSCHES PATENTAMT

PATENTSCHRIFT

Nr. 973 626

KLASSE 39c GRUPPE 25 01

INTERNAT. KLASSE C 08 f ———

Z. 3799 IV b / 39 c

Dr. Dr. e. h. Karl Ziegler, Mülheim/Ruhr,
Dr. Heinz Breil, Oberhausen (Rhld.), Dr. Erhard Holzkamp, Düsseldorf,
und Dr. Heinz Martin, Mülheim/Ruhr
sind als Erfinder genannt worden

Dr. Dr. e. h. Karl Ziegler, Mülheim/Ruhr

Verfahren zur Herstellung von hochmolekularen Polyäthylenen

Patentiert im Gebiet der Bundesrepublik Deutschland vom 18. November 1953 an
Patentanmeldung bekanntgemacht am 4. Oktober 1956
Patenterteilung bekanntgemacht am 24. März 1960

In der deutschen Patentschrift 878 560 werden Versuche beschrieben, bei denen hochmolekulare Polyäthylene aus Äthylen und Aluminiumtrialkyl allein erhalten werden. Hierbei ist es in gewissen Grenzen möglich, den Polymerisationsgrad durch die Wahl des Mengenverhältnisses Aluminiumtrialkyl zu Äthylen zu beeinflussen. Es hat sich ergeben, daß nach dem Verfahren dieses Patent Polyäthylen mit einem Molekulargewicht höher als einige Tausend nur sehr schwer zu erhalten ist, was unter anderem auch daran liegt, daß man für die Erzeugung sehr hochmolekularer Produkte extrem wenig Aluminiumtrialkyl nehmen müßte, beispielsweise

für ein Molekulargewicht von rund 28 000 nur 1 % Aluminiumtrialkyl von der verwandten Äthylenmenge. Damit werden diese Versuche aber außerordentlich empfindlich gegen Spuren von Verunreinigungen im Äthylen, wie Sauerstoff oder Wasserdampf, die die Aluminiumtrialkyle zersetzen. Außerdem laufen die Versuche sehr langsam, weil die Menge des Katalysators in der gesamten Reaktionsmischung zu klein ist.

In der belgischen Patentschrift 527 736 ist die Beobachtung beschrieben worden, daß man den Verlauf der in der deutschen Patentschrift 878 560 beschriebenen Reaktion zwischen Aluminiumalkylen

109 761/4

Fig. 3.1 (B) page 1

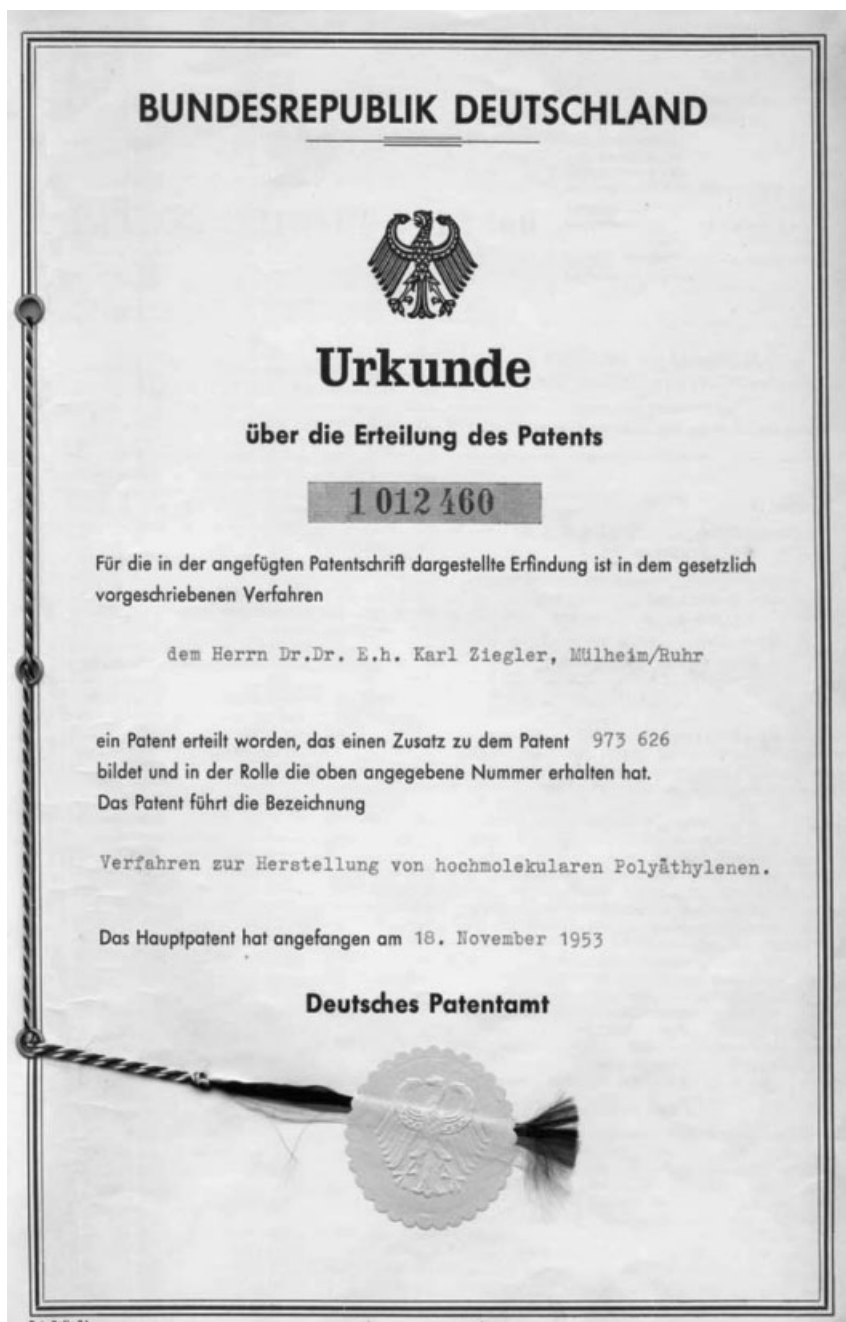


Fig. 3.2 German Patent No. 1 012 460: (A) cover page;

BUNDESREPUBLIK DEUTSCHLAND

DEUTSCHES PATENTAMT

PATENTSCHRIFT 1 012 460

DBP 1 012 460

KL. 39c 25/01

INTERNAT. KL. C 08f

ANMELDETAG: 19. JANUAR 1954

BEKANNTMACHUNG DER ANMELDUNG UND AUSGABE DER AUSLEGESCHRIFT: 18. JULI 1957

AUSGABE DER PATENTSCHRIFT: 13. OKTOBER 1960

STIMMT ÜBEREIN MIT AUSLEGESCHRIFT 1 012 460 (Z. 3941 IVb/39a)

1

Gegenstand des Hauptpatents 973 626 und dessen Zusatzpatente 1 004 810 und 1 008 916 ist ein Verfahren zur Herstellung von hochmolekularen kunststoffartigen Polyäthylenen aus Äthylen durch Zusammenbringen von Äthylen mit Katalysatoren, die aus Mischungen von Aluminiumtrialkylen mit Verbindungen der Metalle der Gruppen IVa bis VIa des Periodischen Systems, nämlich Titan, Zirkon, Hafnium, Vanadin, Niob, Tantal, Chrom, Molybdän, Wolfram, Thorium und Uran, bestehen. Die Umsetzung kann bei beliebigen Drücken und Temperaturen von -20° aufwärts durchgeführt werden.

Es wurde nun gefunden, daß die als eine Komponente des beschriebenen Katalysatorsystems für die Polymerisation von Äthylen vorgeschlagene Aluminiumverbindung gegenüber den zunächst vorgeschlagenen Aluminiumtrialkylen noch abgewandelt werden kann.

Wesentlich für die Brauchbarkeit zur Herstellung der neuen Mischkatalysatoren für die Polymerisation von Äthylen ist die Bindung mit mindestens einem Kohlenwasserstoffrest oder mindestens einem Wasserstoffatom an das Aluminium. Es können also für die Herstellung der neuen Katalysatoren Aluminiumverbindungen der allgemeinen Formel $RAlX_2$ verwendet werden, worin R Wasserstoff oder einen Kohlenwasserstoffrest, X einen beliebigen anderen Substituenten, darunter auch Wasserstoff oder einen Kohlenwasserstoffrest, bedeuten, wobei R und beide X nicht gleichzeitig Alkyl bedeuten dürfen. Beispielsweise werden wirksame Polymerisationskatalysatoren ganz allgemein dadurch erhalten, daß man Verbindungen der in den Patenten 973 626 und 1 008 916 beanspruchten Schwermetalle mit Aluminiumverbindungen der folgenden Zusammenstellung zusammenbringt:

Aluminiumhydrid — AlH_3
 Alkyl- oder Arylaluminiumhydride — $RAlH_2$
 Dialkyl- oder Diarylaluminiumhydride — R_2AlH
 Alkyl- oder Arylaluminiumhalogenide — $RAl(Halogen)_2$
 Dialkyl- bzw. Diarylaluminium-monohalogenide — $R_2AlHalogen$
 Alkyl- bzw. Aryl-Aluminiumdialkoxy- oder diaroxyverbindungen — $RAl(OR)_2$
 Dialkyl- bzw. Diarylaluminiumalkoxy- oder aroxyverbindungen — $R_2Al(OR)$

R soll hier stets einen aliphatischen oder aromatischen Kohlenwasserstoffrest bedeuten.

Mit besonderem Vorzug werden Verbindungen des Typus R_2AlX verwendet, unter denen wiederum die

Verfahren zur Herstellung
von hochmolekularen Polyäthylenen

Zusatz zum Patent 973 626

Das Hauptpatent hat angefangen am 18. November 1953

Patentiert für:

Dr. Dr. E. h. Karl Ziegler, Mülheim/Ruhr

Dr. Dr. E. h. Karl Ziegler, Dipl.-Chem. Dr. Heinz Breil,
 Dipl.-Chem. Dr. Heinz Martin
 und Dr. Erhard Holzkamp, Mülheim/Ruhr,
 sind als Erfinder genannt worden

2


Dialkyl- bzw. Diarylaluminium-monohalogenide die besten Ergebnisse liefern. Selbstverständlich können die erfindungsgemäß zu verwendenden Aluminiumverbindungen neben Aluminiumtrialkylen verwendet werden.

Es ist für das erfindungsgemäße Verfahren keineswegs notwendig, daß die Verbindungen dieser Art im völlig reinen einheitlichen Zustand zugesetzt werden. Es genügen auch Rohprodukte oder Lösungen, wie sie etwa im Zuge der Herstellung der Aluminiumverbindungen der genannten Art zunächst erhalten werden. Weiter kann man geeignete Polymerisationskatalysatoren auch mit Mischungen der erfindungsgemäßen Aluminiumverbindungen herstellen. Beispielsweise lassen sich in besonders bequemer Weise solche Katalysatoren auf der Grundlage der sogenannten Alkylaluminiumsesquihalogenide aufbauen, d. h. den Mischungen der Verbindungen $R_2AlHalogen + RAl(Halogen)_2$, wie sie leicht durch Auflösen von Aluminium-Metall in Halogenalkylen gewonnen werden können.

Beispiel 1

20 g Diäthylaluminiumchlorid werden vorsichtig mit 1 g Titantrichlorid vermischt, wobei sich diese Lösung braun färbt und einen Niederschlag abscheidet. Man setzt (alle derartigen Operationen unter Stickstoff) 200 ccm luftfreies Hexan zu, füllt einschließlich des Niederschlags in einen 500-ccm-Autoklav um und preßt Äthylen bis zu 100 Atm. auf. Beim Schütteln erwärmt sich der Autoklavinhalt spontan auf 60 bis 70° , und gleichzeitig fällt der Äthylenruck langsam ab. Man wiederholt das Auf-

Fig. 3.2 (B) page 1

81 82 10 11 21 22 43 44 45	81 82 26 27 28 29 74 75 76 77 78 79 80	BUNDESREPUBLIK DEUTSCHLAND DEUTSCHES PATENTAMT  Deutsche KL: 39 b4, 3/02 Int. CL.: C 08 f, 3/02 Patentschrift 1 257 430 Aktenzeichen: P 12 57 430.0-44 (Z 4348) Anmeldetag: 3. August 1954 Offenlegungstag: — Auslegetag: 28. Dezember 1967 Ausgabetag: 18. Juli 1974 Patentschrift <u>weicht</u> von der Auslegeschrift <u>ab</u> Ausstellungspriorität: — Unionspriorität: — Datum: — Land: — Aktenzeichen: — Bezeichnung: Verfahren zur Homopolymerisation von Propylen und α -Butylen Zusatz zu: — Ausscheidung aus: — Patentiert für: Ziegler geb. Kurtz, Maria, 4330 Mülheim Vertreter gem. §16 PatG: — Als Erfinder benannt: Ziegler, Karl, Dr. Dr.; Breil, Heinz, Dipl.-Chem. Dr.; Martin, Heinz, Dipl.-Chem. Dr.; Holzkamp, Erhard, Dr.; 4330 Mülheim Für die Beurteilung der Patentfähigkeit in Betracht gezogene Druckschriften: DT-PS 878 560 J. Inst. of Petrol. Technol., Bd. 23 DT-PS 874 215 (1937), S. 679 bis 687 DT-PS 960 268 J. Inst. of Petrol. Technol., Bd. 24 US-PS 2 220 930 (1938), S. 471 bis 495 US-PS 2 567 109 Zeitschrift für Elektrochemie, Bd. 46 In Betracht gezogene ältere Patente: (1940), S. 106 Deutsches Patent 973 626 Brennstoff-Chemie, Juni 1952, S. 193 bis 200
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DT 1 257 430

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Fig. 3.3 German Patent No. 1 257 430: (A) cover page;

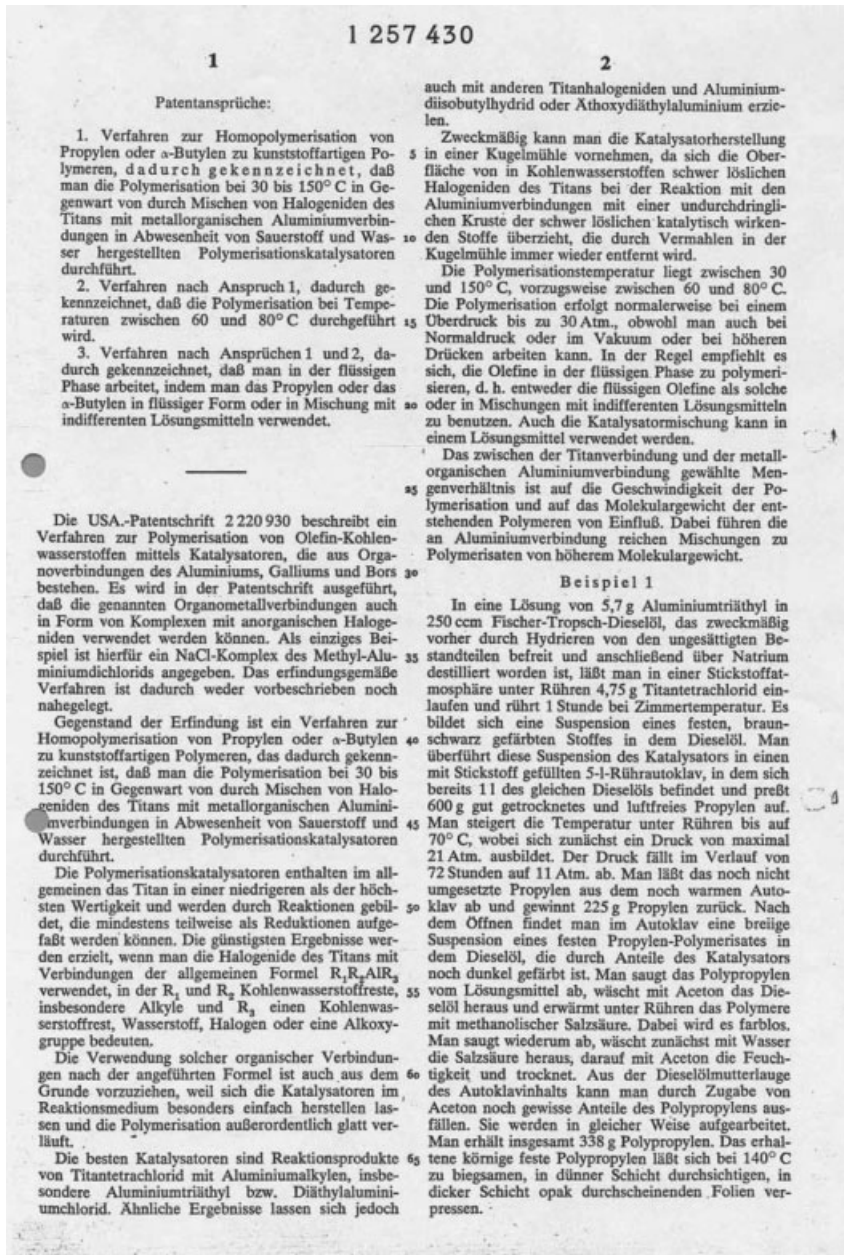


Fig. 3.3 (B) page 1

tures, and the Patent Office eventually ruled that the application should be restricted to the embodiment of the actual examples provided. Given this situation and the anticipated short 2-year term of protection remaining (related correspondence was meanwhile dated in the second half of 1970), it appeared desirable to limit the claim to those elements which were actually being utilized by German industry, i.e. synthesis of polypropylene and polybutene with the aid of a catalyst produced by admixing “halides of titanium with organometal aluminum compounds.” It was after all important to assist the German licensees in curtailing importation of unlicensed products into Germany. The German Patent Office, however, initially refused to grant a patent, and it was only on appeal that the Federal Patent Court [23], in late 1973 – that is, only after Karl Ziegler’s death – approved the issuance of a valid patent.

Since Ziegler was considered to be the formal owner of the patent rights, the patent reverted to the estate, so that it was his wife Maria, néé Kurtz, who appeared as the new owner on the deed. All rights in this and other patents were subsequently assigned to the Max-Planck-Institute for Coal Research and/or Studiengesellschaft Kohle mbH, as Trustee for the Institute.

The patent prosecution process in Germany represented only a small sample of the number of obstacles which had to be surmounted at home and abroad in order to provide a secure basis of operation for Ziegler’s licensees as well as to safeguard royalty income for the Max-Planck-Institute for Coal Research.

In terms of patent law, it should be noted that while conflicts arising between researchers and their discoveries on the one hand, and approval by the various patent offices on the other, were and are being handled differently in different countries around the world, they are essentially triggered by the same situation. When researchers recognized that their work may form the basis for a new process, or when they discovered a new product, in most cases the claims that they submitted were not supported by a sufficient number of examples, which led the various patent offices to refuse to allow the broad claims of the language being used by the inventor; this situation continues to occur today. Any hastily filed patent application concerning an interesting research discovery remains problem-ridden unless the specification is drafted in such broad terms that a competitor would have little chance of making inroads into the field covered by the patent, based on his own patent applications. The reason for these problems is that most research scientists find it tedious to carry out ancillary experiments to clarify all aspects of the invention.

In the case at hand, the patent application originally filed included nine Examples, of which eight mentioned titanium tetrachloride and one specified zirconium tetrachloride as a catalyst component, four of the Examples given referred to the polymerization of propylene, while a further four illustrated the co-polymerization process. The application had certainly been drafted under time constraints. One fact that no-one at Mülheim had been aware of at the time was that the priority patent and the subsequent Du Pont application disclosing similar findings were filed only days apart (see Chapter 5, reference [271]).

As will be reported during the discussion of further events, Karl Ziegler, nevertheless, obtained broad patent protection in foreign countries, but the Patent Office in the Nobel Laureate's own country, oddly enough, allowed only a single claim, limited to the use of titanium halides, an adequate if humiliating result, which, to make matters worse, took 20 years to achieve. In any event, titanium trichloride, the preferred titanium halide, was at least covered by the claim. Additionally, it may be noted that, in Germany, it was Ziegler and not Natta, who was awarded the priority.

As will be shown later, a US court granted Ziegler a broad catalyst claim for the polymerization of olefins despite the fact that the patent failed to set forth a single example describing the polymerization of propylene, although such an example was actually included in the priority application. The appeal court concluded that the invention in question was a pioneer invention which, by definition, called for the allowance of a broad claim. The judgment rendered against Phillips Petroleum Co., Oklahoma [24], led to the further result that, with respect to the United States, the novel feature of using titanium trichloride in place of titanium tetrachloride, as claimed by G. Natta/Montecatini, was clearly encompassed by Ziegler's pioneer invention.

3.2

Foreign Patent Protection for the Inventions of Ziegler and Co-Workers

In order to obtain worldwide patent protection, it was necessary to ensure that within 1 year from the date that any German application was filed, the so-called priority or Convention year¹³⁾, corresponding applications relating to the same invention also be filed in foreign countries. Certain countries examine merits of the subject matter of an application in detail, while others do so with varying degrees of exactitude.

The initial five patents¹⁴⁾ had matured to corresponding foreign patents in most countries¹⁵⁾ between 1955 and 1958, and in Brazil, France, Great Britain and The Netherlands, for the most part, by 1960, in the United States and Denmark by 1966, and in Canada by 1970. With hindsight, it would appear that in terms of territory, such broad and costly patent protection was excessive. Most manufacturers did not,

13) The Agreement of Paris, March 20, 1883 stated that a priority date in one country must be acknowledged by all members of the Agreement.

14) The polyolefin application i.e. polymerization of higher α -olefins, polypropylene etc. is discussed on the end of this chapter.

15) The first four applications were filed and the patents issued relatively soon thereafter covering Egypt, Argentina, Australia, Belgium, Belgian Congo, Bolivia, Botswana, Chile, Colombia, DDR, Ecuador, France, Finland, Greece, India, Iraq, Iran, Ireland, Israel, Italy, Japan,

Yugoslavia, Lesotho, Luxembourg, Morocco, Mexico, New Zealand, Norway, Austria, Pakistan, Peru, Poland, Portugal, Sweden, Switzerland, Spain, Syria, South Africa, Swaziland, Czechoslovakia, Tunis, Turkey, and Venezuela. The 5th application in which the combination of magnesium or zinc alkyls together with titanium chloride is mentioned, was only filed and the patents issued in Argentina, Belgium, Canada, France, Great Britain, Italy, Japan, Netherlands, Austria and USA.

after all, export the basic product itself, but rather the industrially finished articles made therefrom.

It was interesting to observe how industrial nations in which the Patent Office had reservations about the merits of patent applications, were handling the above-mentioned applications, because it was here that some of the early conflicts of interest were being argued or litigated. The sheer number of scientific publications and improvement patent applications from third parties which emerged after the Max-Planck-Institute for Coal Research had published its findings, was truly impressive. Licensees in particular, but also research divisions of companies which did not receive a license, endeavored to acquire an improved patent base of choice so as to establish a strong starting position, especially in the event of Ziegler being denied patent protection by various Patent Offices around the world. The dates on which Ziegler's patents were first published were significant with respect to third-party patent rights that were filed later. It was well-known that, at the time, both in Belgium and Israel, patents were disclosed to the public relatively soon after they were filed and were, therefore, heavily researched. An interested party could ascertain at an early stage, both the contents and scope of any patent application on file in those countries.

The fight for patent rights was very costly, both financially and in terms of time, and, in hindsight, was draining the resources of all parties concerned. Even the most loyal "friend", in the end hoped only to strengthen his own starting situation.

The reader should by now begin to have a clearer picture in mind of how Patent Offices around the world had handled the subject matter in question, particularly those in Japan, The Netherlands, Great Britain, Switzerland and, the United States.

The first three German patent applications had been filed abroad as a single application (Combination I) because they covered similar subject matter, i.e. the use of aluminum trialkyls and titanium halides as catalyst components. As could be reasonably expected, the next patent application filed in a foreign country disclosed and claimed the production and use of catalysts and their application in the production of polyethylene, using compounds of the formula R_2AlX (X = halogen, alkoxy, etc) + titanium halides or other transition metal compounds¹⁶⁾ (Combination II). The third foreign-filed application centered around catalysts composed of magnesium alkyls and/or zinc alkyls and titanium halides (Combination III), and, finally, the fourth application, as previously mentioned, disclosed a method for polymerizing higher olefins (propylene, butene, etc.; Combination IV).

The Patent Examiner in Japan allowed the applicant to submit Examples during prosecution and also accepted the belated filing of a description of the polymer products. As had been done in Germany, the Japanese Patent Office granted a broad

16 The combination partly contained in many countries also the application of components of metals of group VIII (Egypt, Belgium, Canada, Chile, Ecuador, France, Greece, Iraq, Iran, Israel, Yugoslavia, Luxemburg, Morocco, Mexico, Pakistan, Peru, Portugal, Spain, Switzerland, South Africa, Syrien, Turkey, Tunis, USA, Venezuela). This part originated out of

the German priority application of Dec. 1954 which in other countries was followed by itself and issued as patent. (Argentina, Australia, Brazil, Denmark, DDR, Germany, Finland, Great Britain, India, Ireland, Japan, New Zealand, Netherlands, Norway, Austria, Sweden).

scope of protection in the field of polyethylene/catalysts. Switzerland [25], 26, too, allowed a broad claim. But in this case, the Examiner had recognized that the application actually encompassed two separate inventions, i.e. aside from the production of polyethylene, it also disclosed the production of the polymerization catalysts *per se*, a viewpoint which was to attain special significance particularly in the United States.

The ready willingness of the Examiner in The Netherlands to grant patents to Ziegler was stalled by an opposition filed by Resinova of Milan. Aside from the opposition arguments publicized in the German proceedings, Resinova relied on an older British patent and two older US patents as references¹⁷⁾. But this combination, too, failed to support a legitimate argument. The subject matter involved was utilization of the so-called Friedel-Crafts catalysts in connection with isobutene and/or isobutene/isoprene mixtures. A formula set out in the above-mentioned patents would only be of limited relevance to the use of organometal compounds of aluminum¹⁸⁾. The opposition countered with the following argument: if the aluminum trichloride of the BASF/Fischer patent [5] (as applied to ethylene) was replaced with the organoaluminum compound of the Standard Oil patent (as applied to isobutene/isoprene), in other words, if the two patents were combined, a Ziegler catalyst for olefins would be obtained.

The Netherlands Examiner did not accept this argument because

"The skilled artisan would have no reason to combine the German patent ... with the US patent, and would certainly not do so in order to produce solid polyethylene."

As the Examiner further stated, while it is not difficult to form solid products from isoalkenes, it would be doubtful whether, in accordance with the German patent (BASF), solid polyethylene could be produced by using aluminum chloride and titanium tetrachloride. On the contrary, the literature indicates¹⁹⁾ that adding titanium tetrachloride to aluminum trichloride would be ineffective with respect to the formation of solid polymers from ethylene.

The Netherlands Application Section, in an interim decision, gave detailed consideration to the opposing arguments, but rejected them *in toto*. The patent was issued with a broad claim [27]. Practically simultaneously, the Netherlands Patent Office granted Ziegler's subsequent patents on the polymerization of ethylene [28, 29].

Aside from the established process protection, Anglo-Saxon patent law also provides for product claims directed to a novel product. It, therefore, behooved Ziegler to claim not only the newly discovered catalysts, but also the novel polyethylene product *per se*. Since, at the time the application was filed, so-called high-pressure

17) Standard Oil Dev. Co., GB P 587,475, issued Apr. 28, 1947; Standard Oil Dev. Co., US P 2,446,897 (SN 470,030), D.W. Young and coworkers, issued Aug. 10, 1948; Standard Oil Dev. Co., US P 2,220,930 (SN 238,561), Ch. A. Kraus and co-workers, issued Nov. 12, 1940.

18) Dimethyl aluminum (dimethylamine)-complex or etherate of aluminumtriethyl.

19) Dr. H. Zorn, *Angew. Chemie*, 60, 185 and IG Farbenindustrie AG, DE P 718 130, Zorn and coworkers, issued Aug. 18, 1935.

polyethylene was already known in the art, the product claim referring to the Ziegler polyethylene had to be limited to a product with novel characteristics. But even that was unacceptable to the chemical industry, led by ICI (Imperial Chemical Industries), Esso Research and Engineering Co, Phillips Petroleum Co., and Standard Oil Company of Indiana. Citing more than 20 older references – some familiar and some unknown – the opposing parties vehemently contested the issuance of the British patents for the polymerization of ethylene, the catalyst product claims, and the patenting of the novel polyethylene. The opposition arguments presented in the German proceedings – BASF/Bayer v. Ziegler – had become common knowledge around world, but apparently, so too had the news of the final outcome of the dispute. It was eventually agreed that in return for Ziegler dropping his polyethylene product claims, the opposition would withdraw their remaining arguments. Thus, in 1960, based on this arrangement, the first two combination applications were issued as patents in Great Britain [30, 31], carrying strong catalyst product claims and process claims for the polymerization of ethylene.

The third foreign-filed application, relating to the use of magnesium alkyls and/or zinc alkyls as organometal components, contained only process claims, since the references cited against it, particularly those relating to purely academic studies of the reaction of Grignard compositions and titanium tetrachloride²⁰⁾, came too close to the instantly claimed catalysts, and/or anticipated them. The comparatively weaker patent was issued [32] in 1961. Industrial utilization of the magnesium alkyls and/or zinc alkyls was never considered over the years, which proved to be fortunate for Ziegler.

In the Pool Agreements with Montecatini (cf. Section 2.9), as far as “polypropylene” was concerned, Ziegler had accepted the stipulation that the first two applications by Montecatini/G. Natta in Italy and the application by Ziegler/H. Martin be jointly filed in foreign countries, with the aim of providing support for a broad claim directed to the polymerization of olefins higher than ethylene. The unsatisfactory developments in Germany were not perpetuated in other countries. By 1960/61, patents had been issued in 31 countries.²¹⁾ The patent claims allowed by the various Patent Offices were directed to a process for the polymerization of propylene and other olefins using Ziegler catalysts, co-polymerization of such olefins, *inter alia* with ethylene, as well as the characterization of the polymers in terms of their structural regularity and/or their varying degrees of crystallization, their usefulness in being shaped specifically into foils, threads and other plastic materials and, as far as possible, polymer product claims. Solid polypropylene was, after all, a novel product, and this was especially true in terms of the product’s crystallinity and varying stereo structures.

20) Such mixtures had never before been tested on olefins.

21) Argentina (1956), Australia (1958), Austria (1958), Belgium (1955), Brazil (1958), Chile (1956), Columbia (1957), Egypt (1961), France (1957), Finland (1960), German Democratic Republic (1958), India (1957) Ireland (1961), Israel (1957), Italy (1955), Japan (1959), Lux-

emburg (1956), Mexico (1957/1960), New Zealand (1958), Norway (1959), Pakistan (1958), Peru (1961), Poland (1960), Portugal (1956), South Africa (1957), Spain (1955), Switzerland (1961), Turkey (1959), United Kingdom (1959), Venezuela (1956), Yugoslavia (1957/1958),

US-based Standard Oil of Indiana launched a broad-based attack involving numerous countries, against the issuance of “polypropylene patents”, particularly in Europe in the form of opposition proceedings to obstruct the process of granting patents, or by bringing invalidity actions against prior issued patents, especially in Norway, Switzerland, Denmark and The Netherlands and – as discussed earlier – in Germany.

Standard Oil’s strong opposition to the Ziegler and/or Ziegler/Montecatini, patents rights was, as previously mentioned, a clear indication that this conglomerate was producing polypropylene on an industrial scale and/or that production facilities were under construction.

On September 26, 1959, a patent for the combination application was issued in Norway. With a letter dated December 24, 1964, Avisun Corporation, of Philadelphia, objected to a warning issued by Montecatini to the distributors and customers in Norway, urging them to stop selling or using Avisun polypropylene. Avisun simultaneously brought an action for patent invalidity, supporting its arguments with three expert opinions which claimed that olefins could not be polymerized with a catalyst combination consisting of titanium tetrachloride and ethylaluminum dichloride. I. Pasquon from the School of the Polytechnic Institute in Milan submitted an opposing expert opinion demonstrating that ethylaluminum dichloride in admixture with transition metal chlorides was very well capable of polymerizing propylene to crystalline polymers – even though the polymerization reaction was relatively weak.

Avisun’s compromise proposal to forego patent protection for the titanium trichloride/ethylaluminum dichloride catalyst combination was rejected by both Ziegler and Montecatini. By 1967, no end to the controversy was yet in sight. Final arguments before the Court of Appeals in Oslo had been scheduled for November 17, 1970, but the appeal hearing never took place. Parallel developments in other countries led to a more complete solution.

Because of an opposition filed by Staatsmijnen in The Netherlands, the scope of the claim ultimately allowed in the combination application was limited, *inter alia*, to aluminum compounds with fewer than two halogen atoms per atom of aluminum. Nevertheless, in 1964, Standard Oil of Indiana filed a complaint at the District Court in Amsterdam to have the patent declared invalid.

The patent invalidity action brought by Standard Oil of Indiana in 1965 against the Montecatini/Ziegler patent in Switzerland was tried in the District Court for the Canton of the Town of Basel and dismissed, based on an expert opinion submitted in 1968 by the independent expert Professor H.G. Elias of the ETH Zurich. Aside from challenging whether the alkylaluminum dihalides/titanium trichloride catalyst combination was, in fact, an effective catalyst, a question expressly answered in the affirmative by the expert, the opponents intended to weaken the patent by proposing a formal limitation of the claim. Standard Oil of Indiana appealed in the Federal Court at Lausanne, but withdrew their claim shortly before the court reached a verdict.

The complaints filed in Norway, Switzerland, The Netherlands and Denmark were settled by an agreement [33] dated January 1, 1970, to wit: as consideration for withdrawal of the complaints and subsequent issuance of valid patents [34], [35], [36],

the Head of Standard Oil of Indiana's Patent Department, Arthur Gilkes, negotiated a non-exclusive license in Belgium for the production of polypropylene, but not for its sale. In Belgium and The Netherlands, an exclusive license was owned by Shell (Rotterdamse Polyolefinen Maatschappij N.V.). For a payment of \$200,000 by Montecatini/Ziegler, Shell relinquished its exclusive rights and allowed Standard Oil of Indiana to sell limited amounts of polypropylene. The compromise terms, which applied to all of Europe, furthermore called for Standard Oil of Indiana to obtain a non-exclusive license for Norway, Switzerland and Denmark. The negotiators at Montecatini who were hammering out the agreement, disregarded Ziegler's requirement that Standard Oil of Indiana withdraw its opposition in Germany. There, the problem was solved by limiting the scope of the allowed claim (see above).

In Great Britain, the process of granting a patent [37] for polypropylene (1959) progressed smoothly. The patent which was issued contained broad process claims as well as corresponding product claims covering the new polymers. It was not until 1 year later that the US-based Phillips Petroleum Company brought an action for invalidity of the aforementioned product claims by citing their own product claims, and simultaneously prosecuted the British application which claimed an earlier priority date, i.e., January 1953. Thus, Phillips Petroleum and Ziegler's paths crossed for the first time. Since the Phillips application was itself embroiled in an ongoing opposition proceeding, the case never came to trial, nor was a decision ever reached. After that, there were no further attacks on the validity of the British patent.

The events which occurred in Japan were much more dramatic. Even though the Examiner had allowed the application as early as 1959, 2 years later, Avisun Corporation and also Sun Oil Company, American Viscose Corporation and Eastman Kodak, all based in the United States, as well as Tokoyama Soda and Shin Nippon Chisso Hirjo Company, of Japan, brought actions for patent invalidity in Japan. In mid-1964, the patent was declared invalid by the Japanese Patent Office [38], 39] after a petition which had been filed a few days earlier, requesting that the scope of the patent be limited, had also been denied. In its decision, the Board followed the line of reasoning urged by the opponents that certain catalysts which would be encompassed by the existing claim were ineffective. To be sure, while experiments conducted by the defendant demonstrated that a large number of the disputed catalysts did indeed, work, they also showed that a handful actually did not [40]. These findings were, however, not allowed to affect the decision.

The international press and all parties involved were alarmed and immediately brought out statistics to show that Japan, for nationalistic and protectionist reasons, prevented foreign patent owners from exploiting their patent rights in Japan, the current case in point involving the pioneer invention of two Nobel Laureates. Now, for the first time, the cohesiveness of the worldwide polypropylene monopoly built up by Montecatini and Ziegler had been punctured. The situation was additionally aggravated by the fact that three Japanese licensees²²⁾ had meanwhile started using the patent to produce and process polypropylene, and three further licensees²²⁾ were using it to produce fibers, and all of them had already paid substantial advance fees for their licenses.

22) Mitsui Chemical Industry Co. Ltd., Tokyo;
Mitsubishi Petrochemical Co. Ltd., Tokyo;
Sumitomo Chemical Co. Ltd., Osaka; Tokyo

Rayon Co. Ltd., Tokyo; Mitsubishi Rayon Co.
Ltd., Tokyo; Toyo Spinning Co. Ltd., Osaka.

Montecatini urged that, aside from presenting the necessary legal and technical arguments, international diplomacy officials should become involved. Ziegler strongly opposed such intervention as he feared it would adversely affect the situation. Nonetheless, the Departments of State in Rome and Bonn were called on to intercede through their respective Tokyo representatives. Exposés and letters were drafted which were, in fact, answered by the respective Foreign Offices. Ziegler regarded his actions as a gesture of good will towards Montecatini – in fulfillment of his obligation, albeit grudging. Additionally, the Presidents of both the German and Italian Patent Offices were approached in an effort to persuade them to use their influence through the respective Tokyo authorities, to protest the biased preferential treatment of Japanese economic interests over the legal rights of foreign nationals.

On the other hand, Japan was now seeing the formation of conglomerates, such as Asahi Chem. and Showa Denko, which, based on the known decision, announced programs for building their own propylene production facilities.

Tokoyama Soda and Shin Nippon Chisso had already started production and had thereby asserted the independence of their processes vis-à-vis Montecatini/Ziegler.

The complexity of the economic situation was not in any way eased by the difficulties encountered at the patent end. To begin with, a patent claim had to be presented which clearly differentiated the catalyst combinations which were effective from those that did not work. Time was of the essence. There was no possibility of undertaking a major experimentation program. Communication was severely hampered due to the language barrier created by the Japanese attorneys' poor command of the English language. Uncertainty and differences of opinion dogged the discussions over the next few months. Through slow and tentative development the situation was somewhat clarified by limiting several aspects of the claim, a process that was not only necessary but that should also be acceptable.²³⁾ Reading between the lines of the correspondence, it seems that the situation was clearly causing enormous anxiety. All parties concerned recognized the danger of the claim being limited too severely, this would create an opportunity for competitors to avoid the claim altogether. The Japanese attorneys Ushida, Irigana and Homma pushed for a speedy resolution. Nevertheless, Montecatini established a test program to develop a more solid factual base which could be used in future decisions, should they become necessary.

Meanwhile news spread that the delegates of the Italian government agencies in Japan had caused quite an uproar which precipitated a sharp reaction from the

23) A limitation of the claim was proposed as follows (December 1964):

1. The starting material for the polymerization of olefins was changed from unlimited α -olefins to propene, butene-1 and styrene in addition to mixtures thereof and mixtures with ethylene.
2. Both components for the production of catalysts were limited by a change from transition metal compounds of metals of group IV–VII to halides and oxy halides of titanium and vanadium. The second com-

- ponent was limited by a change from metals, alloys, metal hydrides or organo metal compounds of metals of group I–III to organo metal compounds of lithium, sodium, beryllium, magnesium, zinc and aluminum. In the case of aluminum one ligand could be hydrogen, alkoxy or halide.
3. Also hydrides of the named metals should be included. Finally complex compounds of alkali metals with organo aluminum compounds should be included.

General Director of the Japanese Patent Office. In January of 1965, the President of the German Patent Office [41] contacted his colleague in Tokyo to ask on what legal grounds a patent would be rejected in its entirety given the fact that only one part of the claim was apparently not workable. Two months later, the tests which had meanwhile been carried out at Montecatini led them to demand further limitations to the claim.²⁴⁾

A timely appeal against the Japanese Patent Office's final rejection was filed in the "High Court" (Regional Court of Appeals) in Tokyo. Attorneys for both Montecatini and Ziegler rushed to Japan in order to ferret out options to avoid disaster for their clients in the next higher, final review court. Ziegler remained calm. He spent his usual August vacation in Sils-Maria in Switzerland and merely sent word from there that he would be available again in September.

The exchange of views taking place among the individuals involved on Montecatini/Ziegler's side assumed frenzied proportions. There was no way of judging what mandatory, yet moderate claim limitations should be presented to the court to guarantee success. Discussing this question with the judges on the case was not allowed under the rules.

There was thus, nothing left to do but submit a request for limitations to claim to be implemented using clearly defined language. The Japanese group of attorneys threatened to withdraw as representatives if the newly introduced claim limitations were not sufficiently severe. It took a major effort of persuasion on the part of Montecatini to effectively defend the limitations to vanadium chloride and titanium chloride for the preparation of the catalyst, and the limitation of the starting olefins to ethylene, propylene and their mixtures. The correspondence exchanged in the Spring of 1966 was entirely devoted to discussions of the pros and cons of the proposed claim limitation. The major focus here was the question of whether the use of vanadium oxychlorides, which were important in connection with co-polymerization, should be argued, particularly since the specification of the original application did not contain any examples illustrating this combination. In a subsequent Montecatini application, specifically directed to the co-polymerization of ethylene and propylene, the Japanese Patent Office rejected this catalyst combination on the grounds that it was anticipated by the patent presently involved in the invalidity litigation.

In 1967, the claim was allowed in a further limited form (chlorides, bromides and iodides of titanium as transition metal components). Thus, the patent [42] was issued without opposition from the opponents.

The scope of the patent issued in Japan could be regarded as basically satisfactory as was ultimately the case in Germany. It could not have been anticipated that the producers would significantly change their catalyst systems during the time that remained before the patent expired in 1972. As may be gathered from the foregoing

24) Thereafter zinc alkyl as well as divalent titanium and vanadium compounds were excluded, because it was established that while trivalent vanadium and titanium compounds worked as catalyst components, titanium and vanadium compounds with a

valance below three did not. Finally metal hydrides including aluminum hydride were excluded as the light metal compounds, because in practice such starting materials would not be employed.

account, more than 10 years of concerted effort were required to obtain valid patent protection. During this time span the earliest patent infringement actions were filed but these will be discussed later.

In the country with the most powerful market, the United States, significantly greater efforts were being made to obtain valid patent protection and, to shield licensees and/or stop any infringements by going to court.

3.3

The Situation in the United States

As in the other countries, Ziegler's patent attorneys, R. Dinklage and A. Sprung in New York, filed each of the three combination patent applications (I-III) in the Patent Office in Washington within 1 year of the priority date in Germany, i.e. one on November 15, 1954 [43] and the other two on January 17, 1955 [44]. One of the combination applications was broadened to incorporate metal compounds consisting of metals from Group VIII of the Periodic System, claiming a German priority date of December 1954 [45]. The application relating to "higher olefins", particularly polypropylene (IV), will be discussed later.

The initial Office Actions on the merit referred to supposedly "older" US patent applications originating from the research team of A.W. Anderson and co-workers at Du Pont. These claimed priority dates of August 16, 1954 and later and are evidence of the type and scope of the experiments being undertaken at Du Pont in the same area.²⁵⁾ But they also document the fact that 9 months and/or a few days after the patent application was filed for the data produced at Mülheim, Du Pont also filed patent applications relating to the same subject matter. After Ziegler submitted the texts of his early applications filed in the German Patent Office, the Examiner could no longer sustain his arguments in that respect. Arguments relying on other overlapping, older publications could also be dismissed.²⁶⁾

25) US P 2,905,645, issued Sep. 22, 1959 (priority SN 450,243 of Aug. 16, 1954), Anderson and co-workers. Catalyst product claim: $\text{TiCl}_4 + \text{LiPhenyl}$ or LiAlR_4 or Sn- or Cd-alkyls as catalysts for the polymerization of ethylenical unsaturated compounds. Reduction partly below Ti^{3+} . The combination $\text{TiCl}_4 + \text{AlMe}_3$ (Example 18) was not claimed.
US P 2,721,189, issued Oct. 18, 1955 (priority: SN 433,144 of Aug. 30, 1954), Anderson and co-workers. Product claim: polybicycloheptene, catalyst: $\text{TiCl}_4 + \text{EtMgBr}$.
US P 2,900,372 issued Aug 18, 1959 (priority: SN 453,146 of Aug 30, 1954), Gresham and co-workers. Process claim: polymerization of ethylene, catalyst: molybdenum pentachloride + Grignard compounds or LiAlR_4 or tinitetraalkyls.

US P 2, 862,917 issued Dec. 2, 1958 (priority SN 470,812 of Nov. 23, 1954), Anderson and co-workers. Process for the polymerization of ethylene, catalyst: $\text{TiCl}_4 + \text{aluminum alkylhalides}$, above 150°C .

26) US P 2,691,647 issued Oct. 12, 1954 (priority SN 324,610 of Dec. 6, 1952) and US P 2, 731,453 issued Jan. 17, 1956 (Priority SN 324,603 of Dec. 6, 1952, both Field and co-workers. Catalyst: metal oxides of metals of group VI + reduction agent such as alkali hydrides or alkali metals.
US P 2,567, 109 issued Sep. 4, 1951 (priority SN 174,139 of July 15, 1950), Howard, catalysts: $\text{TiCl}_3 + \text{hydroxylamine}$ as reduction agent to Ti^{2+} .

No other references will be mentioned here, except for the product claim directed to a linear polyethylene, which caused Ziegler to cancel his own proposed claim covering Ziegler-polyethylene.²⁷⁾

The debate with the Examiner went on for at least 3 years, creating the definite impression that he thoroughly enjoyed exercising the decision-making power vested in him. All the same, Ziegler was ultimately successful in submitting claims which being acceptable to the Examiner, appeared to be allowable on a broad basis – a feat also accomplished through verbal interviews with him. None of this would have been possible without the skillful assistance of the US attorneys who knew how to expertly present the information derived from Ziegler and Martin.

But the decision-making latitude enjoyed by the US Patent Examiners could also lead to peculiar situations – in this instance taking on far-reaching significance in connection with future exploitation of the invention. While the Examiner in charge of the first combination application initially required a division of the application into process and catalyst claims, but four years later called this ruling inadvertent and, thus, catalyst claims, the same Examiner repeated his division (restriction) requirement [46] when it came to the second combination application, even though the reason why he did so remained a mystery. Nevertheless, the grounds for division were clear in both cases – the catalyst itself, on the one hand, and its utilization for the polymerization of ethylene, on the other, were two separate and distinct inventions. The result of this was that the two divided applications which emanated from the original case (II) became separate patents issued almost 15 years apart. In the United States, the period of protection of issued patents started with the issue date, that is, the second patent expired 15 years after the first. Some of the licensees and infringers saw this as a welcome opportunity to go before the High Patent Court to challenge the propriety of the Examiner's action. The court decided in Ziegler's favor – for the Max-Planck-Institute for Coal Research a probably uniquely favorable development with long-term effect (see p. 216/217).

Another situation of far-reaching consequences in the United States emerged from the fact that, in the individual US applications, Ziegler had not only named himself as an inventor but also the particular co-worker who had conducted the experiments on which the application was primarily based. In the case of a consolidation application, the US Patent Rules required that each individual application list the same inventors. This would insure that the subject matter of the consolidation application did, in fact, incorporate all aspects of the underlying inventions. It became apparent that, in naming the inventors, Ziegler had neglected to take US patent law into consideration, so that the inventorship entities had to be amended accordingly. A review of the history of the invention reveals that each of the individual inventors – Ziegler, Holzkamp, Breil and Martin – had actively participated to varying degrees in the conception and/or development of the catalysts and their utilization. Thus, the requirements for a “joint invention” had been met. The time-

27) E. I. Du Pont, de Nemours and Co, USA, US
P 2,816,883 (SN 240, 044) Larchar and Pease,
Priority Apr. 3, 1947, (see [71]).

frame within which the catalysts for producing high molecular polyethylene had been developed was rather narrow (see p. 10, 16, 24, 25). Given these circumstances, the same inventors had to be assigned to each of the relevant US applications, a task completed in 1957/1958. Correction of the inventorship became the target of fierce attacks by future opponents.

The opposing arguments advanced by the Examiner in connection with the aforementioned consolidation applications (I–III) were successfully overcome by the end of 1958/early 1959. The subsequently negotiated patent claims had been accepted by the Examiner, or, rather, they had not been rejected further. Actually, as matters stood, nothing should have prevented the claims being accepted at this point. However, certain patent strategy considerations (see discussion below, p. 115) on Ziegler's part compelled him to pursue a different path. The Patent Office in Washington, also insured that any premature euphoria was held in check. The Examiners pulled Office Actions to Ziegler off the assembly line so-to-speak, with each of these initiating an interference proceeding.²⁸⁾

In connection with the first consolidation application, the Examiner cited a further Du Pont Application [47] claiming an August 16, 1954 priority date. The Examiner apparently considered the subject matter of this application to be practically identical to Ziegler's specifications and claims.²⁹⁾ The same situation unfolded with respect to the second and third of Ziegler's consolidation applications.³⁰⁾

First, however, it is important to focus on the examination proceedings regarding the fourth Ziegler application – directed to the polymerization of propylene – and do so within the context of certain applications covering the same or similar subject matter, which had been filed by Montecatini, claiming priority dates of June 8, 1954 (inventor, G. Natta) and July 27, 1954 (inventors, G. Natta, P. Pino and G. Mazzanti).

3.4

Polypropylene: Ziegler/Natta, Conflict as to the Priority

Ziegler's dismay at Montecatini's actions in dealing with the findings obtained by Natta had barely dissipated in early 1955, when impending deadlines for filing the corresponding patent applications in foreign countries forced the two parties to work together under a cooperative arrangement. Montecatini, represented by its patent department, headed by Messrs. De Varda and Pirani, initially played the trump card of Natta's earlier priority and demanded that the Washington-based law

28) See Chapter 1, footnote 1.

29) Interference No. 91 379 DuPont/Ziegler of Nov. 14, 1960; US P 3,541,074, Du Pont, Anderson and co-workers, Example 18: $\text{TiCl}_4 + \text{Al}(\text{CH}_3)_3$ 1 : 1. The first decision was in favor to Du Pont Oct. 24, 1964 (it was of no practical importance, since the catalyst was too limited and too expensive).

30) Interference No. 90 957, Phillips Petroleum Co/Ziegler and co-workers May 09. 1960. Phillips Petroleum Co, Lyons and co-workers: US Application, 495,054, claim 21; Ziegler and co-workers: US Application 482 412, claim 38; decision Feb. 21, 1961, priority given in favor of Ziegler and co-workers.

firm of Toulmin & Toulmin, selected by Montecatini, also handle Ziegler's patent application on polypropylene in the United States.

The first Montecatini application was directed to the use of titanium tetrachloride and aluminum trialkyls as the catalyst mixture; the second added dialkylaluminum chloride as an alternative component in place of the aluminum alkyl, whereby, propylene was already present during the preparation of the catalyst mixture. The third application covered polypropylenes *per se* as new products. The three applications claimed Italian priority dates of June 8 and July 27, 1954. Ziegler's application, which was filed in Germany a few days later, on August 3, 1954, covered a broader spectrum of catalysts, but did not initially encompass any product claims. The parties arranged to file the four applications in the United States on the same day in order to avoid having one US application cited against another. Apart from this, every effort was to be made to have each of the four applications issued as patents separately, whereby any overlapping subject matter was to be deleted during prosecution.

Even though both parties, Montecatini and Ziegler, emphasized that it was their aim to achieve the best possible results in terms of a mutually strong patent position, it became apparent during prosecution that any steps taken by the US attorney, who had been chosen by Montecatini, did, in fact, further Montecatini's interests. The objective of having the individual applications prosecuted separately in the United States, with all applications being handled by the same attorney, was bound to create a conflict of interest.

Even the US attorney himself, Mr. Toulmin, expressed his doubts during the early prosecution stage in a letter to his client Montecatini (Chapter 1, reference [173]):

"An issue that I have long feared might be raised in connection with the question of what Professor Natta contributed over Dr. Ziegler and therefore whether Professor Natta was a genuine inventor has now been precipitated by the attached editorial."
"Therefore, Natta, using the exact catalyst of Ziegler produced polypropylene in his early work. It was not until later that he began to be selective in his selection of the catalyst."
"Dr. Orsoni, in one of his communications, indicated that he thought, as we understood him, that you could avoid this situation because of the selection by Natta of a special catalyst, but unfortunately, in the early invention, which was fundamental, Professor Natta used the exact catalyst of Ziegler, and it was not until later that Natta began his selectivity."

Natta should have named Ziegler and co-workers as co-inventors. The invention was dependent on Ziegler (see Memorandum, A. Sprung, Chapter 1, reference [191]).

In the first US application claiming an Italian priority date of June 8, 1954, the Examiner pointed out, *inter alia*, the United States patent of Field and Feller (Chapter 1, references [14, 15]), and, in particular, Example 21³¹⁾ in that patent. Natta repeated this example, but was unable to produce solid polymers in accordance therewith

31) Polymerization of propylene with a catalyst
 out of sodium metal and cobalt molybdate
 (CoMoO₄).

(Chapter 1, reference [16]). This finding was subsequently confirmed by H. Martin³²⁾ while conducting further experiments. In connection with the second US application, with a priority date of July 27, 1954, no noteworthy references were cited by the US Patent Office.

The Examiner's argument that Ziegler's published Belgian Patent No. 533 362 disclosed the same catalysts for the polymerization of ethylene, and that the substitution of propylene for ethylene as the monomer would therefore be obvious, to substitute propylene for ethylene as the monomer, was overcome by pointing out that the first member in a series of homologs behaves differently as compared to the succeeding members, and it would not have been obvious to obtain the same results when using propylene in place of ethylene. The ICI-high pressure process was unsuccessful in converting propylene into solid polymers – only liquid oligomers were obtained.

After this first exchange, Toulmin deliberately delayed further prosecution of Ziegler's application claiming the August 3, 1954 German priority date, with the intent of initially concentrating on having the Montecatini applications issued as a patent. A telex sent to Montecatini from Toulmin during this period was uncovered some time later [48], and it stated:

"Extension for handling Z four [Ziegler's S. N. 514,068] is both satisfactory and desirable [sic] Examiner indicated he would not be taking up Z four for some time in future. Great advantage your company delay Z four to give us time amend and get allowed MC [Montecatini] cases. This policy confidential recommend no disclosure as to MC policy to Germany [Ziegler]"

This was further expanded in a letter written in 1957:

"As you know, it was our objective to play for time in Z-IV to give you the opportunity to have your meeting with Prof. Ziegler and Dr. von Kreisler."

About mid-1956, Ziegler's patent attorney, von Kreisler, requested a legal opinion from his US associate, R. Dinklage of New York, in order to determine whether it might be useful to consolidate the Ziegler polypropylene application of August 1954 with Ziegler's earlier applications on "polyethylene" filed in late 1953/early 1954, so as to establish a firm priority basis vis-à-vis third parties, particularly Du Pont.

Dinklage gave his expert opinion [49], and in conclusion noted that since the Ziegler/Martin polypropylene application could be described

"as establishing conception and reduction to practice prior to the constructive reduction to practice date of Montecatini",

in his estimation, it would thus be given a priority date prior to the Natta applications, and that Ziegler would thus

"maintain broad generic claims at least in the Z IV case or in a consolidation."

32) See Chapter 1, reference [16].

This analysis was based on the fact that Natta had used a Ziegler catalyst to polymerize propylene, that Ziegler had informed Natta of the catalysts, and that the Patent Office was bound to declare an interference between the Ziegler polyethylene/polypropylene combination, on the one hand, and the Montecatini polypropylene, on the other. It was Dinklage's prognosis that in such an interference proceeding, Ziegler would be able to establish earlier priority dates with respect to the process for the production of polypropylene, but that, regarding the product claims for the new polypropylene, Montecatini would probably be the one to present the more desirable characteristics. (Solid, crystalline, thermoplastic polypropylene³³) was unknown at the time). Attorney Toulmin "charged" against the Dinklage opinion. He believed Montecatini's interests were compromised and argued that Dinklage's opinion would invite fraud, however von Kreisler dismissed this argument [50].

Von Kreisler engaged a previously uninvolved US attorney, Mr. Nelson Littell, to provide a neutral opinion. Littell's evaluation [51] became available by mid-1957 and it confirmed Dinklage's assessment: "In order to protect the mutual interests of Ziegler and Montecatini in the field of" polymerization "of higher olefins against Du Pont Company, it is desirable to put the applications having the earliest dates into interference with the Du Pont applications. This application would appear to be a continuation-in-part application [52] combining the disclosure of the Ziegler applications" (of November 1953 and of August 3, 1954). "The invention claims the polymerization of olefins broadly and the polymerization of ethylene specifically, both of these claimed inventions were made on November 17, 1953, prior to any date which can be claimed for the Montecatini applications." "There is no doubt that Professor Ziegler and his associates were the original and first inventors of these two inventions. The application" polymerization of olefins, August 3, 1954 with Ziegler catalysts "extended the application" (of November 17, 1953) "to higher olefins after having previously applied it to ethylene."

A pre-condition for the continuation was that "the inventors be the same in parent and continuation-in-part applications. Prior to filing the application" polymerization of higher olefins, August 3, 1954) "in Germany, Professor Ziegler and his associates did not know of the applications of Professor Natta in Italy." The oath in the patent application in the USA, filed June 8, 1955 was correct, inasmuch as the inventors "did not know" of the invention of G. Natta before August 3, 1954.

33) In April 1958 a hearing in the US Patent Office was conducted between the examiner, Mark Liebmann and De Varda (Montecatini) in the presence of Toulmin and Peake (from Toulmin's office). The examiner asked De Varda whether or not to his knowledge anybody had produced crystalline polypropylene before Natta. De Varda remembered having answered neither yes nor no, but had pointed out the fact that at the time of the Natta invention nothing had been published to this

fact. Later Phillips claimed that De Varda had given his answer despite knowing better. He must have known that Phillips had produced crystalline polypropylene before Natta. It was later decided by the court in Delaware [53] that De Varda's opinion constituted an act of fraud against the Patent Office and Toulmin and Peake were also implicated in this act. Montedison (Montecatini) were excluded from the legal procedure thereafter and lost any claim to the product.

By 1957 the conflict was initially contained as the Toulmin firm was relieved of its responsibilities in handling the Ziegler patent rights, and representation was transferred to the Dinklage office. As a consequence, a series of consolidation applications³⁴⁾ permitted under US patent law, (internal references A-H) which comprised all Ziegler applications filed up to that point, including the polyolefin application (IV) of August 1954, were subsequently prepared and filed in the US Patent Office. [54]

The various catalyst groups disclosed in each of the original applications were incorporated as catalyst product claims *per se*, on the one hand, (CIP applications A, C, E, G) as well as process claims for the polymerization of alpha-olefins in general, on the other (CIP applications B, D, F, H) and prosecuted as such. Thus evolved a package of eight applications, the subject matter of which represented the state of the art of all possible catalyst combinations developed during the priority period 1953 through December 1954. Thus, to the best of anyone's knowledge, the range of subject matter disclosed in the foreign applications, including the applications filed in the United States, defined a scope of protection which, to all intents and purposes, should have guaranteed comprehensive patent coverage for the invention.

The third Montecatini application, which sought to patent the new polypropylenes *per se*, was rejected by the US Patent Office because of the existence of an even an older patent by Field and co-workers [55] which referred to the same subject (Chapter 1, reference [15]), particularly Example 7. The polypropylene product, the production of which was described in the patent filed by Field et al., was characterized by a CH_2/CH_3 ratio of 8, thus clearly differentiating its structure from that of the newly discovered polypropylenes.³⁵⁾

By the end of 1957, during a verbal interview, the US Examiner acceded to the novel polypropylenes to be characterized as "isotactic" and/or "atactic". After all the Examiner's objections seemed to have been overcome, in 1958 he declared an inter-

34) The so-called CIP – (Continuation in Part) – applications, which, as the name implies, were combinations of the subjects of continuous applications for the same invention combined into one application. The priority of the new CIP application is deducted from the single parent applications, including the first of the combined applications. In this way the application IV (production of polyolefins (of Aug. 03, 1954) was combined with the first parent application (I, polyethylene) of Nov. 17, 1953. The priority date of the combination application (CIP) was anti dated (see Littell's opinion, above). The same practice was employed in combining the parent application (II), (priority Jan. 17, 1954) with application IV of Aug. 03, 1954 etc.

A: Catalysts, combination of aluminium tri-alkyl/transition metal compounds.

B: Polyolefins using catalysts A.

C: Catalysts composed of a combination of aluminum compounds R_2AlX (X = halogene etc.)/transition metal compounds.

D: Polyolefins using catalysts C.

35) The Natta affidavit of 1956 stated that the products according to Field et al. are completely soluble in boiling heptane, vinyl/vinylidene (IR 4 : 1 to 8 : 1), but that the products according to Natta et al. are substantially insoluble in boiling heptane. They have a well-defined melting point of 165 °C, and no vinyl bands (IR 11.7 μm). Further, there is no absorption at 13.5 and 15 micron in the linear and regular head-to-tail-structure. The ratio of methyl/methylene is 1 : 1, the intrinsic viscosity at 135 °C is above 1, and therefore the molecular weight is above 20,000. H. Martin, Declaration 1988, see Chapter 1, reference [16].

ference (Chapter 1, reference [1]) between Standard Oil of Indiana, Montedison (formerly Montecatini), Phillips Petroleum, Hercules Powder and Du Pont. The dispute dragged on for the next 25 years. It was Phillips Petroleum who eventually emerged victorious from this struggle [53] (see Chapter 1, p. 2).

But for now, we shall return to the Ziegler polypropylene application. With the search for a line of demarcation between Montecatini and Ziegler, the dispute over the newly filed consolidation (CIP) applications (see p. 115) continued. Toulmin and Montecatini tried to exert increased pressure on von Kreisler in order to prevent the application from being filed. Ziegler's US licensees assumed – and rightfully so – that they were entitled to a license for polypropylene as well, and once the patent had been issued, would not have to pay additional royalties.

From this viewpoint alone, Ziegler could make no concessions whatsoever to Montecatini. Montecatini appealed to its Pool partner not to put a strain on their relationship.

Montecatini's legal department insinuated that Ziegler considered the interests of his licensees to be more important than the joint Montecatini/Ziegler interests and, thus, ran the risk of a potential confrontation with Montecatini. They demanded that Ziegler withdraw at least his product claims for the α -olefin polymers. Evidently, Montecatini was aware that its method claims would not be allowed. In its arguments, it accused Ziegler of incorrect statements, particularly Ziegler's alleged misconduct pertaining to his declaration, under oath, that, on the date that he filed his first German application, he was unaware that he was not the first inventor. During phone conversations and at a final meeting between the Chairman of the Board, Giustiniani, De Varda, Ziegler and von Kreisler in Zurich in mid-1957, Ziegler remained firm under pressure. His refusal to give up the product claims and insistence on correcting the inventor declaration prompted Giustiniani to threaten a claim for damages. For the time being, however, it was settled that Montecatini would revoke von Kreisler's power of attorney and Ziegler would revoke the power of attorney held by the US counsel, Toulmin.

As mentioned earlier, Ziegler's new consolidation applications (A–H) were filed in the US Patent Office [54] in 1957/58, but prosecution of all the then-active applications was undertaken simultaneously.

The eight consolidation applications eventually matured into patents, although at different years, with some of them being issued only after a long drawn-out prosecution. The last patent issued in 1978 [54], which – as mentioned previously – led to a large number of licensees reluctantly paying royalties to Studiengesellschaft Kohle for years to come.

The different issue dates were a result of the opponents' efforts to prevent patent issuance, and also, to a certain extent due to the actions of the Patent Office. The reason was that the patent could be expected only after the Examiner had made sure not only that earlier publication dates of cited references, that is, dates prior to Ziegler's own priority dates, had been shown to be irrelevant, (this had been established meanwhile), but also that, at the time the prosecution was concluded, there remained no overlapping subject matter with later filed third-party applications. Such expectations hardly seemed realistic however, given the provocative nature and

scope of the invention. Based on fragmentary pieces of information, teams of research scientists, working in the same field, were piecing together the composition and action of the Ziegler catalysts and filed their own patent applications in the Patent Offices of their individual countries. The number of these imitation applications increased logarithmically after the subject matter of Ziegler and his co-workers' inventions had become known among the experts in 1955. An overlap of new findings with existing results could not be avoided and, in the United States, led to multiple interference proceedings, initiated through official channels, which had to be resolved as prescribed by law. Within the parameters of an historical analysis, a discussion of the course and outcome of 22 interferences [56], conducted between 1957 and 1983, is beyond the scope of this book. However, in order to provide some insight into the continuing exploitation of the invention, particularly in the United States, a few of these proceedings will be addressed below.

3.5

Contention Proceedings

3.5.1

Montecatini

The anticipated confrontation between Montecatini and Ziegler over the priority for the production of, *inter alia*, polypropylene was initiated in the US Patent Office in January of 1960. The Office also included Du Pont and Union Carbide as parties to these proceedings, whereby the latter dropped out very quickly because Union Carbide's relevant US patent application had been filed much too late to seriously threaten the priority dates of the other participants.

It was not until 9 years later that the US Patent Office, represented by the Board of Patent Interferences [57], handed down its decision, which turned out to be of the utmost significance for all parties concerned.

The decision stated that Du Pont was entitled to a priority date of August 19, 1954 [58] (see p. 8) – too late with respect to Ziegler's priority date of August 3, 1954.

In retrospect, it has to be said that over this 9-year period, the amount of work which went into following the detailed procedures necessary to present all of the parties' arguments to the Patent Office and enable it to arrive at a decision, was impressive. More than 400 briefs were drafted. Montecatini's patent department and attorneys, and especially their US counsel, fought tooth-and-nail to question Ziegler's correction of the inventor declaration and the lawfulness of this correction in particular. By conducting time-consuming depositions of all of the inventors in New York, the opponents tried to draw out inconsistencies so as to prevent Ziegler, Martin, Holzkamp and Breil from being named as the four inventors.

After amendment of the inventor declaration in the United States, the declarations connected to the priority applications in Germany were also corrected. The US Patent Office had accepted Ziegler's proposed revision without objection, and the Board of Interferences had affirmed this assessment as proper.

Natta and co-workers, on the other hand, had ignored the due diligence required in naming the inventors, with the end result that Montecatini/Natta were denied the benefit of their claimed June 8, 1954 and July 27, 1954 priority dates – that is, dates which were later than the earliest and prior to the latest of the priorities claimed by Ziegler and co-workers. In the first priority application, and the Italian patent [59] maturing therefrom, G. Natta had been named as the sole inventor. This first application was filed in the United States in the form of a consolidation case together with the second Italian patent application [60], naming G. Natta, G. Mazzanti and P. Pino as joint inventors. However, this was not allowed under US law without an appropriate amendment. The circumstances of the case led, on the one hand, to the loss of the June 8, 1954 priority date and, on the other hand, caused the second application of July 27, 1954 to be characterized as an “improvement application”, which did not present a conflict with the application by Ziegler and co-workers.

Priority was unequivocally awarded to Ziegler and co-workers, which caused the Board of Interferences to regard as irrelevant a motion by Ziegler asserting that the priority dates claimed by Montecatini for Natta had been made possible only through unlawful derivation.

Studiengesellschaft Kohle mbH, as Ziegler’s successor in interest, obtained a US patent [61] in 1974, the substance of which reflected the outcome of the interference proceeding.

Montecatini could not allow this decision to stand, particularly in view of the fact that, at that time, many interested companies were either seeking a license from Montecatini or had themselves, attempted to prevent additional patents from being issued to Montecatini. Montecatini had to insure that any decision on the issue of priority was held in abeyance at the very least.

The first logical step in that direction was for Montecatini to file an appeal against the decision of the Patent Office’s Board of Interferences in a regular court of law. In the course of the ensuing legal action [62] against Ziegler, which continued for the next 14 years, the parties exchanged briefs reflecting considerably harsher adversarial positions. This included a counterclaim filed by Ziegler, demanding damages based on Montecatini/Natta’s alleged “unlawful derivation”.

The second result of the Interference decision was that Montecatini severed its contractual relationship with Ziegler [63].

It was not until 1983 that Montecatini and Studiengesellschaft Kohle would reach a “settlement” [64], wherein Montecatini undertook to withdraw all allegations, including its priority claims, and pay a seven-figure dollar sum in damages. This occurred at a point in time when Judge Wright had determined that Montecatini was not entitled to a product claim for the new polypropylene³⁶). This decision was bound to have an effect on the pending court proceedings (see p. 157).

36) See Chapter 1, Section 1.5.1 and [183, 184].

3.5.2

Du Pont

The parties initially confronted each other in November 1957, when the US Patent Office declared an interference [65] between one of Ziegler's first US applications [66] (I, SN 469,059) claiming a German priority date of November 17, 1953, and a Du Pont application (SN 450,244, Chapter 1, reference [28]), filed on August 16, 1954. Even though it should have been obvious from the priority dates which party was likely to prevail, Du Pont continued to fight until the end of 1962 [67] before dropping its claim – 5 years in all. However, Du Pont did not drop its claim until there had been a second interference proceeding [68] in 1960, during which they renewed their attempts to secure their selected claim directed to the aluminum trimethyl subsequently employed by Hyson of Du Pont. In this endeavor Du Pont was partially successful, but the specific 1 : 1 aluminum trimethyl/titanium tetrachloride [69] catalyst mixture³⁷⁾ that they had patented was not commercially significant.

Many years later (in 1982), Judge C.M. Wright [70] held that:

“Despite a major effort, involving many experienced scientists with high priority access to the resources of Du Pont’s experimental station, the Du Pont group explored a number of alternatives before investigating alkyl aluminums.”

The confirmation by a court of law of the described situation, that aluminum ethyls had been employed too late, characterized the historical course of events.

3.5.3

Compromise and Concessions

Ziegler was only able to sustain the financial expenditure required for the growing number of litigations because of the revenue from his monopoly position in granting early licenses for the production of polyethylene. Du Pont had realized this and now, via direct negotiations, attempted to obtain concessions from Ziegler, with a two-fold objective. One of the Du Pont applications [71], filed in 1947, claimed a process for the production of linear polyethylene, using extreme and impractical parameters, such as 5000–20,000 bar ethylene pressure and temperatures of 45–200 °C. The process involved the use of well-known radical catalysts, peroxides, etc. In late 1957, the application was issued to a patent, which now contained a product claim directed to linear polyethylene, whose physical characteristics, such as melting point, tensile strength, molecular weight and density, appeared to be similar to those exhibited by the new Ziegler polyethylene (see p. 110). Du Pont used its claim from this patent as leverage into the negotiations with Ziegler and anticipated that Ziegler licensees, particularly those in the United States, could be compelled to shift part of their royalty obligations to Du Pont, thus compensating Du Pont, partially or wholly, for the price it had to pay Ziegler for a license.

37) Special improvement invention. Ziegler and co-workers did recommend the use of an

excess of organo aluminum compounds in relation to titanium tetrachloride.

D.H. Hounshell [72], of Du Pont, later described the polyethylene produced in 1947 as “an entirely new material.” Neither Hounshell nor Larchar and Pease, the inventors of the “new” linear polyethylene, made any mention of the fact that many years earlier (in 1928/1933), a product with the same characteristics had been synthesized as “polymethylene” by H. Meerwein and W. Burneleit, and W. Werle [73], although, by different means of course (decomposition of diazo methane). In a subsequent publication, dated 1958, H. Hoberg and K. Ziegler [74] conducted a comparison study of the products in question – linear polyethylene according to Larchar and Pease, polymethylene, and Ziegler polyethylene – and came to the conclusion that Du Pont’s chemists were not the first to produce linear polyethylene, since polymethylene, at a sufficiently high molecular weight, is practically chemically identical to linear polyethylene.

In June of 1958, as an inherent consequence of the preceding negotiations with Ziegler, Du Pont filed a suit against Phillips Petroleum Co. for infringement of the Larchar–Pease patent [71]. Phillips Petroleum, – as previously mentioned (p. 106) – had developed its own process for the production of polyethylene (Marlex 50) and was not a Ziegler licensee. A.R. Plumley [75], attached to the Du Pont Patent Department, had sent Ziegler a license offer for his licensees in an attempt to persuade him that an agreement with Du Pont would be desirable. Veiled behind Du Pont’s offer, if not accepted, lurked the threat of legal action against Ziegler’s licensees and, thus, endangerment of Ziegler’s entire royalty income. On July 2, 1958 [76], the deadline for accepting or declining the offer, Du Pont increased the pressure and expected Ziegler’s attorney, Dr. von Kreisler, to come to the United States equipped with suitable powers of attorney for finalizing an agreement. Ziegler knew that refusing the Du Pont offer, on the one hand, or bringing an action for declaratory judgment of invalidity of the Larchar and Pease patent, on the other, would impose a huge financial burden on him. But Du Pont, too, was aware of this and pushed for a speedy resolution.

Du Pont [77] expected to receive royalties of 1.5% for the first 30 million pounds per annum and 1% for any additional production directly from Ziegler licensees and left it up to Ziegler to take any appropriate steps based on his publication [74]. W.H. Salzenberg (General Manager of Du Pont’s Polychemical Department) and Plumley (Head of the Patent Department) announced their visit for August 5 and 6, 1958 [78].

In spite of the publication [74] – Du Pont had received a draft of the manuscript with a cover letter dated June 26, 1958 [79] – Ziegler and Du Pont reached an understanding a few days later [80], to the effect that Ziegler’s licensees were to pay Du Pont up to one-half of the running royalties due to Ziegler for any products coming under the Du Pont patent claim. The letter agreement [79] reflected the prior history insofar as Du Pont compromised by reducing its royalty demand to 0.75%, a concession which accommodated Ziegler and avoided costly litigation. Ziegler’s licensees accepted this decision.

Prior to the settlement with Ziegler, after having filed its Complaint against Phillips Petroleum, Du Pont orally conveyed to the defendant that it had reached an understanding with Ziegler, although that was not the case at that point in time [81], “poker”.

Under the agreement, Ziegler's American licensees [82] collectively paid approximately 2 million dollars to Du Pont between 1958 and 1974 (the expiration date of the Du Pont Larchar–Pease patent).

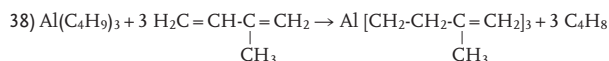
3.5.4

Du Pont Sought Further Advantages

Du Pont exerted pressure on Ziegler by pressing him to concede that not all aluminum trialkyls could possibly be covered by the Ziegler patents, particularly not those whose alkyl chains contained double bonds, since those aluminum compounds had not been exemplified by Ziegler and showed markedly different behavior to the pure aluminum alkyls. Ziegler relied on his factual experience that such aluminum compounds, due to their higher production costs alone, could not be of true commercial significance. He could, however, hardly avoid feeling wary at Du Pont's stubborn attempts to define not only aluminum compounds of the type $\text{Al}(\text{CH}=\text{CH}_2)_3$ and $\text{Al}(\text{CH}_2\text{-CH}=\text{CH}_2)_3$, but also the next higher homolog $\text{Al}(\text{CH}_2\text{-CH}_2\text{-CH}=\text{CH}_2)_3$, as falling outside of the license agreement. No doubt, there had to be a reason why Du Pont pursued this objective during the negotiations.

What appeared to be more important to Ziegler was the promise that as compensation, Du Pont was willing to abandon the interference if, on disclosure of the priority evidence, it was established that the more favorable dates came from Ziegler. By the end of 1958 [83] he had been persuaded to sign an agreement. The results were disappointing. Not only was Du Pont unwilling to acknowledge Ziegler's earlier priority dates, but they also made it clear that in their commercial facility, they intended to use a polymerization catalyst produced from titanium tetrachloride and an aluminum compound which had been synthesized from aluminum triisobutyl and isoprene. In Du Pont's estimation, this compound should fall outside of the scope of the licensed Ziegler claim. Ziegler's basic aim to secure continuous royalty income for himself by terminating the interferences had not been realized. Du Pont's attempt to sidestep any royalty obligations by means of the new agreement created a new problem.

Two chemists associated with a Du Pont research team, J.M. Bruce and I.M. Robinson, had been named as inventors on two patents [84], [85], the applications for which had been filed in the US Patent Office in March of 1960. One of these applications was directed to the production of "Aluminum Diene Polymers", while the other claimed the use of these new aluminum compounds, in combination with titanium tetrachloride, as polymerization catalysts. It was obvious that the definition of the Ziegler aluminum trialkyls originated from the background of the previously mentioned 1958 agreement with Du Pont, except that the two chemists had misinterpreted the structure of the reaction product obtained from aluminum triisobutyl and isoprene. They had presumed that, with isoprene, according to the "displacement method" (Ziegler and co-workers) [86], after the release of iso-butene, a true "aluminum triisoprenyl" would be formed.³⁸⁾



In a commentary [87] written in 1967, Ziegler himself stated:

"It would be correct to designate (the new compound) as aluminum triisopentenyl and would, in fact, correspond to the $\text{Al}(\text{CH}_2\text{-CH}=\text{CH}_2)_3$ -type compound encompassed by the October 8, 1958 agreement. This erroneous opinion may, in part, have been based on the composition of the compound which includes approx. 14% Al. Pure $\text{Al}(\text{C}_5\text{H}_9)_3$ should contain 12.6% Al. The slight excess of 1.4% Al was easily accounted for by the admixture of a small amount of two-valent Al-compound. A (high molecular weight!) material of the type $[\text{al}(\text{C}_5\text{H}_{10})\text{al-}]_n$ should contain 20% Al. In actuality, the substance arising from the reaction of aluminum triisobutyl and isoprene has nothing whatsoever to do with aluminum triisopentenyl, a fact which had meanwhile been recognized by Du Pont. It may, however, continue to be referred to as 'so-called aluminum-isoprenyl' or 'aluminumisoprenyl'."

In response to Du Pont's evaluation [88], a 22-page, illustrated research report [86] was made available to them, which stated, in conclusion:

"From previous discussions with Du Pont personnel, I am aware that Du Pont uses a special form of polymerization, whereby the polyethylene is temporarily dissolved in a suitable medium, and which, in that respect, differs from the original Ziegler-type process (working in suspension). Even discussing this would, in my opinion, be absurd. In terms of technology, it was as a work in progress that each of Ziegler's licensees obtained access to the polyethylene invention. This was being compensated for by imposing a very low royalty rate of 2% (for large scale production). Each successful licensee (Hoechst, Hercules, Dow, Monsanto, Mitsui – to name but a few) has then developed its own modification of the process, - in each case thought to be the 'best' available – without using this as a basis for inferring a special, preferential position relative to Ziegler. There is no reason to believe that Du Pont should deserve to be treated any differently."

It was only later that Du Pont realized that the conversion product formed from aluminumisobutyl and isoprene represented a polymeric aluminum compound of complex structure. This issue was taken up again by H. Martin [89] in 1977 in response to an alternative writ issued during arbitration proceedings between Farbwerke Hoechst and Studiengesellschaft Kohle, and, based on product characteristics and analytical data, suggestions were offered in an effort to define the structure. But by 1966, it had become clear that the structure of this product would not fall under the exceptions of the hotly negotiated formula of the 1958 agreement [83] between Du Pont and Ziegler. In May 1967 - that is, 10 years after the controversy with Du Pont had started – a "settlement agreement" [90] was finally negotiated between Du

Pont and Ziegler, pursuant to which Du Pont paid a settlement amount of 2 million dollars for the polyethylene license in the United States. Moreover, various patents which had meanwhile been issued to Du Pont, *inter alia*, for the production of isoprenylaluminum and its utilization in the polymerization of ethylene, were now assigned to Ziegler on a worldwide basis.³⁹⁾

Ziegler had thus offset the costs of the concessions he had made in settling with Du Pont concerning the “Larchar–Pease polyethylene product patent”. A sizeable profit was, however, realized due to the fact that Du Pont had neglected to include Canada, even though the bulk of their polyethylene production had in the meantime been shifted to that country. Ziegler collected the same amount again as a settlement for Canada. The charming Waldhotel in Sils Maria, Canton Graubünden (Switzerland), where Professor Ziegler and his wife spent the summer, served as a backdrop to this delightful transaction.

The laborious and tiresome detailed work of the past had paid off. On the author’s birthday, in 1967, in the elegantly furnished kitchen at the home of the Head of Du Pont’s Patent Department, Mr. R.C. Kline, in Wilmington, Delaware, USA, glasses of a fine, aged bourbon were raised in a toast to “Nobel Charlie”.

Within a very short period of time, the transfer of the Du Pont patents on isoprenyl aluminum would prove to be a fortuitous arrangement for the Max-Planck-Institute for Coal Research. Farbwerke Hoechst of Frankfurt had decided to use this product for the manufacture of its ethylene polymerization catalysts, being unaware, however, that it was the Institute which, in Germany as well, was now the new owner of these patents (for further details see p. 156/157).

3.6

The History of Patent Issuance from a Year-2000 Vantage Point

Several of the incidents and developments described in the previous pages should be reviewed here because of their extraordinary importance in terms of the successful product exploitation which took place between 1955 and 1995. The scientific community had acknowledged in numerous publications, the significance of the inventions which had originated from the Max-Planck-Institute for Coal Research in Mülheim, particularly the importance of the Ziegler catalysts and their applications.

This did not, however, restrain the commercial sector from repeatedly attempting over the next 40 years, to either prevent or obstruct exploitation of the invention and/or to gain special advantages in licensing agreements. Many further incidents during this period triggered subsequent court battles with for example, infringers, mostly involving issued patents. It was difficult to estimate the expenditure that all parties were willing to invest over the course of the next four decades up to the expiration date of the last patent in 1995. The Institute alone incurred expenses of

39) A single right of utility stayed with Du Pont in the USA.

more than 30 million Deutsch Marks in the defense of its patent rights and to prosecute infringers.

The initial patent applications in Germany had been drafted in haste, without further careful review. With hindsight, the reason for this can only partly be understood. The priority dates of the patent applications filed by research teams around the world working in the same field fell very close together – a fact which was not known at the time. It was, after all, “in the air” at the time for similar findings to be obtained at different venues. On the other hand, it should be noted that any deficiencies in drafting the first application left it vulnerable to later repeated “attacks” by opposing attorneys. With each instance there was an increased risk of losing everything.

As it transpired, the fact that the discoveries of other research groups had for the most part, little commercial appeal, certainly worked to the advantage of the Mülheim Institute. All individuals involved, including the patent attorneys representing the Institute abroad, initially learned how to deal with this extraordinary situation. There were times when moods tended to get depressed, and when it also became clear that it would not be long before the series of accomplishments must reach its limits. Dealing with the events with an eye towards the future required both optimism and perseverance during the entire time that the patents were valid. It seemed impossible to anticipate and prepare for each and every upcoming situation. The certain knowledge of being armed with a good product eventually created a determination in all concerned to look for ways and means of conquering obstacles for many years to come.

Given the potential of the invention, it was, of course, important and appropriate that most of the patents were being maintained for the maximum term permitted by law. This also applied to the so-called blocking patents which were not directly relevant to the commercial production of polyolefins. The anything but modest costs of the worldwide maintenance of the relevant patent rights could be borne only because of the timely negotiation of license agreements and the ensuing commercial operations. These costs were, however, minor in comparison to the legal costs subsequently incurred.

Prosecution of the various applications filed both at home and abroad up to the time of issuance of the first patent did not extend over an unusually long period of time, barring a few exceptions, even though the expectation had been that patents would be granted more quickly. After all, the invention had been awarded the Nobel Prize. Oppositions and/or interference proceedings were legal remedies used to obtain concessions and/or more favorable license terms from Ziegler. There was a sharp increase in the number of interested parties seeking a license, even though it was a well-known fact that BASF, Montecatini and Du Pont were making tremendous attempts to kill the patents and/or obtain preferential terms. No more than 10 years had passed since the invention when it became clear to any interested parties that the Ziegler catalysts constituted a fundamental discovery the efficiency of the catalyst could be controlled in terms of rate and selectivity.

However, despite innumerable proposals for improvements, the underlying principle did not become obsolete nor was it discredited. Neither of the two components

of the Ziegler catalyst – the titanium compound or transition metal compound and the organoaluminum compound or organometal compound – was ever replaced. This statement is corroborated by the fact that actually up to the present day, i.e. for almost 50 years, there has not been a single commercially-operated polymerization plant producing polypropylene that used a catalyst mixture in which these two components were absent.

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4

Innovation, Market Development, Production

4.1

The Market Situation

By the mid-1950s, the scope and significance of the invention of the Ziegler catalysts and their potential utility had been well established, and there was an initial peak in the number of license agreements that had been signed. The Max-Planck-Institute in Mülheim had collected sizeable down-payments for licenses it had granted. The Institute had expected that from then on, commercial interests would focus on developing a usable product. The commercial and technological value of the newly created products was quite certain at the moment of conception of the invention.

The impulse to convert the scientific discoveries into industrially viable products, and the readiness for innovation, following the availability of Ziegler's experimental laboratory findings and test results from early pilot plants, was more hesitant than it had been in connection with competitive products which were already on the market. It was the high-pressure polyethylene ICI process¹⁾ which was commercially in use at the time and which dominated the market. High pressure polyethylene enjoyed a breadth of utility that initially appeared difficult to even partly reverse. The second competitive product, polyethylene synthesized according to the method developed by US-based Phillips Petroleum, was made available through licensing together with the required technical know-how for its production, while preliminary information regarding the properties of this product and any corresponding utility was included in the package. The costs of the catalyst used in the Phillips process were decidedly lower than those incurred in the production of Ziegler polyethylene. This had to be remedied when licensing the patent rights covering the Ziegler polyethylene. Any prospective licensee was obliged to initially gather extensive experience of its own in order to start a commercial operation and conduct additional research for the production, development and marketing of a product line.

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1935; J. C. Swallow was honored with the Swinborne award of the "Plastics Institute".

4.2

The Rush for Licenses 1953–1972, Lucrative Second Half 1970–1990

More than anything, it was the aforementioned competitive situation surrounding polyethylene that was responsible for the moderate license terms which Ziegler imposed on his licensees. On the other hand, the early licensees were quite anxious to be given an opportunity to break the monopoly of the high-pressure polyethylene market and make additional investments towards the development of a high-quality, new product. One major advantage of the Ziegler catalysts was the fact that they were also useful for the production of a wide range of other polyolefins. Thus, there were a considerable number of prospective licensees who intended to run parallel operations for producing polyethylene, polypropylene and polybutene, co-polymers of ethylene and propylene, terpolymers of ethylene, propylene and a diene, as well as the production of polydienes. However, there was no way of predicting which of these additional polymer products would gain acceptance in the marketplace. On the other hand, it was also true that, at the time, none of the other products among the spectrum of materials being produced – except polyethylene – was subject to competition. Thermoplastic polypropylene could then, and can now, be commercially produced only with the use of Ziegler catalysts. There has been no change in this situation for the past 40 years, a remarkable stroke of good fortune for both the producers and the licensors. During this span of time, polypropylene production did eventually outdistance the production of polyethylene.

Table 4.1 ranks the licensees chronologically in the order in which they signed an agreement. It also shows the expansion of the production program and the year in which the first running royalty was paid, in other words, the year of initial sales in the marketplace.

During this period, between 80 and 90 licenses [1] were granted worldwide, spread out over approximately 20 different countries.

Table 4.1 List of licensees in chronological order

Licensor	Product*	Agreement of	Start of first payment out of production/export or paid-up
Petrochemicals Ltd. Shell Chem. UK, GB	PE PP CP	1952/59	1957/59
Montecatini, IT	PE	1953	1957
Farbwerke Hoechst, DE	PE PP CP	1954/59/65	1957/60/66
Ruhrchemie, DE	PE PP	1954	1957
Hercules Powder Co., US	PE PP	1954/64	1958/64

Table 4.1 continued

Licensee	Product*			Agreement of	Start of first pay- ment out of produc- tion/export or paid- up
	PE = Polyethylene, PP = Polypropylene, PBu = Polybutene, CP = Co-respectively. Terpolymers: PB = Polybutadiene, PiP = Polyisoprene				
Ameripol (Goodrich, Gulf Oil) , US	PE		PB PiP	1954/58	1961/67
Koppers, US	PE			1954	1963
Dow Chemicals, US	PE	PP		1954/61	1961
Union Carbide, US	PE	PP	CP	1954/56	1957
DEA, Hibernia (Scholven) Hüls, Rheinpreußen, Mannesmann, Dynamit-Nobel, GBAG (BWV), DE	PE	PP	CP PB	1955/65/60	1957/64
Monsanto, US	PE			1955	1963
Du Pont, US	PE	PP	CP	1955/56	1966/64
Mitsui Chemicals Co. (Mitsui Toatsu Chem.), JP	PE	PP	CP	1955/60/92	1958/62/65
ESSO (EXXON), US	PE	PP	CP	1955	1960
Pechiney (Naphthachimie) FR	PE	PP	CP	1955/58	1958/62
Houilleres (Soc. Normande de Matieres Plastiques), FR	PE	PP	CP	1955/58	1960/62
Staatsmijnen, NL	PE	PP	CP	1955/68/73	1963/76
Koppers, AR	PE			1956	
Hoechst, AT	PE			1956	
Dow Uniquinesa S. A., ES	PE			1956	1972
BASF, DE		PP		1958	1962
Danubia Petrochem. (Linz AG) , AT		PP	CP	1958/68	1961/76/70
ICI, GB		PP	CP	1959	1959
Svenska Esso, SE		PP		1960	1961
Stereo-Kautschuk-Werke (Hüls), FR			PB PiP	1960	1964
Sumitomo Chem., JP		PP	CP	1960/91	1963/64/94
Mitsubishi Petrochem. Co., JP	PE	PP	CP	1960/93	1962/94
Rotterdamse Polyolefinen Maatschappij N.V., NL		PP	CP	1961	1961
Shell Int. Research. Maatsch., NL			PB	1961	
Polymer Corp. (GG), CA			PB PiP	1961	1963
Goodrich-Gulf, Montecatini, Shell, Ziegler, FR			PB	1961	1964
Japanese Geon (GG), JP			PB PiP	1961	1966

Continue ►

Table 4.1 continued

Licensee	Product*	Agreement of	Start of first pay- ment out of produc- tion/export or paid- up
Société Nationale des Petroles d'Aquitaine, FR	PE	1963	
Hoechst, AU u. NZ	PE	1963	1967
Shell, AU u. NZ	PP	1963	1966
Paular, ES	PP CP	1963	1968
Toyo Rayon, JP	CP	1964	
Novamont, US	PP	1964/67	1964
Goodyear Tire a. Rubber, Goodrich Gulf (GG), US		PiP 1964	1965
Esso, AR	PP	1964/69	1969
Shell Oil, US	PP CP PBu	1964/72/ 79/87	1964/74
Austr. Synthetic Rubber Co (GG), AU u. NZ		PB 1965	1967
Hoechst, IN	PE	1965	1968
Uniroyal (US Rubber), US		CP 1965/67	1967
Ube Industrie Inc. (GG), JP		PB PiP 1966/94	1971/94
Safipol (Hoechst), ZA	PE	1966/71	1972
Kuck Tae Ind. Co. Ltd., JP	PP CP	1967	
National Petrochem. (Solvay), US	PE	1967	1971
Copolymer Rubber Chem., US		CP PB 1967/63	1969
Soltex (Solvay), US	PE PP	1967/78	1975/78
Int. Synthetic Rubber (Shell), GB		CP 1969	
Soltex (Solvay), BR	PE	1969	
Hüls, ScholvenChemie, DE	PE	1969	1972
Solvay (Algerien), FR	PE	1969	1970
Chubu Chem., JP	PP	1970	
US Steel (Aristec), US	PE PP	1970	1992
Dainippon Inc. and Chem. Inc., JP		PBu 1970	
SOI (Amoco), BE	PP	1970	1970/72
Diamond Shamrock (Arco Pol.), US	PP CP	1970	1970/84
Kurashiki Rayon Co. Ltd. (GG), JP		PiP 1970	1972
Polysar Inc. (BF Goodrich), CA		CP 1970/81	1982
Mitsui Petrochem. Industries, JP	PE PP CP	1971/69	1972/69

Table 4.1 continued

Licensee	Product*		Agreement of	Start of first pay- ment out of produc- tion/export or paid- up
	PE = Polyethylene, PP = Polypropylene, PBu = Polybutene, CP = Co-respectively. Terpolymers: PB = Polybutadiene, PiP = Polyisoprene			
Compagnie du Polyisoprene Synthetic (GG), FR		PiP	1971	1973]
Chisso Chem., JP	PP		1972	1974
Phillips Petroleum, US	PP	CP	1974/86	1974/86
SOI (Amoco, Avisun), US	PE	PP	1974/73	1976/73
Soltex Polymer Corp. (Solvay), CA	PE	PP	1975/81	1975/81
Showa Denko, JP	PP	CP	1975/94	1977
Exxon, CA	PP		1975	1978
Hercules, CA	PP		1976	1976
Allied (Solvay), US	PE	PP	1976	1978
ICI Holland BV, NL	PP	CP	1976	1979
Celanese (Hoechst), US	PE		1979	1980/83
Shell Ltd., CA	PP		1979	1981
Montefina S. P., BE	PP		1980	1982
Northern Petrochem. (Quantum), US	PP		1986	1986
Dart Industrie, US	PP		1987/88	
El Paso/Rexene, US	PP		1986	1987
Huntsman, US	PP		1987	1988
Idemitsu, JP	PP		1973/94	1994
Mitsubishi Kasei, JP	PP		1994	1994
Tonen, JP	PP		1994	1994
Tokuyama Soda, JP	PP		1994	1994
Tosoh, JP	PP		1994	
Asahi Chem., JP	PP	CP	1965/94	1995

AR = Argentina, AU/NZ = Australia/New Zealand, BR = Brazil,
CA = Canada, BE = Belgium, DE = Germany, FR = France,
GB = United Kingdom, IN = India, IT = Italy, JP = Japan,
NL = Netherlands, AT = Austria, SE = Sweden, ES = Spain,
ZA = South Africa, US = United States of America

4.3

The Marketing of Ziegler Polyolefins

The race to put the first products on the market promised to create windfall profits for their producers. The Ziegler polyolefins possessed unique characteristics: compared to the high pressure polyethylene, they exhibited higher melting temperatures which made them suitable for processing at higher temperatures and pressures, thus making them tougher, with improved form retention, enhanced insulation properties, etc., all of which increased the range of their potential uses. Farbwerke Hoechst in Germany, Petrochemicals in Great Britain, Pechiney (Naphtha-chimie) in France, Hercules Powder in the United States, and Mitsui Chemicals in Japan were the pioneer producers who tested the market and had offered the first polyethylene products for sale as early as 1955–1958. This included the re-selling to future purchasers of the company's own, individually developed know-how. Advance payments previously rendered to Ziegler were being fully or partially offset in this manner.

In 1957, Farbwerke Hoechst went on-stream with a large-scale industrial plant²⁾ for polyethylene, with 10% of the products sold being powder and 90% granulate. In the United States, it was Hercules, who in 1958, opened the first production facility²⁾, with Mitsui following suit in Japan³⁾ [2] in same year. During the same period, Pechiney in France decided to expand its polyethylene license to a capacity of 24,000 tons annually. By 1968, Mitsui Petrochemicals had increased the capacity of its production facilities (four plants) to 72,000 tons annually. For polypropylene, they built a plant with an annual capacity of 24,000 tons.

The quality defects which plagued the early polyethylene products, i.e. excessive crystallinity and any inherent drawbacks, such as flow properties under pressure and brittleness, caused perilous losses and complaints. It was not until these shortcomings were remedied by introducing a small percentage of another olefin into the polymerization process – in other words, co-polymerization – that the market recovered. The race to improve quality was largely fuelled by the “discovery” of the low-pressure polyethylene hula-hoop [3] in the United States.

In terms of the history of the polymers, the growth in the production volume of polypropylene was by far the most rapid. In this case too, it became necessary to upgrade the properties of the polymer over the course of time – the early finished articles were unstable when exposed to heat, light and air, and the areas of use which had been established initially overlapped with those for other known polymers and

2) Hoechst/Frankfurt, capacity 10,000 tons p.a., trade name “Hostalen”; Hercules/Parlin New Jersey, capacity 13,000 tons p.a., trade name for polyethylene “Hi-fax” and for polypropylene “Pro-Fax”. The attempt to open the production plant in the presence of Ziegler a year in advance, was only symbolic in connection with the press release [4]. The ethylene supplier (Esso, New Jersey) was not able to meet the date.

3) Misui/Ivakuni, capacity 12 000 tons p.a., trade name “High-Zex”. The prices in Japan were about DM 1.40/kg for polyethylene and DM 1.50/kg for polypropylene (compared to DM 1.05/kg for high pressure polyethylene). In Europe Ziegler polyethylene was sold at up to DM 1.90/kg



Fig. 4.1 The inventor of Hi-Fax, Karl Ziegler, meeting members of the Hercules Powder team. From left to right, Elmer Hinner, President Forster, Karl Ziegler, Dave Bruce, and Earp Jennings

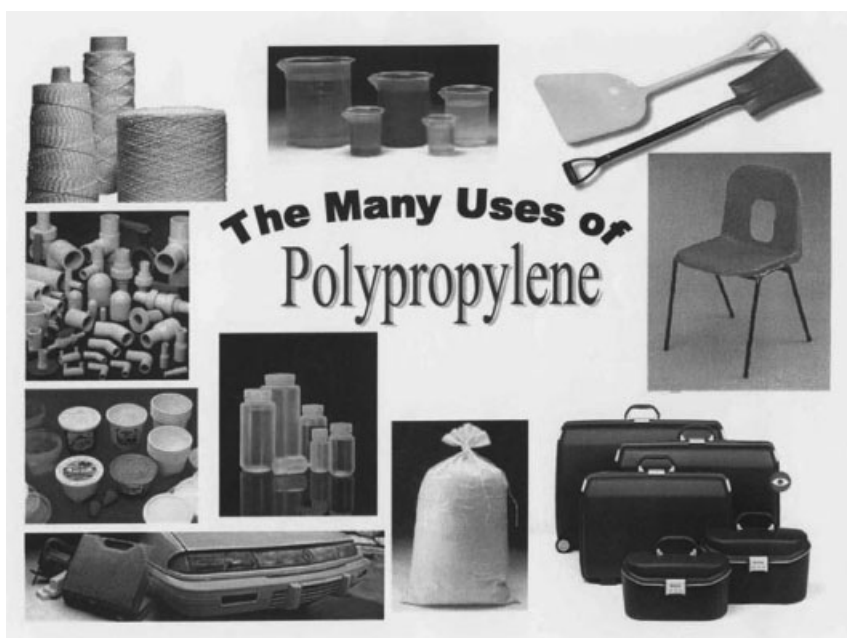


Fig. 4.2 Some examples of the many uses of polypropylene

therefore needed to be redefined to create a competitive edge for the new product. Thus, polypropylene proved to be useful in the production of containers of all shapes and sizes, household goods, toys, furniture (chairs), floor coverings (outdoor carpeting), fibers and filaments, packaging (plastic shopping bags) and auto parts.

There was hardly a licensee who did not manage to start his own operation, whereby the facilities designed for manufacturing one product – polyethylene – could be used equally well for producing another – polypropylene.

At the World Fair in Brussels in 1958, Farbwerke Hoechst set up a display unit, made of glass, to demonstrate the new polyethylene production process for promotional purposes. Despite assurances to the contrary, Hoechst failed to mention the origin of the invention on which this new process was based. Furthermore, no inventor was ever named either later on at the Achema Exhibition in Frankfurt, or in a published pamphlet [5] entitled “From Petroleum to Hostalen” – an omission which Ziegler found annoying.

By the first half of the 1960s, the quality of the products offered for sale had improved to such a degree that the spectrum of utility was now rapidly expanding.

With regard to the license agreements signed by the licensees shown in Table 4.1, which included and/or solely covered the production of polypropylene, Ziegler had generally based the early agreements, i.e. those concluded between 1954 and 1955, on polyolefins. In other words, they had been entered into at a time when the question of priority for polypropylene had not been clarified between Montecatini and Ziegler, therefore, firmly convinced of his independent ownership, Ziegler had granted licenses for the polymerization of olefins. The first two Pool-Agreements between Montecatini and Ziegler (see Chapter 2, references [140, 145]) defined the rights and obligations of the parties with respect to licensing in such a way that, irrespective of the outcome of the priority dispute, the right to grant licenses for polyolefins in Germany was reserved for Ziegler, while Montecatini enjoyed the same right in Italy. For all other countries, Montecatini alone retained the right to grant licenses, after Ziegler’s and Montecatini’s patent rights had been consolidated under the Pool Agreements.

It was Ziegler’s desire, due to the circumstances surrounding the patent applications, to be aligned, worldwide, with a strong industrial partner whose influence on the international market, particularly in terms of granting licenses, appeared to be well established, and with the choice of Montecatini, that desire was satisfied for the time being despite contractual pressures.

Montecatini had undertaken to subsequently grant a Montecatini Pool Polyolefin License [6] under “reasonable” terms to any Ziegler licensee whose contract already covered general polyolefins (see Chapter 2, Section 2.9). Ziegler dutifully advised his older licensees of this choice.

4.4

Montecatini's Pool Polyolefin Licenses

Without first investigating the patent aspect regarding G. Natta's priority, prospective licensees, after 1955, switched to Milan as their travel destination. It was not, however, until the early 1960s that Montecatini decided to grant Pool licenses for the production of polypropylene [7]. Over a long period of time Montecatini carefully searched for appropriate prospective licensees who were not only willing to pay a comparatively higher running royalty, but were also ready to acquire a license under additional Montecatini patents relating to further processing and to pay additional royalties for the use of these patent rights.

In the end, the substantial demands made by Montecatini could only be enforced on the basis of issued patents. Prospective licensees were not willing, as they had been years earlier in connection with Ziegler, to enter into license agreements without the existence of valid patents. The first Montecatini/Ziegler consolidation applications matured into patents in different countries at different times (see Chapter 3, footnote 21).

The delay caused considerable losses, and these losses may possibly have been more substantial than the higher running royalties which were subsequently imposed.

There were notably fewer Montecatini licenses granted up to 1969 than Ziegler licenses, the discrepancy was rooted not only in Montecatini's more severe terms but also in the fact that a good number of prospective licensees doubted that Montecatini's priority claims could be enforced. Initially, Montecatini focused its negotiations in Europe, then in Japan, and finally in the United States.

The dividing line between a company's acceptance of Montecatini's terms, on the one hand, and its readiness to enter into litigation over the priority question on the other, varied from country to country. The latter alternative was chosen predominantly in the United States and led to 20 years of court battles. The industrial giants of the chemical industry were aware of their financial power and were willing to risk major monetary expenditure and to safeguard themselves against potential counterclaims with back-up reserves. The profit balance between the relatively low interest rates charged on damages payable in cases where the litigation proceedings went against them on the one hand, and the current interest rate for the back-up funds on the other hand, always favored the infringer, unless the plaintiff was able to prove the infringer's intention and would, thus, hope for an award of double or treble damages. The court-imposed requirements in this regard are almost impossible to fulfill in the United States. Prospective licensees in Europe and Japan, for the most part opted to accept Montecatini's terms. The legitimate exercise of a license did, after all, assure risk-free production, safe from third-party complaints. Table 4.2 provides an overview of the license agreements [7] for the production of polypropylene entered into by Montecatini up to the year 1969.

Table 4.2 Companies entering into license agreements with Montecatini up to 1969

Licensee	Date of Agreement	Patent Issuance
Polymer S. p.A., IT		1955/57
Du Pont, US	1956	1963
Naphtachimie SA, FR	1958	1957/60
Petrochemie Schwechat, AG, AT	1958	1957/58
Société Normande des Matières Plastiques, FR	1958	1957/60
I.C.I., GB	1959	1959/60
Shell Chemicals UK Ltd., GB	1959	1959/60
Esso Chemical AB, SE, NO, DK	1960	1960
Mitsubishi Petrochemical Co. Ltd., JP	1960	1957/59
Mitsui Chemicals Inc., JP	1960	1957/59
Sumitomo Chemical Company, JP	1960	1957/59
Dow Chemical Co., US	1961	1963
Rotterdamse Polyolefinen Maatschappij, BE, LU, NL	1961	1955/63
Paular SA, ES	1963	1955
Shell Research Ltd., AU/NZ	1963	1957/58
Esso Quimica, AR	1964	1958/63
Hercules Incorporated, US	1964	1963
Novamont Corporation, US	1964/67	1963
Shell Oil Company, US	1964	1963
Kuck Tae Ind. Co. Ltd., KR	1967	

AR = Argentina, AT = Austria, AU/NZ = Australia/New Zealand, BE = Belgium, DK = Denmark, FR = France, GB = United Kingdom, IT = Italy, JP = Japan, KR = South Korea, LU = Luxembourg, NL = Netherlands, NO = Norway, SE = Sweden, ES = Spain, US = United States of America

4.5

Research and Production

A flood of patent applications, in particular filed by the licensees themselves, kept the national Patent Offices in the industrialized countries busy. The value of these improvement patent applications was difficult to assess from an objective standpoint. Each party concerned sought to obtain additional, dominating patent protection in order to have an edge over the competition. Among the large number of proposed improvements, three will be discussed below in greater detail.

When E.J. Vandenberg [8] of Hercules Powder first became involved with Ziegler catalysts after the license agreement with Ziegler had been finalized, he discovered that by varying the amount of hydrogen added during the polymerization process it was possible to control the chain length (degree of polymerization) of the polymers. The UK-based company, ICI, also obtained patents in this same area [9]. The priority dates of the initial patents of both companies were so close that it seemed inevitable

that worldwide conflicts would follow, which, in fact they did, as anticipated. The solutions to these problems varied from country to country.

Altering the hydrogen content proved to be an improvement over Martin's findings, according to which it was the ratio of the two catalyst components – the organoaluminum compound and titanium compound – that needed to be modified in order to adjust the polymer chain to the desired length.

The second case involved the improvement of the activity of the titanium chloride compounds needed to form the catalyst. The particle size of the solid titanium chloride marketed by Stauffer could be reduced by grinding and the active surface area thus considerably enlarged. It is also possible to enhance the porous structure of the titanium chloride component and thereby improve its desired activity by removing the aluminum chloride which inevitably forms during manufacture of titanium trichloride as an integral part of the product. The investigations were conducted at the Esso Development Company in Linden, New Jersey, USA [10]. Despite years of effort, Stauffer was unable to secure a license for the production of the improved product. A license fee on a product (titanium trichloride) which is utilized only in the minutest catalytically active quantities in the polymerization process is understandably of little interest. In order to collect generous royalties based on the sales price of polypropylene, a mass-produced product, it would be necessary to make the compound look initially promising to anyone using titanium trichloride and to the polypropylene producers.

The third development to be highlighted here concerns the use of inert carrier materials onto which the liquid or dissolved titanium component is mounted, whereafter the aluminum component necessary for catalyst formation was, for the most part, added in the presence of the olefin to be polymerized [11]. A more detailed discussion will follow later (see Section 4.6.7, Case 7).

In 1963, the first product claims patent [12] was granted in the United States for Ziegler catalysts, and in the same year, Montecatini was granted product claims [13, 14] for the so-called isotactic polypropylene⁴⁾. This new development greatly strengthened the Montecatini/Ziegler bargaining position in granting licenses. The same year also witnessed the award of the Nobel Prize in Chemistry to Ziegler and Natta as recognition by the international community of the caliber of the invention.

As early as 1963, a number of production facilities were already operating without a license, primarily in the United States. Similar complications threatened in Japan [15]. Once a number of product patents had been issued, as mentioned earlier, the stage was set for infringement actions.

After commercial plants for the production of polyethylene and polypropylene had been planned and gone on stream, the industry, worldwide, became interested in additional ways of using the Ziegler catalysts. The Ziegler patent rights [16] also covered the production of co-polymers, that is, mixed polymers initially comprising ethylene and propylene or butylene and ethylene and the variety of ratios used was limitless.

4) US PS 3,112,300, "...isotactic polypropylene ... insoluble in boiling n-heptane"; US PS 3,112,301, "...Polypropylene ... insoluble in boiling ethyl ether ... which consists prevail-

inglyof isotactic macromolecules being insoluble in boiling n-heptane..."; both patents were issued on Nov. 26, 1963.

The first two Pool Agreements with Montecatini included provisions which governed the handling of licenses on polypropylene and higher polyolefins. In terms of co-polymers, Ziegler had retained control over all ethylene-rich co-polymers. In late 1955, Montecatini filed a patent application [17] covering the production of co-polymers and the processing thereof to form elastomers. This situation required an acknowledgment that the Montecatini application was dominated by the earlier Ziegler priority application. Montecatini, however, characterized the new development as something entirely novel and claimed all rights exclusively for itself⁵⁾. By mid-1958, both parties settled on a very complicated arrangement known as the third Pool Agreement [18]. Once they had filed the new application, Montecatini subsequently pressurized Ziegler into allowing them to retain exclusive control even in cases where the mixed polymers were rich in ethylene. A conflict was inevitable.

All the same, Montecatini initially allowed Ziegler to collect 70% of the royalties from agreements on the production of co-polymers intended for the production of elastomers, while any income generated from utility patents for processing elastomers would go exclusively to Montecatini. They also insisted that, in connection with any existing Ziegler license agreements, 30% of the royalties for products sold after the priority date of Montecatini's "Elastomers" application were to be surrendered.

An agreement between Ziegler and Du Pont already existed at that time [19]. One of the characteristics set forth as part of the Definition of Products was that the co-polymers should contain at least 50 mol% ethylene, this served as a guarantee for Ziegler that he would be allowed to enter into such an agreement as the sole licensor. Montecatini could hardly object to this arrangement. A conflict only arose after Ziegler unilaterally broadened the above-mentioned Du Pont agreement in 1962 [20]. While the required ethylene content remained "at least 50 mol%", the other components which were to be co-polymerized with the ethylene were defined as "at least one other co-polymerizable substance". Since the products according to the agreement were not characterized as elastomers, it was Ziegler's interpretation that they were ethylene-rich mixed polymers, and it was immaterial to him what further components other than ethylene were to be co-polymerized⁶⁾.

Ziegler licensees such as Staatsmijnen [21] and Hoechst now approached Ziegler, pointing out that their respective license agreements covered the production of co-polymers at large, and an additional license from Montecatini for elastomers would not be necessary. They also indicated that, at least in Germany, the important part of the co-polymers (40–60% of either olefin) was covered by the Ziegler application [22], and further, that mention of the term "elastomer" as a characteristic in the patent application was not essential [23]. Montecatini denied this and offered non-exclusive licenses on elastomeric co-polymers. BASF, Chemische Werke Hüls and

5) In countries providing an examination by the Patent Office in Montecatini application problems to be issued. For example, in Germany the parallel patent right was limited to a catalytic mixture of vanadium compounds with

alkylaluminum compounds, in which the alkyl groups contained at least four C-atoms.

6) Under this agreement Du Pont sold a terpolymer of ethylene, propylene and ethylidene norbornene, trade name "Nordel".

Mitsui Chemical now also entered the arena as prospective licensees. The financial demands imposed by Montecatini for a license were exorbitant [24]. Thus, Farbwerke Hoechst and Chemische Werke Hüls were to pay 1.5 million Deutsch Marks each as down-payment for a license in the elastomers field, another 2 million Deutsch Marks each for fibers, they were not to receive any export rights, and were also to pay running royalties of 3–5%. No license agreement was concluded for the time being.

Meanwhile research in the field of co-polymers had been making good progress. Dunlop of Great Britain, was successful at an early stage in establishing a patent priority [25] for the production of ethylene – propylene – diene terpolymers using Ziegler catalysts. The properties of the terpolymers largely depended on the choice for the third component, the diene⁷⁾. The products are useful in the rubber industry as alternative materials for India-rubber. Montecatini persuaded Dunlop [26] to license the production of terpolymers jointly with Ziegler, with a share in the proceeds. The distribution arrangement for royalty income was Dunlop, 28.4%, Montecatini, 40.65% and Ziegler, 30.95%.

In 1965, the three owners of the patent package granted an ethylene – propylene – diene license (EPT) to Mitsui Petrochemical and Sumitomo Chemical in Japan, but ran into difficulties when they tried to obtain permission from the Japanese government agency, MITI [27]. This was because other companies – Mitsui Chemical, Mitsubishi and Asahi – also announced an interest, and MITI was not willing to approve more than two licenses. The Montecatini Co-polymers patent had not yet been issued in Japan, which presented a further obstacle [28]. Mitsui Chemical and Mitsubishi received, “as consolation”, a co-polymers license [29] which included the right to utilize ethylene/propylene co-polymers as plastics.

Rights and obligations between Ziegler and Montecatini regarding the exploitation of patent rights in the elastomers field had so far remained essentially unregulated. A fourth “Pool Agreement” [30] was sorely needed. Even though the distribution arrangement for income from exploitation had been established (70% Montecatini, 30% Ziegler), Montecatini was nevertheless looking for opportunities to further reduce Ziegler’s share. It was agreed that the patent rights dealing with polymerization *per se* should be rated at 60%, while processing would make up the remaining 40%, so that, in granting a pool license encompassing all available patent rights, Ziegler was to receive merely 30% of 60%. The rationale for this solution was based on the large number of processing and/or product-utility patent rights, rather than the contents and significance of the individual applications which Montecatini had filed up to that point. Since Montecatini continued to claim for itself the exclusive right to grant licenses, it was able to avoid having only polymerization patent rights licensed. Montecatini was, of course, intent on securing for itself a backflow from the product-utility patent rights.

7) Bridge-ring compounds, so-called endocyclic compounds containing at least one double bond and seven to 10 carbon atoms are preferred, e.g. bicyclo pentadiene or bicyclo-

heptadiene. Methyl-tetrahydro-indene (MTHI) had advantages over bicyclo pentadiene [34].

This opened the door for licenses, with or without Dunlop patent rights, also being granted to prospective European licensees, i.e. Farbwerke Hoechst, and Chemische Werke Hüls [31] in Germany, Staatsmijnen [32] in The Netherlands, and International Synthetic Rubber [33] in Great Britain – a remarkable achievement. Ziegler's older licensees had apparently abandoned their original position i.e. be-

Table 4.3 Sales under domestic and foreign licenses issued by Studiengesellschaft Kohle mbH in tons/year (1972–1978)

Product	Year	USA	Europe	Japan	Others	Amount
PE	1972	251,162	293,158	200,000		744,320
	1973	241,067	640,708	176,280	–	1,058,055
	1974	176,875	639,168	20,059	–	836,102
	1975	76,840	320,364	17,641	–	414,845
	1976	127,170	73,710	10,706	–	211,586
	1977	189,211	71,207	–	–	260,418
	1978	189,270	70,737	–	–	260,007
PP	1972	166,930	234,028	258,339	–	659,297
	1973	768,168	326,330	284,886	–	1,379,384
	1974	789,875	199,292	223,091	–	1,212,258
	1975	632,023	143,432	179,166	–	954,621
	1976	449,545	121,005	295	–	570,845
	1977	425,318	73,117	25	–	498,460
	1978	531,143	100,183	10	4,418	635,754
CP	1972	64,014	–	134,345	–	198,359
	1973	110,659	–	149,097	–	259,756
	1974	115,797	–	123,349	–	239,146
	1975	80,115	–	97,584	–	177,699
	1976	83,040	–	–	–	83,040
	1977	92,567	–	–	–	92,567
	1978	29,060	–	35	–	29,095
PBu	1972	–	729	–	–	729
	1973	–	995	–	–	955
	1974	–	–	–	–	–
PD	1972	145,913	45,366	114,459	5,060	310,798
	1973	225,228	74,457	92,033	5,494	397,212
	1974	223,691	74,082	97,537	5588	400,898
	1975	174,039	72,313	88,449	3423	338,224
	1976	169,794	45,417	63,331	3841	282,383
	1977	169,222	20,367	31,615	996	222,200
	1978	139,463	–	–	–	139,463

PE = Polyethylene, PP = Polypropylene, PBu = Polybutene,
CP = Co-respectively Terpolymers, PD = Polydienes.

cause they had previously obtained licenses under the Ziegler patent rights they did not need a license from Montecatini.

There were, however, no further running royalty payments. The market was initially tested by way of imports, with only modest results [35]. Finally, in 1970, Farbwerke Hoechst assigned its contract to Chemische Werke Hüls.

Now back to the early 1960s. Having obtained licenses for the production of polypropylene, and because their production capacities were growing rapidly, the Japanese companies were anxious to launch their export business. Export of Japanese polyolefins to Europe was not permitted [36] even though it was Ziegler’s recommendation that export be allowed to any country for a limited period as long as no licensee had rights in that country. In Europe, demand for the new products could not be satisfied, yet, Hoechst, for one, refused to approve Mitsui products for resale, because they did not meet the quality standards of Farbwerke Hoechst’s own products. Hercules [37] took the same position in the United States. Nevertheless, a contractual arrangement regarding export from Japan into the United States was finalized in late 1968 [38]. Meanwhile Montecatini was negotiating with the three polypropylene licensees in Japan to lower the running royalties for production [39] above 60,000 tons to 3.5%, and to 2% for production above 100,000 tons. For exported quantities, the requirement was 5% and for films and fibers an additional 3.5% – certainly a remarkable business accomplishment for Montecatini.

The polyolefin markets around the world exploded with growth rates sometimes running into double figures. The early 1970s saw the expiration of the oldest of the Ziegler patents – a patent in Germany for example, was effective for 18 years commencing on the date that the application was filed. Table 4.3 shows that the sales under domestic and foreign licenses granted by Studiengesellschaft Kohle mbH as the licensing arm for Ziegler, are not entirely representative of the world market situation, since only those production figures are recorded for which running royalties have been paid. The market shifted from Europe and Japan to the United States, because there, the lifetime of the relevant patents – 17 years from the date of issuance – extended until at least 1980.

4.6

Early “New”, So-Called Independent Catalysts

During the early and mid-1960s, the polyethylene and polypropylene market expanded through emerging new production facilities. New markets sprang up around the world, yet some producers managed only limited sales, partly due to the Montecatini’s restrictive licensing policies. A trend developed to either find independent catalysts which would create the same products, or ignore the existing patent situation. There were a number of cases which caused disturbances, but which, from a chemical viewpoint, also stimulated interesting discussions. If the outcome of but one of these cases had been successful, a major part of Studiengesellschaft’s licensing business would have collapsed. Initially prospective licensees were “testing” the limits of Ziegler’s patent protection [40].

There was a question as to whether a certain organoaluminum compound (EtAlCl_2) was suitable for use as a catalyst component. While this component had been mentioned and claimed [41] in two of Ziegler's German priority applications, it had been eliminated from the subsequently filed foreign applications because it was less effective by comparison and, therefore, showed less promise (see p. 102).

The following cases study examples will illustrate this development.

4.6.1

Case 1

In The Netherlands, Staatsmijnen, of Geleen, had obtained a Ziegler license for the production of polyethylene. Montecatini had granted an exclusive Pool-license for the production of polypropylene to their competitor, Rotterdamse Polyolefinen Maatschappij. Staatsmijnen had large stockpiles of propylene available and wished to obtain a license from Montecatini for conversion to polypropylene. Montecatini was compelled to decline. Ongoing discussions concerning this situation began in early 1964 and continued through mid-1966 [42]. Staatsmijnen investigated the patent situation as it existed in The Netherlands and introduced its own, specially developed catalyst [43], which it claimed established independence from Ziegler. This claim was based on the assertion that the aluminum compound of the Ziegler patents had been replaced by an ethylaluminum dichloride, which was not covered by the claims of Ziegler's Dutch patent rights, which, however, required, as an essential feature, the presence of an ether at specific ratios to the aluminum compound (ethylaluminum dichloride to ether at ratios of 1 : 0.8 to 1 : 1.5). Thus, a classic two-component Ziegler catalyst had been modified to a so-called three-component catalyst which supposedly did not fall under any Ziegler patent rights.

At the Max-Planck-Institute for Coal Research in Mülheim, the chemistry of this combination was closely examined. It emerged that the catalyst according to Staatsmijnen worked at peak efficiency only if no more than a deficit amount of ether relative to the amount of ethylaluminum dichloride was present. Then, and only then, through so-called disproportionation, was an equivalent amount of diethyl aluminum chloride, an active aluminum compound according to the Ziegler patent rights formed.⁸⁾ Dominance over the Staatsmijnen variation had been established.

8) $2 \text{ EtAlCl}_2 + \text{R}_2\text{O} \rightarrow \text{AlCl}_3 \cdot \text{OR}_2 + \text{Et}_2\text{AlCl}$, in single cases the ether complex of AlCl_3 was isolated as a crystalline product at room temperature. The solution contained Et_2AlCl . TiCl_3 which was recommended by Staatsmijnen as starting material for the production of the polymerization catalyst contained substantial amounts of AlCl_3 as a result of the method of its production ($\text{TiCl}_4 + \text{Al}$). Ether removes part of the AlCl_3 . The greater affinity of AlCl_3 compared to EtAlCl_2 , for the complex

agent ether, resulted in a further deficiency in the ratio of ether to EtAlCl_2 which drives the reaction to disproportionation as shown above. The speed of reaction and the yield is optimal at a ratio of $\text{OR}_2 : \text{Al}$ of 0.8–0.9. If TiCl_3 produced by the reaction $\text{TiCl}_4 + \text{H}_2$ was used instead of Al , the catalysts were practically inactive. The same result is obtained if an excess of ether is used. The catalyst was therefore inactive regardless of the chosen type of TiCl_3 was used.

In 1972, Staatsmijnen made another attempt to obtain a license from Montecatini for the production of polypropylene. This time, the matter was resolved in that Rotterdamse relinquished its exclusive rights with respect to polypropylene production. Financial compensation received from Montecatini and Ziegler “sweetened” the deal for Rotterdamse. In 1976, Staatsmijnen went on stream with its polypropylene production.

4.6.2

Case 2

The Pool Agreements between Montecatini and Ziegler gave Ziegler the exclusive right to grant licenses in Germany for the production of polypropylene. As opposed to the earlier Ziegler licenses, the newly granted licenses no longer included export of, for instance, polypropylene. Farbwerke Hoechst was, therefore, forced to sell its products in Germany, an impediment based on the fact that Montecatini tried not only to control the export markets and guarantee additional income for itself, but also to protect, at least temporarily, third-party licensees for polypropylene for example, from competition in their own countries. Discussions aiming to liberalize the markets were not initiated until much later.

In 1964, Farbwerke Hoechst AG attempted to persuade Montecatini to open up the markets and threatened to use a supposedly independent catalyst, developed under its own direction, for its future polypropylene production. Infringers, such as Avisun, were free, according to Hoechst, to sell in all countries. Hoechst disclosed to Ziegler the nature of the new catalyst and its production. Here too, the mix of components of choice, as stated by Hoechst, was ethylaluminum dichloride, this time, however modified with the addition of potassium acetate, and titanium trichloride. Again, disproportionation took place, causing the formation of Et_2AlX , an organoaluminum component covered by the basic Ziegler patents, which, together with titanium chloride, was acknowledged to be responsible for the effective polymerization of propylene [44]⁹.

Ziegler sent a warning to Hoechst, since experts at Montecatini had meanwhile confirmed the chemistry involved after conducting their own experiments. The license situation whereby Hoechst was granted only limited export rights (Eastern Europe, The Balkans, and South America) initially remained unchanged. But even this right was withdrawn by Montecatini in 1960.

4.6.3

Case 3

Eastman Kodak, USA, had more than 70 patents issued in its name, all of them directed to additions to, or modifications of, Ziegler catalysts. One of these patents disclosed the combination of titanium chloride, ethylaluminum dichloride and a

9) $2 \text{ EtAlCl}_2 + 1 \text{ KOOCCH}_3 \rightarrow \text{KAl}(\text{OOCCH}_3)\text{Cl}_2 + \text{Et}_2\text{AlCl}$, the diethyl aluminium compound can be identified in the organic solution, and is free of potassium. The potassium com-

pound formed deposits as an insoluble precipitate. It is the solution together with TiCl_3 rather than the precipitate that is active in the polymerization process.

donor. Ziegler suspected that Eastman Kodak¹⁰⁾ was trying to circumvent his patent with the help of these catalyst combinations. As will be discussed later, Eastman Kodak would eventually choose another, more promising combination.

4.6.4

Case 4

In the Fall of 1962, “Kunststoffwirtschaft” [45] published a press release, in which Philadelphia-based Avisun Corp. (a subsidiary of Sun Oil) pointed out, in connection with the issuance of a Hercules Powder Co. US patent, that the catalyst systems disclosed in that patent were “Ziegler catalysts” and were distinct from the catalysts developed by Avisun, so that the Avisun process should not be regarded as constituting infringement.

Avisun had emerged on the polypropylene market when it had granted a license to Shin Chisso in Japan in mid-1961. Montecatini had filed suit in Japan [46].

Rumors abounded regarding the composition of the Avisun catalyst. Initially, it was assumed that the catalyst included indium chloride in addition to a “non-functioning” combination (presumably) of alkyl aluminum halide and titanium tetrachloride. But, according to experiments conducted by Martin, the indium chloride effect could not be demonstrated [47].

Meanwhile, Avisun exported polypropylene to Germany. Traces of a titanium compound were detected in a sample polymer [49]. Montecatini tried to reach a settlement with Avisun, the terms of which included Avisun ceasing its export of polypropylene to countries where valid Pool patents existed, and breaking off its contractual relationship with Chisso and, in consideration thereof, would receive a Pool license in the United States. Avisun stated its belief that its three-component catalyst comprising ethylaluminum dichloride, titanium halide and a third, as yet undisclosed component, was independent of Ziegler. There was a growing belief that the third component was a tetraethoxy silane [50]. With this in mind, experiments were being conducted and the product of the reaction between the aluminum compound and silicon compound was recovered and its properties determined [51] (see also Chapter 3, footnote 12). In early 1964, von Kreisler advised the Head of the Patent Department at Sun Oil Co., Mr. Church, that even though the chemical experiments being conducted by Martin had not yet been completed, there was no doubt that Ziegler’s corresponding German patent was being infringed by the Avisun catalysts [52]. For the time being, Montecatini intended to continue its negotiations with Avisun.

The situation escalated, Avisun exported to The Netherlands. Meanwhile a valid Pool patent had been issued in that country [53].

10) Among others a compound of the composition $(Me_2N)_3PO$ [48] was preferred as a donor. Experiments in the Max-Planck-Institute confirmed that ethylaluminum dichloride and the phosphorus compound lead to disproportionation into aluminium trichloride and diethylaluminum chloride, whereby the phos-

phorus compound is found as a complex with aluminium chloride. The insoluble complex could be isolated before addition of the titanium compound. This reaction was preferential if the donor compound was used in a molar deficiency, as in the previous cases.

Even after the test results¹¹⁾ had been made available, any attempts to convince Avisun to take a license failed – a small foretaste of American aggression [54]. On the contrary, Avisun officially disclosed the composition of the catalyst components to Montecatini and expressed an expectation that both Ziegler and Montecatini would have to accept the fact that this combination was not covered by any Ziegler/Montecatini patents [55]. A description of the Avisun three-component catalyst appeared in an Indian patent [56] which had been issued meanwhile. The catalyst did indeed, encompass, as the third component, the addition of alkyl disiloxanes or alkoxy silanes to the mixture of ethylaluminum dichloride and titanium trichloride.

Brenntag, a subsidiary of Stinnes AG, the German importer of Avisun’s polypropylene, announced that the imported product, intended specifically for export, was indeed, produced using the disclosed catalyst, but that, because of increased production costs, only a limited amount of product could be supplied [57].

In early 1965, Montecatini advised that the negotiations with Avisun had failed, primarily because Avisun refused to accept the standard license terms [59].

Avisun stepped up its efforts to prevent issuance of Montecatini’s and Ziegler’s patents. It was not until 1970, that a broad-based settlement was reached with respect to the opposition and invalidity proceedings¹²⁾ pending in numerous European countries. Meanwhile a Dutch patent was issued to Avisun [60]. Avisun gave up import in Austria [61].

Not only did the choice of the ethylaluminum dichloride serve as a point of departure in attempts to avoid the Ziegler patents, but improvements in the area of catalyst preparation, which had been developed by existing licensees, such as Hoechst and Hercules, but also precipitated threats by non-licensed parties that they would utilize such improvements if a license from Ziegler appeared to be out of reach.

4.6.5

Case 5

Asahi Chemical in Japan, tried to obtain a license under Japanese patents owned by Hoechst [62] and Hercules [63]. Hoechst and Hercules declared that their patent rights were dominated by Ziegler, so that a license could be obtained only with Ziegler’s consent. Asahi asserted that the patents in question were independent of the Ziegler patent rights¹³⁾. Ziegler recommended that his exclusive licensee in Japan, Mitsui Chemical, grant Asahi a sub-license, since Asahi might arrange a license from Phillips if its license request were refused [64]. Mitsui [65] declined Ziegler’s

11) The experimental program of Martin, partly carried out in the presence of a Netherlands expert, Dr. Napjus, (Plastic Institute TNO, Delft) showed that the ratio of Cl/Al in the mixture was decreased from 1.4 to 1.7 before addition of the titanium component, i.e. pure EtAlCl_2 was no longer in existence. Later these experiments were repeated by TNO confirming the results [58].

12) See p. 105.

13) The catalyst preparation $\text{TiCl}_4 + \text{Et}_2\text{AlCl}$ was varied by heating the insoluble reaction product (TiCl_3) to 60–100 °C. The product was washed. The treated TiCl_3 (change of $\beta\text{-TiCl}_3$ into $\alpha\text{-TiCl}_3$ i.e. change of crystalline structure) was now mixed with Et_2AlCl .

wishes, pointing out that Mitsui Petrochemical, as a sub-licensee with an on-stream operation, would not tolerate any competition since they had just recently expanded their production capacity [66] (48,000 tons at Ciba and an equally large plant at Otake).

A patent attorney, Mr. Tanabe, was engaged as an expert who came to the conclusion that the modified catalysts patented by Hoechst and Hercules were independent of Ziegler's patent rights¹⁴). Asahi threatened to bring a suit for patent invalidity.

Even an attractive financial package tied to Asahi obtaining a license failed to soften the hard-line position taken by Mitsui Petrochemical and Mitsui Chemical [67]. These companies relied on the assumption that, in the face of such a high risk factor, Asahi would not start up its own polyethylene operation. Mitsui Petrochemical formally declined to grant Asahi a sub-license [68].

4.6.6

Case 6

Furthermore, there were research findings, produced by the industry, which focused on using starting materials free from organoaluminum compounds for the initial phase of catalyst preparation. Wacker Chemie, together with British-based ICI [69], attempted to have these catalyst mixtures together with their applications made available by license. The components considered for use were special silicon compounds, for example, HSiR_3 and methyl hydrogen polysiloxanes [70], which, in combination with titanium tetrachloride and aluminum trichloride, were said to form effective polymerization catalysts. This involved, in an initial step, reacting the polymeric silicon compounds with aluminum trichloride and admixing the reaction product with titanium tetrachloride. The period from 1957 [71] through 1961 witnessed the issuance of patents directed to the most diverse variations. What aroused suspicion in every single case was the required presence of aluminum chloride¹⁵).

There has never been any recognized production of polyolefins on the basis of such "Wacker catalysts". Legal action on Ziegler's part was, therefore, not necessary.

A different variation of the same or a similar system was being patented by Solvay & Cie. In place of the previously described silicon compounds, the modification em-

14) The opinion of patent attorney Tanabe that Ziegler uses an excess of alkyl aluminium compound in the catalyst mixture was commented on by von Kreisler as follows: Ziegler rights are not limited to the use of excess. Ziegler works at 70–90 °C in one step, Asahi in two steps. Von Kreisler: No independency. Asahi uses only the precipitate of the first step in order to change in its structure, Ziegler uses the total reaction mixture. Ziegler uses only one titanium component, Asahi a solid solution of $\text{TiCl}_3/\text{AlCl}_3$. Von Kreisler: this solid solution comes under the definition "titanium compound".

15) Martin proved that the reduction of titanium tetrachloride is only possible in the presence of aluminum trichloride, but not using the pure silicon compounds, under the given conditions. The hydrogen of H-polysiloxane is stable against hydrolysis. Substantial amounts of this hydrogen are found if the hydrolysis is conducted in the presence of aluminum trichloride, a clear indication that, the hydrogen is first transferred to the aluminum compound.

ployed was a combination of tin tetrabutyl and aluminum trichloride [72]¹⁶⁾. Here, too, formation of Ziegler catalysts could be established.

In Italy, it had become known that Solvay was using this catalyst for the production of polyethylene. In 1968, Montedison – Montecatini had merged with Edison in 1966 – contemplated bringing an action for patent infringement against Solvay.

Ziegler decided not to join this action [73] since his Italian patent rights in this field were set to expire in 1969. The best information available on the capacity of the Solvay production facility in question was imprecise. The rumor was 11,000 tons of polyethylene annually [74].

4.6.7

Case 7

In the early 1960s, the industry focused its research efforts, *inter alia*, on the technical optimization of the polymerization process by exploring the use of so-called carrier catalysts. The costly removal of the catalyst residues from the polymerization product was replaced by employing large-surface-area, inert carrier substances, whose surfaces were coated with a very thin layer of liquid titanium tetrachloride. The required amount of titanium halide could thus be substantially reduced in comparison to the titanium trichloride process. It was, after all, only the surface areas of the titanium halide which were accessible for catalyst formation. As a result of this modification of the process, the catalyst washing step could be eliminated. On the other hand, the process did fall back on the use of aluminum triethyl as the organometal component in the catalyst. Later on, in the 1980s, the class of catalysts thus created formed the basis for the so-called “high-speed catalysts” developed at that time, which were eventually used in the 1990s, particularly by a segment of the industry in the United States (cf. Chapter 5, p. 226). Patentable advancements were achieved, *inter alia*, by Solvay & Cie. in Brussels. Initially, the preferred inert carriers used were phosphates of calcium [75], strontium and barium, later on, aluminum oxide [76], and finally magnesium compounds such as magnesium oxychloride [77, 78], magnesium alcoxide [79] and magnesium oxide [80] types.

Solvay tried to sell the new technology worldwide through licensing, but the rest of the industry had serious misgivings because of the technology’s potential dependency on Ziegler patent rights and the inherent danger of the ordeal of an infringement litigation initiated by Ziegler. Years of negotiations ensued in an attempt to resolve the problem amicably, this culminated in Solvay taking a license under Zie-

16) The proof that butyl aluminum compounds are formed first was relatively easy, since the pure tin tetrabutyl is stable against hydrolysis. The presence of butane formed during hydrolysis originated from the butyl aluminum compound formed. The expected reduction of titanium tetrachloride in the formation of the catalyst is only possible if in addition to

tin tetrabutyl, aluminium trichloride is also present. The inventor of this catalyst system added ether at a certain time during the reaction and in a preferential amount, in order to disproportionate butyl aluminum dichloride into dibutyl aluminum chloride as described in Case 1.

gler's patent rights [81], which included the right to grant sub-licenses in conjunction with the licensing of its proprietary carrier technology – a profitable solution for all concerned.

4.6.8

Case 8

After 1962, rumors began to spread regarding the value of sodium hydride as a reducing agent for titanium chloride in connection with polyolefin catalysts. According to these rumors, which had been circulated by Avisun and Shin Chisso as well as Tokoyama Soda [82], the combination of sodium hydride and titanium tetrabromide and propylene, or sodium hydride together with aluminum trichloride in combination with titanium tetrachloride, or sodium metal plus hydrogen and titanium trichloride for the polymerization of propylene had aroused interest as a means of avoiding the use of Ziegler catalysts [83].

Here, too, it seemed obvious to suspect that the presence of propylene would cause the formation of sodium propyl and/or propylaluminum compounds, i.e. organometal compounds, the use of which was anticipated by Ziegler's patent rights. Initial results derived from a test program carried out at the Max-Planck-Institute for Coal Research in Mülheim, confirmed this assumption. There was, however, no follow-up in this direction, because there was no interest in employing these low-efficiency catalyst combinations in commercial processes.

4.6.9

Case 9

The French company Safic-Alcan in La Garenne-Colombes, imported rubber from Anic, an Italian company and a subsidiary of ENI, which had its headquarters in Ravenna, and sold this product in France under the name "Europrene Cis". Anic had a production license from Phillips Petroleum, USA, for the raw material 1,4-cis-polybutadiene¹⁷⁾, (plant capacity 10,000 tons).

In 1963 a rubber sample taken from Safic Alcan's production line was tested in the Laboratories of Shell Internationale Research Maatschappij [84]. The results indicated the presence of a significant amount of aluminum, titanium and iodine as well as a composition consisting of 89.8% cis-1,4-polybutadiene, 5.9% trans-1,4-polybutadiene, and 4.3% trans-1,2-polybutadiene.

Pursuant to the experience of Goodrich Gulf, this composition corresponded to a polybutadiene material produced using a catalyst composed of aluminum trialkyl and titanium tetraiodide [85]. Goodrich Gulf and Ziegler decided to take Safic Alcan and Anic to court. Maître Mathély of Paris was engaged to represent the plaintiffs.

The action was based on the French patents of Ziegler [86] and Goodrich Gulf [87]. Ziegler's patent covered a polymerization catalyst which included both aluminum trialkyl and titanium iodide (priority 1953). The catalysts – as is possible in France –

17) See Chapter 2, p. 76.

were covered by product claims. The Goodrich Gulf patent was directed to the polymerization of butadiene to 1,4-polybutadiene using Ziegler catalysts, and included a disclosure of the selective effect of the catalysts for the production of trans-1,4-polybutadiene and mixtures of trans-1,4- and cis-1,4-polybutadiene. The amount of the cis isomer could be increased to 50–70%. If the court affirmed that Goodrich Gulf had been the first to selectively produce polybutadiene using Ziegler catalysts, and Phillips had improved on this selective production by further boosting the yield of cis-1,4-polybutadiene, then Phillips had to be dependent on Goodrich Gulf.

The trial proceeded before the Third Chamber of the “Tribunal Civil de la Seine” in Paris, beginning in mid-June of 1965. The opponents pointed out that Ziegler’s patent was limited to the polymerization of ethylene, and nowhere did it mention the use of diolefins, and that the Goodrich Gulf patent disclosed only the production of mixtures of trans- and cis-polybutadiene, whereas it was the cis-polybutadiene alone which exhibited rubber-like characteristics and was useful for industrial exploitation. Mathély responded by pointing out that according to Ziegler’s patent, in addition to ethylene, the gases or gas mixtures derived from the cracking processes were also suitable as monomers, so that co-polymerization of ethylene and butadiene would take place. This argument had been furnished by the defendants themselves when they stated that, in addition to ethylene, “at best”, gas mixtures from cracking reactions, which, along with ethylene, might also contain some butadiene, would be suitable as starting olefins. Moreover, Phillips had documented in its own patent disclosure that according to a comparison test using Goodrich Gulf catalysts, 67.5% 1,4-cis polybutadiene could be recovered.

Phillips highlighted the Ziegler–Montecatini Pool patent 1,138,290 issued in France, which described the polymerization of higher olefins, proof that the Ziegler patent cited in the Complaint was intended only for ethylene. And furthermore, that 1,4-trans-polybutadiene was similar to balata (balata is, in fact, 1,4-trans-polyisoprene, a rather inflexible, rigid material) which would be useful for industrial purposes, at best in a different direction.

On June 16, 1966 the “Tribunal” decided against Arnica and Safic-Alcan. Both parties made representation to the Appeal Court in Paris (“Cour d’Appel de Paris, 4ème Chambre”). At this stage Phillips Petroleum entered the proceedings, in order to assist its customers [88].

The catalyst system used by Phillips was prepared by initially admixing aluminum trialkyl with diiodine butene – which, as has been demonstrated (cf. Chapter 5, footnote 10), yielded dialkylaluminum iodide – and the mixture reacted with titanium tetrachloride. This system was claimed in one of Phillips’ French patents [89]¹⁸⁾.

18) Phillips had four French patents which protected the various catalysts: 1,231,993, aluminum trialkyl, TiCl_4 and TiI_4 ; 1,247,307, aluminum hydride or organo aluminum compound + tri- or tetrahalide of titanium (also TiI_4); 1,259,291, aluminum trialkyl + titanium halide + iodine; 1,426,111, titanium halide + aluminum alkyl + 1,4-diiodine-butene. The cis-1,4-content of caoutchouc is not an absolute value for the quality of the products. Yet, one condition was that this content should be above 50%. The Firestone

product (lithium catalyst) contained about 50% 1,4-cis, and practically no 1,2-product, and the Phillips product (titanium iodide catalyst) 93–95% 1,4-cis, and practically no 1,2-product. Goodrich Gulf (titanium chloride and cobalt catalyst) was the first to develop 1,4-products. Phillips did improve the products by enlarging the cis-1,4-content, but should have been dependent on Ziegler and Goodrich Gulf. The three products competed on the market.

In December of 1968, the court [90] handed down a decision in favor of Ziegler and Goodrich Gulf, affirming that Ziegler's patent disclosed a catalyst which, in addition to ethylene, was also suitable for polymerizing other olefins, for example, butadiene, and that the Goodrich Gulf patent disclosed a 1,4-rubber consisting of polybutadiene with a 60–70% cis-content, and furthermore, that the cited references were irrelevant. The defeated parties appealed this decision in the Court of Cassation [91] (Cour de Cassation, Paris) in March of 1969.

Five months later [92], the opponents signaled their willingness to settle. The prevailing parties engaged in lengthy discussions [93] to determine the amount of the damages called for. This involved the export of 1000 tons, with an intrinsic royalty value of approximately 120,000 Francs. The settlement agreement was signed by all parties in May of 1970. Since Shell and Montedison had shared in the litigation costs, they were entitled to an equal percentage of the proceeds. Ziegler [94] received 8,400 French Francs. The purpose of the legal action had been to issue a stern warning to Phillips Petroleum, rather than to make a financial gain.

4.6.10

Case 10

Aside from the indirect activity of selling polybutadiene rubber in Europe through licensees and import companies, Phillips Petroleum, USA, also attempted to push direct sales in Europe. As early as 1962, Phillips Petroleum International AG, a distributor of Phillips products in Zurich, put out a promotional flyer in trade magazines [95] advertising a “new, revolutionary Phillips cis-4-rubber.” With information obtained from Goodrich Gulf Chemicals [96], Phillips Petroleum manufactured this product with the use of Ziegler catalysts consisting of titanium tetraiodide and trialkylaluminum and offered it for sale in Germany, Switzerland and Austria. In Germany, Ziegler owned not only his own patent rights but also those of Goodrich Gulf in this area¹⁹⁾.

Phillips launched the same type of advertising campaign in France [97]. Moreover, in 1961, Michelin obtained a license from Phillips in France for producing 1,4-cis-polybutadiene in a facility in Bassens near Bordeaux. Their plan was to build a plant with an annual capacity of 20,000 tons [98].

Goodrich Gulf, Shell, Montecatini and Ziegler decided to bring an action in France to have the Phillips patents declared invalid [99] and to sue Phillips for infringement [100].

The Complaints were filed in the Fall of 1967. Maitre Mathéley appeared again for the plaintiffs. Phillips was represented by Maitre Foyer, France's former Minister of Justice. The actions were again based on the French Ziegler patent [101] and Goodrich Gulf's French patent [102]. The case in the Paris court was also based on the same issues. The debate before the court [103] revolved around a highly sophisticated line of reasoning, according to which the polybutadiene covered by the Goodrich Gulf patent, although a 1,4-cis-polybutadiene, had only a trans-configuration,

19) See Chapter 2, p. 79/80.

“at best” being a mixture of trans- and cis-, with up to 50% cis, while Phillips was claiming a polybutadiene with an 85% 1,4-cis-configuration, and furthermore, that the halogen attached to the titanium had an important function, whereby iodine exhibited a particularly high selectivity with respect to 1,4-cis-polybutadiene.

In December of 1967, the case [104] went to trial. As reflected by the record, Phillips had initially worked with a mixture of aluminum trialkyl and titanium tetraiodide. The method was then modified: The new mixture consisted of titanium tetrachloride and titanium tetraiodide with an alkylaluminum compound. Finally, the titanium tetraiodide was replaced with free iodine and, in admixture with titanium tetrachloride and aluminum trialkyl, used as a catalyst. The last method employed in 1967 included a mixture of aluminum trialkyl and titanium tetrachloride with the addition of diiodine butene (cf. footnote 18 in this chapter).

Furthermore, during the trial [105], Phillips documented the fact that, analysis of Phillips’ product showed it to consist of 95% 1,4-polybutadiene and 87% 1,4-cis-polybutadiene. It contained aluminum, chlorine, iodine and titanium. Accordingly, both the catalyst and the resulting product were clearly dependent on the Ziegler and Goodrich Gulf patent rights. The opponents insisted that it was impossible to obtain a 1,4-cis-polybutadiene product by following the Ziegler and Goodrich Gulf patents.

In view of the earlier judgments, Phillips was bound to lose this case and the appeal which they launched [106]. The court handed down a 99-page Opinion document examining the facts of the situation, and, again, reached the pivotal conclusion that the disclosed Ziegler catalysts were suitable not only for ethylene alone, but also for ethylene containing butadiene impurities. Additionally, any use of the patented Ziegler catalyst by a third party, even a novel use, constituted infringement of the Ziegler patent, thus including the use of the catalyst for the polymerization of butadiene [107]. The judgment also conceded that even though Ziegler had initially focused on ethylene, his catalyst was, nevertheless, a valuable broad-spectrum agent, the use of which also covered the polymerization of other olefins, including butadiene; this viewpoint would continue to be important as events unfolded and would be taken into consideration by other courts of law.

The use of iodine compounds and the chosen ratio of aluminum compound to titanium compound were certainly an improvement, but did not constitute a basically new invention. It was interesting to note that the court did not accept Phillips’ arguments with respect to the Goodrich Gulf/Phillips arrangement concerning Phillips Petroleum’s US patent²⁰), stating that the resulting legal consequences in the United States and in France would be different. The court found Phillips Petroleum guilty of omission and assessed damages.

An attempt by Phillips Petroleum to have the verdict overturned by the “Cour de Cassation” [108] failed on April 14, 1972.

This was followed by an attempt to come to an arrangement with respect to the damages. The initially promising figures regarding Phillips Petroleum’s sale of pol-

20) US P 3,178,402, see Chapter 2, p. 76 and reference [157].

ybutadiene in France were eventually reduced to a total of what could be expected in terms of damages of approximately one-third of a million French Francs [109]. After 5 years in all, the parties agreed that Phillips Petroleum had to pay damages [110] in the amount of 300,000 French Francs.

At that time, certain dissenters claimed that a US company had no chance whatsoever of prevailing over a European plaintiff in a patent infringement suit brought in France. An additional fact to be considered was that the European plaintiff, Ziegler, had granted numerous licenses in France.

But what might be expected in a situation where a French producer, Michelin, operating under a license from an American company, Phillips Petroleum, was infringing a Ziegler patent in France?

The royalties due from Michelin's annual production of approximately 20,000 tons, at a net sales price of 2.17 French Francs per kilogram [111], could be calculated at about 1 million Deutsch Marks. Under the effective terms of the Ziegler patent in question, a total of 5 million Deutsch Marks could be expected as royalty income [112]. Thus, there was a clear incentive for suing Michelin. Given the legal position established at the time, according to which the French courts had held that the Phillips' process infringed the patents of both Ziegler and Goodrich, an optimistic view of the outcome of a suit against Michelin was justified.

The Complaint was filed in 1973. Ziegler's French Patents 1,197,613 and 1,235,303 as well as Goodrich's 1,139,418 patent were cited as grounds for the action. In considering the arguments presented by both sides, the court [113] rejected Michelin's reasoning that, first of all, the reduction of titanium tetrachloride with the use of aluminum powder was known. The court found that the Ziegler catalyst resulted from a reduction of the titanium with organometal compounds. Secondly, so Michelin argued, Hall and Nash²¹⁾ had already disclosed the formation of aluminum triethyl from aluminum chloride, aluminum and ethylene as early as 1937. The court pointed out that this statement had been described as doubtful and that the aluminum compound was said to have been formed "in trace amounts."

Thirdly, Gaylord and Mark, in 1959, had taken the position that the Fischer process encompassed the *in-situ* formation of Ziegler catalysts. The court viewed this as a strictly hypothetical opinion. As a fourth point, in 1970, Hopff and Balint [114] had conducted experiments following the teachings of the Fischer patent, according to which, under the conditions of Fischer, aluminum triethyl had been formed. The court concluded that, after close scrutiny of the Balint experiments, it transpired that Balint had used comparatively larger amounts of aluminum and had actually demonstrated the formation of aluminum triethyl in only one of six further experiments.

The court found, in closing, that the Ziegler catalyst was a new product, but concluded that the Goodrich patent failed to disclose the formation of predominantly *cis*-1,4-polybutadiene. Concerning the polymerization of butadiene, the court stated

21) Hall and Nash as well as M. Fisher, BASF; see Chapter 1, p. 19, Chapter 3, foot note 2.

that the Goodrich patent was limited to either trans-1,4-polybutadiene or 1,4-polybutadiene having a 50% cis- and 50% trans-configuration. Samples obtained from the Bassens plant, produced with a catalyst consisting of triisobutyl aluminum, titanium tetrachloride and diethyl aluminum iodide, showed a 92% cis-1,4 content. While the product, according to the Goodrich patent, certainly exhibited an overall 1,4-structure, the Michelin product had a predominantly cis-configuration. Michelin’s additional arguments that Ziegler used two while Michelin used three components for the production of the catalyst were rejected by the court on the grounds that the effectiveness of two components could very well be equivalent to the effectiveness of three. The argument that the Michelin catalysts were homogeneous whereas Ziegler catalysts were heterogeneous was also rejected. The court affirmed that Ziegler did not limit his catalysts to the heterogeneous type alone²²⁾.

The court, which had also tried the previously mentioned cases, Ziegler/Goodrich versus “Phillips” and “Anic/Safic-Alcan”, found that the Michelin catalyst was the equivalent of Ziegler’s catalyst in its preferred form and, at best, would constitute an improvement. The damages were to be determined by expert opinions. J.C. Combaldieu was engaged to assess the damages and render an expert report. As a result of the court’s decision, it was not the net sales value of the polybutadiene which served as the basis for determining the amount of damages, but rather the production costs of the catalyst and its market value accruing during the remaining term of Ziegler’s French patent. Michelin and BF Goodrich each paid half the court costs.

The amount of damages due was established by the parties in the face of robust resistance from Michelin. Several years passed before Mr. Combaldieu – meanwhile appointed President of the Patent Office – handed in his expert report [115]. In late 1985, the court [116] ruled that Studiengesellschaft Kohle was entitled to just under 10 million French Francs. The calculation resulting in this value was initially based on the price of the catalyst components. The court subsequently established a royalty of 15% which, at 10% interest, compounded up until the end of 1984, determined the above-mentioned total amount.

The solution sought by the court represented a compromise in that the damages were to be calculated on the basis of an increased royalty premised on the value of the quantity of catalyst used, on the one hand, and the amount of polybutadiene produced with the catalyst, on the other. The compromise as indicated was based on the consideration that it was, admittedly, only Ziegler’s catalyst patent, and not the production of polybutadiene according to the BF Goodrich patent, which had been infringed, but also that the catalyst must be appraised at a greater value than merely the sum of the current market prices of the individual catalyst components. The court no doubt, had been very accommodating to Michelin. Finally, one of Europe’s biggest tire manufacturers continued to use Ziegler catalysts to fabricate top quality products in the tire field.

Looking ahead, it will be shown that the controversy between Ziegler and Phillips Petroleum in the United States ended quite differently. (cf. Chapter 5, p. 174).

22) Page 3, column 2, line 3 of the Ziegler patent:
Homogeneous polymerization catalyst.

The outcome of the action against Michelin could not be regarded as satisfactory, particularly because a few years earlier, the litigation against Phillips Petroleum in France had led to a verdict of infringement which included the Goodrich patent. An attempt to have the Appeals Court modify the decision in the Michelin case by also ruling in favor of BF Goodrich, in other words, a further appeal to the highest civil court, the Cour de Cassation, failed. In 1988, the decision of the lower court was affirmed [117].

4.6.11

Case 11

In Germany, a conflict developed in the early 1970s between Ziegler and Hoechst regarding the use of so-called “isoprenyl” aluminum as a catalyst component in the production of polyethylene. This material had figured notably in connection with US-based Du Pont during the first half of the 1960s²³). Hoechst argued that the aluminum compound which they were using did not exhibit the characteristics described in Ziegler’s patents.

The license agreement between Studiengesellschaft Kohle mbH (Ziegler) and Farbwerke Hoechst AG, that was current in 1962 [118], provided that, if a dispute arose between the parties, the case should be heard by an arbitration panel rather than a regular district court. In its Complaint [119] of July 1976, filed with such an arbitration panel, Studiengesellschaft Kohle pointed out two German patents of US-based Du Pont [120], for which Studiengesellschaft had acquired the right of ownership. The first of these patents was directed to the production of polymeric organoaluminum compounds and the second to the polymerization of olefins, utilizing, *inter alia*, the polymeric aluminum compounds of the first patent. The chemistry of these polymeric aluminum compounds and the characteristics of these products had previously been discussed in detail and were further augmented by Martin during the course of the arbitration proceedings [121].

The isoprenyl aluminum was produced by Schering in the early 1970s pursuant to the teaching in the first above-mentioned patent. This production was licensed under an agreement [122] entered into with Studiengesellschaft Kohle in 1957, and any sales were subject to royalty payments. Hoechst was the main purchaser of this product, which was being utilized in the production of polyethylene as disclosed in the second of the above-mentioned patents. Thus, the basis for an infringement action against Hoechst had been established.

Based on the annual royalties which Hoechst had paid Studiengesellschaft Kohle for the production of polyethylene in the late 1960s, it was now possible to estimate the royalties currently due and owing for the period 1971 through 1976 (expiration of the two Du Pont/Ziegler patents) and project that Studiengesellschaft was entitled to payment of approximately 18 million Deutsch Marks in back royalties from Hoechst. For the next 9 years, the arbitration panel (three arbitrators) convened several times a year, presided by retired Chief Justice of the Appellate Court Duessel-

23) See Chapter 3, p. 121.

dorf, Heinz Winkler. In 1977 Professor Dr. H. J. Sinn of the University of Hamburg, after approval by the arbitration panel, agreed to render an independent expert opinion, as had been requested by the Chief Arbitrator, and to respond to the panel's questions. According to this expert opinion, the products which Schering delivered to Hoechst, identified as "IPRA" (isoprenyl aluminum), were highly viscous, honey-like substances, containing several aluminum atoms in each molecule (polymer), exhibiting improved resistance to hydrolysis, and in which the presence of unsaturated moieties could be demonstrated, all of which were characteristics described in the first Ziegler/Du Pont patent. The products could be manufactured following the disclosure of the above-mentioned Du Pont/Ziegler patent. An expert opinion of more than 100 pages long [123], was presented to the arbitration panel, and in 1980 was discussed on several occasions before both the panel and the parties' representatives. Under consideration of the findings submitted in the expert opinion, both parties filed petitions to the arbitration panel in early 1981. A further deadline which the Chief Arbitrator proposed for March of 1981, was, however, not observed by the parties, because parallel ongoing settlement negotiations were about to be concluded [124, 125], according to which Hoechst agreed to pay Studiengesellschaft a substantial portion of the sum that had been asked for. Thus, all disputes were settled.

The results of the same chemical experiments, which in the early 1960s, had led Du Pont to acknowledge that the "isoprenylaluminum" material was an aluminum trialkyl as covered by the Du Pont/Ziegler license agreement and that use thereof without a license constituted an infringement of Ziegler's basic patent rights, now, upon supplementation of the test results, became instrumental in establishing a royalty obligation on the part of Hoechst under the longer running Du Pont patents in Germany which Ziegler had acquired.

4.7

The Split between Montecatini and Ziegler in the United States

It became obvious in the late 1960s that Natta/Montecatini were unable to prevail against Ziegler in enforcing the priority right of their first Italian application for the production of polypropylene in the United States [126] (cf. Chapter 3, p. 118). In September of 1969, the US Patent Office rendered a decision awarding priority to Ziegler²⁴). With great satisfaction, Ziegler congratulated both his US attorneys and Martin [127], now that a decade-long fight had been resolved in his favor. Montecatini had tried since mid-1968 to sever its contractual relationship with Ziegler. The Pool Agreements did not contain a termination clause. Thus, a severance agreement [128] had to be negotiated. In accordance with the objective pursued by Montedi-

24) Interference 90833 and the decision thereto [130]: "Priority of invention of the subject matter of the count at issue is awarded to Karl Ziegler, Heinz Martin, Heinz Breil and Erhard Holzkamp". The count being "The process for homopolymerizing propylene to form polymers that are solid at normal

temperatures which comprises (1) forming a catalyst by mixing a halide of a metal selected from the group consisting of titanium, vanadium and zirconium in which the metal has a valence higher than three and an aluminum alkyl and (2) only then contacting said catalyst with propylene."

son's US attorneys, they advised that all existing contractual commitments between Montedison and Ziegler be declared null and void *ab initio*. Ziegler vehemently opposed this. The contract [129] of July 30, 1969 initialed and sealed the split between the parties in the United States as of June 30, 1968.

Montecatini's US attorneys had insisted on this split, since they felt that any association between Montecatini and Ziegler in the United States would negatively impact on attaining the objectives they had set in terms of litigation. Numerous documents were drafted in which they accused Ziegler of fraudulent conduct during previous patent prosecution proceedings [131]. Despite Montecatini's assurance to Ziegler that, in pursuit of their "common" goal, the severance demanded by Montecatini-Montedison's US attorneys would apply only to the United States, and was immaterial in terms of the rest of the world, Ziegler protested against such promises. Montecatini followed the recommendations of their attorneys in order to have the Patent Office's decision reversed by the regular courts. It was obviously Montecatini's intention to keep this secondary law suit at least pending [132] despite the high risk factor.

Implementing the July 1969 agreement meant that the rights and obligations emanating from the joint license agreements in the United States had to be divided up. In the polypropylene field, this involved the Hercules, Dow Chemical, Shell Oil and Novamont as well as Esso, Goodrich Chemical, Du Pont contracts, while in the terpolymer field, it was the agreements with United States Rubber Corp. (Uniroyal) and Copolymer Rubber and Chemical Corp. (CRCC) which were affected, but all of them only to the extent that royalty payments had been made under these agreement. There was distribution of accrued royalties, and in terms of the future, the licensees were directed to enter into separate license agreements with Ziegler. Any royalties which had already been paid were to be retained by the recipients. Interestingly enough, it transpired that in this connection, both Uniroyal and CRCC had used only Ziegler patent rights in their production of terpolymers and, therefore, only Ziegler would have been entitled to royalty payments. Here too, Montecatini withdrew its protest actions against Ziegler concerning the Ziegler/Du Pont Terpolymer agreement.

For 15 years, from 1954 through 1969, Montecatini and Ziegler sustained with a somehow joint licensing policy. It was not by accident that Montedison's Director for Technical Development, Orsoni, sent Ziegler an antique dagger made of bronze as a birthday present [133].

Further consequences of the US Patent Office's decision were that, on the one hand, Montedison attempted, without success, to have the priority issue resolved in its favor by bringing suit in a civil court (District Court) [134] and, on the other, Ziegler obtained a US patent [135] granting him protection rights to the polymerization of propylene and higher olefins (cf. Chapter 3, p. 118).

The suit was settled in 1983 and included a second interference proceeding between Montedison and Ziegler²⁵⁾. In its declaration, Montecatini stated: "Abandon

25) Interference 99 478, count: "Method for the polymerization of alpha-olefins, which comprises contacting such *olefin* with a catalyst formed from an organometal component comprising an aluminum trialkyl and a heavy metal component comprising a

compound selected from the group consisting of salts and the freshly precipitated oxides and hydroxides of metals from the Groups IV-B, V-B and uranium, and recovering the high-molecular weight polymer formed."

the contest as to all counts ...,” “such dismissal is not a concession of priority by either party to the other ... and further ... is not an admission of misconduct ... by either party ...”. In compliance with the settlement terms, Montedison paid 1 million US dollars “in full payment for all past royalties from Montedison that were due to SGK (Studiengesellschaft Kohle) under all of SGK’s presently issued US patents” [136]. The amount of damages paid by Montedison to Ziegler/Studiengesellschaft was more symbolic than representative of the total monies collected by Montedison up to that point in time²⁶⁾.

So much for the clarification of the priority issue regarding the process claims for the production of polyolefins as between Natta and Ziegler. Starting in the early 1970s and up to the expiration of the US patent rights, Studiengesellschaft Kohle granted licenses for the production of polyolefins on a broad basis (cf. Table 4.1). But in terms of the product claims, that is, the protection of the new “polypropylene” product itself, the picture was also changing.

Montecatini’s US product patents issued in 1963, claiming “isotactic polypropylene”, had been challenged in the US Patent Office in an interference [137]²⁷⁾ and was followed by a civil court case brought by Phillips Petroleum, Du Pont, Standard Oil of Indiana and Hercules (cf. Chapter 1, p. 32; Chapter 5, footnote 1). The reason for this was that the aforementioned parties believed that that these patents had been granted in error and that their own priority dates preceded those of Natta [138]. Ziegler was not a party to this interference. The product claims covering Ziegler catalysts appeared to be sufficiently adequate to enforce Ziegler’s interests.

4.8 Review

The observer of these events would be justified in having a deep sense of satisfaction with their outcome even in the present day. An ideal situation had evolved for all concerned – inventors, patent attorneys, licensing attorneys and marketing personnel, namely, the monopoly of an invention, the reassuring ownership of a package of issued patent rights worldwide, revenue from numerous license agreements in the form of fruitful and expanding industrial exploitation, and an uninterrupted boom of requests by prospective licensees, plus an unlimited variety of research activities and, as the crowning event, the award of the Nobel Prize to Ziegler.

The strain of the initial infringement actions in Europe, and the combative rivalry among the licensees over differences in license terms, seemed to pale into insignificance in comparison with the outcome.

Finally, the 1960–1970 decade drew to an auspicious close as the battle between Natta and Ziegler for priority regarding polypropylene, at least in the United States –

26) See Chapter 3, Section 3.5.1.

27) Claim: “Normally solid polypropylene, consisting essentially of recurring propylene

units, having a substantial crystalline polypropylene content.” (see Chapter 1, p. 1, part 3).

the only country where it had become subject to adjudication by a court of law – was decided in Ziegler's favor.

Subsequent publications addressing these events always suppressed these facts, whether due to lack of awareness regarding the outcome of the litigation or for other reasons remains a mystery. According to the prevailing opinion – which could not be refuted as a fact – Natta had produced solid, crystalline and thermoplastic polypropylene prior to Martin. However, the fact that Natta had previously acquired information regarding the composition of the catalyst from Mülheim was of primary importance in the determination of the priority dates by courts of law. With respect to the experimental results obtained by Natta, Chini, Pino and Mazzanti, on the one hand, and Ziegler and Martin, on the other, no exchange of information had taken place prior to August, 1954. The lawful combination of the priority dates and disclosures of the corresponding patent applications for the Ziegler catalyst and production of polypropylene at the Max-Planck-Institute was unbeatable. Thus, the contribution by Ziegler and his co-workers was clearly not limited to the discovery of the Ziegler catalysts and their utilization for the polymerization of ethylene into linear, crystalline, high density polymers. It was, in particular, the ensuing victorious fight and the dispute with Natta and Montecatini that constituted an important part of Ziegler's life and work. While the extraordinary financial success enjoyed by the Institute up to that time was remarkable in and of itself, an added impulse, effective for the next 25 years, was provided by the resolution of the problem situation between Natta/Montecatini and Ziegler in the United States. Ziegler and Studiengesellschaft Kohle had regained their free hand.

There remains to be mentioned that, in 1969, Ziegler was retired from his position as Director of the Institute, but not as Manager of Studiengesellschaft Kohle. Pursuant to the by-laws, the new Director of the Institute, G. Wilke, became new manager of the Studiengesellschaft. Martin was appointed Manager in 1970.

All seemed well with the world of polyolefins. However, in the United States at this time, there were at least three industrial plants producing polypropylene which belonged to companies that neither owned nor desired to obtain a license from Ziegler. They were Phillips Petroleum, Eastman Kodak and Rexall (later Dart).

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5

The American Challenge

It has been mentioned previously that the statutory life of patents was regulated differently in the US and Germany. The 17-year effective term of patents granted in the United States began on the date on which the patent issued, rather than on the date on which the application was filed in the Patent Office. If the period between the filing of an application and the patent issue date was lengthy – as in the instant cases – then the industry could go on producing, free from challenge, even without a license, up to the date of patent issuance, albeit not without risk. Even after relevant patents had been issued to Ziegler, some production plants continued to operate without a license. The producers insisted on first examining the language and breadth of the patent rights granted before acknowledging any obligation to pay royalties. The climate turned increasingly bitter. Licensees recalled the steep down-payments they had made years earlier and aimed for an amortization as early as possible. The era of selling monopoly rights to prospective US licensees at high prices gave way to demands by licensees to stop unlicensed competitors.

Not only did the different statutory regulations governing the term of the life time of a patent cause a shift in the US licensing business, but also the application of Anglo-Saxon law demanded a rethinking of strategy and counseling from American attorneys. Beginning in 1963 – the year that the first US patent was issued to Ziegler – until 1995 when the last Ziegler patent utilized by polymer-producers expired, one dispute followed another, predominantly in the polypropylene field, which, in certain cases, dragged on for well over 10 years until a decision was reached. Ziegler and Studiengesellschaft found themselves exposed to endless confrontations with unlicensed producers. It was only the immensity of the US market which caused Ziegler and Martin as representatives of the Institute, and A. von Kreisler, A. von Kreisler, Jr., R. Dinklage, A. Sprung and N. Kramer as attorneys acting on behalf of the Institute to name only the most intimately involved individuals, to persevere with these battles for these many years.

The time and money necessary to secure royalty income and develop new sources in the United States were considerably more substantial than that expended in Europe. Additionally, as compared to Europe, a markedly different mind-set prevailed in the conduct of American business activities and practice of the legal system – a mixture of New England Pilgrims' piety – a guarantee for fair and ethical conduct – and Wild West readiness to play poker – a guarantee of single-minded avarice. This

conduct was fuelled further by the underlying arrogance of the wealthy Americans which led them to believe that they were superior to a German university professor, whose titles (Herr Doctor, Professor, Director) became the butt of jokes and whose presumed naivety in business matters could be readily exploited to the Americans' advantage.

5.1

Hercules, Esso, Phillips, and Dart seek Advantages

Hercules Powder had launched its production of Ziegler polyethylene and Ziegler polypropylene in 1958 and had paid the royalties due to Ziegler under the contract into an escrow account, since it was, after all, not until late 1963 that Ziegler's first US patent was issued and Hercules' royalty obligation was contingent upon the issuance of a first patent whose subject matter was being utilized in their production processes. An attempt by Ziegler, through his attorney von Kreisler, to have at least partial payments released to him at an earlier date was unsuccessful [1]. Even a veiled threat to arrange for importation into the United States of Ziegler polyethylene produced by Mitsui in Japan initially had no effect [2]. Now, however, royalties began being accounted to Ziegler for product sales in countries where patent rights had already been issued – a partial success at least [3]. In late 1962, Hercules made a lump-sum settlement offer for polyethylene in the amount of 1 million dollars. Hercules had decided to concentrate on the production of polypropylene alone. Based on the remaining effective period of the polyethylene agreement up until 1971, and the established production figures, it was possible to approximate the value of the license and to make a counterproposal in the amount of 1.5 million dollars [4]. The parties settled at 1.2 million dollars, whereby the additional accounting for export production would be retained [5].

In 1963, a US product claims for "isotactic polypropylene" was issued to Montecatini¹⁾. In the Spring of 1964, Hercules dissolved the escrow account and paid the accrued royalties (1957–1963) to Ziegler [6]. Immediately thereafter, Hercules negotiated a polypropylene license agreement with Montecatini, which, in conjunction with its contract with Ziegler, was to guarantee unchallenged production. Given the

1) US PS 3,112,300 and 3, 112,301 (see Chapter 4, p. 139 and references [13, 14]). Parallel to the issuance of the patents the US Patent Office had started an Interference procedure (No. 89634, Chapter 4, p. 159 and reference [137]), during which five parties claimed priority for the product, "Normally solid polypropylene, consisting essentially of recurring propylene units, having a substantial crystalline polypropylene content." Finally in 1971 the US Patent Office decided in favor of Natta. The victory did not last very long. The defeated parties: Standard Oil of Indiana,

Phillips Petroleum, Du Pont and Hercules (Ziegler did not take part) appealed the decision with a Civil Action (US District Court of the District of Delaware, Civil Action 4319, see Chapter 1, Section 1.1 and reference [3]). In 1980/81 the final decision was made in favor of Phillips Petroleum. A substantial effort was made to push the action forward between 1971 and 1981 so that Montecatini's success of the issued patent rights was only limited. At the time of the decision in 1981 the two patents had expired.

prevailing legal positions of the parties at the time (*inter alia*, Pool Agreements), Montecatini managed to obtain payment of enormous royalties, based on a scale between 5 and 3.5 % of the net sales price. In parallel negotiations with Ziegler, amidst harsh confrontations, the royalties were reduced to a scale of 1.2, 0.9 and 0.6 %, whereby a combined value of 0.73 % was to be the lower limit. To balance this concession, the hitherto limited payment period of 15 years, beginning at the time of the first sales, was cancelled and the ceiling on the production capacity was also dropped, that is, Hercules had to continue its payments up to the time of patent expiration, albeit at comparatively lower rates than other licensees [7]. The quantity of polypropylene sold during the first quarter of 1967 reached 40 million pounds ²⁾, while the amount exported during 1967 was 7 million pounds [8].

Montecatini conducted similar negotiations with Esso Research and Engineering Company in Linden, New Jersey. Despite Montecatini's undertaking to Ziegler to grant single licenses in the United States to among others, Esso Research (Pool Agreement of January 24, 1956), the negotiations broke down after 2 years due to the exorbitant demands made by Montecatini. In late 1965, Montecatini brought a suit against Esso Research [9] alleging infringement of the polypropylene product claims [10] by Esso's subsidiary Humble Oil. Moreover, the catalyst which Humble Oil used for its propylene polymerization was a mixture of the type protected under Ziegler patent rights [11].

Esso suspended payments to Ziegler. It was entitled to do this in the event that a third party filed a suit for infringement of a third-party patent³⁾. This gave rise to a bizarre situation. In the Pool Agreements, Ziegler had undertaken to share the costs of defending any patent rights covered by the Pool⁴⁾. Thus, Ziegler paid part of the expenses of both parties to the Montecatini v. Esso suit, without himself being a party to it.

5.1.1

Infringement Action Ziegler versus Phillips Petroleum

Throughout 1966, Montecatini put pressure on Ziegler to file his own suit [12]. The chosen target was Phillips Petroleum because this company had used Ziegler catalysts without a license not only once, but on two separate occasions: for the production of polybutadiene using titanium tetrachloride/aluminum triethyl catalysts – with the addition of elemental iodide – and for the production of polypropylene using titanium halide/diethyl aluminum chloride – the titanium compound employed was titanium trichloride ⁵⁾. The license offers [13] that Ziegler extended to Phillips Petroleum through von Kreisler were rejected. Phillips took the uncompromising position that it was not infringing [14] any of Ziegler's patent rights, since

2) First quarter of 1969, 58 million pounds;
fourth quarter of 1969, 68 million pounds.

3) Licence Agreement Esso/Ziegler 1956, par. XI.

4) Pool Agreement Aug. 27, 1955, par. 11.

5) Phillips used a $\text{TiCl}_3\text{-}1/3 \text{ AlCl}_3$ complex which was formed by the reduction of TiCl_4 with aluminum powder (Stauffer).

it was not using the catalyst system covered by Ziegler's patent [15]. Even a proposed meeting in New York was rejected as futile by A. Young of Phillips. Phillips had already decided not to take a license.

Pointing out the favorable outcome of the court actions in France made no impression on Phillips⁶). In 1967, a Complaint was filed against Phillips Petroleum in the District Court for the Northern District of Texas in Dallas, where the production plant was located [16].

Any chemist studying the opponent's arguments would have been dumbfounded. Here was Phillips Petroleum boldly claiming that there were no instances in which they used a Ziegler catalyst, despite the fact that on the one hand, butadiene was being polymerized with a catalyst consisting of aluminum triethyl and titanium tetrachloride (US Patent 3,257,332; the '332 patent, cf. Chapter 3, reference [66]) "improved" by the addition of elemental iodine, and, that on the other hand, propylene was being polymerized with a modified catalyst consisting of diethyl aluminum chloride and titanium trichloride (US Patent 3,113,115; the '115 patent, Chapter 3, reference [54]), which contained aluminum trichloride left over from its preparation. Though numerous further arguments were advanced, they added but little new substance. In the final analysis, the aim was to challenge the validity of the Ziegler patent rights.

The rules of court procedure in the United States are different from those in Germany in terms of their procedural features. Although the judge assigned to the case issues a preliminary pre-trial and trial schedule after conferring with the parties, the proposed dates may be postponed or changed several times.

On the other hand, during the pre-trial, known as the "discovery" period, the parties question each other in the form of written interrogatories, about the minutest technical, contractual and chemical details, in addition to their lines of argument and underlying rationale. The parties are obliged to produce to the opposing party any and all documents that have been designated as relevant. In the instant case, there were thousands. With each new litigation the number of documents would naturally escalate, since not only would the parties in support of a similar line of argument, fall back on documentation relied on in the previous law suit, but new arguments also needed to be documented.

The next phase calls for members of both parties to be deposed orally by their own attorney, in direct examination in the presence of the opposing attorneys, and subsequently to be cross-examined by the opposing attorney, whereby a court reporter, who has jurisdictional authority, takes down every word uttered. These proceedings may go forward at any desired location and may continue for weeks and, thus, are without restrictions on time or place, a virtual paradise for lawyers. Any attorney present in the room, even if he remains silent, earns a fee. Since highly qualified, experienced attorneys are engaged for such proceedings, the costs will skyrocket. Certainly, each party's attorney had to make sure that the rules were being observed during cross-examination so as to protect his own witness. Thus, numerous ques-

6) See Chapter 4, p. 150

tions would be posed during cross-examination aiming to elicit more information than permitted by law.

The whole procedure is then repeated in court one more time, since the judge will need to read not only both parties' arguments (pre-trial brief), but also to hear them spoken. Again, each word is taken down by the court reporter and two or three hours after adjournment at the end of the day, the daily trial transcript is delivered to the attorneys' hotel room. There, the transcript is carefully scrutinized and questions and/or explanatory notes prepared for cross-examination the following day. Getting to bed before midnight was out of the question.

Each party engages at least one expert, who, under oath, testifies to the technical aspects of the party's position in the case. A question arises as to how it would be possible for a judge to prevent perjury in connection with the testimony given by both parties' sworn experts. Either he interrupts the testimony, or any false testimony goes unpunished. Here, the judge must rely on his tact and sensitivity.

In the case at hand, the discovery period, up to the actual trial before the court, took $2\frac{1}{2}$ years to complete, a relatively short time when compared to the court battles that were to follow during the next few years. In May of 1970, Judge Sarah Hughes⁷⁾, an amiable lady well into her seventies, presided over the proceedings. Hermann F. Mark⁸⁾ the "Polymer Pope" from Brooklyn, New York, presented convincing arguments as Ziegler's expert witness. It was, however, due to the skill of the opposing attorney that a lay person with little knowledge of chemistry, such as the Judge, was able to accept the attorney's self-serving explanation of the "fundamentally" different nature of the Phillips catalyst as compared to the range of the Ziegler catalyst and thus to be convinced that Phillips had something "entirely new" in its hands. It was important to the Judge to collect the evidence in a very meticulous manner, being well aware that, given the momentous consequences of the decision, the losing party would file an appeal.

7) The judge had become known to the public 8 years earlier, when after the assassination of president J. F. Kennedy she took the oath from the successor L.B. Johnson.

8) Professor Dr. H. F. Mark, Director of the Polytechnic Institute, Brooklyn, New York.

On June 22, 1971, she rendered a decision [17], declaring both of the Ziegler patents involved in the litigation “good and valid in law”, but finding neither of these patents infringed by Phillips Petroleum. In her Opinion, she acknowledged the importance of the patents and held that Phillips Petroleum had failed to convincingly demonstrate that the disclosure of the two Ziegler patents would have been obvious from any prior art publication.⁹⁾ US Patent 3,257,332 (the ‘332 patent), the court continued, was, however, limited to the polymerization of ethylene and was, therefore, not broad enough to encompass a catalyst system such as that used at the polybutadiene plant at Borger (Phillips). And further, to interpret US Patent 3,113,115 (the ‘115 patent) as covering also polypropylene would mean exceeding the scope of the Ziegler invention.

The Judge recognized and confirmed the overall significance of the Ziegler catalysts as well as their commercial success, including the opening up of an entirely new field in chemistry, but she nevertheless strictly held to the language of the Ziegler patents being litigated and adopted the position that, according to the ‘332 patent, the catalyst was prepared from two components rather than three, and had been formed prior to coming into contact with the ethylene, whereas the production of polybutadiene rubber as carried out by Phillips Petroleum involved the use of a

9) Essentially three publications were used by Phillips to disqualify the Ziegler patent rights. The first citation was the well-known patent of Max Fischer, DE PS 874 215 (see Chapter 1, p. 6/7, p. 18 and references [30, 136]; chapter 3, reference [5], and p. 90 and p. 103; and Chapter 4, p. 154). The arguments used in the German prosecution were that under the conditions described in the Fischer patent (catalyst: $\text{Al} + \text{AlCl}_3 + \text{TiCl}_4$) aluminum triethyl, the now claimed Ziegler-catalyst, should have been formed. Phillips cited the book by Gaylord and Mark [18] in which the authors (H.F. Mark was now an expert witness for Ziegler, see above) wrote: “This process appears to contain the necessary ingredients for the in situ preparation of a Ziegler catalyst and undoubtedly involves the formation of aluminum triethyl by the reaction at elevated temperatures and under pressure of ethylene and the powdered aluminum. The aluminum alkyl then reacts with the titanium tetrachloride in the usual man-

ner”. This would have been a deadly “own goal” had H.F. Mark not later revoked this entire passage in his publication in front of the judge when he was giving his testimony [19]. Using the amounts of titanium tetrachloride, aluminum trichloride and aluminum powder described by Fischer neither aluminum triethyl nor diethyl aluminum chloride could be formed. The possible presence of ethyl aluminum dichloride was not proven. The second and third citations were two US patents of 1936/1942. These by A.J. van Peski [20] and V. Ipatieff [21] described so-called alkylation reactions by using catalysts which could theoretically include combinations of Ziegler catalysts in a limited manner. Nothing was said about any polymerization reactions involving those catalysts. As a witness Mark offered calculations of how many of the described catalyst combinations could be included in the limits of the Ziegler patent ‘115. The calculation convinced the judge as described above [22].

three-component catalyst¹⁰), formed *in situ* in the presence of the butadiene to be polymerized.

The addition of iodine as a third component¹⁰) was necessary in order to control the polymerization process so as to selectively produce *cis*-1,4-polybutadiene. The fact that, at that point in time, the iodine had already undergone reaction with the aluminum triethyl and, thus, was no longer present as a free component was beyond the Judge's comprehension.

The court went on to say that although the '115 patent was generally directed to polymerization catalysts, the specification only mentioned the polymerization of ethylene or co-polymers of more ethylene and small amounts of propylene. Here again, contact with the monomer did not occur until after the catalyst had been preformed from two components. Moreover, there was no mention made in the patent of titanium trichloride, much less a complex of titanium chloride/aluminum trichloride. The latter, according to the court, was not the equivalent of titanium tetrachloride. The Judge also failed to be impressed by the fact that one of the two Phillips polypropylene plants (Monument, at La Porte, Texas¹¹) had been taken over by Diamond Shamrock in 1967, i.e. prior to the start of the litigation, and that, in 1970, Diamond Shamrock had deemed it necessary to obtain a license from Ziegler [23] to operate the plant, at a time when the Dallas decision had not yet been handed down. Phillips Petroleum's second plant (Adams Terminal) was operated with the same catalysts, using the same technology.

The shock caused by this decision was deep-seated, and Ziegler was afraid that his profitable licensing business had come to an end. None of his licensees would pay another penny under the existing license agreements. Given the nature of the court's Opinion, one thing was certain to happen: the judgment had to be appealed [24].

10) Depositions by H. Martin, and H.F. Mark, in May 1971 [25]: From experiments carried out by Martin it was concluded that iodine (also in the form of diiodine butene by the reaction of iodine and butadiene) under the conditions used in Phillips' technical plant in Borger, does react with AlEt_3 in few seconds to form Et_2AlI and EtI , and this before the reaction mixture makes contact with TiCl_4 . (Iodine does not react with TiCl_4 .) The use of AlEt_3 in excess reduces TiCl_4 in 30–40s at 5 °C, faster than Et_2AlI (in this case 20% of the TiCl_4 had not yet reacted after 3.5 min). From 6 AlEt_3 per 1 TiCl_4 , originally entered into the reaction, 3–4 EtAl_3 were not used up (1 AlEt_3 reacted with iodine, while 1.5 AlEt_3 reacted with TiCl_4). Elemental iodine or TiI_4 show the same effect in selectively forming *cis*-1,4-polybutadiene (percentage).

11) Phillips' subsidiary Alumo Corp. (50/50 Phillips/National Distillers), ran this plant during the period 1963–1968, after which time Alumo was liquidated. Diamond Shamrock decided to enter the polypropylene business with the aid of Phillips by using an hold harmless agreement. Nevertheless Diamond Shamrock had no right to reclaim prepayments to Ziegler from Phillips, as the license agreement was signed without the consent of Phillips. The agreement between Ziegler and Diamond Shamrock was the first of a number of so-called "standard polypropylene license agreements" which expressed harmonization of the license conditions, specifically for Ziegler's US licensees.

Two years later, in April of 1973, the Court of Appeals in New Orleans rendered a decision [26] affirming Sarah Hughes' ruling on polybutadiene, but overturning it on polypropylene, that is, Ziegler's '115 patent was found to have been infringed. The validity of both patents was also upheld. Certain passages from the 44-page Opinion are noteworthy and are briefly set forth below. To begin with, the Judge categorized the '115 patent as a "pioneer patent."

*Under the doctrine of equivalents, the broadest protection is reserved for "pioneer" or "generic" patents. A pioneer patent is "a patent covering a function never before performed, a wholly novel device, or one of such novelty and importance as to mark a distinct step in the progress of the art, as distinguished from a mere improvement or perfection of what had gone before."*¹²⁾

Following this definition, the Judges concluded that the specification of the '115 patent was not limited to use of the catalysts for a single monomer, ethylene, and definitely included the polymerization of propylene. Furthermore, there was no doubt that the patent encompassed the use of titanium trichloride¹²⁾. Titanium trichloride, the court noted, was a titanium salt within the meaning of the definition set forth in the patent. Nowhere, however, was the addition of aluminum trichloride described as fundamentally altering the polymerizing action of the Ziegler components:

"Here, the evidence is clear that Phillips is an infringing improver".

The Ziegler examples, the court held, did not exclude the possibility of the Ziegler catalyst components being admixed in the presence of the monomer to be polymerized. The '115 patent encompassed both methods.

The evaluation of the '332 patent was very different. The opinion was that it was not a pioneer patent, and that there was no convincing evidence that the catalyst components claimed had, indeed, been present in the Phillips process. The presence of iodine caused a complicated series of reactions, and, finally, the polymerization of butadiene was not disclosed, nor had it been envisioned in the '332 patent. Rather, the '332 patent described a catalyst system which was useful exclusively for the polymerization of ethylene. The catalyst product claims were not sufficient in this case to assert "all use". Moreover, testimony had shown that butadiene was very different from ethylene in terms of its chemical reactivity.

This portion of the decision is certainly unsatisfactory, because the pioneer character had originally been ascribed to the action of the aluminum trialkyl/titanium halide catalyst, while the diethylaluminum chloride/titanium halide had been seen as an additional invention. Catalyst product claims covering any desired use, without mention of such use in the specification of the patent in question, would accordingly be inconceivable.

¹²⁾ See Chapter 2, p. 68 and Chapter 3, p. 101.

Phillips paid a seven-figure dollar amount in damages for its past polypropylene production and signed a license agreement with Studiengesellschaft. After the interference decision¹³⁾ in 1969 in favor of Ziegler, Montecatini was no longer a partner in this area, i.e. production of polyolefins, and, in light of the civil suit brought by Phillips against Montecatini, was able to enforce its polypropylene product patent rights only to a limited extent.

Ziegler lost the Phillips alternative for the production of cis-1,4-polybutadiene. Unlike the situation in France, the US court gave Phillips a free hand in the production of cis-1,4-polybutadiene using titanium catalysts. The production of cis-polybutadiene¹⁴⁾ with Ziegler catalysts based on cobalt-containing catalysts had previously been discussed. Here, Goodrich-Gulf did not engage in a court battle with Ziegler. It respected the pioneer character of the Ziegler catalysts¹⁵⁾.

5.1.2

Hercules forces Ziegler into Legal Action against Dart

The decision of the Dallas trial court had not yet been handed down, when, in May of 1969, Hercules sent Ziegler a letter notifying him that Diamond Shamrock Corporation and Dart Industries were infringing Ziegler's US catalyst patent. The Head of Polymer Production, Mr. Giacco, requested that Ziegler put a stop to the infringement, as called for by the license agreement, that is, within 6 months [27]. Ziegler and his German attorney, A. von Kreisler, believed that the long-standing friendship with Hercules as one of the pioneers involved in the industrial exploitation of the Ziegler chemistry, would create enough goodwill on the part of Hercules to allow Ziegler extra time beyond the 6 months to arrive at a suitable arrangement, that is, on the one hand, to negotiate and enter into a license agreement with the alleged infringers and, on the other, to request proof of the infringement from Hercules [28].

Initially, the President of Dart Industries Inc., Mr. R.M. Knight [30], responded to von Kreisler's inquiry by pointing out that they had previously received an offer from Montecatini to acquire a license under Ziegler's US patents and had declined the offer, which meant that Dart did not need a license for its operations. He took note of the fact that Ziegler and Montecatini had severed their contractual ties and believed that a meeting with von Kreisler was not necessary. Von Kreisler persevered and demanded a detailed explanation as to why the catalyst used by Dart was considered to be independent [31]. Meanwhile Hercules extended the period agreed upon [32]. In January of 1970, von Kreisler requested a further extension, Hercules accepted [33], and in June, long after the granted extension had expired, Hercules provided the samples from Diamond Shamrock and Dart's operations [34] which Sprung had requested on Ziegler's behalf.

13) Chapter 4, Section 4.7.

14) See Chapter 2, p. 79.

15) See Chapter 2, p. 75, see also agreement with Goodrich Gulf "Basis of Agreement", Aug. 06,

1958, article II, page 6: "Goodrich Gulf recognizes that operation under its patent rights ... is not possible independently of Ziegler's patent rights" [29].

The results of an analysis of the polypropylene samples, carried out by Gesellschaft für Kernforschung GmbH in Karlsruhe, at Ziegler's request, revealed¹⁶⁾ that in Dart's sample, aluminum and titanium, in addition to other elements such as chlorine, bromine, iodine, sodium, manganese, vanadium and copper, were found only in trace amounts, while as expected, in the Diamond Shamrock sample, titanium and aluminum were detected in amounts which were substantially larger by comparison – an ambiguous result with respect to Hercules' allegation [35].

In September of 1970, Hercules discontinued payment. Ziegler objected and proposed an amicable discussion to resolve any unsettled issues [36]. The problem of Diamond Shamrock's infringement had meanwhile been resolved by the latter signing a license agreement [37].

Ziegler filed a complaint against Dart in the District Court at Wilmington, Delaware, on July 29, 1970. The date took on special significance because Dart attempted to bring a suit against Ziegler in California in order to safeguard the "California" venue for itself [38]. The rules of procedure, however, required, *inter alia*, that a copy of the written Complaint be handed to the defendant in person. Ziegler avoided receiving the Complaint by taking a non-stop flight to Hawaii for a vacation. The process server, attorney Helmut Mewes of Düsseldorf, rang the bell at the Ziegler's garden gate in vain. Dart then tried to prove that Ziegler was conducting business in California, a further prerequisite for shifting the venue to California. However, no relevant business dealings could be attributed to Ziegler in California.

In late 1970, Judge Wright ordered the case to be tried in Wilmington [39].

Since the subject matter of the litigation was practically the same as that tried in Ziegler's suit against Phillips, the parties, after being faced with the unfavorable Dallas decision in the Ziegler versus Phillips case, agreed that it would be best to await the outcome of the appeal [40] before investing any more time and money. This decision was taken in mid-1971. However, after the appeal the interests of the parties had shifted, to a position where both parties were no longer interested in settling the dispute under the changed conditions.

5.2

Between Dallas and New Orleans

When assessing the impact of the Dallas court's unfavorable decision against Ziegler, in general it would have been better to await the decision of the court of appeal in New Orleans so as to have a legally-binding judgment to work with. However, the different license agreements in the United States lent themselves, even at that point, to a case-by-case evaluation of the consequences.

One contract which proved interesting in this context involved Novamont [41] a wholly-owned subsidiary of Montecatini in the United States. Novamont had taken a license only under the '115 patent, since the company had decided that this was the only patent they would be using. The Dallas decision included, *inter alia*, a declara-

16) Neutron activation analysis.

tion that the '115 patent was valid. Whether the process used by Novamont was the equivalent of the Phillips process had yet to be determined. Should Novamont now believe that they were not utilizing the '115 patent, they had the option under the terms of the contract, to terminate the agreement. Initially, Novamont discontinued payment [42], because the court had considered use of a catalyst based on titanium trichloride beyond the scope of the '115 patent. Ziegler referred to the existing agreement [43] and cancelled in the Spring of 1972 [44].

Hercules had decided that the '115 patent covered its polypropylene production and declared that the Phillips process and its own operation were not in the least analogous. Moreover, they believed, the Phillips' decision would not trigger any repercussions until after the decision from the court of appeal became available. Finally – in the event that the Dallas decision was affirmed – they should then check if perhaps any other claims of any Ziegler US patents or patent applications were being infringed. The issue as to whether or not Hercules was entitled to discontinue royalty payments in light of the Dart action remained unresolved.

Ziegler's US attorneys recommended that he cancel the Hercules agreement [45]. Ziegler hesitated and did not wish to make a decision during his August vacation [46]. On his return, he asked von Kreisler to approach Hercules' President, Mr. W. C. Brown, who offered to negotiate so that the parties could arrive at a compromise [47]. At a meeting between Arnold Sprung and Giacco of Hercules, arguments were being exchanged back and forth. For the first time, a "paid-up license" for polyolefins was discussed. Hercules was aware that, in the event of Ziegler winning the appeal, they would be in a poor position if Ziegler had previously cancelled the existing agreement [48]. Giacco made an initial offer proposing a 1.25-million dollar [49] lump-sum settlement payment for polypropylene and co-polymers produced up to December 3, 1980 (the expiration date of the '115 patent) for amounts up to 600 million pounds per year, 1 % royalty for amounts in excess thereof, and 0.75 % for export. For the period after 1980, Hercules requested an option under terms no less favorable than those granted to other paying licensees.

Meanwhile, Ziegler had set sail on a cruise around the world. Hercules supplied its most recent production figures, which made it possible to calculate a settlement figure of approximately 1.7 million dollars to be demanded for the time period in question up until 1980. Ziegler now wished to discuss details and asked Martin to meet him in Hong Kong at the end of February to talk over any unresolved issues [50]. During the next leg of Ziegler's journey, from Hong Kong to Singapore, he and Martin planned their future strategy. Martin met Sprung in New York in early March and, together with Sprung, embarked on negotiations at Hercules in Wilmington, Delaware. The parties reached an agreement. The settlement figure was to be 1.6 million dollars. Ziegler was relieved [51]. In late April 1972, the arrangement was formalized in a letter agreement [52].

Shell had taken a license under a patent package. Royalty payments were, however, limited to the '115 patent as applied to the process Shell used for its polypropylene production. In light of the Dallas decision, Ziegler anticipated that Shell would push for a reduction in royalties.

Shell planned to stop royalty payments altogether. Ziegler threatened to terminate the agreement [53], and in March of 1972, Sprung, acting on Ziegler's behalf, declared the contract cancelled [54]. Shell was disturbed at this and expressed a desire for an amicable solution [55]. Sprung proposed, while withdrawing the cancellation, that Shell pay a reduced royalty up to the time that the appeal court rendered its decision [56]. In mid-1972, Shell and Ziegler signed an agreement [57], under which Shell would now pay only one-eighth of any royalties due.

Shell, wanting to exploit the situation, attempted to gain a further financial advantage by having their license converted to a paid-up contract at a low lump-sum settlement figure and thus to obtain the highly favorable settlement terms granted to Hercules, but they were unsuccessful in their attempt [58]. The settlement amounts required from Shell, for a production capacity of 3 million pounds per year, were between 2.3 and 2.5 million dollars depending on the average royalty scale to be applied [59].

After the Court of Appeals for the Fifth Circuit in New Orleans had rendered its favorable decision in April of 1973, Shell continued payment under the so-called "Standard Polypropylene License Agreement". The nail-biting was over.

Esso's conduct before and after the Dallas decision remained unchanged. They believed that under the terms of the 1955 license agreement, they were allowed to produce polypropylene free from any royalty obligation to Ziegler, because they had been sued by Montecatini for infringement of Montecatini's "Isotactic Polypropylene" product patent¹⁷. In their opinion, the Dallas decision, if anything, would bolster their argument in support of non-payment. But they were also not paying royalties for the production of a terpolymer rubber made of ethylene, propylene and diene, since in that case, too, they had been sued by Hercules for infringement of a patent Hercules had acquired from Dunlop¹⁸.

Royalty payments emanated only from the production and sale of an ethylene/propylene co-polymer rubber. In their accounting for propylene – accountings were obligatory – they eventually deducted payments to Hercules for the use of Hercules' Vandenberg patent (Chapter 4, reference [8]). Clearly, a conflict situation of major proportions was about to arise here.

Dow Chemical was practically exclusively concerned with the production of polyethylene, so that the Dallas decision did not impact on their operation.

While Diamond Shamrock had actually taken a license and delivered the first installment of the down-payment, now, after the Dallas decision had been rendered, the second installment became due. Sprung reminded Diamond of the outstanding obligation and threatened to cancel the agreement [60], [61]. Ziegler felt encouraged. He authorized Sprung to terminate the agreement [62]. Diamond Shamrock faltered under the pressure and paid [63].

Analyzing [64] the licensing situation as it presented itself following the decision of the New Orleans Court of Appeal in April of 1973 created a euphoric picture in

¹⁷) See p. 169.

¹⁸) Chapter 4, p. 141 and reference [25].

terms of future developments in the polypropylene field¹⁹⁾. First, however, the close of the formal proceedings in the Phillips suit was still pending: the Court of Appeals, in its decision, remanded the case to the District Court in Dallas which was to address the issue of damages. Prior to that, Phillips tried to have the Supreme Court overturn the judgment, but to no avail.

The Diamond Shamrock/Ziegler agreement was cancelled for the second time [65]. One year after the decision on appeal, Diamond Shamrock had still not resumed their payments. Moreover, Diamond Shamrock had been engaged in production prior to entering into the license agreement, but had failed to render an accounting. Legally, it was Studiengesellschaft Kohle mbH, Trustee for the Max-Planck-Institute for Coal Research, which now took the place of the late Karl Ziegler, who had died in August of 1973 [66]. In May of 1974, Studiengesellschaft and Diamond Shamrock reached an agreement regarding the payment of back-royalties [67] for the period 1967 through 1970. Diamond Shamrock finally paid, after rendering a full account of its production.

Termination of the Novamont license agreement by Ziegler now proved highly beneficial, since any newly negotiated contract could include more favorable conditions for Studiengesellschaft [68]. The negotiations progressed only sluggishly, since Novamont relentlessly insisted on enforcing its demand for more attractive terms²⁰⁾. Tedious, small steps eventually led to an agreement by mid-1974, only for it to fail a week later. Studiengesellschaft authorized Sprung to withdraw the current offer, affirm Novamont's status as an unlicensed producer, and threaten to take further legal action. [69].

The period for finalizing a contract was extended to July 1, 1974. Novamont now signed a license agreement for future production and an another agreement settling the back-royalties for past production from 1971 through July 1974, whereby an interest rate of 10% sweetened the damages even more. The cancellation imposed by Ziegler was withdrawn, and the old 1967 contract replaced by the agreement of 1974 [70]. By mid-1974, Studiengesellschaft had collected more than 5 million dollars based on the decision of the New Orleans court, a situation that was as comfortable as it was necessary to meet the challenges of the future.

The New Orleans decision also provided Ziegler with a basis for challenging Esso. In March 1973, Martin and Sprung met with Mr. Chasan, Head of the Patent Department at Esso Research, in order to discuss any relevant issues. Ziegler's argument was that there was no justification either for Esso refusing to pay royalties or for reducing their royalty payments [73]. Here too, the most effective approach proved to be cancellation of the agreement [74]. Esso's management was indignant, asked for an extension of time [75], and hired an outside attorney, Mr. G. A. Burrell [76], who was to represent Esso in any future negotiations.

19) Ziegler congratulates the author on his 50th birthday: "Hopefully your successes will be as spectacular in the future as in the past."

20) Thereto Novamont had requested the extrapolation of the paid-up arrangement with Hercules to the production figures of Novamont's own production and had also

requested further license agreements between Ziegler and US licensees in order to compare the various conditions [71]. Martin refused and offered negotiations to Novamont and Montedison (Montedison supported the interest of its subsidiary Novamont [72]) in order to solve the problems.

It was not until February of 1974 that an agreement [77] was finally reached, after both parties who were always aware that infringement litigation would be the alternative, had battled with each other relentlessly. Not only did Esso pay damages in an amount considered steep even under then prevailing circumstances, but also agreed to pay any future running royalties promptly when due, whereas Studiengesellschaft undertook to accept less than the full amounts due, in view of the two ongoing court actions brought against Esso. But the agreement included safety provisions, taking effect upon conclusion of the litigations, which called for a secured portion of the withheld royalties to be surrendered. In 1975, the dispute between Esso and Montecatini was settled [78], but it was not until 1977, and even then “only under pressure from the judge” [79], but then with interest, that Esso finally dissolved the blocked account and, as required by the agreement [80], paid the surplus to Studiengesellschaft²¹⁾.

Following the decision of the Dallas District Court, Standard Oil of Indiana, later re-named Amoco Chemical, of Chicago, expressed an interest in obtaining a license for the production of polypropylene and polyethylene. Of course, they were hoping for favorable terms prompted by the Dallas decision which had been adverse to Ziegler. A licensing offer made by Ziegler in 1972 was treated with extreme caution by the Americans. Actually, Amoco had intended to combine their US interests with a solution for import rights to Germany, since Ziegler had made preparations to sue Amoco for unlicensed import into that country [81]. The dispute with respect to Germany was resolved by the parties signing a mutual agreement [82].

During a conversation with Mr. A. Gilkes (Amoco) in early 1973, it eventually became clear that Amoco’s real interest was focused on a settlement for polypropylene [83].

In the United States, the negotiations dragged on until the Spring of 1973, when Amoco, in an the eleventh-hour effort before the appeal decision (New Orleans) in the Ziegler v. Phillips matter was reached, presented an executed draft for an option and license agreement, in order to lock in favorable terms through signed and sealed documents. The option was a limited-term option, effective for up to 6 months following the Ziegler v. Phillips appeal decision [84], signaling a play-safe policy.

The cover letters were dated April 9 and April 17, 1973 [87], obviously for a good reason. The appeal decision was dated April 16, 1973. It was important to Amoco to have the option become effective immediately, but not the license agreement.

At Ziegler’s request, the negotiations were re-opened [88], thus sparking initial annoyance [89]. After the situation had calmed down, Amoco wished to obtain a license not only for polypropylene but also for polyethylene, but in any case requested confirmation that the option agreement they had signed was valid. They forwarded the option payment [90], which Martin promptly returned. Not until March of 1974 did

21) Before starting the action against Esso Studiengesellschaft had cancelled all contractual relationships with Esso. The cancellations were not revoked after the court decision so that this status still existed after Esso had made the license payments [85]. This sit-

uation was favorable to Studiengesellschaft as it enabled them to exact the excess payments from the second suit in addition to the negotiated paid-up sum. In 1979 Esso paid more than half a million dollars [86].

the parties finally meet on common ground. Studiengesellschaft offered the Standard Polypropylene License Agreement, effective as of January 1, 1973, and a Polyethylene License Agreement at a 2% royalty rate [91]. Both contracts were finalized in mid-1974, with Studiengesellschaft assuring Amoco in a separate letter, that it would waive all royalties for sales prior to 1973 [92].

Thus, the US situation appeared to be resolved to the point where royalty income from license agreements looked secure once more. However, with regard to at least one infringer, Eastman Kodak, who had kept a low profile with respect to its polypropylene operation for at least 8 years, the situation was far from settled.

5.3

Eastman Kodak

In the early 1960s, Eastman Kodak went on stream with its commercial polypropylene operation with a capital expenditure of 18 million dollars. Over the course of the next few years, the product appeared on the market and became well established through Montecatini also to Ziegler. Both in mid-1966 and late 1966, Ziegler offered Eastman a license under his '115 patent²²⁾ but Eastman refused the offer, insisting that the Eastman catalysts were outside of the scope of the '115 patent and that they did not use an alkyl aluminum halide component. A year later, Ziegler tried to sell Eastman a license under his '332 patent²³⁾. Again, Eastman refused. The same procedure was repeated in January and June of 1970. Eastman divulged no additional information. Ziegler's warning remained in effect.

On March 20, 1974, approximately 6 months after termination of the Phillips litigation, Studiengesellschaft brought suit against Eastman in Texas [93]. Eastman's polypropylene operation was located in Longview, Texas. In the view of the plaintiff, Studiengesellschaft, both Ziegler patents had been infringed.

Eastman responded by filing a declaratory judgment action in the District Court for the District of Delaware [94]. Only now did Eastman reveal the catalyst composition it was using: a suspension of titanium trichloride, admixed first with a solution of lithium butyl and then with a solution of aluminum triethyl [95]. Based on such use of lithium butyl, Arnold Sprung, on behalf of Studiengesellschaft, introduced two further Ziegler patents into the Texas proceedings [96], i.e., the '515²⁴⁾ patent and the '162²⁴⁾ patent (see Chapter 3, reference [54]).

22) US PS 3,113,115, issued 1963: Catalysts for instance, diethyl aluminum chloride and titanium halide (see Chapter 3, reference [54]).

23) US PS 3,257,332, issued 1966: Catalyst for instance, aluminum triethyl and titanium halide (see Chapter 3, reference [66]).

24) US PS 3,232,515, issued January 25, 1966: Catalyst for instance, alkali metal alkyl and titanium halide; US PS 3,392,162, issued July 09, 1968: Process for the polymerization of

ethylenically unsaturated hydrocarbons with catalysts for instance, alkali metal alkyl and titanium halide; US PS 3,826,792, issued July 30, 1974: Process for the polymerization of ethylenically unsaturated hydrocarbons with catalysts for instance, $AlEt_3$ and titanium halide (see Chapter 3, reference [54], issued on the basis of interference 90,833 which Ziegler et al. won against Montedison/Natta.

In late 1974, Studiengesellschaft was granted a process patent (the '792 patent²⁴), which, among other features, covered the polymerization of alpha-olefins – in other words, propylene – using a catalyst consisting of, among others, aluminum triethyl and titanium halide. This patent, too, was made part of the action.

Soon after the Ziegler catalysts and their use had become known in 1955, scientists at Eastman Kodak realized the significance of these catalysts. Eastman promptly started looking for catalyst systems [97], [98] which, particularly in the estimation of Eastman's attorneys [99], would not be covered by subsequently issued Ziegler patents, even though some of Eastman's personnel had expressed doubts during their deposition [100].

Thus, H.J. Hagemeyer (Eastman Kodak), Director of Research, stated:

"Our work really begins with Ziegler and his discovery that you could make high density polyethylene at low temperature using a transition element halide ..."

and

"The Ziegler discoveries were at once recognized by Kodak as probably the most significant development in the high polymer field in recent years."

The most successful catalyst system developed by Kodak appeared extreme due to the choice of starting compounds and starting conditions, yet, even so, any expert chemist would have to consider it to be dependent on Ziegler's systems: lithium butyl and aluminum triethyl admixed in equivalent ratios in a mineral oil base, together with violet titanium trichloride, free from aluminum chloride, that is, obtained by reduction of titanium tetrachloride with hydrogen, heated to 160 °C, and propylene polymerized at a pressure of 70 atm, whereby the polymer formed remained in solution. The Kodak catalyst, in their own view, was more cost effective than the previously used catalyst made from lithium aluminum hydride and titanium trichloride.

The decision handed down by Judge Fisher in 1977 was disappointing for Studiengesellschaft [101]²⁵. In his Opinion, the Judge adopted practically the entire set of facts and arguments put forward by Eastman [102].

25) The essential, for the decision important arguments were:

1. The so called Eastman-409 catalyst is prepared from three components: lithium butyl, aluminum triethyl, and "hydrogen reduced α -titanium trichloride", in a mole ratio of 0.3 to 0.3 to 1.0 (Eastman patents [97]). Ziegler's patents disclosed two-component catalysts.
2. Defense of laches and estoppel is granted, since there has been an unacceptable delay of 10 to 5 years between the time of issuance of the Ziegler patents ('115 in 1963, '515, '332, '162 in 1966 and 1968) and the actual assertion against Eastman ([101], p. 8) (6 years is the limit according to the statute of limitation).
3. Key participants are deceased (Ziegler, von Kreisler sen., Toulmin, US Attorney for Montecatini).
4. The '792 patent was prosecuted for 20 years in the patent office before it was issued, and Studiengesellschaft was responsible for at least 8 years of this unreasonable and prejudiced delay.
5. The '332 patent claims a catalyst formed from two components and is limited to the polymerization of ethylene. Polypropylene is a product having its own unique properties which distinctly differ from polyethylene (structure). Eastman produces solid highly crystalline polypropylene or co-polymers (called polyallomer). Eastman uses a catalyst composition formed by three ingredients not described in '332. There only TiCl_4 is mentioned. Eastman's " $\text{H-}\alpha\text{-TiCl}_3$ " having a new crystalline structure is insoluble in the solvent used contrary to TiCl_4 . Therefore it is not a titanium chloride and does not come under the '332 patent.
6. '332 does not disclose the "high temperature process" described by Eastman.
7. '792 discloses the limits of the molecular ratios of the aluminum compound to titanium compound in the preparation of the catalyst, as 1–12 : 1 and the upper limit of the temperature as 150 °C. These conditions are outside of the Eastman process.
8. At the beginning of this action Ziegler patents '515 and '162 were entered. These patents do not contain any examples of the polymerization of propylene. The catalysts are prepared again from two components (alkali alkyls and titanium halides). Complex compounds from lithium butyl and aluminum triethyl formed in the Eastman catalyst are excluded from the Ziegler patents '515 and '162 because of the earlier priority date of US patent 2,905,645 naming Anderson et al. of Du Pont (Aug. 1954, Ziegler Priority Dec. 1954). In addition to this fact, the claims of the patents '515, '162 and part of the claims of '792 are invalid.
9. Natta's US patent 3,582,987, Italian priority date of July 27, 1954, was filed earlier than the Ziegler priority of Aug. 03, 1954 and describes the polymerization of propylene in the same manner as in Ziegler's '792.
10. In the view of the judge, US patent 2,867,612 by Pieper et al, Bayer, German priority Oct. 08, 1954 was filed earlier than the Ziegler rights '515 and '162.

Any arguments presented by the plaintiff, Studiengesellschaft, were ignored²⁶. The team at the Longview polypropylene plant in Texas, had every reason to be pleased with the judge from Texas.

Due to the complex nature of the chemistry involved, law suits dealing with chemical patent rights were by and large unpopular with lower court judges. The judges would allow the parties great latitude in introducing their evidence into the record.

The decision was appealed, and the appeal court, the same court which had heard the Phillips Petroleum case, but with different judges presiding, rendered its decision in May of 1980 [103]. Very little was retained from the long list of arguments relied upon by Judge Fisher in support of his Opinion. In this case, the judges were required to consider only the Ziegler '332 patent (covering the aluminum triethyl/ titanium chloride catalyst) and the '792 patent (covering the polymerization of olefins, including propylene, with the use of the aforementioned catalysts) in arriving at their decision. The verdict was that the patents were valid but not infringed by Eastman.

26) Rebuttal to point 1: Two of the three components according to Eastman do react before the addition of the titanium component forming lithium aluminum triethyl butyl. The effective catalyst is formed from two components therefore.

Rebuttal to point 2: As to laches, the period of 6 years was interrupted by the litigation against Phillips 1967–1973. The validity of the '332 patent was part of the decision in this case, as did Ziegler's offer in 1970 to negotiate a license with Eastman.

Rebuttal to point 3: The deceased witnesses were deposed several times in the past by various parties. No answers during those depositions as to same questions in this case were considered by the court.

Rebuttal to point 4: The delay in the issue of the '792 patent – if Ziegler was responsible at all – was no disadvantage to the defendant because the life-time of the patent was limited to the expiration date of the '332 patent ("terminal disclaimer").

Rebuttal to point 5: The '332 and '792 patents describe identical catalysts. In addition patent '792 describes in one example a process for the production of polypropylene. The "H- α -titanium trichloride", used by Eastman is a titanium halide according to the Ziegler claims. Titanium tetrachloride is also transformed into an insoluble titanium halide during formation of the catalyst. "H- α -TiCl₃"

is produced from TiCl₄ and was bought by Eastman on the market for the purpose of producing the catalyst.

Rebuttal to points 6 and 7: The high temperature and different mole ratio of the catalyst components used in the Eastman process are included in the plaintiff's patent '792, column 3, lines 59–69, temperature "Above 250 °C is not advisable" and as to the mole ratio, in patent '792, figures 1–5 as well as Tables II–V and VII the mole ratio is under 1 : 1.

Rebuttal to point 8: Complex compounds from alkali alkyls (lithium butyl, sodium methyl) and AlEt₃ (AlMe₃) are indeed claimed in the Ziegler patent rights '332 and '792 as catalyst components. These patent rights had an earlier priority date than the Du Pont US patent 2,905,645. Disclosure of the limitations of the Ziegler claims was never requested by the Patent Office. The corresponding complex compounds are first disclosed in an experiment of Feb. 1954 (see Chapter 1, references [123, 124]).

Rebuttal to points 9 and 10: The cited patents of Natta and Du Pont are not effective prior art according to US patent law. Natta's improvement according to the patent under point 9 was that Ziegler catalysts might be more effective if the catalyst components are mixed in the presence of the olefin to be polymerized.

The Court of Appeals explicitly stated that “Construing the reach of a patent of this nature is not easy, particularly for non-scientist judges”. The judges acknowledged that pioneer patents enjoyed a broad scope of protection, but noted that Studien-gesellschaft’s patent must describe essentially the same results obtained in essentially the same way with the same function. This was, however, not the case when comparing the ’792 patent with the method practiced by Eastman. This statement specifically referred to the crystalline content present in the solid polypropylene formed. The early applications by Ziegler and his co-workers lacked such comparison figures. Only subsequent tests, using Eastman components - aluminum triethyl and titanium chloride both with and without lithium butyl - produced an equivalent crystalline content, 70%, in the solid polypropylene product [104].

No consideration was given to the fact that the ’792 patent disclosed the formation of solid polypropylene and its use for making sheets and films.

To begin with, as noted by the court, the Eastman process incorporated as a third component lithium butyl, which was not mentioned in the ’792 patent²⁷⁾. Moreover, Eastman was using a special titanium salt, “hydrogen-reduced alpha-titanium trichloride.” This, too, was not mentioned in the ’792 patent²⁸⁾. Finally, the court considered that the external conditions of temperature and relative ratios of the catalyst components fell outside the scope of the ’792 patent, since the latter described such conditions as being of low effectiveness²⁹⁾.

When comparing Eastman-Kodak’s method of polymerization in the production of polypropylene with that of Phillips – the court did not undertake such a comparison – i.e. in the case at hand, when comparing the asserted Ziegler ’332 and ’792 patents, on the one hand, with the ’115 patent, on the other, the court’s findings in the Phillips case would be equally applicable to Kodak [108]³⁰⁾.

In sum, the court held that the Eastman catalyst was not equivalent to the method taught by the ’792 patent. The court did not address the ’332 patent in any detail as

27) Thereto the plaintiff, Studiengesellschaft Kohle, had earlier argued that during the action against Phillips the same court had decided that the addition of aluminum chloride as third component – aluminum chloride forms a complex compound with titanium chloride – does not alter the fundamental polymerization effect of the catalyst according to the ’115 patent. In the Eastman process lithium butyl, the third component, forms a complex with $AlEt_3$. At 20–40 °C the complex, together with titanium trichloride, is only weakly active, and is sufficiently active in the polymerization at 160 °C. Lithium butyl alone decomposes above 80 °C and therefore would not be useful at 160 °C [105], 106].

28) In the case against Phillips Petroleum the court had decided that the ’115 claims unquestionably included $TiCl_3$. Titanium trichloride is a titanium salt in the sense of

the given definition. Yet, the definition of the titanium salt is identical in both ’115 and ’792 [107].

29) See footnote 26, “Rebuttal to points 6 and 7”.

30) The following statement appeared on page 42 of the appeal decision in the Ziegler v. Phillips appeal (it would also apply to Kodak): “The testimony is clear that the Phillips [Kodak] catalyst performs the same function (polymerization of propylene) in the same way (with a catalyst relying essentially on the Ziegler combination) but in a better fashion (more polymer and less undesirable by-products). This catalyst, though, does not avoid infringement. Without doubt, Phillips’ [Kodak’s] catalyst is an improvement, but an improver does not escape infringing the dominant patent just by improving it.”

the decision against Phillips had characterized it as limited to the polymerization of ethylene.

Two further arguments presented by Eastman were rejected by the court. The first was the allegation of laches i.e. exceeding the 6-year limit from the time that Ziegler had first become aware of the infringement by Eastman to the year in which he had taken them to court. The court considered the time limit issue to be a moot point because Ziegler had made positive overtures to Eastman (license offers), the most recent of which took place in 1970, and because he had filed a Complaint against Phillips Petroleum, since the validity of the litigated patents was expected to be determined in that case.

The second line of argument focused on two US Patents, 3,582,987 [109], with a priority date of July 27, 1954, naming the inventors as G. Natta and co-workers, i.e. it had an effective date of filing a few days earlier than the patent application by Ziegler and co-workers which was directed to the polymerization of propylene and other alpha-olefins, and 2,867,612 which was attributed to Pieper et al., assigned to Bayer AG, and had a German priority date of October 8, 1954. Here, the court followed Studiengesellschaft's reasoning that a prior art reference should become effective solely from the date on which it was filed in the US, that is, June 5, 1955 and/or October 1955, rather than from the date that any foreign priority was registered, which, in this instance, would make it irrelevant.

There would be no point in further analyzing the decision. Eastman Kodak had scored a victory, which was painful for Studiengesellschaft. The chemistry involved could be discussed *ad infinitum*. There was an extremely fine dividing line between instances where a catalyst that was being used by an infringer only just fell under any Ziegler patent rights and when it did not. The subjective review of complex chemical processes by lay judges can virtually never be objectified. It is impossible to anticipate complicated variations in catalyst production. For the Eastman catalysts to be effective, they necessarily needed to include a mixture of a Ziegler catalyst – an organoaluminum compound and a heavy metal compound, practically speaking, an organoaluminum compound and titanium halides. If any component was missing, polymerization could not be expected to occur. The addition of further ingredients served to gradually alter the characteristics of the solid polymer product obtained. The court, however, never referred to a dependent improvement of the invention. Thus, there must have been a loophole in the Patent Law that Kodak had taken advantage of.

It remains unclear whether Kodak has indeed, up to this day, carried out its operations in the professed manner; but what is certain is that no competitor, not even a potential infringer, has adopted the Kodak process to become independent of Ziegler.

Was Eastman's discovery something completely new after all? Now that the courts had affirmed that the Eastman-Kodak catalysts were independent of Ziegler's patent rights, surely, this would have guaranteed that an entirely new field of chemistry would be opened up with the help of Eastman's scientists, this is, of course, absurd. Eastman conceded that without aluminum triethyl or titanium chloride, the typical Ziegler catalyst components, polymerization would be impossible [110]. None of the

further, incredibly diverse and costly efforts undertaken by each of the parties, Eastman and Studiengesellschaft, were taken into consideration by the court of appeal in rendering its decision. Nonetheless, one or the other development and clarification would be of interest, because of their critical importance in connection with future litigations (e.g. Dart; see p. 196).

5.4

Max Fischer, Over and Over

Any defense against a charge of infringement of an issued patent most often includes a counterclaim challenging the validity of the patent being enforced.

From the very beginning, those who challenged the Ziegler patent rights had cited the BASF-owned patent, (Chapter 1, reference [30]), priority 1943, inventor Max Fischer, as a prior art reference over the invention by Ziegler and his co-workers. Yet, starting with the oppositions filed by BASF during the patent prosecution proceedings in Germany up to the final decision in the Ziegler v. Phillips case, the adjudicating panels had consistently rejected the argument.

Under the conditions of the Fischer patent, it was not possible to show the formation of organoaluminum compounds, and thus the formation of a Ziegler catalyst. Yet, the components which Fischer used for his catalyst mixture – aluminum chloride, aluminum and titanium tetrachloride – did approximate the Ziegler catalyst mixture simply because the components selected were an “aluminum compound” and titanium tetrachloride and it had been established in the state of the art literature (Max Fischer had not been aware of this literature) that Hall and Nash [111] had produced organoaluminum compounds from aluminum chloride, aluminum and ethylene. On the other hand, the amount of aluminum powder chosen by Max Fischer was so small by comparison that any organoaluminum compound formed, if at all, could certainly not have been of the type claimed by Ziegler. The fact that the catalyst according to Max Fischer was incapable of polymerizing propylene to solid polypropylene had not even been considered, even though this fact alone would have shown that Max Fischer had not synthesized any Ziegler catalysts.

Obviously, the same prior art argument cannot be repeated over and over, especially not in connection with successive litigations. It would require a fresh viewpoint, new evidence and particularly new witnesses to compel a judge to re-examine the issue.

On April 7, 1975, H. Hopff³¹⁾ gave a lecture at the Spring Conference of the American Chemical Society in Philadelphia entitled:

“Polymerization of Ethylene with Catalyst Systems of Anhydrous Aluminum Chloride, Aluminum and Titanium Tetrachloride”.

31) Professor Heinrich Hopff, the Laboratory for Organic Technical Chemistry at the “Eidgenössischen Technischen Hochschule” in Zürich (ETH), was the former leader of

polymer research at BASF, (Max Fischer was a coworker), and before that he had worked with H.F. Mark (group leader at IG Farben AG).

Hopff reported that an experiment according to Max Fischer had yielded a reaction product from which both a mixture of diethylaluminum chloride and ethylaluminum dichloride as well as pure aluminum triethyl had been distilled off. He concluded his lecture with the remark:

“These experiments show that the so-called Ziegler catalysts were formed in Max Fischer’s work 10 years prior to the sensational invention of Karl Ziegler.”

The cynicism of this statement could hardly be overstated. The lecture itself and the published articles [112] on the same subject, all failed to disclose any details of the experiment or any analytical results.

Hopff’s lecture and the two corresponding published articles were based on experimental work carried out by Hopff’s doctoral student Nikolaus Balint in 1970 [113]. As part of his dissertation, in response to suggestions made by H. Hopff in 1969/1970, Balint had conducted roughly 35 experiments, whereby only one test strictly repeated the parameters prescribed by Max Fischer. According to Balint, ethylaluminum chloride and aluminum triethyl had been distilled off during the experiment.

Herman F. Mark had been asked to lead the discussion after the lecture. He started by pointing out that the findings presented at the lecture were both unexpected and surprising and would have to be based on extremely careful experimentation [114]³². During the discussion, E. Tornqvist [115] (Exxon) asked to be heard and expressed his amazement at how it was possible for aluminum triethyl to exist in the presence of aluminum trichloride. He stated that the aluminum triethyl would have been destroyed. According to his opinion, it would be impossible to obtain aluminum triethyl. In his experience and after conducting numerous tests, he had obtained ethylaluminum dichloride only when the aluminum and aluminum trichloride had been introduced in equivalent amounts, in other words, considerably more aluminum had been present than in the Fischer example.

But if the reported results were nonetheless accurate, then the body of evidence presented would be new, unexpected and show great promise³³.

32) In November 1976, Hermann Mark (now the expert witness for the plaintiff in the *Studien-gesellschaft v. Eastman* action) was cross-examined by the legal representatives of Eastman with regard to Hopff’s lecture and the results of N. Balint. As a witness during the action against Phillips Petroleum, Mark had already stated that because of the small amount of aluminum used in the single experiment described in the Max Fischer patent, if any compound were to be formed it would only be ethyl aluminum dichloride. Comparing the Balint report with that of Martin as to the Balint experiment Mark concluded that Balint did not add any scientific work and if Hopff gave a lecture on this subject, he, Mark, had to accept that Hopff knows what he is talking about [116]. The description of the Balint experiments did not contain any analysis of aluminum, no detailed information about the apparatus used for the distillation, no repetition of the most important

experiment, although Hopff mentioned a number of repetitions. Mark doubted that the given amount of pure ethane would be formed during the hydrolysis of the so-called aluminum triethyl [117]. The only experiment, characterized as positive by Balint, test 3, was conducted between April and August 1970. The polyethylene produced by Balint had a density of 0.928. The softening point of the same material was given as 131 °C. This result differs from those of Ziegler and Phillips who produced polyethylene with a melting point of 130 °C and a density of 0.95. The Balint polyethylene would have a melting temperature of 112–115 °C [118]. Martin demonstrated that solid polyethylene produced according to Max Fischer, but using a larger amount of aluminum, had a softening point at 120 °C [119].

33) See the evaluation of the Appeal Court, Paris, action of Ziegler against Michelin 1981 (Chapter 4, p. 154, and references [112, 113]).

What motivated Hopff to take up this subject, almost 30 years after Max Fischer and 15 years after Karl Ziegler's priority?

In late 1967, initial contacts [120] were made between Kenneth Kaufman, Technical Director of the Plastics Division of US Steel, and von Kreisler, Ziegler's attorney, with the idea of US Steel obtaining a license for the production of polyethylene under Ziegler patent rights. Then, in 1968, personnel at US Steel came across BASF's Fischer patent. In late 1968, Messrs. Kaufman, Anspen and Pegan of US Steel met with Hopff to seek his assessment [121] of Max Fischer.

Hopff sent a bill for this consultation services and proposed a contractual arrangement under which, for a \$10,000–\$20,000 fee, he would conduct an experimental program for US Steel.

At a subsequent meeting [122] with von Kreisler in Cologne in early 1970, a license agreement between US Steel and Ziegler was initialed³⁴⁾. Afterwards, the visitors traveled to Zurich to meet with Hopff and discuss his proposed arrangement, which called for Hopff to determine the existence of aluminum triethyl and the amount present as well as the formation of "high-density polyethylene" when using the Fischer process [123]. Shortly afterwards, Hopff wrote a letter [124] to Anspen, of US Steel, stating that:

"First experiments of polymerization of ethylene with anhydrous aluminum chloride and titanium chloride according [to] Max Fischer patent were successful. AlEt₃ formed and can be isolated in a pure state, contrary to Hall"³⁵⁾.

Anspen quickly flew to Zurich and offered Hopff \$500 per month up to a total of \$12,000, with the proviso, however, that any publication be subject to approval by US Steel [125]. Hopff refused, but visited US Steel in November of 1970. There, his report was discussed, and Hopff mentioned, among other things, that after listening to a lecture given by Ziegler in Zurich in 1955 or 1956, he himself had repeated the Example of the Fischer patent but had failed to find any aluminum triethyl.

By the end of 1972, Hopff wanted to publish the findings. Pegan [126] promptly went to see him in Zurich, approved the publication of Balint's findings, and signed an agreement with Hopff, effective as of January 1, 1973. The first payment under the contract was not made until July of 1974. US Steel insisted, however, that its financial support be kept confidential.

34) The final signature on this an agreement was delayed by US Steel's expectation that they would sign a Ziegler license directly or that this would be achieved via Solvay. The final agreement was concluded in November 1970 [127] (see also Chapter 4, p. 149). In view of the citation of M. Fischer and the results of

Hopff, US Steel expected to receive specific favorable conditions in the agreement. Studiengesellschaft refused, but offered to repeat experiments according to Balint in the presence of representatives of both parties [128]. US Steel were reluctant to agree to this.

35) Hall and Nash, see reference [111].

It has been unequivocally shown that the experimental results compiled in Balint's final report in late 1970 were incorrect and replete with inaccurate conclusions³⁶⁾. What's more, they completely contradicted earlier findings published by other authors³⁷⁾.

36) In his report of 1970, and in his capacity as a witness, Balint had stated the following [129] (Hopff successfully avoided examination in court):

1. At this time Balint had been employed by Amoco Chemicals in the Naperville Research Center since October 1971. Prior to this time, from 1966 to September 1971 he had prepared his doctoral work and was an assistant to Professor Hopff in Zürich [130] at the Swiss Federal Institute of Technology.
2. According to Balint's report, during the polymerization of ethylene (according to Fischer), the aluminum trichloride together with the aluminum is transformed into triethyl aluminum which acts together with the titanium chloride as a catalyst system of the Ziegler type [131].
3. Distillation of the products according to Max Fischer was conducted using a Vigreux column (50–60 cm, 42–45 plates) in a vacuum at 160 °C [132].
4. The pure aluminum triethyl separated in "fraction 5" (free of chlorine, no analysis of aluminum) was identified by hydrolysis with water at room temperature and analysis of the gas products formed. Glass apparatus was used for the hydrolysis. The gas collected was pure ethane [133] and the volume attained corresponded to that calculated.
Balint did not now of any literature describing the reaction of aluminum chloride with aluminum triethyl [134].
6. Balint never investigated the reaction of aluminum triethyl with aluminum trichloride in heptane.

37) Contradictory to points 2, 5 and 6: The single example in the Max Fischer patent describes the use of a large excess, 30 g, of aluminum chloride in relation to the 1 g of aluminum powder. The aluminum triethyl and diethyl aluminum chloride which are eventually formed cannot exist in the presence of an excess of aluminum trichloride under the conditions used [135] i.e. exothermic disproportionation.

If the aluminum powder is used up in the reaction with aluminum chloride and ethylene which is the case according to Fischer, a mixture of aluminum trichloride and ethyl aluminum dichloride (mole ratio 4 : 3) would have been found as the product assuming that no further reaction with the titanium tetrachloride, present in the Fischer mixture, had occurred. But under the conditions described the ethyl aluminum dichloride which eventually formed would react at once. Contradictory to point 3: Aluminum triethyl cannot be separated by distillation from the C₁₀–C₁₂-olefines formed [136, 137]. The vapor pressure curves of pure alkyl aluminum compounds, which according to Balint could be separated, overlap with those of C₁₂-hydrocarbons so that separation of pure aluminum compounds is impossible by the distillation method described [138]. The Vigreux column used by Balint had at most six plates. A column having 42–45 plates would be 10 feet high [139].

According to the Balint report sample no. 3 produced fraction 4 by distillation, from which pure ethyl aluminum sesquichloride was isolated and pure aluminum triethyl was isolated from fraction 5. The next higher boiling fraction, 6, contained mainly C₉-hydrocarbons. But it is known that these boil at 40–50 °C lower than aluminum triethyl under the pressure used. The separation described by Balint would be a physical absurdity [140]. Separation of pure aluminum triethyl, if deliberately added to the product of fraction 5 is not possible.

Contradictory to point 4: The isolated products described by Balint were not analyzed with regard to their aluminum content. The conclusions were solely derived from C-H-analysis, ¹H NMR-spectra and separation by gas chromatography.

The hydrolysis carried out as described at room temperature would result in an explosion. Isolation of the calculated amount of pure ethane is impossible [141].

According to his testimony, Balint had recorded the experiments in his laboratory journal [142] but had left the journal behind at the Institute in Zurich. Efforts to find it were unsuccessful. Around Easter of 1976, Balint, accompanied by Mr. Lawrence, an attorney with the Pennie and Edmonds firm in New York, returned to Zurich once more to locate his papers documenting the Max Fischer experiments [143]. Whatever papers still existed were inspected and then destroyed [144].

Both the District Court and the Court of Appeals in the Kodak case had failed to consider the implications of Balint's, Mark's and Martin's testimonies on this issue.

Judge Fisher found this part of the evidence inconclusive because the depositions had been taken outside the court, that is, not in his presence. The court was unable to form a conclusion [145]. The appeal court declared the Studiengesellschaft patents at issue to be valid and did not deal with this area of the testimony.

Thus, a substantial portion of the documentary evidence and an essential part of the witness testimony had become worthless regarding the present Eastman Kodak litigation, but not so, as previously mentioned, in terms of future controversies. Some of the depositions had been taken by the attorneys for Eastman Kodak jointly with the lawyers representing Dart, and Studiengesellschaft's attorneys, Arnold Sprung and Nat Kramer had, of course, been present at all these sessions. Thus, in connection with any depositions involving both defendants, it was inevitable that at least six attorneys would charge their fees to their respective clients.

One aspect which significantly increased Studiengesellschaft's litigation expenses was complying with the opposing parties' request to depose numerous executives of the Max-Planck Institute for Coal Research, such as Director G. Wilke, R. Köster, and H. Breil, as well as H. Martin in particular. For "reasons of economy" these depositions could take place only in Germany. Thus, in the Summer of 1976, the opposing attorneys with their assistants, and accompanied by a court reporter and an impressive number of steel cabinets filled with myriads of documents, flew to Germany and took up residence at the Castle-hotel Hugenpoet, Essen-Kettwig, in the beautiful Ruhr Valley. The air-freighted documents and filing cabinets completely filled an entire unfurnished "double bedroom" at the hotel. The original plan was for the depositions to be held at the nearby Max-Planck-Institute in Mülheim. But Studiengesellschaft succeeded in having their witnesses moved to the more neutral venue of the hotel for their depositions. During the dog days of summer, the assembled company gathered in the hotel's Red Salon where there was no air conditioning, and for several weeks tortuously struggled from one cross-examination to the next. Studiengesellschaft barely managed to prevent the opposing attorneys from rummaging through the Institute's filing cabinets in search of "useful", i.e. self-serving, documents.

Apparently, with the exception of the "Hopff/Balint" story, the results of the search were sparse. They did, however, provide grounds for future litigations.

5.5

Amoco, Arco and Novamont launch New Attacks

In late August of 1976, a discussion took place between Ralph Medhurst of the Patent Department at Amoco, and Arnold Sprung, which the latter confirmed [146] by stating that Amoco apparently wished to deposit any future royalties accruing under its 1974 license agreement with Studiengesellschaft in an escrow account until a decision was reached in the ongoing Ziegler v. Dart litigation. The rationale for this request was that in view of the Hopff findings – counterarguments on the part of Studiengesellschaft were simply ignored – Amoco now questioned the validity of Ziegler's '115 patent. However, under the aforementioned license agreement, Amoco was not entitled to proceed in this manner without requesting that Studiengesellschaft cancel the agreement.

Despite Studiengesellschaft's offer [147] to cooperate with Amoco by providing a scientific evaluation to help clarify the Hopff findings, Amoco filed a declaratory judgment action in the District Court in Wilmington, Delaware, on August 30, 1976 [148]. As grounds for the complaint, the plaintiff initially pointed out the clause in the license agreement which, as mentioned earlier, prohibited the licensee from challenging the validity of the patent being licensed. Amoco sought to suspend this provision. Since Amoco was producing polypropylene in a plant at New Castle, Delaware, the court appeared to have jurisdiction. Amoco then went on to charge that, in view of newly uncovered information, the licensed '115 patent was invalid and requested that it be allowed to deposit all future royalties in an escrow account until a decision could be reached in the parallel action against Dart, which also probed the validity of the '115 patent (see above). Amoco demanded a decision to clarify the contested positions.

In its initial response, Studiengesellschaft offered not to cancel the license agreement if Amoco continued to pay royalties to them, plus, as a further consideration, Studiengesellschaft would consent to a declaratory judgment action to determine the validity of the '115 patent. Sprung further offered that Studiengesellschaft and/or the Max-Planck-Institute for Coal Research would make their entire experimentation resources available for re-examining the results obtained by Hopff and Balint [149].

No doubt, Amoco was the most potent licensee at the time. Studiengesellschaft expected to collect approximately 8 million dollars in royalties from polypropylene sales and 4 million from polyethylene sales [150] under the '115 patent until its expiration in December of 1980. In light of these figures, of which Amoco was, of course, well aware, they were able to justify the loss of 1 or 2 million dollars in litigation expenses. On the other hand, Amoco did not want to risk losing its license. But then, they couldn't have their cake and eat it too!

In view of the large, non-returnable running royalties due to Studiengesellschaft, Amoco urged the court to render a decision as soon as possible (Motion for Summary Judgment) [151] on the further grounds that judgment was warranted as a matter of law and that there were no new facts which could be debated in court. Then, at a hearing [152] before the court in November of 1976, Amoco's representatives

pointed to the publications by Hopff and Balint to illustrate the risk of continuing royalty payments. The judge urged the parties to settle.

By the end of November, the parties signed an initial settlement agreement [153], pursuant to which Amoco would continue to make royalty payments to Studiengesellschaft, while the Max-Planck-Institute, through its Director, G. Wilke, undertook to guarantee repayment of the royalties in the event that the declaratory judgment action was decided in Amoco's favor. The settlement effectively eliminated Amoco's first Complaint. Amoco, however, reserved the right to file a new action charging invalidity of the '115 patent and, in fact, did so in June of 1977, with submission of a modified Complaint [154]. All of the previously presented arguments focusing on BASF's German Patent 874 215 (Max Fischer) as a prior art reference were re-introduced.

To streamline the proceedings, the parties agreed [155] between each other and with Dart, that the two pending cases involving Studiengesellschaft should be combined concerning the discovery phase. Two months later, Amoco disclosed the composition of the catalyst it was using [156] for the production of polypropylene: virtually the same components as employed by Phillips, i.e. titanium trichloride and diethylaluminum chloride. After that, the Amoco controversy was "put on the back burner"; the parties decided to await the outcome of the Dart litigation. It was not until late 1979/early 1980, that is, approximately 1 year prior to the expiration of the '115 patent, that negotiations were initiated which ended with a settlement agreement [157]. Amoco subsequently settled the remaining effective period of its license agreement for the production and sale of polypropylene with a lump-sum payment of 1.2 million dollars. It was, however, not only the '115 patent which was being paid up, but also similar rights deriving from other US patents owned by Studiengesellschaft, as recited in the license agreement. This latter aspect would become significant later on during the ongoing dispute with other parties (see p. 236). In the same agreement, Amoco, for now, relinquished any claims asserted in the aforementioned action.

Any news regarding the production of polypropylene in addition to production capacities, prices, law suits and scientific discoveries quickly circulated throughout the industry. In 1977, Diamond Shamrock, a licensee, had sold its production facilities for the manufacture of polypropylene, including its sales business, to Arco Polymers [158] (a subsidiary of Atlantic Richfield). Arco was well aware of any developments relating to its competitors, Amoco and Dart, as well as the lawsuits that those using and infringing the '115 patent were embroiled in. In August of 1978, 2 years after Amoco, Arco brought a declaratory judgment action against Studiengesellschaft in the District Court of Pennsylvania [159], the grounds for which were that Arco was not using the '115 patent, and furthermore, the patent was invalid. Arco was represented by the attorneys R.E. Hutz and Paul E. Crawford. The latter would, in the future, be associated with other opponents of Studiengesellschaft.

After adopting the existing Diamond Shamrock license agreement, Arco also tried to deposit any accruing royalties in an escrow account in order to avoid the risk of not having the royalties paid under the license, refunded in the event that one of the challengers in the ongoing litigations was successful.

No later than 1978, Arco ceased paying royalties, which resulted in cancellation [160] of the license agreement. In the end, Arco did pay the outstanding amounts, but payment was made under protest [161]. The same dance was repeated in 1981, missing royalty payments and cancellation [162]. Arco brought a suit, and during the proceedings, Sprung petitioned the court [163] to dismiss the complaint as “res judicata” on the grounds that the issue raised by the plaintiff, Arco, had already been decided in the previous Ziegler v. Phillips case (the ’115 patent is valid and infringed by the production of polypropylene at the “Monument Plant”, which was now operated by Arco). In his decision [164], rendered late in 1982, Judge Hannum adopted the rationale set out in Studiengesellschaft’s petition. Arco settled their outstanding debts [165], but appealed the decision [166] and lost. The Federal Circuit [167] affirmed the District Court’s findings, but ordered that any further review must await the outcome of the Studiengesellschaft v. Dart proceedings. After deciding that case (see p. 202), the same court [168], in March of 1984, once again affirmed the verdict of the District Court in the Studiengesellschaft v. Arco matter.

Novamont, which was paying, with great reluctance, any royalties due to Studiengesellschaft under its license agreement, was looking for new ways to rid itself of the obligation. They seemed to have found a self-serving solution when they ceased payment in 1977. Studiengesellschaft responded by bringing a suit [169]. In their Answer to the Complaint [170], Novamont highlighted the most-favored licensee clause (Article IX) of its 1967 license agreement, which required the licensor – Ziegler at the time – to notify Novamont of any third-party licenses that he had granted in the same field which contained more favorable royalty terms than those accepted by Novamont, and which also gave Novamont the right to adopt such new, and more favorable terms. Novamont then referred to the Diamond Shamrock agreement which had been entered into in 1970, and which had been supplemented by a letter of agreement with Diamond Shamrock³⁸⁾ containing considerably more favorable terms, of which Novameont had, however, not been advised. Attached to the Answer was a copy of the “secret” letter of agreement. Novamont furthermore demanded the same terms as those in the Hercules settlement agreement (see footnote 20) but extrapolated to its own production capacity.

The first instance involved the terms Ziegler had granted with respect to sales made prior to the date of the Diamond agreement, in other words, for the time period during which Diamond Shamrock had been infringing Ziegler patent rights. At the time, Ziegler had conceded that any belated payments for this period would be

38) The license agreements with Diamond Shamrock as well as Hercules were extended to Novamont in autumn 1970 (under the most-favored clause). Novamont allowed the set time limit to expire before they agreed to the conditions set forth in both agreements. The conditions of the new agreement were less favorable in total than those in their own original license agreement. (From the Diamond agreement they gained the advantage of

slightly lower royalty payments but the disadvantage of a pre-payment sum of \$200,000 with no deductions for payments to third parties. From the Hercules agreement on the other hand, they gained only disadvantages: the sum that had been paid up by Hercules was \$1.6 million, a figure that was too high for Novamont to accept).

creditable against future royalties and treated as an additional advance payment under the license agreement. Diamond Shamrock had requested this arrangement for settling its past infringement, not least in order to avoid a strained relationship with Phillips Petroleum³⁹⁾. After detailed negotiations, Diamond Shamrock was now willing to pay \$750,000 for past infringement based on its sales prior to 1970⁴⁰⁾.

In his decision [171], Judge R. W. Sweet determined that he would have to find essentially against Novamont, since most-favored-licensee clauses were not applicable to third-party settlement terms involving past infringement, that is, they could not be applied to Ziegler's settlement arrangement with Diamond Shamrock for the period prior to 1970⁴¹⁾. Moreover, Studiengesellschaft did not have an affirmative duty to advise Novamont of the letter agreement. This rule of law was meant to facilitate settlement arrangements between litigating parties and to avoid burdening them with third-party contractual provisions such as, for example, a most-favored-licensee clause.

The appeals court [172] affirmed that Novamont was not entitled to information regarding the so-called secret arrangement with Diamond Shamrock. Moreover the most-favored-licensee clause was not applicable to third-party license agreements entered into prior to and after the effective period of the Novamont license agreement. Furthermore, Novamont had no right to information relating to how the settlement amount for Hercules had been arrived at, nor was it entitled to a similar arrangement. For the remainder, the court affirmed the District Court's findings.

The case was remanded to the District Court, and before the year was out, Judge R. Sweet handed down his final decision [173], assessing the damages at a little over 2 million dollars. Meanwhile, news had spread that Novamont had been taken over by US Steel Corp. in 1982 and was continuing operations under the name of US Steel Polypropylene Division. Thus, in the end, it was against US Steel that judgment was rendered [174].

Novamont's attempt to have the decision overturned by way of an appeal to the US Supreme Court [175] was rejected.

Each of the lawsuits simultaneously in progress against Amoco, Arco and Novamont took between 4 and 6 years to complete, a relatively short time compared to what was to come.

39) See p. 173.

40) Altogether Diamond paid \$950,00 for their license. They were able to credit 50% of the royalties paid per annum against this pre-payment.

41) Representatives of Novamont together with their US Attorney, Mr. Finnegan, had previously visited Studiengesellschaft to discuss with Ziegler, Martin and von Kreisler Jr. the

same problem – application of the most-favored clause to Novamont/Diamond Shamrock. At that time Mr. Finnegan advised that under the US law the most-favored clause of the existing Novamont agreement could not be applied to a settlement agreement in a court procedure dealing with a past infringement [176]. Thus Novamont was aware of the legal situation.

5.6

18 Years of Feud with Dart⁴²⁾

5.6.1

Determination of Liability

The decision of the appeal court [26] in the Ziegler v. Phillips Petroleum Co. case, rendered in 1973, should actually also have disposed of the Ziegler/Studiengesellschaft Complaint against Dart Industries Inc.⁴²⁾ filed in 1970 [39]. In 1973 the court of appeal (5th Circuit) in the Phillips case had, as described earlier, declared US Patent 3,113,115 to Ziegler (the '115 patent) to be valid, that is enforceable, and infringed by Phillips: the catalyst consisting of a mixture of diethylaluminum chloride and titanium trichloride, used by Phillips for the commercial polymerization of propylene, was covered by the '115 patent. The catalyst [177] that Dart had been using since 1964 for the production of polypropylene was practically identical to the Phillips catalyst. Since the beginning of the Dart controversy, Dart's attorneys, T.F. Reddy and S. T. Laurence, had, understandably, not only been watching any progress in the Phillips case, but had also attended the depositions in the subsequent Kodak litigation and gathered material from the Amoco and Arco suits in order to go over each document with a fine tooth comb in search of helpful details.

Intentionally or unintentionally, preparations for the actual trial dragged on for 12 years [178]. Then, for 6 weeks, starting on January 6, 1982, the parties convened before Judge Wright at the District Court in Wilmington, Delaware. The participants had to sit through the direct and cross-examination of numerous witnesses, including five experts hired by the parties, who were authorities in either chemistry or patent law. During this period, more than 5000 pages of record were generated and more than 800 documents, some 1900 pages in all, were admitted as evidence, perused, and explained by witnesses [179]. After the trial, each party summarized its position in the form of a post-trial brief [180]. Certain information was not put before the judge for his consideration including those documents which had been discovered in the files of the respective opposing parties but excluded as privileged, correspondence and depositions generated in connection with earlier proceedings, and Patent Office papers. It was hard to believe that any one judge would be able to review and organize all of this material and reach a decision which would not lead to an appeal.

Seven months after the conclusion of the trial and 12 years after the Complaint had been filed, Judge Wright rendered his decision in a 106-page Opinion [181]. It was a stroke of good fortune to have found in Judge C. Wright an individual who was not only a highly competent authority on US law, but who had also developed remarkable expertise in dealing with the chemistry under review. Rumor had it that his next-door neighbor, a chemist at Du Pont, was tutoring him at weekends; but, on the one hand, this would not have been against the law, and on the other, the noticeable chemical expertise demonstrated by the Judge throughout the daily court ses-

42) Successor to Rexall Drug and Chemical Co.

sions proved to be helpful and expeditious. Such technical competence on the part of a judge should by no means be taken for granted.

Right at the beginning of his Opinion, the Judge excluded from consideration all those arguments which had no bearing on the determination of the case and would, therefore, be without significance [182].

Initially, Dart's attorney, Mr. Reddy, was called on to establish any differences in the evaluation of the '115 patent in the context of the Phillips case, on the one hand, and the current case against Dart, on the other. Mr. Reddy claimed that not only were there numerous such differences, but these differences were also materially significant [183].

What actually was at issue here? To begin with, there was the renewed attack on the validity of the '115 patent, involving the assertion that the earlier Fischer patent had anticipated the invention by Ziegler and his co-workers, and further, that according to papers by Anderson and co-workers (Du Pont), Natta, prior publications by Ipatjeff and van Peski, and finally prior patents issued to Standard Oil of Indiana, the '115 patent was an obvious outgrowth of these earlier publications and, therefore, its contents was no longer patentable – thus read the summary of Dart's arguments in support of its invalidity charge.

The Judge was hardly swayed by these largely unsubstantiated allegations. When comparing [184] the prior Fischer patent with the disclosure of the '115 patent, he found that the catalyst mixture described by Fischer consisted of aluminum powder, aluminum trichloride and titanium tetrachloride, used for the polymerization of ethylene. No mention was made of any organoaluminum compound. The latter is, however, an essential element of the '115 patent. It was beyond the purview of any skilled chemist in 1953 to appreciate and predict that organoaluminum compounds would be formed *in situ* during the Fischer process, if they were actually formed at all. The aluminum powder was not mentioned as a component of the active catalyst, but rather as an auxiliary ingredient intended to bind any hydrogen chloride which might form. The Judge cited Martin's testimony, where he pointed out that, in place of aluminum, Fischer had also considered iron and zinc to be effective materials. Ferro-organic compounds do not, however, exist. Fischer would, therefore, teach away from the assertion that organometallic compounds were formed *in situ*, and were responsible for producing Ziegler catalysts.

During the preliminary stages prior to trial before the court, Dart's attorney, Mr. Reddy, had, in fact, highlighted the findings of Hopff and Balint [185], who had claimed that while repeating the single Example in the Fischer patent, they had isolated not one but three different organometal compounds (ethylaluminum dichloride, diethylaluminum chloride as well as aluminumtriethyl). Yet, not only did he neglect to call Balint as a witness (Hopff had meanwhile died), but he also failed to introduce the results of those experiments into the evidence. He rather hoped, with the help of an eminent US expert witness, George A. Olah [186] of the University of Southern California (Nobel Laureate for Chemistry in 1994), to confirm Hopff's findings and thus insure that the Court took note of his argument.

It was on a Friday morning that Olah was asked to take the stand for his direct examination and cross-examination in open court by Studiengesellschaft's attorney Arnold Sprung. His most important statement was as follows:

Question (Sprung): "But you never did isolate or detect ethyl aluminum chloride using the Fischer proportions?" Answer (Olah): "That's correct." [187].

The Dart camp was stunned, and requested a recess until Monday morning, during which time Olah was sent off cross-country to California in a standby private jet so that he could conduct further experiments. On his return to the witness stand, he introduced spectra⁴³ which led the Judge to conclude that, in his experiments repeating the Fischer Example, Olah had never clearly confirmed the formation of organoaluminum compounds. The spectrum, according to Olah, pointed to only a very small amount of organoaluminum compound. He was, however, unable to determine its exact composition, nor had he maintained the original parameters [188] of the Fischer Example, nor, on cross-examination, was he able to convincingly demonstrate to Studiengesellschaft's designated experts that the spectrum presented did at least *suggest* the presence of an ethyl aluminum compound. The Judge [189] concluded that even 30 years after the invention of the Ziegler catalysts, not even top-ranking experts in the field were able to prove that ethyl aluminum chlorides were formed during the Fischer process. It would be highly unlikely that any skilled chemist, on reading the Fischer patent, would come up with a catalytically effective mixture consisting of diethylaluminum chloride and titanium tetrachloride.

Nor did a combination of the disclosures of the Fischer patent and the Hall and Nash publication from the 1930s prove to be helpful. Hall and Nash described the formation of ethyl aluminum chlorides from aluminum, aluminum chloride and ethylene and, thus, were not concerned with the production of polyethylene, while Fischer failed to mention organometal compounds altogether. The conditions under which Fischer and Hall and Nash had conducted their experiments were materially different. Fischer, by comparison, chose only one-tenth of the amount of aluminum, with an entirely different purpose in mind. Neither Fischer nor Hall and Nash described the polymerization of propylene. What is more, the Fischer catalyst was completely unsuitable for forming solid polypropylene from propylene. Nor did any of the other, older references that were cited mention such combinations.

The issue of the Hopff/Balint experiments, debated back and forth for many years, was, thus, finally laid to rest, as was the speculative evidence offered by Mark in 1959, Hopff in 1960, Tornqvist between 1959 and 1969, Kennedy in 1974, Lenz in 1975, and Boor in 1979 [190], the latter without any experimental support whatsoever. None of these individuals had been able to furnish any information based on their own experience (all of it, therefore, hearsay).

Martin's statement that Breil's conclusions in his doctoral dissertation had been purely speculative was persuasive [191]. There were no experiments to back up these conclusions (cf. Chapter 1, Section 1.2.4).

The possibility that under the conditions chosen by Fischer, a small amount of ethylaluminum dichloride might have formed in conjunction with large quantities of excess aluminum chloride but only in the absence of titanium tetrachloride, was

43) C¹³- and Al²⁷-NMR-spectra.

not taken into account; neither was the fact that Olah obtained improved results in polymerizing ethylene according to Fischer whenever he increased the amount of aluminum and worked at comparatively lower temperatures, that is, employed parameters approaching the conditions described by Ziegler [186].

After careful consideration of all the facts, it was the court's opinion that Dart had failed to demonstrate that the '115 patent had been anticipated by the Fischer patent. The court further noted that in a recently published article addressing the Ziegler/Fischer question, Tornqvist (Esso) [190] had stated that the notion of the invention by Ziegler and his co-workers being a logical next step to Fischer's disclosure was completely absurd. The Fischer conditions were incapable of producing an effective Ziegler catalyst⁴⁴).

The findings published by Du Pont, Judge Wright continued, rather than constituting prior art that anticipated the Ziegler '115 patent, actually ran parallel to Ziegler's work. The court rejected Dart's arguments that Du Pont, by utilizing information obtained from independent sources, had developed a polymerization system which worked in a manner similar to Ziegler's system⁴⁵).

In August of 1954, the Judge noted, Du Pont obtained information on the Ziegler process and attempted to find realms which were not dominated by any Ziegler patent rights. In doing so, Du Pont mainly focused on using aluminum trialkyl compounds or lithium aluminum tetraalkyls for the production of catalysts in conjunction with titanium chlorides. Du Pont did not bother with the use of ethyl aluminum chlorides as catalyst components, which the court viewed as a further indication the Ziegler process was not obvious. The Du Pont researchers were, after all, "skilled and highly motivated scientists" [193].

Dart was the only company known to have obtained a license [194] (license agreement of August 21, 1962) under Du Pont's '471 patent [195], because they believed that this was the basic patent covering the use of titanium trichloride as a catalyst component for the production of polypropylene. It is, however, not possible to partially reduce titanium trichloride with diethyl aluminum chloride [196], as set out in the patent claim. Once Dart realized this, they discontinued their payments to Du Pont. On the other hand, all of the principal olefin polymer producers in the United States obtained licenses under the Ziegler '115 patent, and, what is more, even under the patent application before it matured into a patent.

44) In a publication [192] in 1985 H. Martin and co-workers reported their experimental results regarding the Fischer process. Without titanium tetrachloride small amounts of ethyl aluminum dichloride were identified under the same conditions in addition to an excess of aluminum trichloride. Diethyl aluminum chloride cannot be formed under these conditions. During this reaction ethanediylbis dichloro-aluminum compounds are formed as intermediary products.

45) See Chapter 1, Section 1.1.3.1. They referred here to US patent application 450 243, filed by

Du Pont on August 16, 1954, which became US patent 2,905,645 [26] (see Chapter 1, reference [27]). In the action against Kodak this application was wrongfully described by the defendant, Kodak, as an application having an earlier priority than Ziegler's. The application was followed by Du Pont in a CIP application which was issued as patent in 1962 [194]. The claims of both US Du Pont patents are limited to the quantity of the organometallic compound being sufficient to lower the valence state of the titanium, at least in part, to below three.

The fact that the problem of polymerizing olefins had previously been the focus of active and worldwide investigations and was finally solved by Ziegler and his co-workers, and that their discovery then immediately sparked widespread interest throughout the industry, supports the court's finding that the solution was not obvious [197].

Dart's accusation of fraud made to the Patent Office based on Ziegler's failure to disclose the previously discussed Fischer patent to the Patent Office Examiner, and their further accusation that Ziegler had incorrectly named the inventors in his applications filed in the various Patent Offices around the world, were both dismissed by the court. As noted by the Judge, numerous adjudicating panels had already decided these issues in previous proceedings. The court held that each of the named inventors had played a part in the overall program [198].

The catalysts developed by Ziegler and his team revolutionized the production of high molecular weight polymers from simple hydrocarbons. Prior to Ziegler, it had been doubtful, according to the Judge, whether it was possible at all to convert propylene into solid polymers [199].

Diethyl aluminum chloride as the aluminum compound and titanium tetrachloride or titanium trichloride as the heavy metal compound are the catalyst components which came under the claims of the '115 patent. As stated by the court: this also applies to different varieties of titanium trichloride and furthermore does not depend on how the titanium trichloride was formed from titanium tetrachloride, i.e. by reduction with aluminum powder, with diethyl aluminum chloride, with aluminum triethyl or with hydrogen, and also does not depend on whether the titanium trichloride is an α -, β -, γ - or δ -type [200]. All titanium trichloride compounds had been formed from titanium tetrachloride [201] and admixed with diethylaluminum chloride and had then been brought into contact with propylene⁴⁶⁾.

In this context, the Judge found, the titanium trichloride was always "preformed", including the titanium trichloride commercially available from Stauffer, as well as the titanium trichloride which, as described in the '115 patent, was formed from titanium tetrachloride and diethylaluminum chloride. Thus, all of these variations were similar in the way they functioned. The '115 patent explicitly covered all forms of titanium trichloride⁴⁷⁾.

46) Dart argued that the '115 patent did not describe the application of "preformed" titanium trichloride and illustrated to the Judge the difference between titanium tetrachloride (Ziegler) and preformed titanium trichloride (Dart) by pointing out that titanium tetrachloride is a clear, non-crystalline liquid, and titanium trichloride is a solid insoluble crystalline material. The Judge did not step into this trap.

47) Indeed, the '115 patent does not contain the words "titanium trichloride" (nor the formula TiCl_3) nor "propylene". It was mentioned because titanium tetrachloride was reduced in the reaction with ethyl aluminum compounds. In order to compensate that deficit

Studiengesellschaft filed a patent application in the US in 1972 naming the same inventors and the priority dates (as '115) with the title "Polymerization catalysts" using the same examples for the polymerization of ethylene and propylene, the claims being directed to a catalyst for the polymerization of olefins being a mixture of titanium trihalides and an organo aluminum compound. The patent [203] was issued in 1975, an indication that the examiner agreed. The patent expired on the same date as the '115 patent. The examination by the patent office included the two cited Du Pont patents, Anderson et al 2,905,645 and 3,050,471.

Judge Wright stated [202] that

“Through application of Ziegler’s discovery Natta at Montecatini, Martin at the MPI and subsequently many others were able to produce crystalline polypropylene...”

The catalysts were then called “Ziegler catalysts” [204]⁴⁸⁾.

Even though the application for the ’115 patent, based on the ’332 patent application, had been filed several months later, the court held that the ’115 patent was a pioneer patent because it

“represents a significant step in the progress of the art” [205]

in its own right.

The invention and the work done by the Ziegler team had been a continuous activity, the team itself being relatively small. The development of the catalyst system was a brilliant achievement which was new and of great significance for the commercial world of polymers [206].

During Martin’s cross-examination, the opposing attorney, Mr. Reddy, explained the difference between diethylaluminum chloride as disclosed in the ’115 patent and aluminum triethyl as taught in the ’332 patent, in other words, the substitution of an ethyl group by chloride. Not being a trained research chemist, Mr. Reddy downplayed the significance of this difference. The court, however, adopted Martin’s testimony, according to which the reaction of ethylene for example, with the two compounds is such that diethyl aluminum chloride causes practically no reaction at all, while aluminum triethyl by itself is capable of polymerizing ethylene⁴⁹⁾, and when it is employed together with titanium chlorides⁵⁰⁾, not only will the reaction capacity be different, but also the grade of the resulting polymer product. Thus, the production of crystalline polypropylene was carried out almost exclusively with the use of a diethylaluminum chloride/titanium chloride catalyst (’115 patent) [207].

Dart believed it to be necessary to obtain a license from Esso under the Tornqvist patents⁵¹⁾. These patents described the dry grinding of titanium trichloride in order to enhance the polymerization reaction. The court held that patent ’115 generally taught moist grinding, so that the Dart catalyst treated according to Tornqvist could not be considered as falling outside the scope of the ’115 patent. Tornqvist’s objective

48) Natta did this in 1954 [208].

49) Also forms high molecular weight polymers [209].

50) In November 1953 H. Breil tested the catalytic efficiency of a mixture aluminum trialkyl together with titanium tetrachloride (see Chapter 1, reference [84]) and in the middle of December 1953 H. Martin tested the efficiency of the mixture ethyl aluminum chlorides together with titanium tetrachloride in the polymerization of ethylene (see Chapter 1, references [89–94]). The priority application of August 3, 1954 enlarged the invention to the polymerization of olefins generally

such as propylene and disclosed both classes of aluminum compounds. However no example describing the polymerization of pure propylene to polypropylene by applying diethyl aluminum chloride as a catalyst component was given. The judge asked Martin to translate the relevant example [210] from his laboratory journal in order to prove that the application of a ’115 catalyst to propylene produces a good result. There was a high tension in the court room, a cross-examination did not follow.

51) US P 3,032,510; 3,814,743; 3,128,252 and 3,252,960 chapter 4, reference [10].

had been to improve the existing catalyst technology, and specifically the Ziegler catalyst disclosed in 1953/54. Esso used titanium trichloride and had obtained a license from Ziegler under the '115 patent. Dart, by the way, was the only company to take out a license under the Tornqvist patents. The judge found that the quantitative differences asserted by Dart, between dry ground catalyst components and wet ground '115 components were not sufficient to place the Dart catalyst outside the scope of the valid claims of the '115 patent. The catalyst used by Dart produced the same effect in substantially the same manner, yielding essentially the same result.

On the question of laches, the judge held that between 1962 and 1966, Ziegler had received insufficient information on the nature of Dart's commercial production of polypropylene⁵². The court rejected Dart's allegation of laches asserting that Ziegler had forfeited his patent rights by not acting them.

After the '115 patent had been incorporated into the Phillips action, Ziegler also offered a license to Dart [29]. Conversely, Dart knew no later than August of 1969 that Ziegler had asserted the '115 patent against Phillips. That is to say, Ziegler was quiescent with respect to Dart between early 1966 and mid-1969 which was too short a period of time to justify laches.

In his conclusions, the Judge found that Studiengesellschaft's '115 patent was valid and infringed and could thus be stringently enforced.

While the losing party, Dart, sought review by the next higher court with submission of a detailed appeals brief [211], reiterating the basic arguments which had been rejected by Judge Wright, the plaintiff Studiengesellschaft, also formally filed an appeal.

In early 1984, the Court of Appeals handed down its decision [212]. The panel credited the District Court, and thus Judge Wright, with extraordinary diligence, a meticulously detailed decision, as well as an extensive and painstakingly conducted investigation. The court concluded that all points dealt with in the Opinion were clear of reversible error, affirmed the lower court's judgment, and remanded the case to the District Court for a determination of the damages to be awarded.

After 14 years of litigation and 20 years of infringement by Dart, Studiengesellschaft had still not received a single penny. Nor was a settlement concerning the amount of damages anywhere in sight.

After 30 years, counting from the time the Ziegler catalysts had first been discovered, a decision was rendered in the United States which, in clear language, sent a message to all US infringers that catalysts based on a mixture of diethylaluminum chloride and titanium halides, including all titanium trichloride compounds known at the time, constituted Ziegler catalysts, claiming a priority of January 19, 1954. With the exception of Kodak, all polypropylene producers, including producers of high-crystalline polypropylene, were using this Ziegler catalyst and were thus obligated to pay royalties. All known infringers who had challenged the '115 patent, i.e.

52) In December 1965 a Dart patent [213] was issued. The subject was an improvement in the technical polymerization procedure. It

was not obvious that the patent disclosed the preferred catalyst used by Dart, as claimed.

Phillips, Dart, Arco, Novamont and Amoco, had either been ordered to pay damages or had settled with Studiengesellschaft.

The '115 patent had expired on December 3, 1980. The current decision came too late to provide an umbrella under which Dart's operation could be threatened with an injunction. During the entire effective period of the '115 patent, Dart had infringed that patent.

The decision was in accord with the judgment of the 5th Circuit court in the *Ziegler v. Phillips* matter. Titanium trichloride, as used by Kodak, also fell within the scope of the patented titanium component of the Ziegler catalyst, since the definition of the titanium salts⁵³⁾ was identical in both the '115 and the '792 patent (the latter providing the basis for the Kodak action). In that respect, Judge C. Wright revised the decision in the *Studiengesellschaft v. Kodak* case without any further ramifications.

Two years earlier, in 1980, in the dispute between Standard Oil of Indiana, Phillips Petroleum, Du Pont and Montecatini, involving "polypropylene" product patent rights, Judge C. Wright had found in favor of Phillips Petroleum. Up to the time of that decision⁵⁴⁾ (affirmed 1 year later by the Court of Appeals for the Third Circuit), Montecatini had tried to enforce its product patent with all polypropylene producers, particularly because the previously concluded interference proceeding [214] had been decided in its favor. Thus, Montecatini had sued Dart, after unsuccessful negotiations, as early as 1965 and settled the action in 1975.

5.6.2

Decision Concerning the Amount of Damages

Judge C. Wright had been directed by the court of appeals to determine the amount of damages to be paid by Dart Industries for its patent infringement. To expedite proceedings, the overburdened judge proposed to the parties that a Special Master be appointed – at the expense of the parties – to render a preliminary finding in the form of a Final Report, following negotiations with the parties. The Special Master, a renowned attorney, Victor F. Battaglia, was charged, in March of 1985, with a mandate to determine the amount of damages to be assessed and to ascertain whether or not interest should be paid for the period prior to the decision, and at what rate and for how long. He was furthermore ordered to determine whether Dart's infringement had been willful and, if so, whether the damages should be increased, and finally whether Studiengesellschaft should be reimbursed for its attorneys' fees.

It was not until January of 1986, that the parties were sufficiently prepared to start negotiating before the Special Master, exchange arguments (28 days) and, after conclusion of the negotiations, to summarize their positions in their respective briefs [215]. Ten months later, Mr. V. Battaglia delivered his Final Report [216] to the judge.

With respect to the royalty rate, the parties haggled about a figure between 1.5 and 5.5%. Mr. Battaglia initially determined that no appropriate royalty rate was availa-

53) See foot note 26, points 5–7, and footnotes 27–29.

54) See Chapter 1, reference [3].

ble from parallel Ziegler license agreements for the period around 1964 which might have served as a guide. The sliding scale of 4, 3, 2% from the 1950s, on the one hand, and the 1.5% rate pertaining to contracts signed after 1970, on the other, were the boundaries within which an appropriate figure could be arrived at. Much more appropriate was the figure derived from the Phillips settlement, i.e. 5% of the net sales price from sales handled during the period of past infringement, since Dart's catalyst was essentially the same as that used by Phillips. Both production plants – Dart and Phillips – were comparable in size, and the dollar value of the sales made by each party during the period 1964/65 was approximately the same, that is, slightly above 4 million dollars each [217]. Added to that, as a general formula for calculating royalties, was the consideration that the owner of a pioneer patent could demand one-quarter of the projected income (Dart had indicated 35–36% in this connection), that is to say, a royalty of approximately 8% [218].

The “Special Master” sought to determine an appropriate royalty rate by exploring existing license provisions:

1. The Ziegler licensing program (see Chapter 2, Section 2.4) from the 1950s i.e. the 4, 3, 2% sliding scale, based on the quantities produced and the down-payment called for under the license agreements, including partial crediting against future royalties and most-favored licensee clause.
2. The Ziegler/Montecatini Pool licensing terms calling for 5.5% royalties, of which Ziegler's share was 30%.
3. The Standard terms, granted as of 1970, calling for a maximum royalty of 1.5%.
4. The settlement arrangement following the successful conclusion of the Phillips litigation, which specified 5% for past infringement and 1.5% for licensed future production. From these figures, an effective mean rate of 2.15 % could be calculated.

Since none of the proposed and actually effective license terms was directly applicable, the law provided for hypothetical negotiations to have the patentee and the infringer agree on a mutually acceptable rate.

Aside from the familiar 4, 3, 2-sliding scale, from which an average rate of 2.2% was calculated, the most-favored licensee clause, incorporated into all of Ziegler's earlier license agreements, indicated that Dart – as a hypothetical early licensee – would have been required to make a down-payment also, in addition to the 2.2% royalty payments, which – calculated in terms of annual royalties prorated over the infringement period – amounted to between 1.5 and 1.8%. The Special Master considered a royalty of 4% of the net sales to be appropriate.

Much more complex was the inquiry into whether or not Dart had acted willfully in infringing the patent rights in question. If so, then the law would allow the court to award as much as treble damages (a figure three times as high as the “normal” amount). Showing that the infringer had obtained an expert opinion from an outside independent source is considered *prima facie* evidence in the defense against an

accusation of willfulness. Mr. Valles, Dart's in-house counsel, had not done this. It was the opinion of the Special Master that Mr. Valles' own memorandum on the '115 patent had not examined the issues carefully enough [219].

Moreover, after the decision in the Phillips case, Dart continued to infringe the Ziegler patents for another 7 years, and did so without any significant evidence that it was avoiding infringement of the '115 patent. Battaglia determined willful infringement, scaled up the level of damages by 50%, and awarded Studiengesellschaft a return of reasonable attorneys' fees.

During the entire period of their infringement, as discussed above, from February 1964 through December 1980, Dart had a turnover of roughly 454 million dollars. This figure was ascertained from the accountings furnished by Dart, which were the subject of dispute for a long period of time, and was eventually settled on by mutual consent between the parties. The figure encompassed the amount of product produced but not sold prior to December 2, 1980 (expiration of the '115 patent), product samples, internal shipping costs, and product processed by Dart itself, etc.

Equally controversial was the question of how much interest to be applied. Dart tried to reduce the infringement period by 11 years, arguing that Ziegler had failed to bring suit during that time. Judge Wright's decision provided, however, that the entire period was subject to a calculation of interest. A straightforward 6% interest, as proposed by Dart, was considered unfair by the Special Master. Instead, he determined that the appropriate measure would be the prime interest rate, applied to the quarterly payable royalties, including compounded interest.

Summing up, the Special Master concluded that a royalty rate of 4% should be adopted based on the mutually agreed sales volume, plus the value of any internally processed material, polypropylene samples, stock quantities not sold prior to December 2, 1980, and internal shipping costs. Because of the deliberate nature of Dart's infringement, the basic amount, so calculated, was to be scaled up by 50%, on top of which interest was to be paid at the prime rate in effect at the time each quarterly royalty payment became due, including compounded interest. Finally, Studiengesellschaft was to be reimbursed for its attorneys' fees. In October of 1986, Kraft Inc. had joined Dart Industries Inc. as a co-defendant in the proceedings⁵⁵.

In 1987, Judge Wright handed down a final judgment [220], wherein he initially explained the applicable law governing his decision with respect to the Special Master's Report. The Report, he stated, merely constituted a recommendation, with the court being free to change any of the Special Master's stipulations, although within limits. Any material facts determined by the Special Master would have to be adopted by the court unless they were clearly in error.

Concerning the issue of what royalties would be appropriate for Dart, the Judge found that combining the damages for past infringement with the running royalties for future production had been a reasonable attempt at a solution in the Phillips con-

55) The defendant, Dart Industries Inc., was a subsidiary of Dart and Kraft Inc. Since Kraft reorganized the company at this time there was a danger that Dart Inc.'s liabil-

ity would no longer be in question if the subsidiary was closed. The judge therefore ordered that both Dart and Kraft Inc. entered the action as defendants [221].

troversy. He failed, however, to take into account, for the hypothetical negotiations, the settlement amount paid by Phillips for past infringement.

The court furthermore held that the Special Master's recommendation of the down-payments rendered by the early licensees to be applied as royalty payments was clearly in error, since the underlying license agreements called for the down-payments to be credited – at least partially – against accruing royalties.

Following the concept of hypothetical negotiations, the court found a royalty of 2.5% of the net sales to be appropriate, but also imposed a down-payment⁵⁶⁾ of 1.18 million dollars, 50% of which was creditable against royalties that had actually been paid.

The Special Master's finding of deliberate infringement was considered by the court to be based on a faulty premise: The absence of an impartial expert opinion (by an outside attorney) does not prove willfulness. Dart's designated in-house counsel, Mr. Valles, the court stated, had advised Management in an appropriately competent manner of the patent situation at hand. Under the law, Dart was only expected to have made their decision in good faith.

In the court's opinion, the Special Master had also failed to take into consideration that the patentee had less than vigorously enforced his patent rights. In conclusion, the court decided to reject the 50% punitive increase in damages due to willfulness of the infringement. As a result, the award of attorneys' fees was also reversed.

The court did, however, adopt the Special Master's recommendation on the interest and compounded interest payments to be made. Compiled as figures, this amounted to 450 million dollars in sales at a royalty rate of 2.5%, a 1964 down-payment of 1.18 million dollars, creditable against 50% of the annual running royalties, and interest and compounded interest payable with each quarterly royalty payment.

The fact that the Special Master's rulings which were now drastically reduced by the Judge, were clearly appropriate became apparent from the subsequent decision on appeal, wherein one of the three justices, Judge Newman, voted in favor of the figures proposed by the Special Master. As he stated, the 4% rate determined by the Special Master was not an obvious error, 4% was a clearly appropriate royalty. The same, according to Judge Newman, applied to the 50% extra damages, as provided for by the Special Master. All in all, he considered the Special Master's recommendations to be reasonable. In the end, Judge Newman was voted down by the other two judges.

Judge Wright left it to the parties to calculate the damages, and in late September of 1987, after the latter had come to an agreement, he rendered a decision [222], pursuant to which Dart and Kraft had to pay a total of 43.7 million dollars. The court's instruction that this sum should be paid to Studiengesellschaft immediately, met with resistance by El Paso, Dart's partner in the El Paso/Dart production plant, who requested that the money be deposited with the court until the decision on appeal became available. Judge Wright then ordered [223] that the amount be divided up, with

56) An early licensee, Union Carbide, did not start production until the termination of the agreement. Therefore they were unable to

benefit from the arrangement whereby running royalties could be credited against the large prepayment.

one half being paid over to Studiengesellschaft and the other half deposited in escrow with the court.

As expected, both parties filed an appeal [224]. The timely submission of their briefs [225] took place in early 1988, and in May the parties filed reply briefs [226] in rebuttal of the opponent's arguments. In late 1988, the appellate court pronounced judgment [227], affirming Judge Wright's decision.

Early in 1989, Studiengesellschaft learned that Judge Wright had now also released the monies held in escrow [228].

This marked the end of the 18-year litigation with Dart (Dart/El Paso and Kraft Inc.) – the '115 patent had been subjected to quite a remarkable journey.

5.7

A Tentative Record of the '115 Patent

A series of experiments carried out over a 15-month period during the years 1953 to 1954 had laid the foundation for the '115 patent. It all started with two experiments conducted in May of 1953 (Chapter 1, references [43, 44]), during the course of which Holzkamp observed the first signs of a high molecular polyethylene being formed under the influence of a catalytically effective mixture of aluminum triethyl and chromium acetylacetonate. In October and November of 1953, subsequent experiments, with Breil in charge, unequivocally proved that solid, high molecular weight polyethylene was being formed from ethylene using a catalyst mixture of aluminum triethyl and zirconium acetylacetonate (Chapter 1, references [62, 84]). During the course of further developments, in December of 1953, Martin was able to demonstrate experimentally the polymerization of ethylene at normal pressure, and with the aid of a highly active catalyst mixture of diethyl aluminum chloride and titanium tetrachloride (Chapter 1, references [90–92]). Then followed the co-polymerization of ethylene and propylene and, in March of 1954, it was shown that commercially-available titanium trichloride, together with diethylaluminum chloride, was useful as a polymerization catalyst (Chapter 1, reference [114]). A mechanism for predetermining a desired chain length for the polymers was discovered (Chapter 1, reference [174]) and, finally, utilization of the Ziegler catalysts for the polymerization of propylene and other alpha-olefins was successfully demonstrated in July of 1954 (Chapter 1, references [178–181]).

While the experimental work was still continuing, and once the results had been made public, Ziegler was deluged by frenzied prospective licensees clamoring for a piece of the action, which allowed him to exploit his monopoly at steadily increasing rates (down-payments). Initially, payment of running royalties was sluggish, because it took approximately 10 years for the situation concerning existing patent rights to be clarified and for industrial utilization to go proceed under the governance of a proper patent framework. It took all this time for Ziegler to largely rid himself of any competitors trying to squeeze into areas presumably unprotected by existing rights, who were scouring the field for prior art publications in order to prevent patent grants. The European and Asian markets initially developed at a quicker pace than comparable activities in the United States.

There remained a long line of interested parties who were unable to obtain a license from Ziegler, and a growing number of producers who, at great cost to themselves, tried to shake off Ziegler's dominance by challenging him in court. Patent protection was relatively narrow in the United States due to Ziegler's forced partnership with Montecatini. The drawbacks of this liaison were successfully eliminated in the US in 1969 following a decision by the US Patent Office regarding the question of priority between Ziegler and Natta⁵⁷⁾ – this decision was made approximately 15 years after the first experimental results had been obtained and 6 years after the first basic US patent had been issued.

To safeguard their royalty income, Ziegler and Studiengesellschaft were compelled to pursue a string of legal actions. Although these litigations temporarily came to an end with the expiration of the first basic US patent – approximately 30 years after the invention had been made – the objective remained the same as ever, namely to dominate production, particularly in the United States which was the country with the largest production plants. Europe and Asia posed no threat because any relevant patents had expired in these parts of the world.

For the polymerization of propylene, the industry worldwide largely made use of the catalyst combinations claimed in the '115 patent and in countries other than the United States utilized corresponding patent applications and the patents which matured from them. Since polypropylene, with two-digit annual production growth rates, had been effectively established worldwide as a mass-produced plastic, the US '115 patent and its foreign counterparts proved to be by far the most successful patents owned by the Mülheim-based Studiengesellschaft/Max-Planck-Institute for Coal Research.

Throughout the entire length of the “life and effective term” of the patents obtained within this framework, up to the very end, attempts were constantly made to invalidate the patents, minimize their impact⁵⁸⁾, circumvent them, and “willfully” infringe them. However, the superior skill of the attorneys that Ziegler and Studiengesellschaft had engaged to represent them, proved to be tremendously important. It was certainly a stroke of good fortune that during the entire time that the Ziegler catalysts were undergoing further scientific development, research failed to discover a more commercially and economically favorable catalytically-effective combination to replace the “organoaluminum compound/titanium halide” system. Here, an in-depth knowledge of the local patent laws of each country was also necessary so that appropriately precise terminology could be used to close any potential loopholes in any future patent applications. This proved to be not entirely successful in every case. In any legal disputes, starting with adversary proceedings in the Patent Offices,

57) See decision by the US Patent Office (interference 90 833) 1969 i.e. acknowledgment of the priority for Ziegler catalysts as polymerization catalysts and their application for the polymerization of propylene. Natta's earlier dates for the polymerization of propylene were admitted but were found not to be independent of Ziegler.

58) The opponent's attorneys said that Ziegler's invention was a laboratory curiosity, at best and by using a few experiments he had tried to control the total polypropylene industry. The contribution of other inventors should be valued much more highly.

and ending, for the most part, with decisions by various national patent courts, Studiengesellschaft was successfully represented by patent attorneys of the caliber of Arnold Sprung, to name just one example. However, no one individual could have handled this workload by himself.

The sheer number of documents to be processed, as shown earlier, was too overwhelming.

In the United States particularly, a team emerged which included, on the one hand, patent attorneys such as A. Sprung and N. Kramer and, on the other, witnesses such as Martin, and together they perfected their cooperation with each new case. Each piece of evidence supporting an attorney's arguments had to be introduced into the proceedings by that attorney through his witness. The witness was additionally called upon to analyze the chemical – technical aspects of the various strands of the argument and to advise his counsel of the weaknesses and strengths not only of his own party's position but also that of the opponents⁵⁹. Furthermore, he needed to be able to deal with the contents of the voluminous documentation effectively enough to face cross-examination, which was not always an easy task. Any current testimony had to avoid contradicting any statements that had been made during previous depositions.

Aside from the unfolding patent and licensing situation, the scientific community, as mentioned earlier on several occasions, also took full advantage of the new catalysts, eagerly trying to develop them further. In this context, the school around Natta propagandized the so-called Ziegler–Natta catalysts. This was intended to highlight the fact that by selecting the appropriate titanium trihalides, it was possible to enhance the crystalline content of polypropylene from 50–70% which was typical for the early Ziegler/Martin products, to better than 90%, and that, in this way a new, independent class of catalysts had been invented. Such a characterization would, however, be contrary to the definition of Ziegler catalysts as set forth in the '115 patent. No doubt, Natta's contribution constitutes an improvement over Ziegler, as do the improvements proposed by Tornqvist and Vandenberg, but all of these are clearly Ziegler-dependent improvements. It is indisputable that the discovery of the stereoregular structure of polypropylene being the cause of its crystallinity must be attributed to Natta. However the use of various titanium trichloride modifications for the purpose of achieving high crystallinity is covered by Ziegler patent rights, as determined by the highest US patent appellate court, and is exemplified by the '115 patent. The final judgments involving the '115 patent had ramifications not only for infringements that had occurred in the past, but also impacted on the future as well.

59) In a hearing [229] ordered by Judge Wright, at which only the attorneys for both parties were present, the Judge and T. Reddy, the attorney for Dart discussed the necessity of Dart obtaining details of the know-how in addition to the license agreement. The conversation was noted in the records and contained a compliment to Ziegler and Martin which reinforced the credibility of Ziegler/

Studiengesellschaft. The Court asked what Ziegler knew about the processes anyhow and commented that he had never built a plant in his life. Mr. Reddy replied "Well, he made the invention. And he had Dr. Martin. Dr. Martin is just as good in the laboratory as he is on the stand. The Court replied "I know he is good in the laboratory. But we are talking about building plant."



Fig. 5.1 US Patent 3, 113, 115: (A) cover page;

United States Patent Office

3,113,115

Patented Dec. 3, 1963

1

3,113,115

POLYMERIZATION CATALYST

Karl Ziegler, Heinz Breil, Heinz Martin, and Erhard Holzkamp, all of Mulheim (Ruhr), Germany; said Breil, Martin, and Holzkamp assignors to Karl Ziegler, Mulheim (Ruhr), Germany

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Claims priority, application Germany Jan. 19, 1954
18 Claims. (Cl. 252—429)

This invention relates to new and useful improvements in polymerization catalysts and is a continuation-in-part of copending applications Serial No. 482,412 filed January 17, 1955, and now abandoned, Serial No. 527,413 filed August 9, 1955 and Serial No. 514,068 filed June 8, 1955.

The polymerization of olefins for the production of polymers ranging from gaseous through solid polymers is well known. When producing solid polymers from olefins such as gaseous ethylenes, high pressures of, for example, 1,000 atmospheres and more were generally required, and oxygen or peroxides were generally used as the polymerization catalyst. The yields obtained by these conventional methods were generally low with, for example, about 15–20% of the ethylene being converted in a single operation into the polyethylene. The highest polyethylene polymer which could be effectively obtained by the prior known methods had a molecular weight of about 50,000.

Another type of polymerization catalyst which has been proposed to polymerize olefins such as ethylene consists of aluminum hydrides, aluminum trialkyls or aluminum triaryls. The polymerization reaction involved in that use, however, normally produces low molecular polymers not ranging substantially above the liquid range. By using extremely small, controlled amounts of the aluminum catalyst it is possible to obtain higher molecular products. With the use of such small quantities of the aluminum catalyst, however, the reaction becomes extremely sensitive to traces of impurity in the olefins such as ethylene and proceeds very slowly since the quantity of catalyst to the total reaction mixture is very small.

One object of this invention is a new catalyst useable *inter alia* for obtaining high molecular weight products which may be used as plastics.

A further object of this invention is a new catalyst useable *inter alia* for the polymerization of ethylenically unsaturated hydrocarbon products.

A still further object of the invention is a new polymerization catalyst for obtaining polymers having molecular weights higher than those heretofore obtainable.

Another object of the invention is a polymerization catalyst for lower olefins up to about C₅ and particularly ethylene.

These and still further objects will become apparent from the following description:

In accordance with one application of the invention at least one ethylenically unsaturated hydrocarbon of the general formula CH₂=CHR is polymerized into high molecular products by contact with our novel catalyst composed of a mixture of a first and second component, said first component being at least one aluminum compound of the group consisting of aluminum hydrides and mono- and di-hydrocarbon aluminum compounds, having the general formula R'₃AlX in which R' is the same or a different member selected from the group consisting of hydrogen, alkyl radicals and aryl radicals, and X is a member selected from the group consisting of hydrogen, alkyl radicals, aryl radicals, halogen atoms, alkoxy radicals, aryloxy radicals, secondary amino radicals, second-

2

ary acid amide radicals, mercapto radicals, thiophenyl radicals,



radicals, and —O—SO₂—R' radicals, said second component being a heavy metal compound selected from the group consisting of the non-ionized salts including organic salts, and the freshly precipitated oxides and hydroxides of metals of groups IV-B, V-B and VI-B of the periodic system including thorium and uranium. R in said formula may be hydrogen or a hydrocarbon radical.

The designation aryl or similar expression as used herein generically, in identification of an organic compound, is intended to include, as is well understood in the art, an organic compound having one or more aryl, aralkyl or alkylaryl substituents.

The term "non-ionized salt" as used herein is intended to designate the true salt as such and which under the conditions of the formation of the catalyst mixture and the contacting with the ethylenically unsaturated hydrocarbon is not reduced to free metal and is not ionized.

Except as otherwise limited herein, the term "salt" or "salts" designating a compound having a heavy metal of the IV-B, V-B and VI-B groups of the periodic system, including thorium and uranium, is employed in its broadest sense, i.e. to connote the reaction product between a base and an acid, including products of the type of acetylacetonates and further including salts in which said periodic system group member is present as a cation as well as those in which such member is present as an anion such as in products of the type of titanates, zirconates, chromates, molybdates or tungstates. The term "pure alcoholates" hereafter used in designation of the said "salts" is intended to connote "salts" having solely alcoholate radicals attached to said heavy metal. "Mixed alcoholates" of said heavy metals as hereafter referred to are such salts having at least one alcoholate radical and at least one nonalcoholate radical.

Particularly good results are produced with heavy metal compounds which are soluble in inert organic solvents such as hydrocarbons.

Using the catalyst in accordance with the invention for the polymerization of olefins such as gaseous ethylene, the same are polymerized into high molecular polymers by contact with the catalyst. The catalyst may be formed by mixing, for example, aluminum hydride, aluminum dialkyl- or aluminum diaryl-compounds with a compound of a metal of group IV-B, V-B, or VI-B of the periodic system of elements.

When the aluminum compounds comprise dialkyl or diaryl aluminum monohalides, a compound of a metal of the VIIIth group of the periodic system or of manganese may be used in place of the group IV-B, V-B, or VI-B metal compounds.

The term "high molecular" as used herein is intended to designate molecular weights of more than 2,000, and preferably more than 10,000.

The herein designated numerical values for molecular weights are based, in accordance with conventional practice, on the viscosity of the solutions of the polyethylene for which the molecular weight determination is to be made. This viscosity is expressed as "intrinsic viscosity" (η) which is to be calculated on the basis of an equation given by Schulz and Blaschke (Journal fuer Praktische Chemie, volume 158 (1941) pp. 130–135, Equation 5b p. 132) and corrected for the therein mentioned specific viscosity according to Fox, Fox and Flory (J. Am. Soc. 73 (1951) p. 1901). The average molecular weight, as for instance that of 50,000 above given, is calculated from such intrinsic viscosity by way of the modified equation of

Fig. 5.1 (B) columns 1 and 2 of the same patent



Fig. 5.2 Arnold Sprung and Heinz Martin

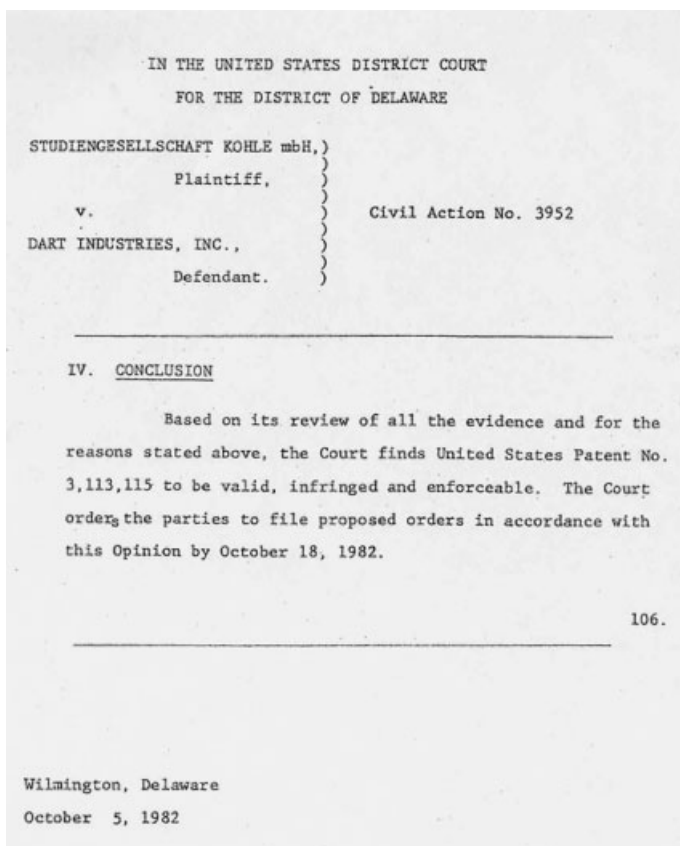


Fig. 5.3 Documentation showing the conclusion of Civil Action No. 3952 against Dart Industries Inc.

In this connection, it is interesting to note that the diethylaluminum species – in place of aluminum triethyl – had become the component of choice in preparing the catalysts not only for polypropylene but also, as pointed out earlier, for polybutadiene, because it allowed the formation of a product having a high ratio of stereoregular polymers.

In the 1980s, a new variation of the diethylaluminum species was introduced which had the highest technically effective activity rate. It had been discovered that it was desirable to replace the chlorine in the diethylaluminum chloride with an alkoxy group (diethylaluminumalkoxy). The utility of such compounds had first been confirmed in experiments conducted by Breil (high pressure experiment) and Martin (normal pressure experiment) in January of 1954 (Chapter 1, references [110, 111]). Combining these compounds, yet again with titanium tetrachloride instead of titanium trichloride, which was a renaissance, brought about an enhancement in polymer yields of such magnitude that trace amounts of catalyst were left in the product⁶⁰). Whether the improvement was simply that or was also an attempt to circumvent Studiengesellschaft's patent rights became the subject matter of future disputes (see Section 5.10.).

Let us now return to the patent situation.

5.8

Lex Ziegler

During the prosecution of one of Ziegler's earliest patent applications, the Examiner in the US Patent Office had, *inter alia*, issued a restriction requirement. This application [230] claimed catalysts consisting of dialkylaluminum species admixed with titanium halides and the use of these catalysts for polymerization. The Examiner at the time took the position that the application covered two separate inventions, one directed to the production of the catalysts, and the other to the utilization of these catalysts. He persisted in his restriction requirement (cf. Chapter 3, p. 110). Both parts of the application were prosecuted separately. The catalyst case matured into US Patent 3,113,115 ('115) (1963), while the second case did not issue as a patent until 1978: US Patent 4,125,698 ('698). The reason why the second case took 24 years beyond the German priority date to mature into a patent was because the application was contested in the Patent Office and became involved in several interference proceedings, and since prosecution had been suspended for a number of years, it

60) Applying solid insoluble titanium trichloride as a polymerization catalysts component it acts by way of the surface of the titanium trichloride-particles (see chapter 1 [119]. In this procedure the internal parts of the particles do not act in the catalysis, is expensive ballast. To reduce the amount of titanium

chloride, the soluble titanium tetrachloride was used to be impregnated on an inert carrier surface (magnesium chloride) and was transferred together with the soluble aluminum compound (diethyl aluminum-species) into the polymerization pot.

simply slumbered in the US Patent Office until issuance. Two years prior to the expiration of the '115 patent, the process patent was granted, with a further 17-year effective term.

Studiengesellschaft's new patent initially attracted little attention, the '115 patent was still in force. It was during the course of Studiengesellschaft's efforts to conclude a license agreement with a new polypropylene producer, Northern Petroleum Corporation (NPC) of Morris, Illinois, that the significance of the new patent was discussed for the first time [231].

The discussions [232] dragged on from the time that NPC went on stream with its polypropylene plant in 1978 (capacity 200 million pounds per annum) to late 1980 without a contractual arrangement being agreed upon. Because of the brief period remaining until the expiration of the '115 patent at the end of 1980, the negotiations focused on payment of a "paid up" sum both for the '115 patent and the '698 patent. The amounts offered by NPC in settlement were of no interest to Studiengesellschaft, and following this rejection NPC declared that it was no longer interested in a license. Studiengesellschaft, therefore, brought a suit against NPC that same year, 1980 [233].

NPC petitioned for a suspension of the proceedings until after a decision had been reached in the Dart case [234]. The court denied the request [235]. Concurrently with these events, Sprung, on behalf of Studiengesellschaft, referred to the previous offer of 1980 and proposed to reopen the negotiations for a settlement agreement [236]. In May of 1982, Studiengesellschaft declared its willingness to enter into a partial settlement arrangement, under the terms of which NPC was to make an immediate payment of \$450,000 and a further payment of \$400,000 once the Dart action had been decided in Studiengesellschaft's favor. This would dispose of any claims deriving from an infringement of the '115 patent. For the new '698 patent, Studiengesellschaft offered a 30-day option period, running from the date of the decision against Dart, during which it guaranteed a royalty-free license for the production of 200 million pounds per year for a lump-sum payment of 1 million dollars, with the pending action against NPC to be suspended until then [237]. Thus, the action for infringement of the '115 patent by NPC had been effectively settled. NPC paid the agreed second installment [238] after the Supreme Court had denied certiorari in the Dart matter and the appeal decision in that case had become final. There was a string of positive ramifications which ensued from the outcome of the Dart case, one of which was relevant to the NPC litigation.

With respect to that part of the action involving the '698 patent, Judge McMillan set up a schedule to be adhered to for any further proceedings in the case [239]. In response, the parties agreed that the option period for a settlement license under the '698 patent was to run from the date of the appeal decision in the Dart case [240]. The parties further agreed that NPC would also concede infringement of the '698 patent, should the appellate court in the corresponding Dart action conclude that Dart had infringed the '115 patent. The double-patenting issue [241] had been excluded from this arrangement and was the subject matter of the controversy with NPC.

This requires further explanation. The specifications of both patents being litigated were practically identical, but while the claims of the '115 patent were directed

to the product itself, the “polymerization catalyst”, the ’698 patent claimed the “utilization of the catalyst” for the polymerization of alpha-olefins. Any parties operating under the ’115 patent viewed the ’698 patent as an attempt to have the same invention patented twice, which of course, the law would not permit. Thus, the heart of the controversy consisted of having the courts either affirm the Patent Office’s judgment that with the two patents, the Office had granted rights to two different inventions, or declare the ’698 patent null and void because of double-patenting. If the validity of the ’698 patent were upheld by the courts, then NPC would concede infringement. The suit was to be strictly limited to the issue of double-patenting, requiring either a “yes” or “no” decision.

Following the appeal decision in the Dart matter, NPC offered [242] to pay \$75,000 in settlement of its unauthorized use of the ’698 patent – not enough for serious consideration.

In June of 1984, both parties submitted their briefs to the court, laying out their closing positions and arguments [243]. The parties initially reiterated that they were in agreement that NPC was infringing the ’698 patent through the operation of its Morris, Illinois plant and that the ’698 patent was valid in terms of novelty and level of invention. NPC furthermore confirmed that, in 1983, it had sold 10 billion pounds (4.45 million tons) in the United States alone and that polypropylene had thus been shown to be a very useful material, for example, for manufacturing containers, fibers and carpet pads. Since the start-up of production, the catalyst of choice had been a mixture of diethylaluminum chloride and titanium trichloride.

NPC now claimed that no utility was either known or had been described for the catalysts according to the ’115 patent, except for the polymerization of α -olefins. To begin with, this opinion was contrary to the position taken by the Examiner of the US Patent Office, on the basis of which a restriction requirement had been issued for the application – with the basic application to be directed to the polymerization catalyst and the divisional application to the polymerization process. There could be no doubt, as the Examiner had argued almost 30 years earlier, that the claim directed to the catalyst was broader in scope than the claim directed to the polymerization of α -olefins using the patented catalyst. A catalyst could be sold and the sale would fall under the catalyst claim, but not under the process claim of the ’698 patent. There were a number of further examples where polymerization would be covered by the catalyst claim but not by the process claim of the ’698 patent, such as, for example, the polymerization of isoprene, 1,3-butadiene, acetylene, trimerization of butadiene, etc. [244]. Admittedly, the ’115 patent did not disclose such examples, but the plaintiff believed this was not necessary.

Six months later, in late 1984, Judge T. McMillen handed down his decision [245]. Initially, the court dismissed the opponent NPC’s, allegation that Studiengesellschaft had intentionally dragged out the prosecution of the ’698 patent in order to substantially extend the monopoly afforded by the ’115 patent. The court pointed out that there was absolutely no evidence to support this allegation. Any deadlines set by the US Patent Office had been unfailingly observed by the plaintiff. The delay in patent issuance until 1978 had been caused by the interference proceeding with Natta (Interference No. 90,833, cf. Chapter 4, p. 157). While the Patent Office had de-

terminated the priority issue in Ziegler's favor, Natta had appealed this decision, in a so-called "§ 146 Civil Action", to the District Court for the District of Columbia, which was settled in 1983 between the successor parties Montedison/Studiengesellschaft. In fact, the Patent Office did not formally terminate this interference until the end of 1984. NPC had, therefore, failed to prove laches on the part of Studiengesellschaft in bringing action.

On the other hand, the court found that the '698 patent was invalid because of double-patenting. When dealing with complicated technical issues, the judge stated that the court would have to rely on the expert opinions presented, knowing full well that each party's expert witness would tailor his opinion to fit the plea put forward by his own party. It was the court's opinion that it was the statement by NPC's patent expert which carried more weight, particularly because all of the Examples disclosed in the '115 patent were directed exclusively to the polymerization of alpha-olefins, that is, no other kind of polymerization was mentioned.

In deciding the "double-patenting" issue, the statute set forth under 35 USC § 121 [246] is crucial. This section states, in part, that in the event that an application contains claims directed to two or more separate and distinct inventions, the examiner could require restriction. If one of the divisional applications matured into a patent, that patent could not be cited against the other as a reference. Judge McMillen did not, however, consider § 121 to be pertinent to the matter in hand, because, in his opinion, the two patents did not involve two separate inventions.

During the appeal proceedings before the US Court of Appeals for the Federal Circuit in Washington [247], each party reiterated its pertinent arguments. If the '698 patent was declared valid, the entire polypropylene industry would have to pay royalties for 32 years. The attorneys for NPC claimed that if this happened the economic scope of the patent system would thus be altered to Studiengesellschaft's advantage. Sprung and Kramer pointed out repeatedly that the inventions claimed in the '115 and '698 patents were not identical, and that the '698 patent was thus entitled to protection under 35 USC § 121.

The decision [248] of the appellate court, dated February 10, 1986, was one of extraordinary importance.

To begin with, the court agreed with Studiengesellschaft that the two patents, the '115 and '698, must be classified under different categories of invention, one being a product patent and the other a process patent, and that viewed strictly in light of that distinction, they did not contain the same invention; this would be sufficient to avoid double-patenting based on "same invention". The claimed invention according to the '115 patent could be practiced without infringing the second patent. It was the appellate court's opinion that the District Court had been in error. There was, thus, no need to further address the issue of § 121. But, in addition, double-patenting based on the obviousness of one patent over the other would not be applicable specifically in view of this section, because the statute expressly prohibited one patent being cited over the other as a prior art reference.

Upon reform of the patent laws in 1952, Congress had refused to modify the expiration date of patents granted after a restriction requirement by the US Patent Office, even though the issue remained open. It was Congress's intention in particular,

not to limit the effective period of the later issued patent compared to the term of the first patent. In the case at hand, an especially long time period had elapsed between the issue dates of the two patents. Studiengesellschaft, however, was not to blame for this as Judge McMillen had previously found, but the fault, instead lay with the interference process practiced at the US Patent Office:

“If the law as it has been written by Congress creates anomalous situations, then it is for Congress to decide whether to change the law.”

The District Court decision regarding the invalidity of the '698 patent was overturned.

In an appendix to the decision, Judge Newman discussed the basis and history of 35 USC, § 121. With the division of the original 1954 application, following a restriction requirement by the Office, § 121 became applicable. The pertinent statutory law had been passed by Congress. Thus, a situation commonplace in 1952 and earlier, where an examiner was allowed to issue a restriction requirement and then reject the divisional application over the disclosure of the parent case had been corrected. Even where two related patents had issued, the courts, at the time, had habitually declared one of the patents invalid due to double-patenting. This was the reason why § 121 had been enacted. The proposal that both patents expire on the same date had been expressly rejected. There were only a very small number of cases after 1952 where § 121 was relied on in connection with double-patenting issues. Not a single case exists, nor did the parties cite any case in which a court had declared a patent invalid due to double-patenting, where that patent was based on an application filed as a result of a restriction requirement according to § 121. Furthermore, § 121 safeguards the applicant from having to challenge the legality of the restriction requirement. On the other hand, the Examiner did not withdraw the restriction requirement.

In the decision no mention was made of the fact that the disclosures of the two patents partly overlapped. Accordingly, an invention could be covered by both patents as far as the overlapping portion was concerned.

The end result of the decision was that the polymerization of propylene with the aid of the catalyst used by NPC was covered by both patents, and Studiengesellschaft was able to enjoy patent protection over a period of 32 years, from 1963 through 1995, instead of merely 17 years as intended by law. Because of its uniqueness, the author has taken the liberty of dubbing the decision “Lex Ziegler”. Thus, the unusually long duration of patent protection was ultimately brought about by the Patent Office’s failure to withdraw its restriction requirement and the different lengths of time it took to issue the two patents.

Certain formal procedural steps followed, such as remand of the case to the District Court [249]. The court, presided over by Judge Newman, denied both a further extension of time to await the decision of the Supreme Court [250] and a further hearing [251].

On June 16, 1986, Judge W. Hart issued an injunction [252] for NPC’s production plant, effective as of 5 p.m., June 27, 1986. The pressure exerted by the court proved

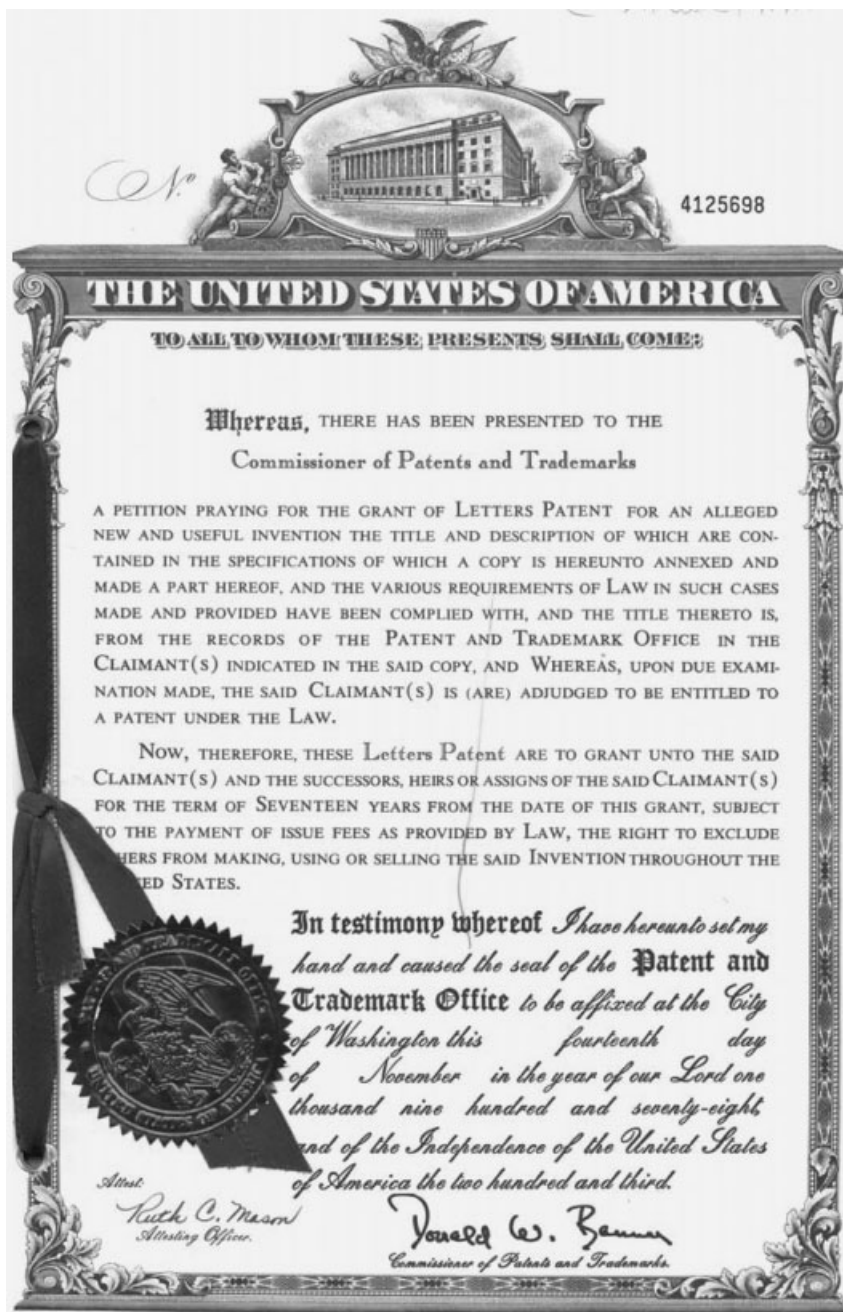


Fig. 5.4 US PS 4,125,698: (A) cover page;

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United States Patent [19]		[11] 4,125,698
Ziegler et al.		[45] Nov. 14, 1978

<p>[54] POLYMERIZATION OF ETHYLENICALLY UNSATURATED HYDROCARBONS</p> <p>[75] Inventors: Karl Ziegler; Heinz Breil; Heinz Martin; Erhard Holzkamp, all of Mulheim an der Ruhr, Germany</p> <p>[73] Assignee: Studiengesellschaft Kohle M.b.H., Muehlim, an der Ruhr, Germany</p> <p>[21] Appl. No.: 770,484</p> <p>[22] Filed: Oct. 29, 1958</p> <p>Related U.S. Application Data</p> <p>[63] Continuation of Ser. No. 482,412, Jan. 17, 1955, abandoned, and Ser. No. 527,413, Aug. 9, 1955, abandoned, and Ser. No. 514,068, Jun. 8, 1955.</p> <p>[30] Foreign Application Priority Data</p> <table border="0" style="width: 100%;"> <tr> <td>Nov. 17, 1953 [DE]</td> <td>Fed. Rep. of Germany</td> <td>..... Z 3799</td> </tr> <tr> <td>Dec. 15, 1953 [DE]</td> <td>Fed. Rep. of Germany</td> <td>..... Z 3862</td> </tr> <tr> <td>Dec. 23, 1953 [DE]</td> <td>Fed. Rep. of Germany</td> <td>..... Z 3882</td> </tr> <tr> <td>Jan. 19, 1954 [DE]</td> <td>Fed. Rep. of Germany</td> <td>..... Z 3941</td> </tr> <tr> <td>Aug. 3, 1954 [DE]</td> <td>Fed. Rep. of Germany</td> <td>..... Z 4348</td> </tr> <tr> <td>Aug. 16, 1954 [DE]</td> <td>Fed. Rep. of Germany</td> <td>..... Z 4375</td> </tr> <tr> <td>Dec. 11, 1954 [DE]</td> <td>Fed. Rep. of Germany</td> <td>..... Z 4603</td> </tr> <tr> <td>Dec. 13, 1954 [DE]</td> <td>Fed. Rep. of Germany</td> <td>..... Z 4604</td> </tr> </table> <p>[51] Int. Cl.² C08F 4/66; C08F 10/00</p>	Nov. 17, 1953 [DE]	Fed. Rep. of Germany Z 3799	Dec. 15, 1953 [DE]	Fed. Rep. of Germany Z 3862	Dec. 23, 1953 [DE]	Fed. Rep. of Germany Z 3882	Jan. 19, 1954 [DE]	Fed. Rep. of Germany Z 3941	Aug. 3, 1954 [DE]	Fed. Rep. of Germany Z 4348	Aug. 16, 1954 [DE]	Fed. Rep. of Germany Z 4375	Dec. 11, 1954 [DE]	Fed. Rep. of Germany Z 4603	Dec. 13, 1954 [DE]	Fed. Rep. of Germany Z 4604	<p>[52] U.S. Cl. 526/159; 526/95; 526/103; 526/105; 526/107; 526/164; 526/169.1; 526/169.2; 526/352; 526/906</p> <p>[58] Field of Search 260/94.9, 93.7; 526/159, 169, 103, 95, 105, 107, 169.1, 169.2, 164</p> <p>[56] References Cited</p> <p>U.S. PATENT DOCUMENTS</p> <table border="0" style="width: 100%;"> <tr> <td>3,058,963</td> <td>10/1962</td> <td>Vandenberg</td> <td>..... 526/159</td> </tr> <tr> <td>3,114,743</td> <td>12/1963</td> <td>Horne</td> <td>..... 526/159</td> </tr> </table> <p><i>Primary Examiner</i>—Edward J. Smith <i>Attorney, Agent, or Firm</i>—Burgess, Dinklage & Sprung</p> <p>[57] ABSTRACT</p> <p>Method for the polymerization of alpha-olefins, e.g., ethylene and its higher homologs, to produce high molecular weight polymers by contacting the alpha-olefin or an alpha-olefin mixture to be polymerized with a catalyst formed from a mixture of a first and second component, the first component essentially consisting of a dialkyl, diaryl organoaluminum compound, or aluminum hydride, and preferably a dialkyl aluminum halide, e.g., diethyl aluminum chloride, and the second component essentially consisting of a salt, freshly precipitated oxide or hydroxide of a Group IVB, VB, VIB or VIII of the Periodic System, and most preferably a salt, such as titanium chloride.</p> <p>15 Claims, 5 Drawing Figures</p>	3,058,963	10/1962	Vandenberg 526/159	3,114,743	12/1963	Horne 526/159
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Fig. 5.4 (B) page referring to the polymerization of ethylenically unsaturated hydrocarbons

effective. Six days before the deadline, the parties, represented by Martin/Sprung, on the one hand, and Mr. R.D. Kinder, Executive Vice President of Enron Corporation (successor to NPC) of Houston, Texas, on the other, agreed on the "Heads of Agreement" [253] with respect to damages, and 1 day prior to the expiration of the deadline, Enron Chemical made a direct payment to Studiengesellschaft [254]. The arrangement led to a license agreement [255], which defined the damages for past infringement, down-payment, and running royalties for production after June 30, 1986.

The decision had far-reaching consequences regarding other producers who had thus far ignored the monopoly afforded by the '698 patent. Initially, it should be noted that the appellate court decision against NPC gave a further positive boost to the licensing business [256]. Thus, Phillips Petroleum Co. [257] and El Paso Products Co. [258] (which had taken over all of Dart's production) entered into license agreements that same year, under the terms of which royalties were established for both past production since 1980 as well as future production. NPC, Phillips Petroleum and El Paso all paid running royalties practically until the day the '698 patent expired.

Arco Polymers (a subsidiary of Atlantic Richfield Co.) had undertaken, with respect to the '115 patent, to be bound by the decision in the Dart matter, and after a decision had been reached, paid damages. Arco believed that this had discharged their obligation, but Studiengesellschaft, again, filed a suit, this time based on the '698 patent [259]. This was all the more surprising to Arco, since the company had intended to divest itself of its polyolefin business. The buyer retreated from the deal for fear of becoming involved in protracted litigation with Studiengesellschaft [260]. Arco wanted to resolve the conflict with Studiengesellschaft in an amicable manner, and in June of 1984, both parties signed an agreement [261], which, for payment of 1 million dollars by Arco, settled all accrued damages for past infringement of the '698 patent up to a maximum capacity of 500 million pounds of polypropylene produced per year. Any excess production was to be subject to a 0.5% royalty.

Shell Oil Co. of Houston, Texas, had owned a license for the production of polypropylene [262] since 1964, with a reduced royalty rate since 1972 [263] (see p. 177/178), and with the then applicable Standard royalty rates since 1974 [264]. The latest contract also covered utilization of the US patent application which subsequently matured into the US '698 patent. Like other polypropylene producers in the United States, Shell, too, expressed its misgivings to Studiengesellschaft as to the validity of the '115 patent and finally stopped paying royalties. As it had already attempted in the past, Shell Oil Co. pushed to have its license converted to a paid-up license, with payment of the lowest possible settlement sum. In late 1979, they did pay 1.8 million dollars and received a royalty-free license [265] to produce up to 450 million pounds of polypropylene per year. Furthermore, they undertook to pay a running royalty of 1% of the net sales of any excess production until the expiration of the '115 patent at the end of 1980, and for the period after 1980, to pay an amount to be negotiated for any excess production. In this connection, it was agreed that for royalties due after 1980, the royalty rate should be no higher than that granted to other paying licensees. It should also be mentioned at this point that Studiengesellschaft accepted Shell's declaration that its polybutene production, which Shell represented as being rather small, "was not covered by any of Studiengesellschaft's patents."

The decision against NPC prompted Studiengesellschaft to cancel all license agreements with Shell on the grounds that Shell Oil had not rendered accountings for its sales nor paid royalties [266]. Shell protested by arguing that, pursuant to the 1979 agreement, no fixed royalties had been stipulated. On the day the cancellation took effect, Shell had sent Studiengesellschaft a last minute accounting for its polypropylene production [267]. It was in this report, with the first occurrence in 1986, that mention had been made of a production practiced without the use of alkylaluminum halide as a catalyst component, a process which Shell had evidently considered to fall outside the license agreement.

Finally, Shell filed a declaratory judgment action [268]. Aside from continuing with the proceedings, which were moving forward rather sluggishly, Shell was interested in an out of court settlement. In late April of 1987, Shell presented its first offer of detailed settlement terms [269]. In negotiations with Shell, the issues were resolved, and in early July of 1987, the parties entered into a license agreement [270].

Cancellation of the 1974 and 1979 agreements by Studiengesellschaft, one of the issues being tried, was withdrawn, and Shell Oil agreed to pay royalties under the '698 patent of 1.5% of any net sales of polypropylene in excess of 450 million pounds per year. They furthermore settled damages for past infringement sales by making a lump-sum payment of 2 million dollars.

Another very significant aspect of the arrangement involved Shell's undertaking to specify those quantities of polypropylene produced which they did not consider to fall under Studiengesellschaft's patent rights, together with enough information to enable Studiengesellschaft to make its own evaluation as to whether this production did, in fact, fall outside the scope of the licensed patent rights, in other words, was independent of the '698 patent. A new conflict was looming on the horizon.

Before venturing into further detail on this, it would be helpful at this point, to address an element of the story which at this time was passing through a critical phase in its historical evolution.

5.9

Product Patent Protection for “Polypropylene”

In the foregoing historical review with respect to the new polypropylene (Chapter 1, Section 1.1), numerous references may be found to the court battle for ownership of the product rights for polypropylene [271]. The pertinent decision between the parties involved, Standard Oil of Indiana, Phillips Petroleum, Du Pont, and Montecatini, was rendered in 1981 in favor of Phillips Petroleum. The origin of the controversy goes back to 1958, when the US Patent Office initiated an “interference proceeding” (cf. Chapter 1, reference [1]) on the basis of the relevant patent applications made by the engaged parties. Ziegler, as mentioned earlier, was not a party to these proceedings (cf. Chapter 1, Section 1.1.1). It was the time of the dispute over the consequences of the Pool Agreements with Montecatini. The parties had agreed that the field in which they shared a mutual interest would be divided up in terms of the patent rights so that Ziegler was to focus on his catalysts and Montecatini on the “isotactic polypropylene” in the United States. Thus, the polymerization catalysts developed by Ziegler and his co-workers were patented, *inter alia*, in the '115 patent, while the isotactic polypropylene products were covered by the '300 (US Patent 3,112,300) and '301 patents (US Patent 3,112,301) issued to Montecatini. Use of the Ziegler catalysts for the polymerization of propylene, among other monomers, was subsequently protected in the '698 patent. The original US patent application directed to the polymerization of, *inter alia*, propylene, however additionally included product claims which Ziegler and co-workers had drafted to cover the new polypropylene product. At no level of prosecution so far had there been a decision on whether these claims [272], still pending before the US Patent Office, were to be allowed. With Ziegler's interests being covered, there was at first no reason to force a decision, since sufficient patent protection existed in the form of the two basic patents ('115 and '698).

Encouraged by the appellate court's decision upholding the validity of the second Ziegler/Studiengesellschaft patent, '698, Studiengesellschaft pushed for prosecution of the pending "polypropylene" product claims in the US Patent Office. One can imagine the position of the Examiner [273] who, so many years later and after prior adjudication in 1980/81, found himself in a situation where he had to render a further decision. He rejected the request by raising massive objections. Even after a number of formal objections had been overcome, the Examiner's position remained uncompromising. He made the rejection of these claims final. Only two prior art references remained as so-called "prior rights" before the Board of Appeals of the US Patent Office [274], thus preventing Studiengesellschaft's claims to be allowed – one being the earlier patent issued to Field of Standard Oil of Indiana (2,691,647, dating back to 1952) and the other the patent to Hogan of Phillips Petroleum (4,376,851, claiming a priority of January 27, 1953) – both patents have been discussed previously herein (cf. Chapter 1, references [4, 14]).

It should be noted here again, that the polypropylene materials produced in accordance with the two above-mentioned patents were not produced using Ziegler catalysts. In connection with both patents, the court in the above-mentioned civil action 4319 had rendered a decision with respect to product protection for polypropylene, stating that the '647 Field patent⁶¹⁾ did not contain any disclosure of a plastic polypropylene material which could be molded into foils and plates. The patent completely lacked any descriptive characterization of the polypropylene. The '851 Hogan patent⁶¹⁾ – as far as the 1953 priority was concerned – was devoid of any disclosure regarding a plastic, moldable, solid polypropylene material. The specification only mentioned that solid, partially crystalline polymers could be obtained.

In early 1987, the Board of Appeals of the US Patent Office [275] affirmed the Examiner's position and denied product protection for Studiengesellschaft in view of the two prior art references, Field and Hogan. As grounds for the denial, the Board found, with respect to Hogan, that the reference described a solid polypropylene material consisting of recurring propylene units with a crystalline content. Regarding Field, the Board pointed out that the patent disclosed the production of solid polypropylene and generally stated that the polymers could be worked into films⁶²⁾. The fact that the decision in the civil action had not affirmed the validity of Field's priority date, but, instead, had moved it up to October 15, 1954, was being disregarded here. It was furthermore not apparent, pursuant to the Board of Appeals, that the polypropylene product according to Hogan had a different structure than that claimed by Studiengesellschaft; the same being true in connection with Field.

Thus, if according to Hogan and Field, both products were structurally identical, it was difficult to see why Field (Standard Oil of Indiana) had not been awarded priority, since the Standard Oil of Indiana application was the older.

61) Civil action 4319, US District Court for the District of Delaware, following the interference 89,634 (494 F. Supp. P. 370–461, 206 U.S. P.Q. p. 676, 1980), decision confirmed by 3rd Circuit court in 1981.

62) But see 494 Fed. Suppl. Civil Action No. 4319, page 397–410.

The case was now appealed to the US Court of Appeals for the Federal Circuit, the highest appellate forum [276]. But here, too [277], Studiengesellschaft was unsuccessful. The judges affirmed [278] the prior decisions. They added, however, that the applicant, Studiengesellschaft, had, in fact had an opportunity to support its arguments by submitting comparison tests in the form of affidavits.

Following the court's hint, the patent application involved in these proceedings was re-filed as a continuation application (identical disclosure) in connection with which Martin now introduced in the form of sworn declarations, the findings obtained from his series of experiments concerning Field and Hogan. From experiments reworking the parameters according to Field [279], no solid but at best, oily products had been obtained. While a repetition of the experiments according to Hogan [280] did yield solid, even partially crystalline products, any solid fractions which had been obtained through solvent extraction according to given procedure, were either waxes or brittle rather than flexible substances, all of which were soluble in boiling heptane (whereas highly crystalline, thermoplastic polypropylene is insoluble in boiling heptane).

In early 1989, the Examiner [281], E. Smith, also rejected this new application. He, again, refused to take into consideration the fact that Studiengesellschaft's claims called for the product to be plastic and/or flexible. Now, however, the Examiner cited an experiment by Baxter of Du Pont [282] from May 1954, that is, prior to the August 1954 priority date claimed by Ziegler/Martin (see Chapter 1, Section 1.3.1.2). The court in the 1980/81 Civil Action 4319 had already ruled that this product⁶³⁾ did not meet the legal requirements for an award of priority since no utility had been disclosed for the product. Du Pont had been awarded a priority date of August 19, 1954, that is, after Ziegler's priority date. The court, so the Examiner now argued, had apparently been unaware of the fact that a few days before Ziegler et al.'s priority date, Baxter's product had been characterized as crystalline in nature and its utility described in at least the same manner as that disclosed in the Ziegler application. If the Du Pont application lacked an adequate utility statement, this would also be true for Ziegler's German priority application⁶⁴⁾.

Initially two further declarations [283, 284] were submitted to the Examiner, which discussed the older patent rights by Field in the context of the decision rendered in Civil Action No. 4319 which has been reviewed previously. The judges, at that time, had not addressed the two Field publications. Instead, they defined the required characteristics for the polypropylene product in question, which were not

63) 494 Fed. Suppl. page 370–461, 1981 Civil Action No. 4319, District Court of Delaware, decision of January 11, 1989, page 390 left-hand column, last line to line 1 in right-hand column: "Du Pont's scientists did not recognize what they had nor did they sufficiently prove the utility of their product." Page 395, left-hand column, lines 29–33: "Therefore, the Board's determination that Du Pont did not recognize utility is affirmed." Page 397, left-hand column, lines 24–27: "This Court there-

fore affirms the Board's Opinion awarding Du Pont a priority day of August 19, 1954."

64) The patent office in its decision in interference 90 833 (three-party-interference, Ziegler, Du Pont (Baxter), Montecatini (Natta), decision in favor of Ziegler 1969) did acknowledge as sufficient the disclosure of the utility of solid polypropylene in the Ziegler priority application. The same statement was made by the court [286] in the action Studiengesellschaft v. Eastman Kodak.

met by Field and/or Standard Oil of Indiana until October 15, 1954. In 1956, Natta had repeated Example 21 of the '647 Field patent, and was not able to produce any solid polypropylene but merely a hydrocarbon oil (see Chapter 3, p. 115, Chapter 1, reference [16]). In a final declaration [285] concerning Hogan, Martin had described seven experiments, in which polypropylene produced with Ziegler catalysts showed melting points above 140 °C, in order to demonstrate that Ziegler polypropylene was different from that making up the Hogan-and-Banks products. In Civil Action 4319, the court found, with respect to Hogan of Phillips Petroleum, that Phillips did not disclose the use of the product as a solid, plastic material, but, instead, as a “wax modifier” (Fed. Suppl., page 418, right-hand columns 12–15 and 16–18)⁶⁵.

The Examiner disregarded the requirement that the polypropylene as claimed by Studiengesellschaft must be plastic and flexible. He evaded further confrontation by maintaining that Studiengesellschaft's claims did not exclude the products of Hogan and Field [287]. This called for the same procedure as was followed some years earlier. In late 1989, Studiengesellschaft, again filed an appeal [288] to the Examiner's Final Rejection. In his Reply Brief [289] answering the appeal, the Examiner's arguments became increasingly bizarre. He thus claimed that a polypropylene material according to Hogan, which had been described by Martin as liquid at 140 °C, could most certainly be processed at that temperature by pouring it into suitable molds. Apart from that, he reiterated that the priority application by Ziegler and co-workers failed to provide an adequate description of the utility, for instance, of polypropylene and, specifically, of processing the solid products at temperatures above about 140 °C. At best, the application indicated that sheets could be formed by pressing the product at 140 °C. Support could not be found in the specification for either “about” or “above about 140 °C”, as called for in the claims. Thus, if in Civil Action 4319 the court came to the conclusion that Du Pont did not provide an adequate utility disclosure prior to August 1954, then the same would be true for Ziegler's German priority application.

In its decision [290], the Board of Appeals accepted the substance of the five Martin Declarations, according to which the polypropylene claimed here was not identical to the product as described by Hogan and Field – a partial victory for Studiengesellschaft. The Board then continued by stating that the invention on which the polypropylene product claims were based had however, first been described by Baxter in the United States. In this connection, it should be pointed out, the Board stated, that the utility of an invention is to be differentiated from the invention *per se*. If a description of the utility as claimed by Studiengesellschaft cannot be found in the specification, then the requirement for priority based on utility has not been met and the filing date of the Baxter application becomes eligible.

65) Judge Langobardi decided in the action Phillips Petroleum v. US Steel that the polypropylene disclosed in the Phillips application of 1953 was a brittle polymer of low molecular

weight. There was no commercial production of this product (page 1125, 6 U. S. P. Q 2d 1065).

It was at this point, at the very latest, that Studiengesellschaft could hardly deny the fact that the decision-making panels both at the US Patent Office and the competent patent courts, were not willing to grant them a further patent with a 17-year period of effect, which would have enabled them to dominate any polypropylene production in the United States through its patent rights. Prior decisions were set aside.

Nevertheless, the claim in question was amended by deleting the word “about” with respect to the temperature of 140 °C, in order to overcome the Examiner’s and the Board’s objections [291]. The Patent Office, however, refused to accept the amendment [292].

Studiengesellschaft appealed the case to the highest appellate court. In 1993, the “US Court of Appeals for the Federal Circuit” [293] held that if it was true, as the Patent Office and lower court had determined, that Ziegler was not entitled to rely on his German priority date, then the invention would be anticipated by the Du Pont (Baxter) ’680 patent [282] of August 19, 1954. The only issue to be decided was whether or not the Examiner and the Board of Appeals had been correct in ruling that Ziegler’s German application did not contain a statement of practical utility. Upon close scrutiny of the specification, the court concluded that Ziegler did not disclose any practical utility for polypropylene or polypropylene film.

This was the crucial finding based on which the German filing date was not recognized as a priority date for the product claim. No doubt, this finding, contrary to all previous decisions, had the desired effect that no further patent would be granted for this invention. The ruling that a film made of polypropylene did not constitute an adequate showing of utility was unusual. It had taken the Patent Office and the courts 8 years to come to this conclusion. The newly rendered decision was in sharp contrast to several earlier decisions that had been handed down by among others, the Patent Office and several courts (footnotes 63–65).

The fact that no further patent could be obtained was not the only outcome of this drawn out process. At this point, there was no longer any concern about patent protection for polypropylene. It should, however, be mentioned, as a further partial result, that no prior publication or any effective older patent right ever existed which anticipated the polypropylene product claimed by Ziegler. The polypropylene described by Hogan of Phillips, although partially crystalline, was a brittle material which, at best, was useful as an additive in candle making. Field from Standard Oil of Indiana, had described a polypropylene material which could be characterized as more oily than solid. Though older than Hogan’s findings, the disclosure could not even stand up as prior art over Hogan. With respect to Natta, the only valid date was Natta’s US filing date, which was clearly later than the German priority date relied on by Ziegler/Martin. Furthermore, Natta’s experiments involving polypropylene, although conducted earlier than Martin’s experiments, were not considered to be an independent invention.

In addition to the available documentation, it is the volume of licensing business conducted with US polypropylene producers which serves as further proof that the situation outlined in the previous paragraph has been portrayed accurately.

5.10

“High Speed” or “High Mileage” or “Ziegler Catalysts of the Second and Third Generation”

In the first half of the 1970s, experimentation by different, predominantly industrial, research teams led to enhanced polypropylene yields with the use of Ziegler catalysts, and to initial patents covering these improvements. The aim of such experiments was to increase the activity of the catalyst to such a degree that removal of catalyst residues from the polymer became unnecessary, with now only minor amounts of atactic, sticky polypropylene by-product being formed [294]. It was developments at Montedison, Italy, in particular, which involved the use of so-called special titanium carrier co-catalysts as catalyst components [295]. The inert carrier consisted of finely ground magnesium chloride. Surprisingly, it was found that, again, it was the liquid or dissolved titanium tetrachloride that proved to be the starting material of choice, which was then applied to the carrier in the form of a very thin coating. For the first time, however, the catalysts were produced with the addition of so-called donors, Lewis bases, which were generally termed “complex formers”. Such complexes were to be formed not only with the titanium compound, but also with the second catalyst component, the aluminum compound. This process allowed for a dramatic reduction in the amount of titanium component required [296]. In Japan, parallel investigations were underway at Mitsui Petrochemical [297]. Subsequently, both companies, Montedison and Mitsui, filed individual as well as joint patent applications [298]. Furthermore, experiments which used this optimized process had been described in numerous publications. Yet only a few of these publications reported experimental results with respect to products obtained from a chemical reaction where the aluminum compound involved included added donors with aromatic esters of the alkyl-benzoate type being the donor of choice.

Those using these “high-speed” catalysts claimed that their work was entirely independent of Ziegler and/or Studiengesellschaft patent rights. They pointed out that there was no court decision which dealt with a system involving magnesium chloride-carrier catalysts with added aluminum triethyl [294].

In any subsequent commercial processes utilizing the improved catalyst systems described above, the aluminum compound, for example aluminum triethyl, was first admixed with the donor and only then was the titanium component added.

Karl Ziegler and co-workers had already published a report which described the reduction of carboxylic acid esters (cinnamic acid ester, benzoic acid) with aluminum trialkyls [299]. The preferred (molecular) ratio employed in the cases described in that publication was two aluminum trialkyls per one ester and more. As described by Karl Ziegler and co-workers, the reduction of aromatic alkyl esters using this ratio produced a dialkylaluminum alkoxyl and a dialkylaluminum arylalkoxyl. In 1983, a paper [300] was published by B.L. Goodall describing research findings obtained at Shell’s laboratory in Amsterdam. At the time of the publication, the author was already working for Shell Development Co. in Houston, Texas. It is important to mention this fact because, in his publication, Goodall stated “that esters, under polymerization conditions, will undergo irreversible chemical reactions with aluminumtri-

ethyl," whereas US-based Shell Oil, in a subsequent litigation, vehemently denied this with regard to its own production plants. Goodall presented a picture outlining the course of the reaction. The products obtained were comparable to those previously described by Ziegler and his co-workers.

The same results were arrived at by researchers at the Gulf Research Center [301] in Pittsburgh. The authors suggested that in the new type of catalysts, it was the diethylaluminum alkoxides which formed that were the actual modifiers in the olefin polymerization process, and which were responsible for the crystalline content of the polymers obtained at a comparatively higher polymer yield.

Aromatic esters were in fact, utilized when this catalyst system subsequently became available for industrial use. In the early 1980s, the large-scale production of polypropylene was based on two alternate catalyst systems, consisting, on the one hand, of the titanium trichloride/diethylaluminum chloride catalysts which had been conventionally employed up to that time, and, on the other hand, titanium tetrachloride/magnesium dichloride/aluminum triethyl and ethyl benzoate.

In the mid-1970s [302] a search began which continued more extensively into the early 1980s, for donors which exhibited the same or improved activity, and which, for various reasons, could be used to replace the aromatic ester (ethyl benzoate). The search led to the silylethers, such as for example, phenylsilicon trimethoxyl or diphenylsilicon dimethoxyl. As early as 1987, concurrently with work carried out by Martin [303] at the Max-Planck-Institute in Mülheim, articles were being published [304] which disclosed identical results inasmuch as the alkoxy groups of the silicon ether reacted with aluminum triethyl, with an exchange taking place between the alkoxy group on the silicon and an ethyl group on the aluminum, and this, in turn, caused the formation of diethylaluminum ethoxyl compounds among others.

These findings were not without consequences for Studiengesellschaft Kohle, Mülheim, in terms of patent rights: the required presence of diethylaluminum alkoxy compounds was covered by the claims of Ziegler's catalyst US Patent 3,113,115 (the '115 patent), as well as the Ziegler/Studiengesellschaft process US Patent 3,125,698⁶⁶⁾ (the '698 patent). Thus, in place of the aluminum component, diethylaluminum chloride, in the catalysts employed up to that time, it was now the diethylaluminum ethoxy compound which had become an essential part of the catalyst. Montecatini was well aware of this situation [305]. Working with the utmost secrecy, Montecatini refined the formula for producing the titanium component and sold the resulting product to polypropylene producers, particularly in the United States, who, in turn, manufactured the catalyst by combining the product with aluminum alkyls, plus a donor, and then used it in their operations.

In early 1985, Studiengesellschaft found itself facing difficult decisions with respect to its licensing business in the United States, particularly regarding its relationship with Novamont, now US Steel. US Steel had paid the damages accrued under the expired '115 patent which had been ordered by the court in the appeal decision [172, 174, 175] against them, but had ignored its royalty obligation under

66) US P 3,113,115 expiration date Dec. 02, 1980,
US P 4,125,698 expiration date Nov. 14, 1995.

the second patent, the '698. At the time, that patent was involved in appeal proceedings [247] seeking to overturn the unfavorable decision [245] (based on a judgment of unlawful double-patenting) which the District Court had rendered against Studiengesellschaft in the Northern Petrochemicals (NPC) case (cf. p. 215).

The 6-year statutory period of limitation in connection with the infringement of the Ziegler/Studiengesellschaft '698 patent by US Steel's operations at both of its plants, i.e. Neal in West Virginia and La Porte in Texas, required an immediate decision [306] as to whether an action should be brought against US Steel/Novamont. In view of the potential ramifications concerning production by other infringers, Studiengesellschaft filed a Complaint [307]. The Neal, West Virginia, plant continued operation utilizing a catalyst consisting of titanium trichloride/diethylaluminum chloride. With regard to this situation, it remained to be seen whether the validity and enforceability of the '698 patent would be upheld. With respect to the nature of the catalyst employed at the La Porte plant, Studiengesellschaft was merely informed that the compound in question was a titanium-containing "Hi-Yield" component of unknown composition, which, however, in admixture with aluminumtriethyl and a donor, formed the catalyst [308]. Apart from this, they were told, any further details would fall under the supplier's "trade secret"⁶⁷⁾. Based on this information, Studiengesellschaft was constrained to amend the Complaint to include both the '332 and '792⁶⁸⁾ patents, which claimed the use of titanium compounds and aluminum trialkyls as a catalyst mixture.

Judge Langobardi ordered [309] that the parties must conclude their discovery within 9 months of the appeal decision concerning the '698 patent (Studiengesellschaft v. NPC, see p. 216). From the evidence collected (such as "discovery"), it became apparent that US Steel produced not only polypropylene but also co-polymers of ethylene and propylene and that the La Porte plant had gone on stream in 1979. US Steel took the position [310] that no valid patent existed covering the production of polypropylene. They had meanwhile acquired a process patent [311] which, according to US Steel's testimony, disclosed the process as practiced by them.

Between September 1986 and February 1987, US Steel changed its name to "US X Corporation" and its associated company "Aristech Chemical Corporation" joined the proceedings as a co-defendant [312].

Concerning the chemical side of the argument, it turned out that, contrary to US Steel's assertion, their catalyst was produced by actually first admixing aluminumtriethyl with the donor, and then introducing the mixture into the polymerization vessel which contained the titanium component suspended in propylene [313]. The

67) In the settlement agreement between Studiengesellschaft and Montecatini 1983 (see Chapter 4, reference [136], article 3, e), Montedison protected themselves against Studiengesellschaft by retaining the right for sale and export of catalyst components including a titanium component which it had developed and which was kept secret.

Studiengesellschaft reserved the right to act against an unlicensed user of such components, for instance, for the preparation of the catalyst in the polymerization of olefins.

68) US P 3,257,332 expiration date Jun. 21, 1983, US P 3,826,792 expiration date Jun. 21, 1983.

fact is, however, that when aluminumtriethyl and silylether are combined, diethylaluminum ethoxyl can be distilled off under mild conditions.

At the same time, USX had outlined 16 defense arguments in the form of answers to interrogatories posed by Studiengesellschaft. It was interesting to learn from these answers that, for the La Porte plant, a license agreement had existed between Montedison and Novamont since March of 1981, covering Montedison's newly developed catalyst system [314]. USX was, indeed, correct in its contention that after the expiration of Ziegler's '332 and '792 US Patents in June of 1983, no infringement could have taken place under these patents. The same was true for the '115 patent after its expiration in December of 1980. Thus, concerning the time period after 1983, the only issue to be decided was whether the '698 patent was valid and had been infringed.

In the specification of this patent the two catalyst components were described as being admixed prior to contact with the olefin to be polymerized in order to bring about the reduction of the titanium compound. USX now argued – without evidentiary proof – that such a reduction was neither necessary nor was it being practiced, and that the mixing of the two components took place in the presence of propylene.

From the deposition of one of US Steel Chemical's consultants, Mr. A. Amato, taken in May of 1987, it was learned, that in connection with polypropylene produced between 1979 and 1987, different donors[315] had been employed in preparing the catalyst, and what these donors were⁶⁹).

In mid-1988, initial settlement discussions got under way. The decision on the validity of the '698 patent had meanwhile been rendered in favor of Studiengesellschaft [248], all available witnesses had been deposed during discovery, and all briefs setting forth the parties' arguments had been exchanged. Particularly in view of the above-mentioned decision in the '698 case, USX offered 0.5 to 1 million dollars to terminate the action and settle all claims under any of Studiengesellschaft's patents covering the production of polypropylene [316]. Kramer and Martin exchanged data analyzing the capacities of the two production plants and the royalties to be derived therefrom. Studiengesellschaft made a counter-offer in the amount of 8 million dollars [317].

USX/Aristech Chemical now changed their strategy. P.E. Crawford, counsel for USX, tried to compel a partially favorable outcome by filing a motion for summary judgment⁷⁰) on issues not involving the chemical aspects of the case, but, instead, dealing with contractual questions. In early 1989, Judge Langobardi rendered his decision on the motion [318]. The court stated that the license agreement with Novamont, dated 1974 (Novamont had been taken over by USX) was void, since Nova-

69) The donors were methyl-paratoluate, phenyl-triethoxi-silane or diphenyldimethoxi-silane.

70) "Summary Judgment" is a legal key (Fed. A. Civ. P. 56 8 c), if "pleadings, depositions, answers to interrogatories and admissions on file, together with the affidavits, if any, show that there is no genuine issue as to any mate-

rial fact" and the party filing the motion has the right to obtain a decision under the law. The opposing party can prevent the decision by proving that there is still a dispute to the material facts and this not only in form of arguments.

mont owed Studiengesellschaft back-royalties and, up to 1981, had failed to make such payments (cancellation effective from July 1981). USX's accusation that Studiengesellschaft had breached the agreement by disregarding the "most-favored licensee" clause⁷¹⁾, was therefore irrelevant.

Once the cancellation became effective, Studiengesellschaft was in a position to claim damages for infringement. Royalties owed prior to the date of cancellation were distinct from damages for infringement. This issue had, however, previously been decided in favor of Studiengesellschaft (*res judicata* [171]).

The decision had its effect. In a letter dated March 1989 [319], Crawford, with authorization from USX, increased the settlement offer to 2.5 million dollars or, alternately, a payment of 0.5 million dollars for La Porte limited to the '698 patent (double-patenting issue), but with the litigation to be continued, that is, a repeat of the Studiengesellschaft v. Northern Petrochemicals case. In other words, the intention was to make the 2.5-million dollar settlement appear attractive. In any event, the proposal included a settlement payment for the operation which utilized the so-called High-Speed catalyst. Meanwhile the production capacity for polypropylene had increased to 650 million pounds annually. Since May of 1988, the entire production had been adapted for utilization of the new catalyst.

In a hearing before the court, the judge ordered that the discovery period, which had meanwhile expired, was to be extended for another 6 months, with the trial date [320] being set for October 28, 1991. Subsequently, Sprung turned over [321] to Crawford portions of a memorandum by Martin, including a description of the experiments conducted by him. Aristech secured Montecatini's support. Crawford submitted to Sprung a data sheet summarizing the results of experiments conducted by A. Zambelli [322] (Montedison), which were intended to show that the diethylaluminumalkoxyl compounds, which Martin had separated during his experiments, did not form active polymerization catalysts in combination with titanium compounds. Martin objected (see Chapter 1, Section 1.2.3) and, as proof [323], submitted the description of relevant experiments, which clearly demonstrated that crystalline polypropylene could most definitely be produced with these catalyst mixtures.

Despite several reminders, Aristech had managed, for more than six years, to keep Studiengesellschaft from obtaining samples of the titanium components which Aristech was using. Not until October of 1991 [324], and then only by court order, was Studiengesellschaft supplied with titanium carrier components. Using the conditions employed by Aristech's polymerization technology, it was now possible to carry out experiments. The experience was, however, that the results [325] were no different than those obtained in previous runs.

71) Many license agreements contain a "most-favored clause" which means that the licensee has an option right (within a time limit) to take over the conditions of a license agreement signed later by the licensor with a third party. It was disputed whether the licensor

was required to present any later license agreement or only those which in his opinion granted more favorable conditions to the third party compared with those to the first licensee.

Meanwhile, the judge had scheduled the trial for the second half of January 1992. Each party utilized the remaining time period to exert pressure on its opponent. Aristech/Crawford urgently demanded that the results of Martin's further test should be produced and that Martin's deposition should be taken prior to the beginning of trial. Studiengesellschaft pressed for further information on the manufacture of the titanium carrier component [326].

The results produced by Studiengesellschaft's test program made a definite impression. In mid-January of 1992, Aristech made a settlement offer [327] of \$5.25 million which was good for 3 days. Before the deadline expired, Sprung, on behalf of Studiengesellschaft, signed a settlement agreement [328], accepting Aristech's offer and withdrawing the Complaint. The settlement figure did not reflect the magnitude of Studiengesellschaft's claim, but further postponement of a final settlement and costly court battles were no longer desirable in view of the suits which had meanwhile been brought against Hercules/Himont and Shell Oil, involving the same subject matter.

At this point, Studiengesellschaft, through Arnold Sprung, wrote a letter [329] to Shell Oil Co., requesting that Shell, under the existing 1987 license agreement, provide information regarding any of its polypropylene production which, according to Shell, did not fall under Studiengesellschaft's '698 patent, i.e. details on the quantities and the characteristics of the process used to produce such quantities of polypropylene. In its reply [330], Shell stated that the production at its "Seadrift" plant was not included in the 1991 accounting since the catalyst used in the Seadrift operation did not contain diethyl aluminum chloride. This assertion was inconsistent with the terms of the existing license agreement (§ 4(d)), under which Shell was obligated to provide Studiengesellschaft with sufficient confidential information to allow Studiengesellschaft to make an independent assessment as to whether that production was, in fact, outside of the scope of the license grant. Initially, Shell obliged by providing the production figures for 1987 through 1991, this time including the "Seadrift" operation, with repeated assurances that the process in question did not use diethyl aluminum monochloride. After deduction of the 450 and/or 300-million pound production paid up capacity⁷²⁾ under license, the only remaining excess production on which an accounting was due was for 1987 and 1990. As a further piece of information [331], Shell offered that in the Seadrift plant, it was employing the "fluidized gas phase UNIPOL PP" process, using a "Shell-SHAC" catalyst. At the heart of this mysterious circumscription and similar to the Aristech case, was the use of a catalyst carrier of titanium chloride on magnesium chloride, whereby a further substance was now being added to the aluminum triethyl, i.e. the aluminum component, which, in Aristech's case, had been called a donor, while here it was described as a "selectivity control agent". Thus, it was true that diethyl aluminum chloride was not being used, but it was not true that, as Mr. Vance claimed, the donor had been injected into the polymerization reactor separately

72) Shell had sold its plant (capacity 150 million pounds) in Woodbury, New Jersey to Huntsman Chemical Co.

from the other catalyst components. Martin had pointed this situation out to Sprung and added that B.L. Goodall, Director of Shell Development Company, had published the result that when aluminum triethyl was admixed with the donor component (for example, ethyl anisate), diethylaluminummethoxyl was being formed [332]. Sprung pointed out these facts to Mr. Vance in a letter. Needless to say, the situation was heating up. Nearly 900 million pounds of polypropylene, produced between 1987 and 1992, were left out of Shell's accounting because Shell believed that its technically improved, so-called gas phase process was an independent procedure [331, 333], whereas Studiengesellschaft took the position that the polymerization method used was one which utilized a Ziegler catalyst as claimed in the '698 patent.

Studiengesellschaft pushed to obtain the titanium components (SHAC) used by Shell, so that it would be able to conduct its own experiments. Shell denied the request and, instead, proposed tests to be demonstrated by its own experts. Shell was playing its "cards" close to the vest. Thus, Martin was to carry out his own experiments only in the presence of a Shell scientist [334] – under the strictest secrecy, of course.

In March of 1993, Studiengesellschaft cancelled all license agreements due to breach of contract and filed suit in New York, because Shell had neither furnished adequate accountings for any production in excess of the royalty-free ceiling, nor paid royalties for such excess production [335].

The first and last official act performed by the judge was changing the venue of the case to the US District Court of Texas [336] on the grounds that the majority of the witnesses resided in Texas and Shell Oil's production plants were located there. Six months later, formal transfer of the case had been completed. The case was now being handled by Judge V.D. Gilmore in Texas [337].

During discussions regarding details of the process being practiced at the Shell plant, Shell maintained that the formation of diethylaluminumalkoxide during the preparation of the catalyst was "undesirable". All flow charts, however, indicated that the donor was admixed with aluminum triethyl prior to its introduction into the actual polymerization vessel. A reaction of the two substances, Shell now argued, would be impossible, given the short time available for mixing prior to introduction into the polymerization reactor [338]. What is more, a diethylaluminumalkoxide together with the titanium carrier component would be utterly ineffective in the presence of donors. This last distinction was not relevant, since the parameters chosen for the test results submitted by Shell were different than those used in their commercial operation.

Spectroscopy tests (NMR) – carried out at the Max-Planck-Institute for Coal Research using test equipment constructed specifically for this purpose, with the aim of determining the second-to-second nature of the reaction products formed from aluminum triethyl and donors – proved that a reaction between these two substances led directly to the formation of diethylaluminumalkoxide [339]. The fact that formation of the diethylaluminumalkoxide occurs after 1–7 min had already previously been ascertained. The amount of diethylaluminumalkoxide was, in each case, larger than the subsequently added amount of titanium component (molecular ratios). It was furthermore possible to establish that it was the diethyloalum-

inumalkoxide formed which controlled the ratio of crystalline polymer being produced. Shell, meanwhile, had made samples of its titanium component available to Studiengesellschaft.

In mid-1986, Martin again summarized the findings of the experimental program in the form of a memorandum [340]. Meanwhile, several test results obtained by Shell's own experts Kilty, McGrath and Goodall had been made public, which - probably inadvertently at best - confirmed Martin's test results to the extent that, when using a mixture of diethylaluminumalkoxide and different titanium components (Shell), it was indeed possible to obtain high yields during the polymerization of propylene⁷³). Concerning the chemical aspect of their argument, Studiengesellschaft was able to show conclusively that in the polymerization process as described by Shell, and as also practiced at the Seadrift plant, the catalyst components as claimed in the '698 patent constituted the essential, effective elements: diethylaluminumalkoxide and titanium halide.

It would be appropriate at this point to revisit the year 1986, because it was then that Studiengesellschaft had filed suit against Hercules, Inc., the subject matter of which was closely related to the issues tried in the Shell case.

As a consequence of the successfully concluded dispute with NPC concerning the validity of the '698 patent, Studiengesellschaft, brought an action [341] in 1986 against Hercules, which was amended to also include Himont, because in late 1983, the latter had acquired Hercules' entire polypropylene operation. At that time, polypropylene and propylene - ethylene co-polymers were produced at the Bayport, Texas, and the Lake Charles, Louisiana, plants, using a conventional Ziegler catalyst, consisting of titanium trichloride and diethylaluminum chloride [342].

Information then became available that Hercules had also been producing co-polymers since 1979, without, however, accounting to Studiengesellschaft on their sales [343]. Hercules argued that these co-polymers, containing 90% and less propylene, did not come under the existing agreements. Studiengesellschaft objected, stating that the 1954 agreement did, in fact, cover the production also of these polymers. Then, in 1983, Hercules and Studiengesellschaft agreed to interrupt the running of the statute of limitations until both parties had sufficient opportunity to examine the facts.

Himont expanded the acquired production plants [344] between 1984 and 1986, and now used carrier catalysts, titanium tetrachloride on magnesium chloride as the carrier, in combination with triethylaluminum and an electron donor as the catalyst mixture⁷⁴). As in the previously described cases, proof had to be collected that the addition of donors would lead to the formation of adequate amounts of diethylaluminumalkoxide.

73) It was published knowledge that diethylaluminumalkoxide exists as dimer but also as trimer. With a higher average degree of association of the aluminum compound its activity decreased. Therefore, the different effectiveness of catalysts using diethylaluminumalkoxide seems to be connected with the degree of association. Under the polymeriza-

tion conditions applied in the Shell plant the aluminum component was present as a dimer at the most, possible also as monomer.

74) Ziegler /Studiengesellschaft US P 3,826,792; US P 3,257,332; catalysts prepared from titanium halides and aluminum trialkyls, expiration date June 21, 1983.

Aside from the familiar defense strategy used in prior cases – laches, invalidity due to double-patenting of the '698 patent – now, for the first time, Studiengesellschaft was accused by Hercules, of having breached existing agreements by disregarding the most-favored licensee clause (see footnote 71), particularly Hercules' right to be offered any more favorable third-party license terms [345].

In late 1986, Studiengesellschaft, through Arnold Sprung, offered Hercules a license under the '698 patent, but Hercules and Himont demanded a retroactive license going back to May 1, 1980 at the same terms as had earlier been granted to Amoco Chemicals Co. in their settlement agreement [346]. Studiengesellschaft refused. There could be no most-favored licensee rights for Hercules concerning past infringement, and for the time period after the expiration of the '115 patent (December 3, 1980), the 1972 Ziegler/Hercules agreement provided that Ziegler would be willing, upon request, to grant a license under terms no worse than those granted to the other "paying" licensees. Up until 1986, when the validity of the '698 patent was upheld by the court, there was, however, no paying licensee. The point of the most-favored licensee clause was to make the polypropylene sales of different producers subject to the same royalty obligation [347].

On this basis, a settlement appeared to be almost beyond reach. Nevertheless, Martin made an attempt, through direct contact with Mr. S. M Turk, Vice President and General Counsel of Hercules, to initiate an offer/counter-offer exchange between the parties. The figures suggested [348], i.e. 1 million dollars offered by Hercules versus 6.6 million dollars requested by Studiengesellschaft, were miles apart.

The parties now embarked on a whole new strategy. Out of the wealth of arguments and counter-arguments available, each party chose the legal position which appeared most promising for its case and tried, step by tiny step, to strengthen its position through partial summary judgement in its favor, or even to force a final decision.

In 1990, Hercules sought to have a judgment rendered on the issue of laches on the part of Studiengesellschaft. For this purpose, they filed a "Motion for Partial Summary Judgment"⁷⁵⁾. Studiengesellschaft responded by filing its own "Motion for Partial Summary Judgment" based on the assertion that Hercules had failed to render an accounting of its production and sale of ethylene – propylene co-polymers.

First, it must be remembered that there were four license agreements with Hercules which governed the royalties to be paid for the production and sale of polyolefins. The first agreement, dated 1954, remained in effect to the extent that the subsequent agreements did not call for any changes. The 1962 agreement confirmed, in letter form, the settlement regarding polyethylene. The 1964 supplement agreement covered the royalties payable for the production of polypropylene and co-polymers (mixed polymers) containing more than 90 mol% of propylene. Finally, the 1972 agreement spelled out the paid-up terms for polypropylene up to a ceiling of 600 million pounds of annual sales. As before, co-polymers containing less than 90% propylene continued to come under the 1954 agreement.

75) See footnote 70.

In the State of Delaware, the statute of limitations for claims based on infringement was 3 years. In their response to Hercules' motion, Studiengesellschaft pointed out that it was not until 1983 that they learned of Hercules' failure to account on co-polymers. It was in 1986, prior to the expiration of the 3-year period and shortly after the court in the *Studiengesellschaft v. NPC* civil action had upheld the validity of the '698 patent, that Studiengesellschaft filed the instant suit.

In late 1990, Judge J.J. Farnan found [349] that the statute of limitations on Studiengesellschaft's claims had run out for the period 1972 through 1979, but not for the year 1980. The statute of limitations started running from the moment adequate grounds for a suit had been established, even if though not all of the facts were known at the time⁷⁶⁾.

In 1992/93, both parties renewed their attempt to have the court reach a partial judgment. While Hercules/Himont believed they had found new arguments [350] with respect to unlawful double-patenting and had identified the invalidity of a number of the claims in the '698 patent, Studiengesellschaft referred to the appeal decision [248, 249] rendered against NPC (Northern Petrochemical Co.), wherein the court, contrary to the opponents' assertions, had decided the double-patenting issue in favor of Studiengesellschaft⁷⁷⁾.

In his decision [350], Judge Farnan denied Hercules' motion on the grounds of the highly divergent contents of the expert opinions. Under these circumstances, it was not within the court's discretion to render a partial decision in favor of the moving party, Hercules. A court order to that effect was issued in late 1993, at a point in time when Hercules, for the third time, had made a stab at proving that Studiengesellschaft was in breach of contract.

Simultaneously with the events as portrayed, Martin conducted an experimental program employing variations of the titanium components that had originally been used by Himont. It should be noted here that Himont, like Shell, did not make samples of its titanium catalyst component available to Studiengesellschaft until compelled by court order [351]. Repeating the parameters as described by Hercules, the experiments yielded the same results as previously disclosed in Martin's Memorandum [321, 352].

In May 1980, Studiengesellschaft had resolved its past differences and had defined its future arrangement with Amoco in a settlement agreement [157]. The '698

76) Studiengesellschaft could not demonstrate that fraudulent concealment had occurred. It was not Hercules's duty to assure that all of Studiengesellschaft's rights were being protected. The Court concluded that Studiengesellschaft was not "blamelessly ignorant" of the facts. The Studiengesellschaft inquiry of 1983 and the agreement reached then according to which the defendant Hercules waived the statute of limitations, caused Studiengesellschaft to bring an action in 1986. Delaware had a 3-year statute of limitation in this case.

77) 1. Protection of the '698-patent in view of 35 U.S. C. paragraph 121.
2. Application of the test to examine double patenting by the Appeal Court, the patent to be "identical" and/or "obvious".
3. The Court accepted Martin's statement as a witness, according to which catalysts covered by the '115 patent could be used differently as described in the '698 patent, i.e. using the catalyst covered by '115 patent without infringing the '698 patent.

patent had already been issued at that time and was incorporated into the settlement agreement.

Hercules now complained that, under the most-favored licensee clause of its agreements with Studiengesellschaft, the latter had failed to offer Hercules the same terms as had been agreed in the Amoco settlement agreement.

Both parties presented their positions in voluminous briefs and lengthy rounds of hearings before the judge (November 1994). In the end, Hercules prevailed. In a decision [353], handed down in mid-1995, Judge Farnan affirmed that Studiengesellschaft had disregarded their obligation under the 1954 agreement to advise Hercules of the Amoco agreement, and that Hercules was entitled to adopt the terms of that agreement with respect to the '698 patent. This decision was upheld 18 months later by the appeal court.

Interesting to note in this context was the courts' position that Studiengesellschaft did not have a right to determine whether the terms of a third-party license agreement were more favorable or less favorable. Thus, regardless of the circumstances, Studiengesellschaft was obligated to advise Hercules of the Amoco agreement, pursuant to the 1954 agreement with Hercules. The latter was also entitled to a guaranteed option under the 1972 settlement agreement. At that time, the '698 patent (and/or the corresponding patent application) had been licensed to Hercules, but only up to December, 1980, when the '115 patent expired. Now, in 1979, prior to entering into the settlement agreement with Amoco in 1980, Studiengesellschaft had cancelled the principal 1954 agreement with Hercules because of non-payment of royalties. In order to have the cancellation withdrawn, Hercules had subsequently made a payment within a contractually-stipulated period, which – as became apparent later on – had been substantially lower than called for [354]. Studiengesellschaft's argument that withdrawal of the cancellation was, therefore, not legally effective and Hercules was not entitled to the terms of the Amoco settlement agreement was now rejected by the courts because Studiengesellschaft, pursuant to the courts, had accepted payment at the time, in 1979, and had failed to verify the accuracy of the amount of this payment through a qualified audit. The burden had been on Studiengesellschaft to insure that the amount paid was accurate. Suspension of the cancellation had, therefore, been lawful in light of a payment having been made, and the most-favored licensee clause of the 1954 agreement was deemed to be applicable.

Furthermore, the court rejected the argument that the 1972 Hercules settlement agreement contained a modified most-favored licensee clause, pursuant to which an option would be granted, after expiration of the '115 patent, for a license under the '698 patent at terms no worse than those granted to other paying licensees, but that Amoco was not a paying licensee. The court stated that the payment made by Amoco under the settlement agreement of 1980, would fall into this category. The lump sum settlement payment was a payment made by a "paying" licensee.

Neither the court nor the parties could know for sure whether Hercules would have adopted the terms of the Amoco agreement in 1980. Studiengesellschaft should have ascertained this information at the time. Confidence in the fair and accurate accounting by a licensee with whom Studiengesellschaft had enjoyed a loyal

business relationship for 30 years was severely shaken because Studiengesellschaft had failed to have Hercules' books examined at the proper time.

Hercules adopted the terms of the Amoco agreement and paid 1.2 million dollars as a settlement sum for the '698 patent.

Now back to Shell. News of Judge Farnan's decision in the Studiengesellschaft v. Hercules litigation had spread. In a letter to Martin, Mr. D.F. Vance (Shell Oil) demanded that Shell retroactively be granted the terms of the 1980 Amoco agreement [355].

Prior to this time, Judge V.D. Gilmore had rendered decisions [356] in the Shell case based on summary judgment motions filed earlier by Shell and Studiengesellschaft. Before going into detail regarding the new accusations, these two decisions must be examined. The first case involved issues of patent law, while the second dealt with questions of contract law. Both decisions were appealed by the parties and the issues were finally adjudicated by the appeal court [357].

The decisions are very interesting, because throughout the history of American jurisprudence, one and the same issue has been adjudicated in different ways, depending on the case.

In the first case, the '698 patent was based on a "continuation-in-part" (CIP) application filed in 1958, which consisted of a combination of three original applications filed in 1954. During the intervening period between the two filing dates, 1954 and 1958, Belgian Patent 538 782⁷⁸⁾ had been issued in 1955, that is, more than 1 year⁷⁹⁾ earlier than the filing date of the '698 application. Studiengesellschaft referred to the patent rule, according to which exceptions existed where the specifications and claims of earlier applications, the so-called parent applications, had been carried over into the CIP applications. This is where the different interpretations came into effect.

The judge cited the rule of law (35 USC Sec. 112) which required that the invention as reflected by the claims of the '698 patent be disclosed in one of the parent applications filed in 1954. While Studiengesellschaft requested that the court compare each patent claim in the '698 patent with the original applications taken as a whole. However, the Judge felt that this request was not covered by law, but rather, that the comparison must be undertaken separately with each individual parent application. Such a requirement had never been issued by the Patent Office during prosecution of the '698 patent. In the Judge's opinion, the first parent application described, on the one hand, the polymerization only of ethylene, rather than that of alpha-olefins generally, and, on the other hand, set forth the use of transition metal compounds of metals from group VIII (of the Periodic System of Elements) only in combination with dialkylaluminum halides, whereas the '698 patent disclosed the polymerization of alpha-olefins much more broadly and the use of the transition metal compounds of metals from group VIII with organoaluminum compounds in general.

78) A combination of the first patent application "polypropylene", Natta/Montecatini with the first patent application Ziegler/Martin.

79) US law, 35 section 102b, US-Code provides that a person shall be entitled to a patent

unless the invention was patented or described in a printed publication more than 1 year prior to the date of application for a patent in the US.

The third parent application made no mention of the polymerization of olefins generally, nor did it specify the group VIII metals. If the '698 patent thus failed to meet the necessary requirements, the priority of the published Belgian patent would preempt that of the '698 patent. The court declared a crucial number of the claims in the '698 patent to be invalid, 17 years after issuance, a decision which in effect, negated the ruling of the US Patent Office.

The second case. Invalid claims cannot be infringed. Shell, however, had entered into a license agreement under the '698 patent and did not challenge the validity of the patent until 1993. Thus, there needed to be clarification as to whether Shell, in acknowledgement of the '698 patent and the license agreement entered into, was obligated to pay royalties up to the time when the Complaint was filed. The Judge was not willing to rule on this issue since the dispute between the parties involved facts which precluded a decision at this point. She ordered the parties to turn this latter issue over to the court of appeals promptly for a ruling, and stated that 10 days after the appellate decision had been handed down she would continue trying the case.

It was not until May 1997 that the court of appeals [357] rendered a judgment which was partially in favor of Shell and partially in favor of Studiengesellschaft, while other outstanding issues were remanded to the District Court. Shell prevailed in having a crucial number of the '698 patent claims declared invalid. It was improper to combine the specifications of two applications which had been filed earlier, 1954 (see above), into a subsequent application in order to secure the benefit of an earlier filing date and/or priority date for a substantially broader application. The individual parent applications did not support the invention as claimed in the '698 patent, and hence, several crucial patent claims in the '698 patent were invalid in view of the Belgian patent application which had been published earlier.

Studiengesellschaft was successful in its demand for payment of royalties for the period starting from the signing of the last license agreement up until the Complaint was filed in 1993. The court of appeals found that Shell had enjoyed the advantages of producing polypropylene free of unlicensed competitors, free of infringement, and, up to the filing of the Complaint, free of paying royalties. Not content with these advantages, Shell now sought to eliminate the existing license agreement. What is more, Shell disregarded its obligation under the agreement to inform the licensor of the full extent of its production. The court remanded the case to the District Court with the mandate to determine the consequences of the license agreement.

Finally, the appeal court raised the question of whether the remaining claims in the '698 patent were being infringed. The District Court had not addressed this issue in its decision, any more than it had ruled on a claim put forward by Studiengesellschaft – also part of the appellate court's mandate – for royalty payments owed for the considerable production of polybutene. On this issue, too, Judge Gilmore had failed to render a decision. The latter problem had only become apparent during the course of the proceedings before the court. After cancellation of their agreements,

Shell did not have a license⁸⁰⁾ under the '698 patent to produce and sell polybutene. The '698 patent expired in November of 1995. A judgment awarding payment of back royalties up to 1993 would have been a good and satisfactory outcome. But this was not to be.

Referring back to 1995 when judgment was rendered in the *Studiengesellschaft v. Hercules* case, Shell Oil demanded that they be granted the same terms as those granted to Amoco under the most-favored licensee provision in their 1980 agreement with *Studiengesellschaft*. In response to this request, N. Kramer, on behalf of *Studiengesellschaft* [358], pointed out that the wording of the individual most-favored licensee clauses was different and rejected the request. He simultaneously noted that the Amoco agreement did not include a license for the production of polybutene, so that, even with the Amoco terms, Shell would still not own any production rights. [359]

Shell brought suit [360] and adopted Hercules' line of argumentation. Before the end of that year, both parties – Shell and *Studiengesellschaft* – moved to suspend proceedings in this new action until a decision had been reached in the first litigation (see p. 232) [361]. The appeal decision [357] in that first suit had been rendered in 1997 and further proceedings remanded to the District Court. Several months later, Judge Hughes transferred the action brought by Shell to the jurisdiction of Judge Gilmore, who, at that time, had resumed proceedings in the *Studiengesellschaft/Shell* case [362]. Both parties were dissatisfied with Judge Gilmore's past handling of the case. She had, after all, been charged with implementing the decision of the court of appeals. The parties agreed to file a motion requesting that the case be re-assigned to a US Magistrate⁸¹⁾. A suitable individual was found in Mary Milloy who accepted the mandate.

But Magistrate Milloy, too, did not bother to implement the appeal decisions. Instead she selected, out of all the arguments advanced by both parties, those which, in connection with the as yet undecided question of *Studiengesellschaft's* breach of

80) In the 1979 agreement the production of polybutene was included in a settlement payment. At that time Shell had characterized the capacity of the production as very small and had claimed that the process did not come under the licensed patent of *Studiengesellschaft*. At that time Shell already knew that this statement was not correct. In the meantime production capacity was substantially raised (sales from 1987 to 1995 \$470 million), a fact which Shell did not mention. Later it was established that the production of polybutene was carried out using a catalyst of titanium halide and dialkyl aluminum halide which came under the claims of the '698 patent. In 1980 Shell signed a license agreement with Montecatini for the production of polybutene. The licensed patents (US P 3,197,452 and 3,435,017) covered the production of “isotac-

tic” polybutene by extraction of polybutene which was produced using Ziegler catalysts [358]. The infringement by Shell of the last license agreement with *Studiengesellschaft* due to their withholding information regarding the production figures and royalties for polypropylene resulted in *Studiengesellschaft* canceling the agreement including the right to produce polybutene.

81) US Magistrate Judge is a legal institution under US law. A United States Magistrate Judge is a federal trial judge appointed to serve in a United States district court. District Judges, supervise the activities of the Magistrate Judges by assigning civil cases for jury or non-jury trial upon consent of the parties and for pre-trial matters. He or she is appointed by the life-tenured federal judges of a district court.

contract, addressed the issue of the most-favored licensee clause of the 1974 Studiengesellschaft/Shell agreement. Out of the entire spectrum of arguments presented by both sides, the Magistrate picked this latter motion because the factual situation there appeared easily comprehensible to her as compared to the arguments put forward on the issues of double patenting, invalidity of claims of the '698 patent, and compared to the chemical aspect of the argumentation regarding infringement of the 1987 license agreement by Shell. Magistrate Milloy rendered a judgment on September 30, 1998 [363]. In her ruling, she adopted Shell's line of argument and found that, in May 1980, Studiengesellschaft had failed to discharge their obligation under the agreement to advise Shell of the license agreement it had concluded with Amoco. The conclusions of the court were interesting and should be kept in mind.

The relevant clauses of the license agreements between Studiengesellschaft and Hercules, on one hand, and Studiengesellschaft and Shell, on the other, spelling out the most-favored licensee provisions, had been couched in quite different language, so that simply adopting the court's opinions in the decision against Studiengesellschaft (Civil Action Studiengesellschaft v. Hercules [353], see p. 233) were inappropriate. In the case of Hercules, Studiengesellschaft, under its agreement, was obligated to divulge to Hercules each and every agreement entered into with third parties, regardless of whether Studiengesellschaft believed that the third-party license terms were more favorable or less favorable. In the case of Shell, on the other hand, Studiengesellschaft was required to inform Shell only if the terms granted to a third party were more favorable than Shell's terms.

Shell had pointed this out in May 1980. Amoco had obtained a license to produce unlimited amounts of polypropylene for a lump sum of 1.2 million dollars, while in contrast, Shell's price for an annual production limited to 450 million pounds had been 1.8 million dollars, with royalties of 1% to be paid for any excess production. In accordance with the 1974 and 1979 Shell agreements, which were effective through 1980, these facts were irrefutable. The Magistrate did not then accept Studiengesellschaft's contention that both agreements, Amoco and Shell, must be compared in their entirety. Studiengesellschaft pointed out that the Amoco license was limited to polypropylene, whereas Shell, in accordance with the 1979 arrangement, had been granted an additional license for the production of polybutene without any further royalty obligation (cf. footnote 80). Shell thus disregarded the value of the polybutene license.

The Magistrate refused to take the royalty-free production of polybutene into consideration when comparing the agreements. In her opinion, neither the agreement of 1974 nor the 1979 agreement contained any indication that a connection could be properly made between Shell's polybutene production and the royalties paid by third parties (Amoco) for polypropylene. The polybutene production was irrelevant with respect to the terms of third-party polypropylene licenses. The most-favored licensee provision of the 1974 agreement referred solely to the polypropylene license. Only this portion should, therefore, be considered in a comparison. The terms of the Amoco agreement were thus more favorable than the provisions of the agreements with Shell. Accordingly, the obligation to advise Shell of the Amoco agreement was enforceable. The right to produce polybutene solely fitted in with the proper application of the royalties paid for the polypropylene production.

If any agreements with third parties were to provide for comparatively lower royalty rates, then such terms must be disclosed under the most-favored licensee clause. Failure to do so carried the risk of violation of the most-favored licensee provisions.

The Magistrate further rejected the claim that if Shell adopted the Amoco terms, they should then make a further payment of 1.2 million dollars in addition to the 1.8 million dollars already paid in 1979. In Studiengesellschaft's opinion, it was exactly this requirement which would preclude the terms of the Amoco agreement from being more favorable. However, neither of these agreements – 1974 or 1979 – according to the Magistrate, contained an indication which would confirm this argument. Studiengesellschaft had failed to cite a case in support of the suggested interpretation.

As in the case of *Studiengesellschaft v. Hercules*, the Magistrate refused to differentiate between running royalties, on the one hand, and lump-sum settlement payments for limited or unlimited production, on the other hand. The court held that both cases involved a monetary compensation for the use of a patent.

Studiengesellschaft was unable to provide proof that Shell had been aware of the Amoco agreement long before 1992. Had Studiengesellschaft been successful in this, laches on the part of Shell would have become a significant issue.

The court's finding in favor of Shell on the question of violation of the most-favored licensee provision as defined in the agreement did not mean that the entire civil action had thus been terminated. The remaining issues of the case, according to the Magistrate, were, however, purely academic. A resolution of the lawsuit as a whole would have to be viewed in light of this violation.

The Magistrate believed that the case was eligible for resolution in an out-of-court arbitration proceeding. She ordered that, within a set period, the parties name a competent arbitrator. At the same time, she ordered that both parties submit in writing, their claims for damages – Shell in connection with the Amoco agreement and Studiengesellschaft with respect to royalties due for the period 1987 through 1993.

Two months later, the feuding parties reached an agreement [364] in the presence of an arbitrator, Mrs. S. Soussan, a former judge, to the effect that the claims for damages and reciprocal demands canceled each other out and that, therefore, neither party was required to make a payment to the other. This arrangement included that Studiengesellschaft would not raise any claims resulting from production at the Seadrift plant. The same was to apply also to Huntsman Chemical Corporation, which had acquired part of the polypropylene plants. Furthermore this arrangement required Studiengesellschaft to waive any claims which had arisen in connection with the polybutene production.

The decision by Magistrate Milloy drew severe criticism, especially from Studiengesellschaft's attorneys [365], Arnold Sprung and Nat Kramer. They felt that the decision was in error on numerous grounds, with the crucial error being that Shell had been awarded most-favored licensee rights for the period after 1980. An attempt to file an appeal was pre-empted because the Magistrate, as described above, ordered the case to go into arbitration.

At this point in time, in late 1998, the '698 patent had already expired. Nevertheless, the bottom line was that after the top-ranking '115 patent, this patent was so positive that it outdistanced all other patent rights in terms of licensing success for the period 1980 through 1995 [366]. In 1980, after the expiration of the '115 patent, the '698 patent seemed to have so little value, that no polypropylene producer in the United States respected it. It took some courage to bring an action against Northern Petrochemical in order to enhance the value of the '698 patent and to make an attempt to recapture control of the market.

Although Studiengesellschaft was successful in proving that producers were indeed using Ziegler catalysts according to the '698 patent, and in also disposing of the double-patenting allegation, their opponents found their "Achilles heel" by ferreting out contractual weaknesses in the license agreements i.e. the most-favored licensee provisions. Nevertheless, it should be borne in mind that the adjudication of that issue was less than persuasive, and that Hercules/Himont and Shell Oil were both using Ziegler catalysts.

5.11

Japanese Export of Automobiles to the United States

In the mid-1980s and shortly after the validity of the '698 patent had been upheld by the court of appeals in Washington, the US government amended its Trade Law [367] (Section 337, 19 USC 1337, Omnibus Trade Reform Bill). In accordance with the new regulations, any sale of products imported into the United States would infringe an existing valid US patent if the products had been manufactured in a foreign country according to the process as described in that US patent. Every automobile imported into the United States, which had been manufactured in Japan, contained between 10 and 30 kg of polypropylene in the form of finished articles, such as bumpers, gas tanks, dashboards, interiors, carpeting, etc.

Studiengesellschaft instructed Sprung to write to the Japanese automobile manufacturers, advise them of the situation, and make them a license offer. In their response, the automobile manufacturers pointed out that they were purchasing the integrated parts from outside sources and did not produce any polypropylene themselves. They named the polypropylene producers who were their suppliers.

The majority of the polypropylene producers fought back, pointing to their respective license agreements, pursuant to which they had been given an assurance that they were entitled to export, royalty-free, even into those countries where Ziegler owned patent rights. This export right was, however, limited to the life of the corresponding Japanese patents. Those had long since expired. Other agreements did not contain this clause on export rights. A third group of licensees was allowed to export regardless of the life of the Japanese patents [368]. Initially, responses by the polypropylene producers and suppliers varied widely, ranging from "we need more time," over "refusal – referring to past agreements," and "please make us a license offer," to "absolute refusal," because the '698 patent was not being used [369]. Given these reactions, it appeared difficult to request that all producers sign identically-

worded license agreements. Then there was the additional task of ascertaining what polypropylene producer had supplied what quantities to what auto manufacturer.

Now, the number of exported automobiles and their manufacturers were known entities, and these manufacturers, in turn, knew how much polypropylene was being used per automobile [370]. Accordingly, the simplest solution in terms of royalties would be to ask for a fixed amount per automobile. But the question still remained of how to allocate the processed polypropylene to the individual polypropylene producers [371].

After visits by Japanese partners to Mülheim and Martin and Sprung's trip to Tokyo, there was an initial exchange of drafts spelling out contractual arrangements between Studiengesellschaft and a number of sincere prospective licensees in the field. Sumitomo Chemical Co., Mitsubishi Petrochemicals and Mitsui Toatsu Chemicals had an expert report prepared which determined the amount of polypropylene utilized for each automobile model exported by Japanese automobile manufacturers [372]. According to this report, in 1989 an average of 12.8 kg of processed polypropylene was used in the manufacture of passenger cars and an average of 10.3 kg in the manufacture trucks. By ascertaining the number of automobiles exported to the US, it was easy enough to calculate the tonnage of polypropylene which had been exported.

The parties then took another 2 to 4 years to resolve the question of how much was to be paid in royalties and the allocation of these royalties among the individual polypropylene producers, in addition to obtaining consent from all the producers to accept conforming agreements. Terms were also discussed for adjustments in the event of price cuts and price fluctuations in connection with the quantities of polypropylene exported.

In settling the above issues, the parties agreed that all polypropylene producers were to pay Studiengesellschaft a fixed royalty per year. The royalty was based on the number of exported vehicles and the amount of polypropylene ascertainable therein, whereby the share allocated to each individual polypropylene producer was calculated in terms of a percentage of the total amount exported. As an accommodation, the majority of the polypropylene producers were willing to work out their individual shares among themselves. Payments started in 1988 and ended in 1995, the year the '698 patent expired.

There were a few companies which claimed that they were not using the '698 patent, as for example, Mitsui Petrochemical. These companies were willing to await resolution of the dispute in the United States before endeavoring to reach a settlement with Studiengesellschaft depending on the outcome of the controversy surrounding the so-called "high-speed" catalysts.

Not least responsible for this positive conclusion was the fact that the negotiations had been overshadowed by Studiengesellschaft's warning that a ban on the import of automobiles was a distinct possibility.

Between 1991 and 1994, Studiengesellschaft signed export license agreements with a prevailing number of the polypropylene producers, each producer being granted a non-exclusive license for the use of Studiengesellschaft's US '698 patent in return for royalty payments starting June 1, 1986. The royalty figure was a fixed-dol-

lar amount, due annually, which was calculated on the basis of the average tonnage of polypropylene that had been exported to the United States in automobiles. The Japanese contract partners strongly emphasized their desire for all producers, if possible, to be included in this arrangement. Execution and implementation of the agreements proceeded smoothly throughout the entire effective period of the '698 patent until its expiration.

5.12

“The Last Chapter”

The Y.C. Wang family had established a major industrial enterprise in Taiwan: “Formosa Plastics Group” [373], Taiwan’s largest producer and vendor of petrochemical products. In the late 1970s, the company expanded its business into the United States (Formosa Plastics Corp.) and set up a wholly-owned subsidiary in Texas, “Formosa Plastics Corp. Texas” with their place of business in Comfort Point, Texas. About 10 years later, the Taiwan-based company entered into negotiations with one of Ziegler’s German licensees to obtain a contract allowing them to produce polypropylene in accordance with a modified process which the licensee had developed. Transfer of the expertise was to occur in exchange for an appropriate payment.

The aforementioned modified process had already previously been licensed in the United States to Northern Petrochemical Company (NPC). Studiengesellschaft had prevailed in an infringement action which it had brought earlier against this company (p. 215). NPC had conceded that it was infringing the Studiengesellschaft-owned '698 patent.

In 1993, Studiengesellschaft learned from a notice in a newspaper that Formosa Plastics, USA, was about to enter the US market as a new polypropylene producer. When it approached Formosa by letter, Formosa responded by stating that their interests were being represented by the German company which had provided the know-how.

Word from Germany was that a recommendation had been made to Formosa that they offer Studiengesellschaft a lump sum settlement [374] for utilization of the '698 patent until its expiration date.

It was well-known at the time that Studiengesellschaft had successfully enforced the 4,125,698 ('698) patent in the United States. Formosa Plastics Corp., USA, however, refused to enter into direct negotiations [375] with Studiengesellschaft. In addition to Studiengesellschaft (catalyst patent), Phillips Petroleum was also known to hold polypropylene patent rights (product patent) at that time.

Simultaneously with efforts to come to an arrangement with Studiengesellschaft, negotiations were underway between Formosa and Phillips Petroleum – without success. Phillips then filed suit against Formosa in 1993 which was terminated in March of 1994 by payment of a considerable royalty [376]. The license involved the production and sale of “crystalline” polypropylene.

In discussions between the German Ziegler licensee and Studiengesellschaft regarding the catalyst employed by Formosa, Studiengesellschaft noted that For-

mosa's process involved the use of a classic Ziegler catalyst – titanium trichloride and diethylaluminum chloride – to which a reduced amount of a third and/or fourth component was added to both the titanium chloride and the aluminum component in order to enhance the productivity [377]. In the absence of either the titanium component or the aluminum component, predictably, there was no polymerization, while polymerization did occur even when the third and fourth additional components had been omitted [378].

Earlier court decisions, for example, *Studiengesellschaft v. Phillips Petroleum* or *Studiengesellschaft v. Dart*, indicated that the language of the claim ('698) should not be interpreted to the effect that "essentially" meant the same as "exclusive", so that using a complex of aluminum chloride with titanium chloride for example, would not avoid infringement. All forms of titanium chloride were covered [379].

Likewise, the feature of the catalyst components being admixed prior to contact with the propylene to be polymerized was interpreted in these decisions within the meaning of the *Studiengesellschaft* patents. The presence of propylene as a carrier and/or suspension agent of the individual catalyst components for instance, was well within the scope of those patent rights.

Arguing back and forth concerning the catalyst did not lead to an agreement [380]. The parties steadfastly maintained their positions. In searching for a solution, pertinent production figures needed to be ascertained. For 1994, the quantities involved were approximately 78,000 tons and for 1995 an estimated 150,000 tons of polypropylene. A meeting in January 1995 between "high echelon executives" of the parties did not lead to a settlement. Even though figures were put out on the bargaining table, any final arrangement was subject to approval by Formosa's management. Management signaled "no", but then, in February of 1995, changed their position and made an initial offer of \$900,000 [381]. Yet, from the sales figures, it was easy enough to calculate that *Studiengesellschaft* was entitled to claim 2.7–2.8 million dollars in back royalties [382].

In mid-March 1995, *Studiengesellschaft* filed suit against Formosa Plastics Co., USA and Formosa Plastics Corp. Texas [383]. The depositions taken in May of 1996 revealed that Formosa Plastics, USA had reached 175 million dollars in sales up to the end of November 1995, when the '698 patent had expired. Using these sales figures to calculate the royalties at the customary licensing terms, it transpired that *Studiengesellschaft* was entitled to 2.64 million dollars. Formosa's legal representative, Mr. Norris, (who was also Shell's attorney in the *Studiengesellschaft v. Shell* action) took the position that the '698 patent had been declared invalid, which was not the case [384]. Some of the claims – those which would exactly come under consideration in connection with Formosa – were indeed valid and had never been dealt with by the courts in the Shell case [385]. As an added bonus, the depositions of Formosa's witnesses revealed that Formosa was also producing polyethylene⁸²⁾.

82) The catalyst consisted of titanium tetrachloride on an inert carrier and aluminum triethyl in addition to aluminum triethoxide as the aluminum components (see reference [381]). The reaction of aluminum triethyl with

aluminum triethoxide formed ethyl aluminum ethoxide compounds (see Houben Weyl 1970, *Methoden der Organischen Chemie*, Vol. XIII/4, p. 80, Georg Thieme Verlag, Stuttgart.

The discovery period ran until mid-1998. In late July, Judge Farnan issued an order regarding further proceedings in the case, wherein, on the one hand, he scheduled the trial date for May 1999, while stating, on the other hand, that he wanted to await the decision in the *Studiengesellschaft v. Shell* case [386].

In February of 1999, the parties agreed [387] on a lump-sum payment by Formosa in amount of 1.65 million dollars.

By the time the agreement was reached, the '698 patent had expired a little over 3 years previously. Because the patent had been challenged by numerous opponents, it could hardly be denied that its effectiveness had been weakened. Further court actions against unlicensed polypropylene producers appeared ill advised. In 1994, 1 year '698 patent was due to expire, the total production capacity for polypropylene in United States was calculated to be approximately 5 million tons. Of this capacity 15% was not governed by contractual arrangements with *Studiengesellschaft*, nor had royalty obligations been established by a court of law.

5.13

Epilog

The foregoing historic reflection on the discovery and worldwide development of polypropylene was born out of the author's desire for clarity and structure. Prejudices and lack of information had led to various distorted viewpoints in judging events, which, in turn, gave rise to uncertainty in dealing with the facts as they were. The time for review was, after all, propitious at this point – not least since the important patents had expired – so that a well-rounded picture of the events in context could be presented.

The exploration is designed not only to help clarify the events, but also to provide any interested reader with an opportunity, if he so desires, to apply for himself any lessons to be learned from the history of polypropylene. Beyond that, there surely was, and is now, an interest in the involved Ziegler chemistry as well as the patent aspects connected with it.

Judgments, particularly by US courts, were, on the one hand, instrumental in bringing about a retroactive clarification of the legal rights attending the use of the catalysts – almost 30 years after the discovery of the Ziegler catalysts – and, on the other hand, provided an impression of the political influence being exerted. The judges involved were not always able to deal with the chemistry aspects of the case. In the course of time, they ignored partial judgments previously rendered on the same subject matter. Since there were no provisions in the rules of procedure for impartial experts, it was left to the attorneys for the individual parties to educate the judge on the scientific facts of the case during presentation of their arguments. As the patents approached their expiration dates, it became noticeable that the courts were exceedingly careful, at best, to maintain the then existing scope of patent protection, but more often to narrow it. In view of the plethora of arguments dealt with by the courts, this trend was to be expected in view of the length of time that had passed.

The enormous commercial interest in the Ziegler catalysts prompted all-out efforts in pursuing any even half-way promising approach to deny Ziegler, his co-workers and his institute their proper share of the fruits of their discovery, or at least to trim down that share with legal as well as scientific arguments, or by simply infringing existing patent rights.

If nothing else, Studiengesellschaft was able to secure, for more than one generation, an acknowledgment of the original invention and the dependency of any improvements. Karl Ziegler never dreamed that for over half a century he would cause so much turmoil.

The stated outcome notwithstanding, some questions still arise. There are some scientists who categorically refuse to have their inventions patented and thus decline any utilization of these inventions for the benefit of their own research organizations. Should an organization such as the Max-Planck-Institute undergo the hardships of engaging in long, drawn-out disputes for 30 to 40 years? The answer, most certainly, depends on the benefit – cost ratio, but not exclusively. Attorneys, and in particular patent attorneys, were presented with new viewpoints during both patent prosecution proceedings and in Opinions issued by lower and appeal patent courts. But chemical research, too, has received much fruitful input from the disputes in the courts and before the Patent Office and not least from the patents themselves.

For more than 40 years, the Max-Planck-Institute for Coal Research in Mülheim had been self-supporting through income generated from the utilization of the 1953/54 patent rights. Most recently, worldwide revenues from sales of polypropylene were in excess of 20 billion Euros annually.

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