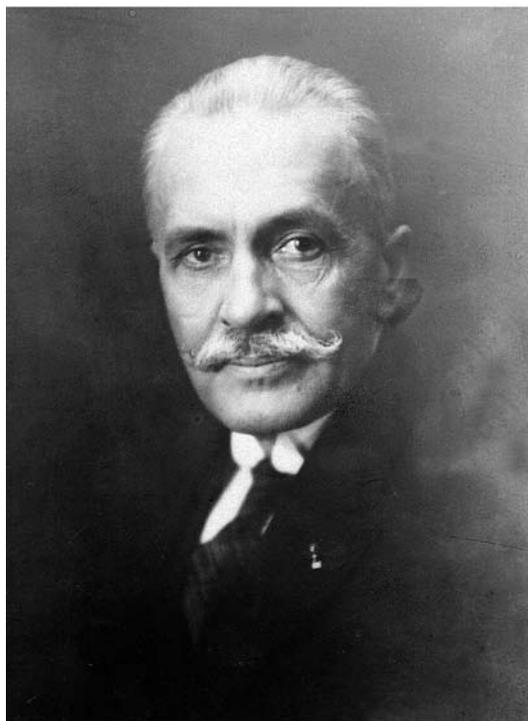


# Technical and technological history of Zakłady Azotowe w Tarnowie-Mościcach S.A.

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The development of Polish chemical industry at the dawn of the Second Polish Republic [II Rzeczpospolita] is connected with the person of Ignacy Mościcki, Professor at the Lviv Polytechnic National University, who became a director of the nitrogen compounds plant which was taken over from the Germans after the Silesian Uprisings, in Królewska Huta (currently Chorzów). The Chemical Research Institute of Warsaw founded by I. Mościcki was the main promoter of chemical industry development in Poland, including nitrogen compounds. The plans for chemical industry development, including an urgent construction of the second large plant of nitrogen compounds, were created in that institute [1]. The final decision on the construction of a new plant near Tarnów was made on 12 March 1927 [2]. On 14 May 1927, land properties with the total area of 670 hectares for 270 thousand US dollars were bought from the Sanguszko family. First works at the construction site started on 5 May 1927 [3]. The State Works of Nitrogen Compounds (Państwowa Fabryka Związków Azotowych, PFZA) in Mościce established in 1927 was one of the largest investments of the renascent Polish Republic, setting the trends in Polish chemical industry. Azoty Tarnów has been continuing the traditions of its predecessor until present days.



Prof. Ignacy Mościcki - initiator of Mościce

## State Works of Nitrogen Compounds in Mościce (Państwowa Fabryka Związków Azotowych w Mościcach, PFZA)

In October, 1927, Poland obtained a so called stabilisation loan in the amount of 62 million dollars and 2 million pounds sterling financed by American banks with a contribution of European banks. A part

of loan revenues in the amount of 23.8 million zlotys supplied the funds for the construction of a new plant of nitrogen compounds [4]. Total preliminaries of its construction were initially estimated to be 75.7 million zlotys [5]. The plant was located near Tarnów, about 4 kilometres from the city centre towards the west. The plant premises within the administration boundaries of two communes in Tarnów province: Dąbrówka Infulacka and Świerczków (then renamed into one commune - Mościce) had a triangular shape. The Dunajec River from the west, the White River from the east and Cracow-Lviv railway line from the south were its sides. The management of individual departments being under construction was held in majority by the alumni of Professor Ignacy Mościcki, the head of the Department of Physical Chemistry and Technical Electrochemistry at the Lviv Polytechnic National University and the rector of this university in the years 1925÷1926. Tadeusz Hobler, one of the most skilled engineers from the Lviv Polytechnic National University, became a head of technical group for the plant construction, and then the Construction Office at the plant [6].



Panorama of factory in Mościce the 30s Twentieth century

The majority of equipment for PFZA production installations was purchased from abroad, and the remaining part from the national manufacturers. The Polish companies with the help of the plant workers usually performed assembly tasks. The Polish engineering and technological solutions applied in the plant were mainly represented by nitrogen oxide absorption towers (nine pieces) designed by Ignacy Mościcki (so called Mościcki towers), a patent for producing solutions of ammonium salts invented by Mieczysław Kalous, Eng., and a design of the technical method for producing ammonium sulphate from gypsum supported by the Chemical Research Institute of Warsaw. A group of engineers from the Construction Office at the plant under the supervision of Tadeusz Hobler was involved in developments concerning the practical use of purchased technologies for ammonia and ammonium sulphate synthesis. Stanisław Hempel, PhD, was appointed a Head of the chemical group. The main objective of this group was to develop the technology for obtaining hydrogen as a basic, besides

nitrogen, component for ammonia production. Meeting with a refusal from foreign companies to grant licences, the group of S. Hempel adjusted the solutions of an installation for water gas conversion using the Haber process, whose pilot plant installation was present in the Łódź gas works from 1928. The works covered the preparation of iron-chromium catalyst and the design of the whole installation, necessary to conduct the process. Technological parameters obtained during the works on the pilot plant installations were used as a basis for T. Hobler, Eng., to develop the technical design for the installation for conversion of CO present in water gas. He also developed the solutions for ammonium nitrate technology [7]. Blast furnace coke was a raw material in the installation of ammonia synthesis preceded by gas cleaning plant. Water gas was obtained from this raw material in six periodic generators of Marischka-Brünn-Königsfelder A.G. system. The ammonia synthesis installation, operating under the method of Fauser, an engineer from the Italian company Montecatini, consisted of 9 units, each with the production capacity of ca. 10 tons of ammonia per 24 hours. At those times, it was a modern production plant based on hydrogen obtained from coke gasification and not on electrolytic hydrogen that was used in many other plants constructed at that time [8]. A combined heat and power plant was built for the needs of chemical production, which was equipped with a fine coal-fired boiler house. The steam section of the combined heat and power plant was equipped with six boiler units. Turbine sets including 3 condensing turbines and 1 back-pressure turbine were mounted for the purpose of producing electricity [9]. An assembly of the combined heat and power plant's machines and equipment was completed on 2 October 1929. That day is recognised as the final date of the plant construction [10]. In 1928, apart from the nitrogen installations, began a construction of the Chloride Department with Billiter electrolysis cells, which had no technological connection with the nitrogen part. This department started working in July 1930 [11].



Home Chemist, 60s Twentieth century

An opening ceremony of the State Works of Nitrogen Compounds (Państwowa Fabryka Związków Azotowych w Mościcach, PFZA) with the participation of the Second Polish Republic President – Ignacy Mościcki, representatives of the Polish government with the Prime Minister – Kazimierz Bartel, and the Minister of Industry and Trade – Eugeniusz Kwiatkowski, took place on 18 January 1930 in Mościce. In the initial period of its operation, PFZA was producing ca. 60 tons of ammonia per 24 hours. Nitrogen required for the synthesis was obtained from air liquefaction using Linde's method, and hydrogen was obtained from water gas. A part of produced ammonia was processed into 50% nitric acid (ca. 170 tons of nitric acid per 24 hours). Ammonium

nitrate, obtained by neutralising nitric acid with gaseous ammonia, was then processed into fertiliser under the name of nitrophos (ca. 240 tons per 24 hours), containing ca. 15.5% of nitrogen [12]. In 1930, the global production of the State Works of Nitrogen Compounds was ca. 57 thousand tons by weight, of which 80% was constituted by nitrogen fertilisers [13]. In 1930, the production included, apart from the above mentioned products, ammonium nitrate for the arms industry and such chloride products as chlorobenzene, chloronaphthalene, chlorinated lime and melted sodium hydroxide.



The first power plant of PFZA in Mościce

The first year of PFZA activity ran parallel to the first year of a serious worldwide economic crisis. At the time of the critical situation for the plant, Eugeniusz Kwiatkowski, the previous Minister of Industry and Trade, became its Managing Director. The Minister arrived in Mościce in the middle of February 1931. He came to grips with a serious task – to maintain the existence of the plant which, only just launched owing to a great amount of work and money, was under threat of closure. The plant in Mościce had to expand its offer of fertilisers and modernise the existing one in order to remain competitive on the market. In 1930, a decision was made to modernise the installation for producing nitric acid and ammonium sulphate [14]. In that period, prilled Calcium saltpetre and coarsely crystalline ammonium sulphate were the most in-demand fertilisers on foreign markets. A licence for producing coarsely crystalline ammonium sulphate with the capacity of 60 tons per 24 hours was bought in England as the powdered sulphate manufactured so far using the Fauser method stopped being attractive for buyers [15]. The decision on producing prilled Norwegian saltpeter being in global demand, which was then manufactured only by two factories in a world, was the most important investment step for the plant in the interwar period. The technology was bought in Norway, and other structural works, construction and assembly of equipment was performed using the plant's own resources. In the late 1930s, the installation with a designed capacity of 80 tons of Norwegian saltpeter per 24 hours was producing much more, that is, ca. 180 tons per 24 hours [16]. In 1934, the first installation in the world for producing concentrated 98.5% nitric acid using the Wendlandt's method (Ho-Ko, "hochkonzentriert") with the productivity of 20 tons per 24 hours was built in the plant in Mościce [17]. The plant offer was also expanded to other products. That year, an installation of calcium chlorate, so called herbatox – a herbicide, was launched. Two years later, an installation for compressing oxygen into cylinders, being in wide demand for welding works, was put to use. The Machinery Maintenance Section was created on the initiative of the

Managing Director – Kwiatkowski, in the late period of his presence in Mościce. Its purpose was to maintain machines and equipment in a good condition [18]. In 1932, efforts taken for PFZA admission to the Fertiliser International Syndicate resulted in success. That was related to, inter alia, the quota given on fertiliser export in the amount of 32 thousand tons a year.

On 1 August 1933, PFZA Chorzów and PFZA Mościce were merged into one commercialised enterprise owned by the State Treasury under the name the United Works of Nitrogen Compounds in Chorzów and Mościce (ZFZA - Zjednoczone Fabryki Związków Azotowych w Chorzowie i Mościcach). "Azot" plant in Jaworzno also became a part of ZFZA. The enterprise was operating under that name until the outbreak of World War II. The decision on extending the plant and building the Research Laboratory was taken in 1934. It had its own research laboratories, pilot plant halls, academic library and its own construction office [19]. The Laboratory also included an analytic laboratory and a catalyst plant. Opening the Laboratory had a great impact on the development of technical progress in the production plant. A development plant for "Mościce" was prepared in 1936. The plan assumed investment expenditures in the amount of 14.8 million zlotys on modernising the installation for producing ammonia, nitric acid, chlorine, Norwegian saltpetre and starting the production of methanol and formalin [20]. In August 1933, the works on delivering natural gas from Lviv and Krosno basins to Mościce through piping began. The investment was a part of gasification programme of the Central Industrial District (COP – Centralny Okręg Przemysłowy) and "Polmin" was a contractor. Poland spent ca. 14 million zlotys on COP gasification in the years 1937 ÷ 1938 [21]. The works on delivering gas to Mościce ended in 1937. At first, the plant used gas to fire the combined heat and power plant boilers, which resulted in reducing the costs of transport and coal storage. Then, tests were made to decompose methane in generators. After achieving positive results, hydrogen content in crude gas increased, which influenced the increase in ammonia production in Mościce by ca. 10% on average [22]. It initiated the process of using natural gas as a basic raw material and simultaneously reduced production costs. At the end of the 1930s, ammonia production in the plant in Mościce was based on gas obtained from coke gasification generators and two additional generators adjusted to coke and methane gasification. Simultaneously, the construction of a new installation (commonly known as methane decomposition I) of natural gas-based production of hydrogen with the use of nickel catalyst began. It was a non-pressure installation of the American company Hercules Powder Co. system. That investment performed by the German company Bamag IG was completed shortly before the war outbreak. However, it was launched only during the occupation [23]. In that time, also a pilot installation for producing a new nitrogen and phosphorus fertiliser – (biphosphate) precipitate was launched. Its production was based on phosphorites imported from the USSR [24]. In 1938, the total production of that fertiliser amounted to about 750 tons [25]. The tests conducted in the laboratory at the plant resulted in activating pilot plant installations of methanol and formalin synthesis. In the years 1933 ÷ 1938, the plant in Mościce produced the following nitrogen fertilisers: ammonium sulphate, nitro-chalk, Norwegian saltpetre and initially nitrophos as well as (biphosphate) precipitate at the end of that period. The following nitrogen industrial products were manufactured: ammonium nitrate (also used as fertiliser), concentrated and industrial nitric acid, mixed acid and liquefied ammonia.

In September 1939, World War II started. When the September Campaign was over, the plant was restarted in October 1939. During the occupation, the plant in Mościce produced the following fertilisers: Norwegian saltpetre (calcium saltpetre), ammonium sulphate, nitro-chalk and a small amount of phosphatic fertilisers: biphosphate and ground phosphate rock and additionally, inter alia, industrial and concentrated nitric acid, mixed acid, ammonium nitrate, compressed

oxygen, caustic soda, hydrochloric acid, liquefied chlorine, and herbatox (calcium chlorate) [26]. The production of many products was reduced or even ceased during the occupation. The occupiers tended to keep mainly the production which could be used under the war conditions. At that time, the production of 20.5% prilled nitro-chalk based on nitrophos installation started. From the beginning of 1944, the Germans, facing their military disaster, commanded the disassembly of the plant equipment and its transport far into the Third Reich. The production was practically stopped.

### Restoration and development towards organic production

After the occupation, in June 1945, the government passed a resolution on restoration and expansion of PFZA as part of the national programme of producing fertilisers for agriculture. The fact that the majority of plant buildings remained undestroyed supported the decision on its restart. Incomplete gas generators, oxygen generation plant, oxygen compressor station, and gasometers were the most crucial installations that remained. The machines and equipment removed from Mościce were consecutively found, inter alia, in Brzeg Dolny in Silesia and in the quarries in Graby on the route Ścinawa – Rawicz, due to the engagement of the plant workers. In June 1945, the combined heat and power plant, the installation for producing compressed oxygen and the machine shop were operating in the State Works of Nitrogen Compounds in Mościce (PFZA Mościce) due to the initiative of the workers and credits obtained from the Province Department of Industry in Cracow [27]. A small-scale methanol production started in the same year [28]. In November 1946, a pilot plant installation for formalin was put to use and a resolution was passed on the restoration of chlorination plant [29]. The years 1947 ÷ 1949 were the period of a 3-Year Economic Plan. Thanks to the recovery of plundered apparatus, the installation for producing iron-chromium catalyst used for carbon oxide conversion was already put to use in May 1947, the installation of synthetic ammonia with the production capacity of 30 tons per 24 hours started to operate in July, and then the installations for producing industrial nitric acid with the production capacity of 140 tons per 24 hours and the installation for producing nitro-chalk (20.5% N) with the production capacity of 214 tons per 24 hours were activated [30]. The first tons of nitro-chalk were produced on 27 August 1947 [31]. The installation of Norwegian saltpetre with the production capacity of 220 tons per 24 hours was restored in 1948 and put to use a year later [32]. In summer 1948, after 8 months of restoration, the chlorination plant was put to use [33]. Chlorine obtained from salt electrolysis was a raw material used in the production of chlorinated lime, hydrochloric acid, chlorobenzenes, and chloronaphthalene waxes. The production capacity of restored Billiter chlorination plant was 2800 tons of chlorine, and that of chlorinated lime – 2100 tons per year [34].

In 1949, the Nitrogen Plant in Tarnów (Zakłady Azotowe w Tarnowie) already produced 20 products [35]. The contribution of nitrogen fertilisers to the total production of the plant was initially kept at a similar level as before the war (about 80% wt.). The production of other chemicals constituted the remaining 20%. It was still a plant that practically produced inorganic products. The demand of national economy was much bigger and still increasing. It was necessary to expand and modernise Azoty and introduce the changes into the technological concept. "Avoiding" coke and replacing it with natural gas in the first phase was among the main trends towards the production modernisation. The plant was predisposed to such an action due to its location on the areas through which ran the southern main east-west gas line. Azoty, located nearest to natural gas deposits among all its greatest consumers, had favourable conditions for using that raw material. Similarly, the table salt resources near Bochnia were considered to enable the development of chlorine-based production. The Institute of Chemical Synthesis, established from the Central Experimental

Laboratory of PFZA, had a great contribution to the technological development of the plant. The programme of intensifying ammonia and fertiliser production, also known as “Tarnów I”, was performed in stages. The initial stage consisted in expanding the installation of ammonia synthesis to the production capacity of 160 tons per 24 hours (in 1953) and then, two years later, to 240 tons per 24 hours [36]. The expansion of nitrogen plant was targeted for the capacity of 480 tons per 24 hours (which was performed in the period of “Tarnów II”) by developing two new gas generators for ammonia synthesis from coke and two generators producing gas for ammonia synthesis based on natural gas, by constructing two new installations of natural gas conversion, and by modernising gas purification plant for ammonia synthesis from carbon oxide by introducing so called copper washing method. Also new gas compressors were arranged along with the interior redevelopment of ammonia synthesis reactors from Fauser type to NEC type. The expansion of nitric acid production capacities included putting to use 5 new units of industrial nitric acid using the pressure method, co-developed by Tadeusz Hobler, Eng. (so called Hydro-Nitro-Hobler), the construction of a second block unit for nitric acid using a non-pressure method, and then the intensification of the installation of concentrated nitric acid to the capacity of 110 tons per 24 hours, and the one of mixed acids to the capacity of 180 tons per 24 hours. In 1953, the production of crystalline ammonium nitrate used for explosive materials with the production capacity of 120 tons per 24 hours was restarted [37].

Efforts devoted to the expansion of the fertiliser line in the 1950s did not mean that the plant modernisation was one-sided. In that period, also the preparations for further development started. They were aimed at expanding the product range and introducing modern highly purified products. In the years 1952÷1953, the installation for methanol synthesis was built and activated on the basis of experience from the pilot plant installation operating in the Research Laboratory from 1934. Produced methanol was a raw material for the production of formalin and hexamethylenetetramine from the new installations built and activated at that time. In 1953, the first organic product – formalin – was produced on the technical scale with the production capacity of 4500 tons per year. In 1954, a pilot plant installation for producing thiocarbamide with the capacity of 30 tons per year was started in the buildings of the formalin production plant [38]. Both the intensified production of ammonia and the installation of formalin were the investments which were designed and performed using the plant's own resources [39]. Starting the construction of caprolactam installation – a semi-finished product for manufacturing fibres and polyamide materials – was the next milestone action tending towards changing the production profile of the company [40]. 1957, the jubilee 30<sup>th</sup> year in the history of “Mościce”, was called “the year of great activations”. Apart from the activation of caprolactam plant, a new mercury cell installation for salts having the production capacity of 18000 tons per year was put to use. Moreover, the production of hydrogen chloride, methanol for pharmaceutical use and aluminium-nickel catalyst for cyclohexanol started [41]. The intensification of production and the construction of new installations resulted in increased demand of the plants for steam and electricity. This demand could not be met by intensifying the operating combined heat and power plant (EC I). Thus, it was decided to build a new combined heat and power plant with the capacity of 50MW and possibility of its further expansion. The new combined heat and power plant (so called EC II) equipped with machines imported from the USSR was being built in the years 1954÷1958. The first stage of EC II construction consisted in installing three steam boilers and two pass-out and condensing turbines [42]. Stanisław Opalko became the Managing Director of the company in 1958 and he was holding that position in the 1960s and the 1970s – these years were considered as a period of the company's greatest development in the second half of the 20th century.

## “Tarnów II” - chemistry of acetylene. Development of caprolactam

In May 1961, the Economic Committee of the Cabinet approved “the fundamental principles for the assumptions about expanding the Nitrogen Plant in Tarnów” (Zakłady Azotowe w Tarnowie), that is, the company transformation into a big chemical industrial complex. For the contemporary amount of 4.8 billion zlotys, it was planned to expand the plants and to intensify the so far offered range of products from the operating installations (including the previously mentioned increase in ammonia production capacity from 260 to 480 tons per 24 hours) in the years 1962÷1967. A few key factors were decisive while making a decision about the location of the biggest investment of chemical synthesis industry in Tarnów. Technical personnel of the plant had a long-term experience in performing investment works not only in Tarnów, but also in other chemical plants in Poland. The company had some undeveloped land situated at the north-east side. Due to this location, it was possible to conduct the land development and provide utilities relatively quickly. Other important factors were as follows: convenient transport connections, developed technical infrastructure, own production plant of chemical apparatus as well as relatively small distance from newly discovered deposits of natural gas in the region of Lubaczów in Podkarpackie province [43]. The programme of plant expansion was named “Tarnów II”. It included the construction of three installations for producing acetylene in the process of methane partial oxidation along with an oxygen generation plant and a new installation of ammonia synthesis (3 units, each with the capacity of ca. 200 tons per 24 hours), a new chlor-alkali production plant with the capacity of ca. 30 thousand tons per year, the construction of vinyl chloride and polyvinyl chloride plant using acetylene and having the capacity of 80 thousand tons per year, hydrogen cyanide and acrylonitrile production plant (a semi-finished product for manufacturing wool-like fibre) with the capacity of 16 thousand tons per year [44] as well as the construction of semi-conducting silicon production plant. The expansion of caprolactam production capacity with the installations for producing such semi-finished products as sulphuric acid and oleum, ammonium nitrite, hydroxylamine sulphate and the installations for processing ammonium sulphate – a side product formed during caprolactam production – were planned as part of that programme [45].



Caprolactam plant, half of the 60s Twentieth century

High-methane natural gas (in the amount of about 450 million m<sup>3</sup> annually [46]) was a raw material base for “Tarnów II” installation. It was used to produce acetylene using the method of pressure partial oxidation, modern at that time. Acetylene was then processed into suspension polyvinyl chloride and acrylonitrile. Gas was delivered to the plant using pipelines from Dashava deposits in Lviv and Krosno districts [47]. The construction of “Tarnów II” started in 1962 and it was the beginning of an over 30-year period of so called “acetylene chemistry”

in Azoty Tarnów. It was characteristic that only two from many newly designed technological processes were based on licences purchased from abroad. This referred to the production plant of acetylene (the contract with Montecatini Company in 1959) and of acrylonitrile along with hydrogen cyanide (the contract with Ugine company in 1961). Many Polish planning and design companies coordinated by the Company's Plant Design Office were involved in preparing the documentation for "Tarnów II" programme [48]. In 1963, Zbigniew Szczypiński became the Technical Director of the plant. He was one of the main creators of the technological profile of the industrial complex for the subsequent decades [49].

In July 1966, the first unit for methane partial oxidation, the most crucial investment of the national chemical industry at that time, began to operate [50]. An urgent need for building another source of acetylene was caused by technological difficulties with activating that installation and consequently the delays in acetylene deliveries to already completed polyvinyl chloride and acrylonitrile production plants. In the third quarter of 1966, it was decided to buy two ready carbide-based installations for producing acetylene from Messer Company from FRG. In 1967, a decision was taken to resign from the assistance of Montecatini in starting up the installation of partial oxidation. Also, it was decided not to build the third unit of partial oxidation and hold for some time the construction of the second unit that was in progress. At the same time, negotiations on buying know-how for that process started. The start-up of the first installation unit of methane partial oxidation was taken over by the plant employees. Gas for producing the composition for ammonia synthesis was to be obtained simultaneously with acetylene from the installation of partial oxidation. A new installation of ammonia synthesis with the capacity of 540 tons per 24 hours (three production lines, each producing 180 tons per 24 hours) for processing that composition was built in the years 1964÷1967, and then put to use [51]. In April 1967, acetylene necessary for initiating at first the operation of the installation for producing polyvinyl chloride, and then the installation of its polymerisation was supplied due to regular improvement of the operational continuity of the installation of partial oxidation [52]. An action against the Italian licensor was brought in the International Trade Tribunal in Switzerland for incurring losses of millions due to the delays. The legal proceedings ended in the judgement in favour of Azoty. The Italian party had to pay compensation [53]. The lack of acetylene was also the reason for delays in activating the first production lines of hydrogen cyanide and acrylonitrile. The start-up of those installations began in August 1967 with the participation of French experts [54]. On 5 September 1967, a 50-ton cistern was filled with the first Polish acrylonitrile produced from the own acetylene of the company [55]. In 1973, acetone cyanohydrin in the amount of 6500 tons per year as well as sodium cyanide and sodium thiocyanate in aqueous solutions started to be produced using the excess of hydrogen cyanide remaining due to the reduced national demand for acrylonitrile. The investment named "Tarnów II" also covered the construction of the first Polish production plant of semi-conducting silicon, whose application as the semi-conductor was getting wider in the electronic industry. The plant started to be built in 1962 and it was completed in 1965 [56].

A significant expansion of the plant infrastructure and a number of ancillary facilities was required to realize the investment "Tarnów II". It included the construction of, inter alia, a water purification plant specially for the needs of new installations, a water intake on the Dunajec River, circulating water pumping stations, and a central air compressing and freezing station necessary for the needs of the pneumatic control system, commonly used in new production plants. Also new supply units, switching stations and electric sub-stations with new cable networks were performed. The further expansion of the combined heat and power plant EC II was realised by adding new steam boilers Nos. 4 and 5 and an assembly of two steam turbines from the Hungarian company –

Lang [57]. As part of "Tarnów II", a new production plant for producing iron-chromium catalyst for CO conversion, iron catalyst for ammonia synthesis and copper catalyst for methanol synthesis was also set in motion (in a new building); and the production of nickel catalysts for methane conversion in the old production plant was intensified [58]. In the years 1962÷1964, a semi-pressurised nitric acid installation with the capacity of 100 tons per 24 hours was built. It was the first Polish installation with the incorporated foam system of nitrogen oxide absorption. From 1964 to 1968, the production plant for nitric acid using the moderately pressurised method, which consisted of 4 units with the total production capacity of 800 tons per 24 hours, was consecutively being put to use. In the first half of the 1960s, a new production plant of ammonium nitrate and granular nitro-chalk using tower granulating was built by the project of "Prosynchem" – the planning and design company from Gliwice. The construction was performed in two stages: In 1963, the first line for producing nitro-chalk with the capacity of 750 tons per 24 hours was activated. The second line for nitro-chalk production was activated two years later. As a result, the production plant capacity increased to 1500 tons of fertilisers per 24 hours. The columns with heat recovery of nitric acid neutralisation by gaseous ammonia were a novelty in producing nitro-chalk and ammonium nitrate [59]. In the 1960s, also Billiter electrolysis and mercury cell I installations were intensified. In connection with the construction of the Polyvinyl Chloride Plant, a decision was taken to build the second unit of mercury cell. It was completed in 1967. In 1971, the second installation unit of partial oxidation was built and set in motion. Technical and technological solutions applied for that installation contained know-how bought in Japan and own improvements of the company which were made after cancelling the cooperation with the Italian company. The operation of the installation was stable and efficient. In 1972, the first partial oxidation unit was modernised by incorporating the solutions following the pattern used in the second installation. Putting to use the modernised first unit of partial oxidation was regarded as the completion of "Tarnów II" investment. The partial oxidation technology including the Japanese know-how and a number of own solutions of the company was so original and the installations operating on its base were so reliable that in the second half of the 1970s, this technology was exported as the engineering solutions and initiated a new activity of Azoty Tarnów which was intensively developed in next years.



Installing the partial oxidation of methane, 70s Twentieth century

The years of “Tarnów II” construction were a turbulent period for the Research Laboratory which had been functioning in Azoty Tarnów from the middle of the 1930s. In 1963, a new building for the Laboratory started to be built [60]. The Research Laboratory went through a few transformations in the post-war period. In 1947, it was transformed into the Central Experimental Laboratory (CLD – Centralne Laboratorium Doświadczalne), in 1952 – into the Branch Department of the Institute of Chemical Synthesis in Gliwice, while in 1958, this Department in Tarnów was designated as an independent research unit named as the Fertilizer Research Institute (INS – Instytut Nawozów Sztucznych) seated in Tarnów. In 1968, INS was moved to Puławy by the decision of the Ministry of Chemical Industry. At that time, the unit, which had been operating in Tarnów for many years, was divided. Its part in Tarnów was transformed into the Research Institute.

Four fundamental production lines were clearly separated when the main installations of “Tarnów II” were put to use. They were: the production line of nitric compounds, organic compounds – methane derivatives, chlorine compounds and caprolactam. The further change in raw material base for ammonia production took place in the first half of the 1970s. This change was affected by economic aspects and the production capacity of the installations producing gas for ammonia synthesis smaller than the capacity of ammonia synthesis installations modernised and built in the period of “Tarnów II” investment. For the purpose of changing the raw material base, a decision was taken to purchase a new installation of methane-steam conversion on the nickel catalyst with the capacity of 500 tons per 24 hours in terms of ammonium. The licence was bought from the Danish company Haldor-Topsøe. The unit was given a number of the consecutive synthesis plant No. 5 and was known by the name of “base change” [61]. The new installation started to operate in September 1973. Its start-up resulted in discontinuing water gas-based production of ammonium from expensive coke. The outdated generators, which belonged to the most important installations of Azoty before the war, were shut down.

The period of “Tarnów II” was also a period of successes of the chemists from Tarnów in improving the caprolactam technology. After the installation start-up in 1957, its designed capacity of 4000 tons was achieved and then exceeded [62]. In subsequent four years, the installation capacity was increased to as much as 16.2 thousand tons per year. Nearly fourfold increase in the production capacity of caprolactam was produced in the operating plants, which required considerable effort and skills from the designers, contractors and all the services. Further works on modernising and intensifying caprolactam production including, inter alia, the construction of caprolactam purification plant by crystallisation, the construction of “Luwa” film distillation based on the Swiss solutions, and the construction of installation for producing cyclohexanone based on cumene-phenol process resulted in achieving the capacity of 55 thousand tons per 24 hours in 1975. The major landmark in caprolactam production was the start-up of a new, innovative method for producing cyclohexanone from benzene on an industrial scale in 1973. This method was based on own studies of the company and the studies of the Institute of Chemical Industry in Warsaw. The process, named “Cyclopol”<sup>®</sup>, was a hit in the field of exporting Polish engineering solutions not only related to the chemical industry, but also to the whole national industry.

At the beginning of the 1970s, a decision was taken to expand the installation for processing polyvinyl chloride which had so far been operating as the pilot plant. The production of PVC perforated drain pipes and telecommunication ducts being in great demand from the agriculture sector began. By the end of 1975, two lines of FRG origin for producing perforated drain pipes were started up in the PVC Processing Department. Two identical production lines were bought a year later. In that way, Tarnów became a potentate regarding the quantity and quality of perforated drain pipes. In the 1980s, Azoty was producing 20 thousand kilometres of PVC drains annually, which was more than

the total production of all other Polish production plants in Oława, Sochaczew and Wąbrzeźno [63]. In August 1975, the production of polytrioxane under the trade name Tarnoform<sup>®</sup> on the experimental pilot installation began in August 1975 [64]. It was an installation of a rather small capacity (about 1000 tons of powder per year), but, due to the scale of technological problems and the qualities of produced polymer, that installation was classified into a group of important technological achievements [65]. The decision on building the technical production plant of polytrioxane was not taken before 1985 [66]. In 1987, the Fertilizer Research Institute in Puławy (INS) and the Institute of Industrial Chemistry in Warsaw (IChP) elaborated the design basis for Tarnoform<sup>®</sup> industrial installation based on the experience from the pilot installation. The Design Office at the Plant performed that design [67]. Starting the production of polytetrafluoroethylene (PTFE) under the trade name Tarflen<sup>®</sup>, an equivalent to a product known in the world under the name teflon produced by “Du Pont” company, was a novelty in Azoty Tarnów at first, the Research Institute team developed the technology for producing semi-finished products: freon and tetrafluoroethylene, and then polytetrafluoroethylene. The pilot plant-scale production of that unique material was mastered already in 1970. The start-up of prototype installation with the production capacity of 500 tons per year, which was designed by the Design Office at the plant, began in 1978 [68]. Its success caught an interest of foreign contractors and made that technology, besides the caprolactam technology, an export object of engineering solutions in the following years. The significance of engineering solutions was recognised in the plant not only as a source of the company’s additional revenue, but also as the motivation for specialists to develop and improve their qualifications.



Installing the oxidation of cyclohexane, 70s Twentieth century

In the 1980s, the availability of investment means was reduced, and consequently, the Nitrogen Plant was only performing the modernisation tasks. At the end of the decade, the new installation of industrial nitric acid began to be built [69]. The improved technology for the installation was developed by the scientists from the Fertilizer Research Institute in Puławy, and the process project was done in “Prosynchem” from Gliwice [70]. The production of nitrogen fertilisers in Tarnów would have been abandoned if the old acid units had not been replaced by the new installation meeting the ecological standards [71]. The installation was activated in April 1992.

#### Period of transformations and restructuring. Towards the privatisation

On 21 February 1991, the act on the Nitrogen Plant in Tarnów (Zakłady Azotowe w Tarnowie) transformation from the State Enterprise into a joint-stock company with a 100% participation of

the State Treasury was signed. The company was registered on 1 March 1991. The fall of so called acetylene chemistry and a significant increase in natural gas prices caused that from the beginning of the 1990s, the "strategic business exit" was gradually incorporated to the majority of the installations which had begun to operate in the period of "Tarnów II". The acrylonitrile installation which did not meet the conditions of the introduced market economy was shut down as the first installation in 1990. Azoty was still producing 11-13 thousand tons of acrylonitrile annually until 1989 [72]. In the middle of 1991, a decision was taken to stop its production [73]. At the end of the 1980s, the government's industrial department plans included the intensification of silicon production to the level of 110 tons annually, but the prognosis for Polish electronics sector failed [74]. In June 1991, the technological start-up was completed and a preliminary operation of a recently built silicon production plant with the capacity of 50 tons per year was in progress. After opening the market, the installation was shut down at the end of 1991 as the production was unprofitable [75]. At the end of the 1990s, problems with selling acetone cyanohydrin came up. At the beginning of 1992, the installation had to be temporarily shut down [76]. Similar difficulties were related to the sale of chloro compounds. The lack of buyers and uncompetitive quality of products caused that the plant, which had been operating since the 1960s, was shut down, and completely liquidated in 1999 [77]. The new economic principles also resulted in difficulties with selling perforated drain pipes and telecommunication ducts at the beginning of the 1990s [78]. In 1990, the production of PVC pipes had to be temporarily reduced even to 20% of the production capacities. The discontinuance of subsidising the land improvement works by the government was the main reason for the reduced demand for PVC pipes.

In autumn of 1990, the construction works of the modern ammonium sulphate installation started. It was designed by the Company's Design Office at the plant and put to use in November 1992, delivering a product of high quality [80]. The new investment not only improved the quality, but also the effectiveness of the product and the amount of sulphate introduced to waste was reduced [81]. In the same year, the production plant of window profiles from polyvinyl chloride started, and a few years later - the production plant of windows from PVC was activated [82]. In 1993, the production of liquid carbon dioxide based on CO<sub>2</sub> emitted from the methane synthesis plant V into the atmosphere began. The available production resources were used for that purpose [83]. The industrial recession was reflected by the decrease in the number of orders for Tarflen® [84]. However, the buyers' interest in Tarflen® products was increasing, so a part of produced Tarflen® powder was processed into a wide range of such finished products as foils, seals, or parts of machines and equipment [85]. In 1995, about 150 tons of Tarflen® finished products were manufactured for sale [86].

In the 1980s and 1990s, caprolactam production capacity was continued to be improved (75 thousand tons per year was reached in 1995) [87] by intensifying the production of cyclohexanone from benzene as well as modernising the installations of ammonium nitrate and hydroxylamine sulphate. The two-step Beckmann rearrangement introduced in September 1993 considerably improved the product quality. In December 1993, the quality of flaked caprolactam and the productivity of its packaging were improved as a result of modernising the installation and activating a new line for its packing and palletising. These actions resulted in increased export of caprolactam, and the product was delivered to the buyers in China, Taiwan, Malaysia, Indonesia, South Korea and Japan [88]. In autumn of 1994, the start-up of the first line of the new Tarnoform® production plant with the specified production capacity of 10 thousand tons annually in two production lines began [89]. It was based on the new installation for producing formalin production, which had been activated in 1993. Putting to use the modern installation of caprolactam polymerisation for producing polyamide PA-6 (Tarnamid®) with the production

capacity of 22,700 tons annually was the most significant event in 1996. The old installation, dating back to the beginning of the Caprolactam Production Plant, was shut down after the activation of the modern one. The construction of a new production plant of polyamides under the licence of the German company Zimmer started at the turn of 1995 [90]. The start-up of that production plant considerably contributed to the increased export sale of Azoty. In the middle of 1996, for the first time in the company's history, the export was higher than the national sale, reaching more than 53% of the total sale value [91]. In 1997, the second line of Tarnoform® production plant was put to use. Consequently, the production of that material in demand was increased by 5 thousand tons achieving 10 thousand tons per year [92]. Product dyeing was introduced in order to optimise the production costs and to widen the product range [93]. Production technology of polytrioxane was included into the group of the company's technologies to be exported as engineering solutions.

In 1996, the installation of concentrated nitric acid started to be built. The technology, process design and basic equipment were purchased from the German company Plinke [94]. The installation was started up at the end of 1997, after less than 15 months from the construction beginning [95]. Due to the start-up of the new installation of concentrated nitric acid with the capacity of 100 tons per 24 hours, the existing and worked out installation was shut down; consequently, the emission of nitrogen oxides to the atmosphere was reduced and the energy consumed during the production of concentrated nitric acid was considerably reduced [96]. In the 1990s, the production of ammonia in the plant was becoming more and more ineffective and it was doubtless that the ammonia synthesis installation dating back to the pre-war period required modernisation. It was determined by not only the economic, but also the ecological aspects. The design works related to the construction of the new installation based on the equipment purchased along with the ammonia installation from the Hungarian plant Varpalota were commenced in 1997 [97]. The hand-over of the installation to the operation in 2000 made it possible to shut down the ammonia synthesis units operating from 1929. That investment was realised in collaboration with the Fertilizer Research Institute (INS) in Puławy and the Design Office at the plant [98].

The plans for modernising the technical and technological solutions were being implemented during the whole decade of the 1990s regarding the reduction of costs and the improvement of competitiveness as well as the reduction of harmful impact on the environment. The effects for the environmental protection were achieved as the result of modernising the existing installations, equipping them with extra protection nodes as well as bringing into operation the new installations based on modern technologies. Even a tenfold reduction of nitric oxide emission to the atmosphere, of dust emission, and reduced discharge of nitrogen and pollutant load in sewage certainly belong to the most significant and noticeable pro-ecological successes. Deleting Azoty Tarnów from the list of the Polish enterprises most troublesome for the environment, so called the "List of 80" [99], in April 2001 [100] was the confirmation of the company's effective pro-ecological actions. The further expansion of the combined sewage treatment plant was realised in the 1990s [101].

In the 1990s, PVC production from own vinyl chloride, which was based on expensive raw material – acetylene obtained by methane partial oxidation, was not profitable apart from exceptional periods. From the second half of the 1990s, the price of natural gas was increasing regularly, which caused the further increase in PVC production costs. In 1991, the company concluded an agreement on importing vinyl chloride to improve the production profitability. It was cheaper than the semi-finished product from Tarnów as it was obtained from ethylene-based production [102]. The existing economic conditions necessitated the further reduction of acetylene production, and, consequently, own vinyl chloride. In 1998, the partial oxidation installation I was shut down

and the partial oxidation installation II ceased operating in 2001. In the second half of 2001, in relation to high costs of buying vinyl chloride and its processing, the production of PVC was discontinued and then, in April 2002, a resolution was passed on putting Polyvinyl Chloride Production Plant into liquidation [103]. The beginning of the 21<sup>st</sup> century was also the fall of acetone cyanohydrin from Tarnów, for which the second half of the 1990s had been the period of dynamic development, in which the top production capacity had been at the level of 50 thousand tons per year using the released production capacities of hydrogen cyanide after stopping the production of acrylonitrile. In April 2002, when the contracts on processing for export expired, a decision was taken to shut down the installation of acetone cyanohydrin [104].

After the time of forced restructuring of the production in the plants built at the time of "Tarnów II", the expanded and modernised caprolactam plant with ammonium sulphate, the fertiliser plant on the basis of the new ammonia synthesis and a number of ancillary and infrastructure facilities remained. The reduced production of chlorine and its derivatives was maintained until 2007. After 2005, the profitability of manufacturing the products from the polyvinyl chloride processing installation was not maintained. A small-scale production and high competitiveness on the market forced Azoty to reduce and consequently, cease their production. In 2005, Tarnoform<sup>®</sup> plant was modernised and intensified, achieving the production capacity of 14,100 tons per year. The experience from designing and performing installations in China as part of the licence contracts were used for the above purpose. Also the second line for producing modified construction materials, so called compounds based on Tarnamid<sup>®</sup> and Tarnoform<sup>®</sup>, started to operate [105]. A new kind of cooperation with the Polish Oil and Gas Company (PGNiG – Polskie Górnictwo Naftowe i Gazownictwo) started as well. As a result of conducted analyses and changes in production processes, Azoty Tarnów and PGNiG signed a mutually favourable contract on delivering natural gas with increased inert content from the local sources located in the immediate surroundings of Tarnów. 2005 was also the year of realising two investments which were crucial for the last period. The intensification of polyamide PA-6 plant to the capacity of 45 thousand tons per year was the first one. The project was realised in 2007 on the basis of purchasing a licence, process project and key equipment from the German company Zimmer AG. As a result, the amount of caprolactam processed in the plant was increased. The construction of a mechanical granulating installation for fertilisers with the production capacity of ca. 400 thousand tons per year was the second investment [106]. Another landmark in the technology for producing cyclohexanone from benzene occurred in the same year. It was related to the implementation of the so called two-stage oxidation process developed jointly with the Institute of General Chemistry and the Warsaw University of Technology. It started the technology named Cyclopol-bis<sup>®</sup>. In consequence of intensifying and modernising actions made in the years 1995 ÷ 2005, the total production capacity of caprolactam reached the level of 95 thousand tons per year.

In March 2008, Jerzy Marciniak became the President of the Management Board. In June 2008, Azoty Tarnów made its debut on the Warsaw Stock Exchange. Financial means from the Warsaw Stock Exchange were to be a considerable part for financing the investment programme covering the modernisation of the Caprolactam Plant along with the construction of the new installation for producing hydrogen, the start-up of fertiliser mechanical granulation, the expansion of the Modified Plastic Production Plant (so called compounds), the increase in polyamide production capacity (by 55 thousand tons per year) and the increase in polyoxymethylene production capacity. By the end of 2009, a modern installation of fertiliser mechanical granulation with the capacity of 1200 tons per 24 hours was put to use. A new product - ammonium sulphate nitrate

(Saletrosan<sup>®</sup>) was introduced to the market and the market position of the plant in the sector of nitro-chalk fertilisers was strengthened as a result of realising the above task. The Nitrogen Plant in Kędzierzyn was a licensor of the installation, and the technology for producing Saletrosan<sup>®</sup> was a solution developed jointly by the chemists from Tarnów and Kędzierzyn. The second realised objective was the increase in production capacities of polyamides. That objective could be realised by changing radically the company strategy, a part of which was not only the internal growth of the company, but also appreciated possibilities of mergers and acquisitions. At the beginning of 2010, Azoty Tarnów acquired 100% shares of the German manufacturer of polyamides from Guben, presently operating under the name ATT Polymers. The acquisition turned out to be a considerably more favourable option than the construction of a new plant in Tarnów. It resulted in a significant advancement of the Azoty Tarnów Capital Group among the European polyamide manufacturers. In 2010, the next objective started to be realised – the construction of Hydrogen Installation in Tarnów with the production capacity of 8000 Nm<sup>3</sup>/h related to the modernisation of the Caprolactam Plant to the capacity of 101.3 thousand tons per year. As a result of that investment, the consumption of natural gas from local sources was increased and consequently, production costs were lowered. The Modified Plastic Production Plant has been intensifying and thus it is possible to increase the production capacities of polyamide PA-6 based compounds.

Striving for the strategic expansion of the operation scale, Azoty Tarnów took the control over ZAK S.A., one of the biggest enterprises in the chemical synthesis sector in Poland, when such an opportunity arose on the market of acquisitions. The next strengthening of the Azoty Tarnów Capital Group took place in 2011 when the controlling interest of Police Chemical Plant was acquired. As a result of the entry of two big enterprises from the sector into the Azoty Tarnów Capital Group, a considerable increase in the scale of actions and efficiency regarding mineral fertilisers, construction materials, Oxo alcohols, flexibilisers and titanium white was possible and thus the Azoty Tarnów Capital Group achieved a strong position not only on the national chemical market.

The information contained in this paper is an attempt to present briefly the facts on developing the technology and technique of the biggest enterprise in Tarnów region during 85 years of Azoty Tarnów operation. The facts at every stage of history were created by people. In regard to established rules, only a few names of people connected with the time periods that seem to be the most important during the whole time of factory existence were mentioned. The author assumed the following periods to be the most important: the period of PFZA formation, the period of so called Tarnów II and the present period after Azoty Tarnów debut on the Warsaw Stock Exchange. The paper only generalises the achievements of the latter period as they are dynamic and still in progress. However, the author would like to emphasize that such great achievements described in the paper would not have been possible and certainly the company's present position would be different without people (often the generations) entirely devoted to the company, who were frequently outstanding specialists and personalities.

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100. Kompromis zdrowego rozsądku i zdecydowanego stanowiska, „TA” nr 1889, 18.04.2001, s. 1.
101. Wspólna GOŚ-ka, „TA” nr 1728, 11.03.1998, s. 2.
102. Polichlorek winylu znów w cenie, „TA” nr 1402, 05.12.1991, s. 3.
103. Z prac Zarządu..., „Biuletyn Tarnowskie Azoty” nr 1927, 04.04.2002, s. 2.
104. Z prac Zarządu..., „BTA” nr 1927, 04.04.2002, s. 2.
105. Rok 2005 w Centrum Tworzyw Sztucznych, „BTA” nr 2026, 21.12.2005, s. 4, 9.
106. Inwestycje stają się faktem, „BTA” nr 2019, 14.09.2005, s. 1.

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