

UNINTENDED TECHNOLOGY TRANSFER: ACETYLENE CHEMISTRY IN THE UNITED STATES*

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Introduction

Peter Morris, recipient of the 2006 Edelstein Award, through several splendid essays focusing on science and industry in Germany and the United States, has shown that it was less preconceived policies that transformed chemical industry during the 1930s and early 1940s than the expediencies of war economies, in other words, political and strategic priorities (1). This is clearly exemplified by the rise of high-pressure acetylene chemistry, as originally described by Morris in his 1982 doctoral thesis (2). Though acetylene chemistry rapidly declined from the 1960s, apart from the production of 1,4-butyne-3-diol, it did have some considerable and long-lasting impact in the United States at the General Aniline & Film Corporation, particularly at its Linden, New Jersey, facility. Until 1942, this corporation, originally a leading producer of indanthrene vat dyes and azo dyes, was under the ownership of I. G. Farben (3).

The corporate behemoth I. G. Farben was the outcome of the 1925 merger of the main German dye-manufacturing firms, BASF, Bayer, and Hoechst, founded in the 1860s. Until 1914, they had dominated the manufacture and supply of coal tar dyestuffs. However, following the outbreak of war in Europe, and in direct response to shortages of the essential chemicals previously available from Germany, the modern US chemical industry emerged. Meantime the German dye firms had begun to diversify, which included work in high-pres-

sure chemistry, particularly the Haber-Bosch ammonia synthesis (4).

In the United States the wartime situation stimulated the rapid development of technologies based on complex aromatic chemistry (5). This led to an advanced science-based industry that during the 1920s also embarked on diversification. However, even then, access to German innovations was still needed, including the new high-pressure Bergius coal-to-oil process. Such was the perceived importance of I. G. Farben's processes that in Germany the corporation was plagued with industrial espionage, and it was not unknown for I. G. Farben's intelligence department to break up spy rings acting for French and American interests.

At the same time, the Germans wished to regain dye markets lost during the war. This led to a singularly important merging of American and German interests, the General Dyestuff Corporation, in 1925. This was predecessor to the General Aniline Works, established by I. G. Farben in 1929, which was renamed General Aniline & Film in 1939. The main manufacturing sites were at Linden, on the Arthur Kill, opposite Staten Island, and the former Bayer works at Rensselaer, New York, on the Hudson River. There was also the AGFA-Ansco facility at Binghamton, New York. The latter was responsible for the word Film in the name. A unique strategy for control of production of dyes, intermediates, photographic products, and detergents was implemented at the German

controlled General Aniline & Film (hereafter GAF), and affiliates.

Reppe Chemistry

During the 1930s, I. G. Farben embarked on a completely novel area of diversification based on high-pressure acetylene chemistry. This had previously been considered far too dangerous since acetylene under pressure is inherently unstable and explodes with extreme violence. This reflects its great reactivity that derives from the triple-bond between the two carbon atoms. As a result of the I. G. Farben investigations, the development of acetylene-derived chemicals in Germany represented a major technology breakthrough and became an outstanding commercial success. The basic research for safely reacting the highly flammable gas with other chemicals under high pressures was done by J. Walter Reppe (1892-1969) at the Ludwigshafen laboratories of I. G. Farben from the late 1920s.

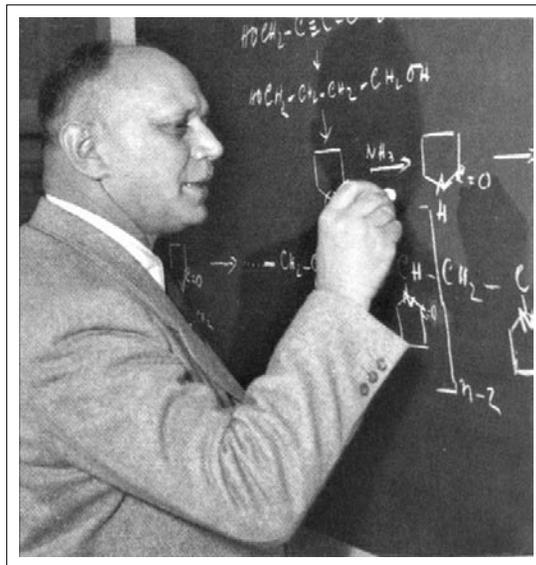
As Morris has pointed out, Reppe "bravely pioneered dangerous research on the reactions of acetylene under pressure, and thereby opened up entirely new fields of industrial organic chemistry (6)." In 1934 Reppe was appointed head of the new Ludwigshafen Intermediates and Plastics Laboratory, and in 1938 of the main Central Research Laboratory. With acetylene, there were certainly daunting technical hurdles to overcome. Engineers and chemists fixed the problems in much the same way that they had overcome difficulties with ammonia reactors two decades earlier. In England, Imperial Chemical Industries (ICI) had undertaken similar studies in the 1930s, but, because of the enormous danger, worked on a very small scale.

Reppe's team, meantime, developed large-scale robust and reliable manufacturing equipment, as well as designs for suitable buildings that were capable of withstanding explosions.

The acetylene research had a tremendous impact on the development of several processes, including the production of synthetic rubber, which became strategi-

cally important once the second Nazi Four Year Plan was inaugurated in September, 1936. The technical potential of acetylene chemistry had been realized following Reppe's 1927 conversion of acetylene into butadiene. This and the directives of the plan stimulated further studies, including reactions in which the triple bond was retained. This was successfully achieved by Reppe in 1937 when he treated acetylene with two molecules of formaldehyde to afford 1,4-butynediol. The reaction provided a new route to butadiene, the building block for Buna synthetic rubber. As a result of this and other developments, as Morris has pointed out (7):

At I. G. Farben (in contrast to American companies), a central role was played by acetylene. By the late 1930s, acetylene underpinned most of I.G.'s heavy organics (with the exception of methanol), most notably the copolymer synthetic rubbers.



Walter Reppe at the blackboard preparing a diagram of the vinyl pyrrolidone process. BASF Archives.

Such was Reppe's fame that in 1945 the American Chemical Society nominated him a major target for the Allied investigators that were hunting down leaders in German science and technology. Four years later the Department of Commerce published its eagerly awaited Reppe Report. Industrial acetylene chemistry based on Reppe processes thrived for a time in Germany and, as summarized here, at GAF in the United States. How this came about requires some nonchemical explanation.

Patent Transfer and US Government Ownership

During the late 1930s, concerns over possible Nazi influences on American industry led investigators at the Securities and Exchange Commission to investigate the ownership of GAF. The situation was confused by the existence of dummy companies set up by I. G. Farben in Holland and Switzerland that claimed ownership of GAF and other facilities outside of Germany. The investigations were intensified after war broke out in Europe during 1939. Fearful of loss of important assets in the

largest chemical-consuming market on earth, the patent committee of I. G. Farben on April 30, 1940 convened to discuss assignment to GAF and U.S. affiliates of certain of 2,208 out of 5,500 American patents and applications. According to the minutes of the meeting, August von Knieriem, chairman of the board of directors, supported the measure (8):

[I]n considering the transfer, it should not be asked whether the transfer is a profitable business venture, but the transfer should be regarded above all as a protective measure taken for the safe-guarding of the I.G. patents. These patents will be in the possession of a friendly business enterprise.

New York based Walther H. Duisberg, son of Bayer's Carl Duisberg, "a patent attorney and consulting chemist," would be the intermediary in the transaction. Duisberg also supported transfer, since Americans would be expected to honor the arrangement and were unlikely to sequester patents belonging to a foreign corporation whose ultimate ownership, unlike Bayer during World War I, was uncertain: "American opinion takes a much more legalistic view of such assignments as we believed hitherto." Moreover, the chairman explained, "German public opinion has changed and does no longer, as was the case at the beginning of the war, regard any such safe-guarding as defeatism. The Reichs Minister of Economics urges again and again that I.G. should follow the example of other German business enterprises and should assign its patents to American business enterprises. It is the task of the Patent Committee to fix the specific terms under which such a transfer may appropriately be made." Mr. Redies of the Lower Rhine factory group, however, was against transfer of the patent rights, in part because he doubted that the United States would enter the conflict:

There are two possibilities; either we make a genuine sale of the I.G. patent rights or we refrain from doing so and bear the risk inherent in such action. If the patent rights are not sold, disadvantages to I.G. will arise only if the United States enters the war and if later on German patents will be seized as was done in the First World War I doubt very much whether this possibility will materialize and therefore the risk existing at present is a very doubtful.

The transfer, however, was considered the safest strategy, since, as von Knieriem emphasized (8):

the Reichs Ministry of Economics expects that the transfer of our most important patents and patent applications will be made...Furthermore, many American inventions are protected by German patents. The United States owns valuable patents in Germany. In the event of war, these patents will be subject to seizure

by the German Government and for this reason the American Government will be compelled to exercise considerable restraint.

The transfer of patents, including the acetylene-based processes, was made a few days later, on May 4 (9).

However, this did not turn out to be in the best interests of I. G. Farben, whose influence in the United States came to an abrupt end following American entry into World War II in December, 1941. GAF was now a corporation belonging, even if in a convoluted way, to an enemy country. The US Treasury Department, unable to sequester the corporation immediately because of the uncertainties concerning ownership, installed seventeen secret service agents in the main offices and plants of the corporation to ensure American control of all activities, and to prevent disclosure of sensitive information to Germany. Then in January, 1942, the Department ousted five German-born executives, all naturalized American citizens, for personifying the Nazi domination of the company. Treasury Department agents closely monitored the activities and communications of all research staff. FBI agents conducted extensive interviews with all employees (10). Former GAF chemist Barry Bochner, for example, remembers that at Linden quite a few were "Ready to die for Hitler," and were removed by the FBI.

Fifty executives and key workers, regarded as un-dependable, or as security risks, were fired or taken into custody. On February 16, 1942 the Secretary of Treasury issued an order for transfer of stock to the government, that is, formal seizure of the assets of GAF as enemy property. Four American businessmen were put in charge as appointees of the Treasury and began redirecting activities to the war effort.

The only former German chemists allowed to remain at GAF manufacturing sites were the few who were Jewish, were married to Jewish women (which was the reason that Dr. Paul Nowialski was sent by I. G. Farben to America, as was probably also the case for Dr. Werner Freudenberg; both worked at Linden), or who had expressed strong anti-Nazi sentiments. They included Dr. Wilhelm Von Glahn, director of process research at Rensselaer, who "had a very thick German-Jewish accent" and bore the ultimate Prussian mark of honor, "terrible looking dueling scars all over his face (11)." Several I. G. Farben chemists of Jewish origins had already been assigned to posts outside of Germany, including at AGFA-Ansco, by sympathetic managers at certain divisions (12). As a result, the ethnic composition at former I. G. Farben-owned factories in America would

henceforth be quite unlike that found at other American chemical factories. This was perhaps ironic, since US chemical firms tended not to hire Jewish chemists until after 1945.

Patents for America

As a result of the 1940 transfer of patents to GAF, an important strategic and military asset came into the hands of the Americans. Under US government ownership, GAF engaged in diversification based on prewar German innovations, particularly in the new Reppe acetylene-based processes.

A principal contributor was Hans Beller (1901-1984), one of the German chemists assigned to a GAF manufacturing site. Beller was born in Munich, where in 1924 he received a doctorate in chemical engineering from the institute of technology. He then joined BASF at Ludwigshafen as chemical engineer, and then he was transferred to GAF. He was highly vocal in his opposition to the Nazis, and for this reason was assigned to Linden. There his first challenge, in 1942, was development of carbonyl iron, also based on patents and know-how previously acquired from I. G. Farben. His endeavors enabled the Linden plant to produce carbonyl iron powder suited to the manufacture of radio frequency electrical cores needed by the military (13).

Prior to 1942, GAF had depended on I. G. Farben for research to support all of its business areas. Though GAF had not duplicated I. G. Farben research in the United States, details of a few innovations made at Linden were sent to Germany. These related mainly to dye-stuffs. A small amount of research was carried on in the photographic film plants for the AGFA-Ansco division. Certainly, no fundamental research or expansion into new fields was done. In 1945, it was reported that (14):

the result of this policy was the complete subservience of [GAF] to [I. G. Farben], for the results of the German research were never disclosed to [GAF].... in many cases important material was only communicated verbally to the most trusted employees of [GAF] on the occasion of their visits to Germany.... The information thus obtained was not disclosed to other employees of [GAF in the US]. Thus on several occasions when the man in possession of information died [GAF] was obliged to send another employee to Germany for instruction in the particular process.

In other words key processes carried out in the United States were carefully controlled from Germany.

The GAF and the I. G. Farben Central Research Laboratories

The outcome was that after the government takeover in 1942, GAF was in possession of almost 4,000 I.G. Farben patents but lacked the technical staff to commercialize the inventions. The American management team then committed \$10 million to create a first-class research organization, known as the Central Research Laboratory, established at Easton, situated in Pennsylvania's Lehigh Valley, in the summer of 1942. By the autumn, some fifty chemists, engineers, physicists, and technicians had been brought together both from within the corporation and from leading scientific institutions. The laboratory was among the largest industrial research centers in the United States. Several German chemists at GAF manufacturing sites, including certain senior managers believed to have close ties with their homeland but not considered to be high security risks, were reassigned to the Easton laboratory.

The early effort at Easton was focused on dye chemistry but was soon extended into broader fields, including research into photographic materials. The constitutions of the important AGFA color formers, rivaled only by those of Kodak, and previously kept secret, were quickly worked out and their production was commenced in 1942. The hot topic was high-pressure acetylene chemistry. The Easton laboratory developed thirty acetylene-derived products that showed potential commercial applications.

Progress was also made in Germany. At the end of the 1930s, Reppe's chemists at the Ludwigshafen Central Research Laboratory had developed an acetylene-based process that afforded vinyl pyrrolidone in five steps. This was then converted into its polymer polyvinylpyrrolidone (PVP), in the presence of catalysts, according to the first patents filed during 1938 and 1939 (15). Suggested uses included as textile assistants and thickening and sticking agents (16). However, PVP was transformed into a blood plasma substitute, called Kollidon by Helmut Weese and Gerhard Hecht of the Elberfeld (Bayer) pharmacological laboratories (17). It was chosen after Weese had tested colloidal substances received from all divisions of I. G. Farben in response to the needs of the Wehrmacht. For intravenous use Kollidon was dissolved in water containing inorganic salts and named Periston. Trials were carried out by H. Bennhold at the University of Tübingen. Introduced probably at the end of 1940, Periston was considered highly important by the German military. The product was improved by increasing the

PVP content, leading to greater efficacy in maintaining blood pressure. During 1943 a similar product, Subtosan, was introduced in France by Rhône-Poulenc.

After the war it was reported that Periston (18):

is said to have saved the lives of tens of thousands of German soldiers. In addition, because of the difficulty of obtaining plasma in wartime Germany, it was used extensively and successfully in civilian hospitals in shock and similar conditions.

One of the chemists engaged in this work was Curt Schuster, who from 1939 had participated in studies on the reactions between acetylene and carbon monoxide that afforded acrylic esters. In May, 1943 he was arrested by the Gestapo; he had probably been under suspicion for some time, as a member of a group helping Jews and opposed to the Nazi regime. On February 15, 1944, Schuster was sentenced to three years in prison. His colleagues at BASF had testified on his behalf as character witnesses, emphasizing his important role in the invention of Periston (19). This apparently led to a relatively mild sentence; though a prisoner he was forced to work at the I. G. Farben Oppau works. After the war Schuster returned to Ludwigshafen and prior to retirement became an outstanding historian of the synthetic dye industry (20).

When the war ended, the Easton Central Research Laboratory employed 107 research workers, including chemists and physicists, 67 of whom had Ph.D.s. GAF manufactured dyestuffs and miscellaneous chemical products, including detergents, carbonyl iron powder, and resins; the Ansco Division manufactured photographic films, papers, and chemicals, as well as cameras; and the Ozalid Division produced sensitized materials and machines for printing and developing.

The December, 1945 monthly research letter at Easton provides a useful summary of activities directed at exploiting certain patents held as a result of the 1940 agreement (21):

These patents deal, to a very large extent, with the chemistry of acetylene and for new methods of handling acetylene under pressure and at high temperatures. Under these patents two products have been developed [that]... require the same general technique for handling acetylene under pressure.

Further research had been hampered under the wartime conditions, but it was subsequently placed on a systematic footing, particularly the polymerizations involving methyl vinyl ether, and other ethers.

Studies into the role of peroxides as initiators in vinyl polymerizations were conducted, and, as a result, the first polymerisations of vinyl pyrrolidone were carried out at Easton using hydrogen peroxide at 100° C.

The product was PVP. Information from Europe "on new method for synthesis of acrylic acid esters ... fits nicely with our work on the development of acetylene chemistry (21)." Infor-

mation had also arrived about Periston, the blood plasma substitute made from PVP.

As elsewhere, the research work was not without incident. In 1945 there was a massive explosion in the high pressure acetylene section (22):

It blew out windows and wrecked equipment in the research building... The blast buckled the ceiling of the basement and the floor of the first floor, knocking over and destroying analytical balances.

The cause was a miscalculation, namely addition of fifty times the required amount of catalyst.

Reppe Chemistry in America

As a result of the successful acetylene research at Easton, in May, 1946 GAF announced that work had commenced on a new \$1,250,000 building at Linden that would serve as a semi-works and pilot plant for the manufacture of chemicals from acetylene. Opened in 1949, this was the first and probably only unit of its kind in the United



Hans Beller, at right, and Abraham Zoss, his chief assistant, at GAF Linden examine a sample of PVP. Photograph by Russell C. Aikins. From PVP. Polyvinylpyrrolidone. Preparation, Properties and Applications in the Blood Field and in Other Branches of Medicine, General Aniline & Film Corporation, New York, March 1951.



Linden factory of GAF, July 1947. The new high-pressure acetylene building is at center, closest to the camera. Courtesy of Newark Public Library.

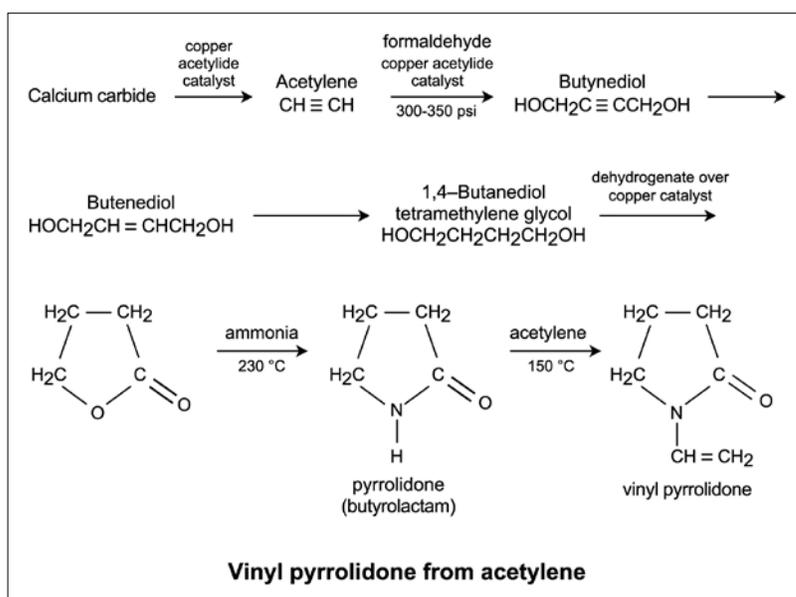
States. The two-story building was located on a vacant area at the southeast corner of the site. Some 640 piles supported the foundations. The building incorporated a reinforced explosion-proof stall for high-pressure acetylene experiments. The acetylene was produced from calcium carbide in a nearby building, since natural gas was then too expensive as a source.

The starting point for many of the new products was the reaction of acetylene under pressure with formaldehyde to form butynediol. This was reduced to butenediol and then to butanediol. During World War II, GAF produced two acetylene products for the military. One was Pollectron, poly(vinyl carbazole), also made in Germany as Luvican. It was useful as insulation for electronics and where high operating temperatures were employed. It was similar to polystyrene but exhibited improved heat resistance; mass polymerization gave almost clear glasslike castings. However, for peacetime use it suffered from high cost, lack of uniformity, poor color and poor mechanical properties. Nevertheless, copolymers of vinyl carbazole and styrene were found to have good molding properties. The other product was Koresin, also first developed in Germany.

This was a condensation product of acetylene and *p*-*t*-butylphenol. It was a very effective tackifier for GR-S synthetic rubber. John W. Copenhaver at Easton, a leading GAF expert in acetylene chemistry, and Maurice H. Bigelow, affiliated with Linden, undertook extensive investigations, including interviews of Reppe, in postwar Germany as members of an Allied commission. Later they wrote what would become the authoritative volume on the technology of acetylene chemistry (23).

The most important early product arising out of the acetylene work at Easton and Linden was PVP, originally discovered by Reppe's group at Ludwigshafen. A white powder, soluble in both alcohol and water, it was the basis of the valuable blood plasma extender.

Marketing of the vinyl derivatives and polymers was taken over by Jesse Werner, a former research chemist at Linden. In 1952, Werner was appointed director of commercial development, a post he held until 1959, when he was appointed vice president of the corporation. A GAF executive working closely with Werner was Juliette M. Moran, notable since she was one of



Flow chart for *N*-vinyl pyrrolidone (*N*-vinyl butyrolactam).

the first women to rise to a position of leadership in the chemical industry.

Moran had joined GAF at the end of 1943, since, as elsewhere, the shortage of male recruits during the war years hastened the admission of women into research and technical laboratories. Trained as an organic chemist, Moran was assigned to the GAF Process Development Department in New York, which in 1945 was transferred to Easton, where she became involved in administration. Subsequently she returned to New York, taking up a post with the marketing division, prior to becoming assistant to Werner. With Werner's support she was appointed, successively, vice-president, senior vice-president, and executive vice-president, followed by posts that led right to the top of the senior management team (24).

Werner, as part of his program for diversification away from dyes, and ably assisted by Moran, pushed the marketing of GAF's version of the blood plasma substitute (25). He also promoted the use of PVP in other medical applications. Though PVP underwent clinical trials in hospitals and was tested by the US military, it did not succeed commercially in medical fields as a blood substitute. One reason was that there was little perceived demand, since during World War II the Allied armies, unlike the Germans, organized the efficient collection of human blood on a vast scale and developed successful processes for handling plasma and other blood products.

However, the research did pay off in other ways. PVP formed transparent films on glass, plastics, and metals and found application in the formulation of cosmetics, particularly hair sprays. During 1949-1951, Herman A. Shelanski, a consultant to GAF, established through clinical trials that PVP combined with iodine was a useful germicide (26). GAF invested \$6 million in an acetylene chemicals plant at Calvert City, Kentucky, that came on stream in 1956. Linden's Hans Beller, who had earlier cooperated with Easton in acetylene research, was project director during the construction phase and the first plant manager. As in Germany, the technology was difficult and there were two serious explosions in the early years. However, GAF was the only producer of acetylene and its products in the United States, at least until the Dow-BASF process was introduced in 1958. The Calvert City plant lost money until 1962, after which the business became highly profitable. In 1965, after much legal wrangling over ownership, GAF was released from US government ownership and returned to the private sector, this time in American hands.

To fill the demand for the many new applications of acetylene products a second manufacturing unit was built at Texas City, Texas, in 1968 (27). There the acetylene was produced from petrochemical fractions. Another polymer based on PVP, known as Polyclar, used in production of beer and wine, was manufactured at Linden until the 1980s, and the copolymer Gafquat, for hair care products, was manufactured until 1991, half a century after Reppe acetylene chemistry was adopted at GAF.

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