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FIAT FINAL REPORT 729

THE GERMAN HIGH TEMPERATURE COAL TAR INDUSTRY

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OFFICE OF MILITARY GOVERNMENT FOR GERMANY (US)

FIELD INFORMATION AGENCY TECHNICAL

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THE GERMAN HIGH TEMPERATURE COAL TAR INDUSTRY

BY

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Joint Intelligence Objectives Agency

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FOREWORD

Germany's war-time, coal tar industry had two main branches which can best be considered individually because their raw materials, operations and distributions were almost completely separated.

One branch, old and well established, was concerned almost entirely with the processing of coal tar produced by the high temperature carbonization of caking, bituminous coal. The carbonization was effected principally in by product coke ovens and to a lesser extent in vertical, horizontal and inclined retorts. The high temperature tar was all processed in tar distilling plants that handled high temperature tar almost exclusively.

The other branch, newly established, dealt with low temperature tar which came principally from brown coal, a small amount from non-caking bituminous coal and very little (1.5%) from caking bituminous coal. Over ninety percent of the low temperature tar was produced in Lurgi-Spülgas retorts. The balance came from Krupp-Lurgi, Geissen, Borsig-Geissen and Rolle retorts. Most of the low temperature tar went directly to hydrogenation plants or was used, without processing, for fuel purposes. Only a small amount was processed in tar distilling plants and they handled low temperature tar exclusively.

Based on quantities of tar involved the two branches were about equal in size in 1943. However, they differed so much in all other respects that they were, virtually, two separate industries. Therefore, this report which deals only with matters pertaining to the processing of high temperature coal tar and the use of products derived therefrom is entitled "The German High Temperature Coal Tar Industry."

PURPOSE AND SCOPE OF THE INVESTIGATION

The following report covers an investigation of the German high temperature, coal tar industry conducted between September 15th and January 15th, 1945.

The primary purpose of the investigation was to determine to what extent the processing of high temperature coal tar and utilization of products derived therefrom advanced in Germany in the years immediately preceding and during the last war and to collect information concerning new or improved processes or products. Such information is summarized in the following portion of this report designated, Part I.

Of secondary importance was the collection of general information and data concerning German manufacturing procedures, equipment and uses for high temperature coal tar products even though they might not have been developed during recent years. Information of this kind is contained in the individual target reports that comprise Part II.

The quantity of high temperature coal tar processed anually in Germany by fifty-six tar plants during the period 1941-1943 was approximately two and one quarter million metric tons. About seventy thousand tons additional was used by small roofing plants for the manufacture of saturants and coatings. An appendix to this report contains a tabulation of the tar plants with the quantities of tar processed by each in 1941, 1942 and 1943. The table also gives the status of each in November 1944. The latter information was based on observation so far as possible but in those cases where the status could not be checked, the most reliable information available at the time was used.

The appendix also contains a table showing the origins of the tar supplies received at the tar plants. They came from bituminous coal carbonized at (a) coal mines in by-product ovens (b) steel works in by-product ovens and (c) gas plants in by-product ovens and vertical, horizontal and inclined retorts.

All documents collected during the investigations have been microfilmed. An index will be included with the indexes covering documents microfilmed at Griesheim by JIOA that will be forwarded to Washington, D. C.

To collect the information for this report forty targets were visited in the British, American, French and Russian zones of occupation. They included producers and users of high temperature coal tar products, selling organizations, engineering companies, equipment manufacturers and research organizations. A list of the targets visited with the location of each follows this statement. While it is realized that there are other targets of each type that might have been included if time had permitted, it is reasonable to assume that the information received and conclusions reached from the targets actually visited can be extended to the German high temperature coal tar industry as a whole since they represent a major portion of it.

TARGETS VISITED

Name	Location
Gesellschaft für Teerverwertung	Duisburg-Meiderich
" " "	Castrop-Rauxel
" " "	Bochum-Gerthe
Rütgerswerke Aktiengesellschaft	Castrop-Rauxel
" "	Berlin
Chemische Fabrik Weyl (Rutgerswerke)	Mannheim-Waldhof
Gelsenkirchener Bergswerks A.G., Carolinenglück	Bochum
Gelsenkirchener Bergswerks A.G., Pluto-Wilhelm	Wanne-Eickel
Bergbau A.G., Ewald König Ludwig	Herten
Gutehoffnungshütte, Zeche Sterkrade	Oberhausen-Sterkrade
Teerdestillation Mathias Stinnes	Essen-Karnap
Hanseatische Teerproducten Fabrik, Haltermann & Co.	Hamburg
Aktiengesellschaft Johannes Jeserich	Hamburg
Binne und Sohn	Hamburg-Pinneberg
Nord Deutsche Kohlen und Cokes Werke A.G.	Hamburg-Freihafen
Dr F. Raschig G.m.b.H.	Ludwigshafen
" " " " , Zweiniederlassung	Bochum-Langendreer
Teerdestillation Stuttgart	Stuttgart
Ebano Asphalt-Werke Aktiengesellschaft	Hamburg-Harbourg
Emschergenossenschaft	Essen
Lothringen Bergbau Aktiengesellschaft	Bochum-Lothringen
Verkaufvereinigung für Teererzeugnisse	Castrop-Rauxel
Mannesmannrohrenwerke, Königen Elizabeth Mine	Essen-Frillendorf
Heinrich Koppers G.m.b.H.	Essen
Dr. C. Otto & Co.	Bochum-Dahlhausen
Firma Carl Still	Recklinghausen
Didier Werke A.G.	Berlin-Wilmersdorf
Schumachersche Fabrik	Bietingheim
Aktiengesellschaft der Kohlenwertstoff-Verbände	Bochum
Phenol Verband	Berlin
Verkaufsvereinigung für Teererzeugnisse	Berlin
I.G. Farbenindustrie	Ludwigshafen
" "	Uerdingen
" "	Schkopau
Chemische Werke Albert	Wiesbaden-Biebrich
Deutsche Gasrusswerke, G.m.b.H.	Dortmund
Ruhröl, G.m.b.H.	Bottrop
Ruhrchemie	Sterkrade-Holten
Gasinstitut der Technische Hochschule	Karlsruhe
Kaiser Wilhelm Institut für Kohlenforschung	Mülheim

PART I

RECENT ADVANCES IN GERMANY IN THE PROCESSING OF HIGH TEMPERATURE COAL TAR AND USE OF PRODUCTS OBTAINED THEREFROM

SUMMARY

In the years immediately preceding and during the last war, Germany's need for fuel oils, especially for the navy, motor fuels for Diesel and gasoline engines and briquette fuels for transportation and industrial heating purposes caused the old, and well established branch of the German coal tar industry, which deals with the processing of high temperature coal tar, to make an all-out effort to produce maximum quantities of fuel oils, oils and pitch for the hydrogenation plants, pitch for fuel briquettes and pitch and pitch coke for carbon electrodes. Such peacetime activities as the manufacture of creosote for wood preservation and refined tars for road construction were almost completely abandoned. Few changes in manufacturing procedures or equipment were made at the tar plants and such changes as were made resulted largely from the increased production of fuel oils, hard pitches and pitch coke brought about by the war.

In this summary advances in equipment, methods, products and uses of products that have taken place within the past ten years in the high temperature coal tar industry of Germany are enumerated and briefly discussed. Most of the items mentioned in this manner are discussed in greater detail in the individual target reports which follow this summary. In some cases they are discussed in greater detail in reports submitted by other investigators. In such cases reference is made to those reports wherever possible.

NEW OR IMPROVED TAR PLANT EQUIPMENT

TAR STILLS

Probably the most important improvement made in any of the German tar plants was the installation of a continuous tar still of somewhat novel design at the Duisburg Meiderich plant of Gesellschaft für Teerverwertung. Prior to the installation of that unit, this company, the largest tar distilling organization in Germany, had used only batch stills in all of its plants. In fact, tar distillation in practically all of the German plants is carried on in batch distilling equipment and the installation of the new still by Gesellschaft für Teerverwertung was more significant and noticeable on that account.

Tar Stills, General. The various types of batch stills used in Germany are illustrated in Figure 1. Vertical, pot type stills without columns, (Teerverwertung, Meiderich), predominate. Teerverwertung, Rauxel, the largest tar plant from the standpoint of volume of tar distilled, and others, use horizontal stills (without columns) heated through two large tubes. At least one plant, (Carolinenglück) uses horizontal stills like those at Rauxel connected to offset fractionating columns. Carolinengluck, and most of the other tar plants, use vertical stills with offset fractionating columns for the redistillation of oils. Only one company, Rütgerswerke, uses vertical stills with columns mounted directly on top. Most of its plants are provided with installations of that kind. All the different types of batch stills mentioned above are operated under vacuum, usually about 600-650 m.m. of mercury.

Continuous tar stills are used in only a few of the high temperature tar distilling plants in Germany. Installation of the Koppers, atmospheric type, illustrated schematically by Figure 2, are used by Nord Deutsche Kohlen und Cokes Werke A.G. at Hamburg and by Röchling'sche Eisen und Stahlwerke G.m.b.H. at Völklingen, Saar. The largest installation of this type is located at Mahrisch Ostrau in Tschechoslowakei. The Ludwigshafen plant of Dr. F. Raschig G.m.b.H. uses a Raschig, continuous, vacuum still arranged as shown in Figure 3. It is the only installation of this type in Germany.

For comparison with the above mentioned systems the arrangement of the new continuous, vacuum still of Gesellschaft für Teerverwertung at Duisburg-Meiderich is shown schematically in Figure 4.

G.f.T. Continuous Tar Still. In principle the G.f.T. still is similar to other continuous stills employing pipe heaters with fractionating columns operating in series but it differs from them principally in the use of vacuum rather than atmospheric pressure in the vaporizing and fractionating columns. Coal tar dehydrated in other equipment is heated in a pipe heater under pressure, released through a constricting cock into a cast iron, vacuum flash chamber where the pitch is separated from the vapors. The briquette pitch flows by gravity through a pitch-to-tar heat enchanger to a pitch receiver. The vapors pass from the flash chamber, successively, through three steel columns packed with Raschig rings, through a final condenser and through two scrubbers ahead of the vacuum pump. From the three columns and final condenser are discharged, by gravity, through barometric legs, anthracene oil, "Solvay" wash oil, naphthalene oil and carbolic oil.

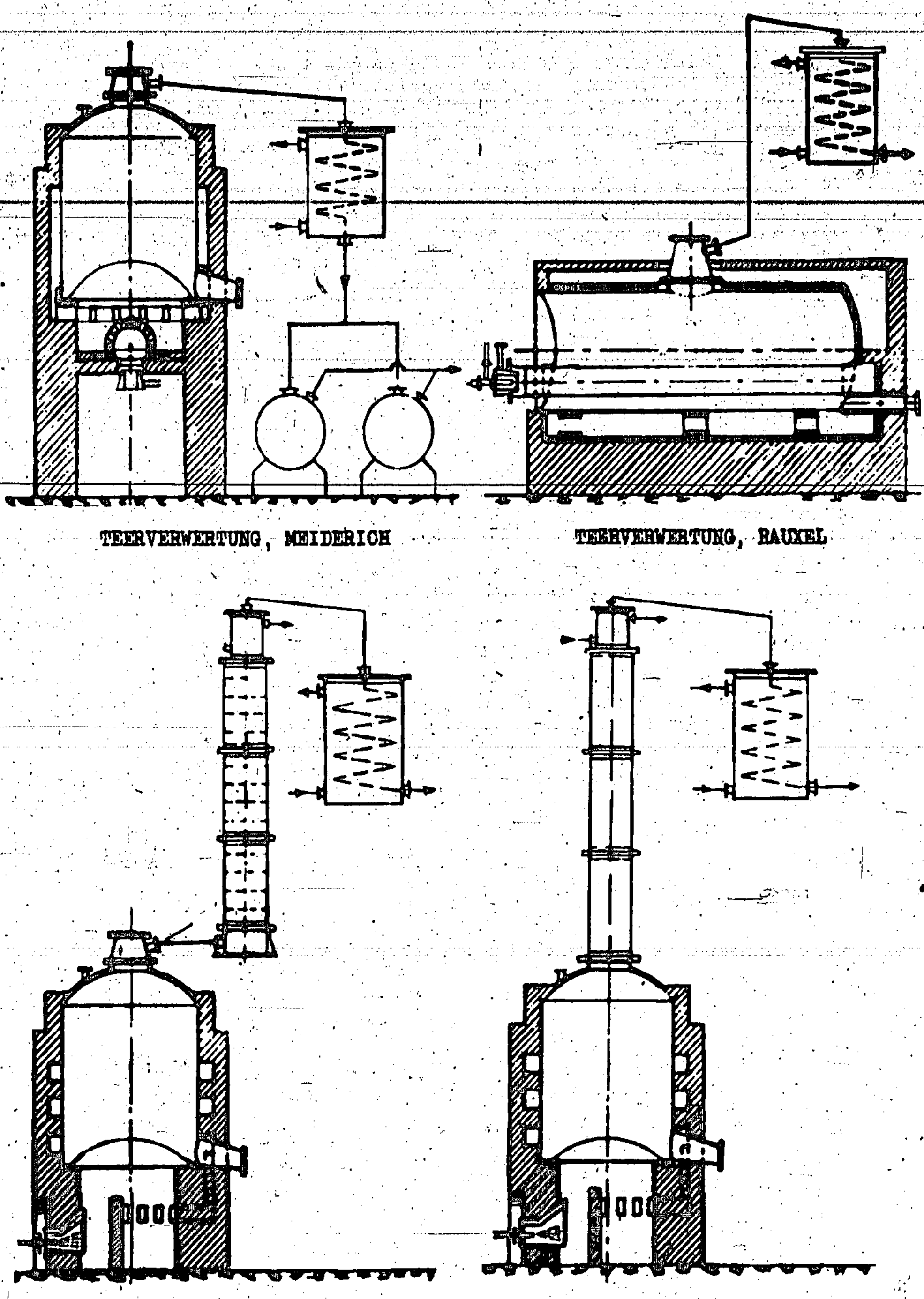


Fig. 1 - VACUUM-BATCH-SYSTEMS

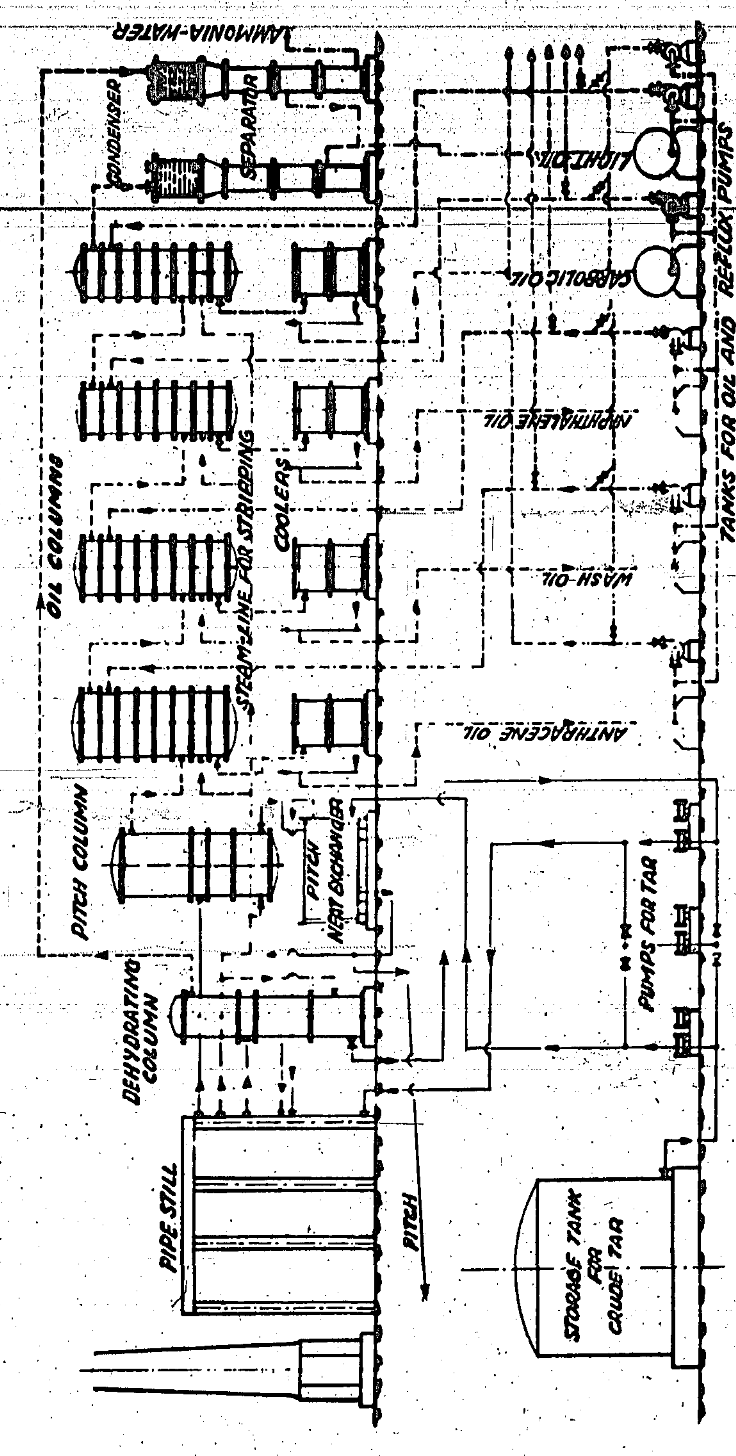


Fig. 2 - CONTINUOUS KOPPERS ATMOSPHERIC SYSTEM

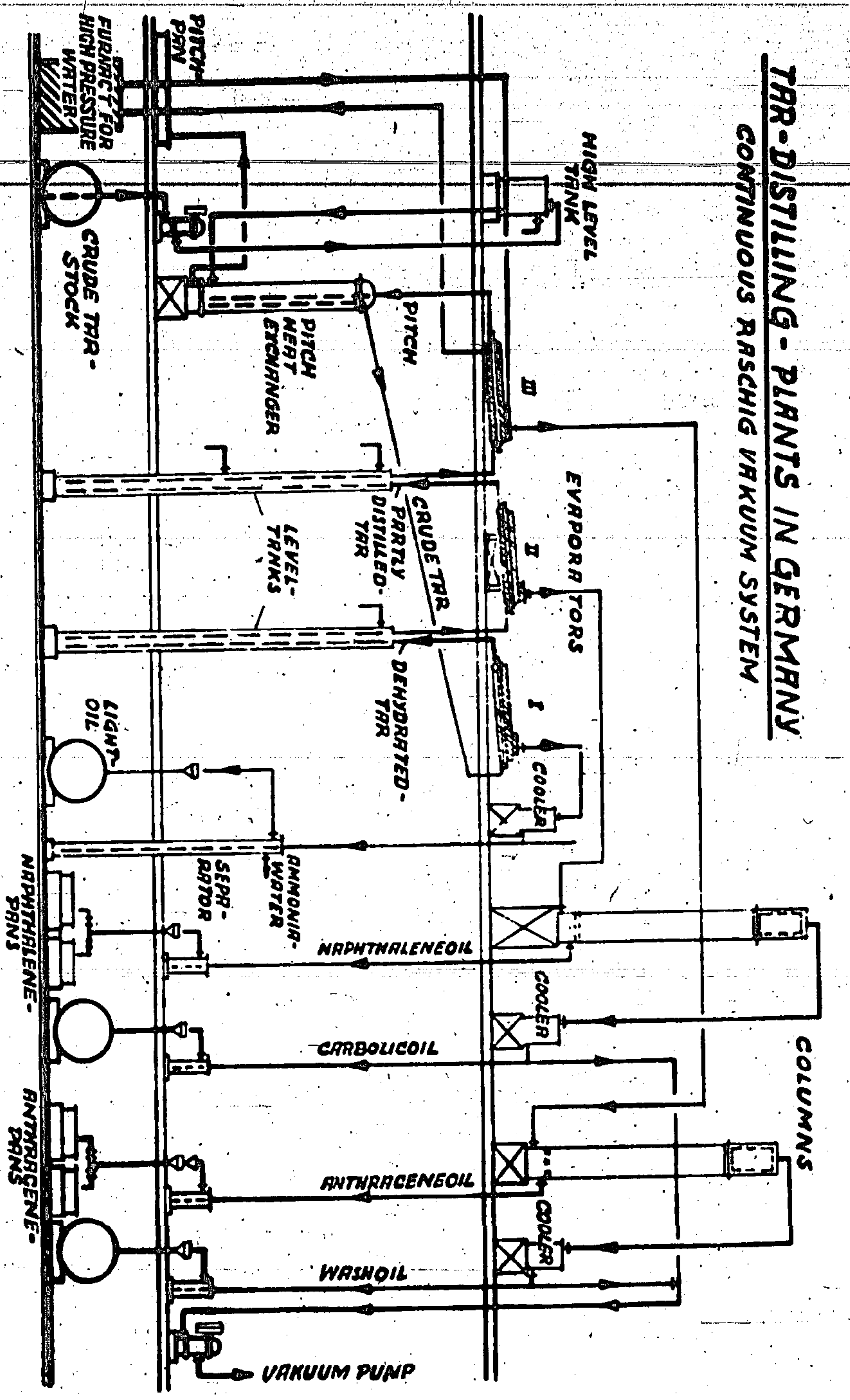


Fig. 3 - CONTINUOUS RASCHIG VAKUUM SYSTEM

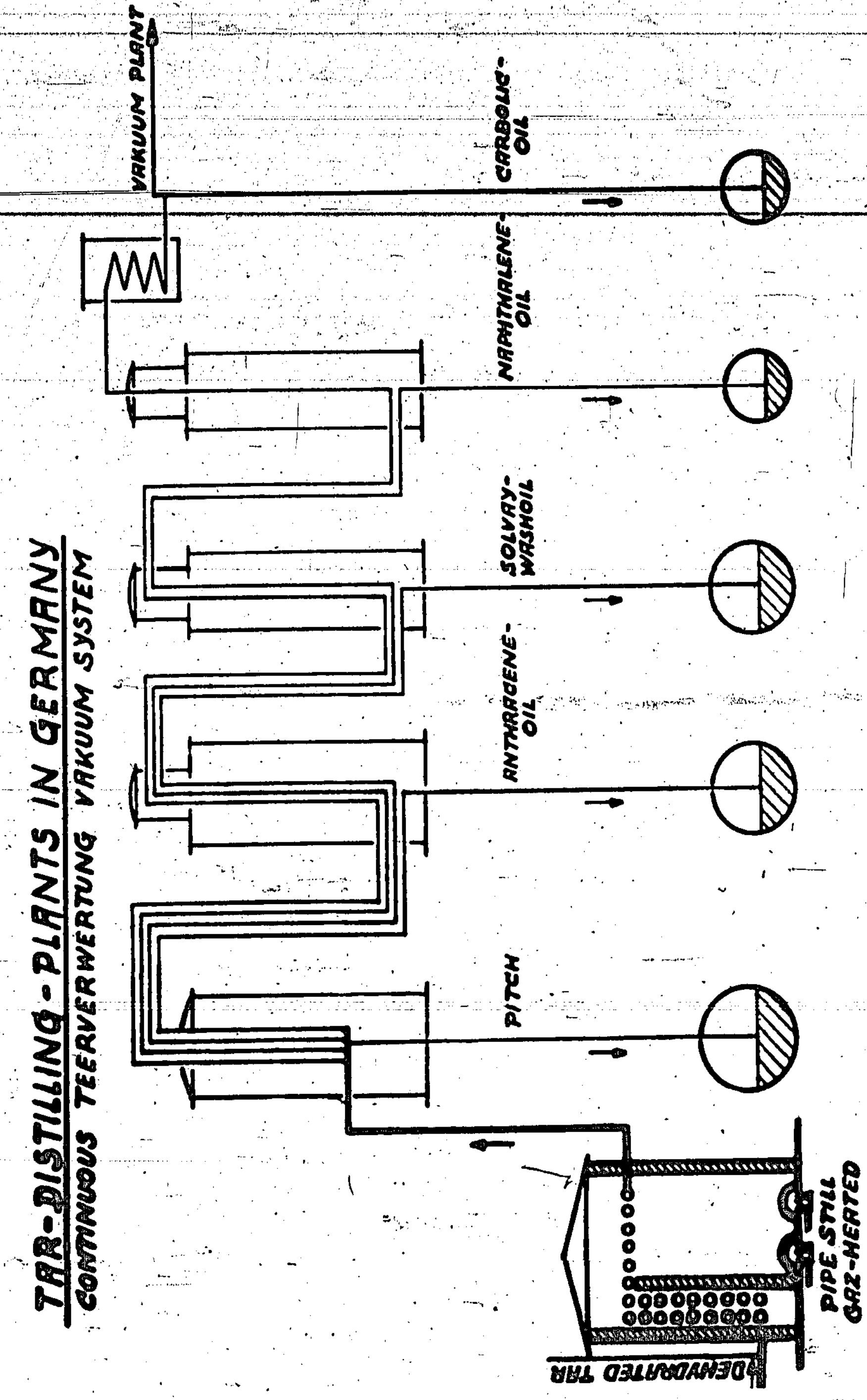


Fig. 4 - CONTINUOUS TEERVERWERTUNG VACUUM SYSTEM

In addition to the use of vacuum in the columns, other items of interest in this installation are: the location of the gas burners in the floor of the pipe heater instead of in the usual position at one side, a constricting or pressure reducing cock between the pipe heater and the flash chamber; iron Raschig rings in the upper part of the flash chamber to protect subsequent steel parts of the equipment against corrosion, a unique method for distributing the reflux from the dephlegmator onto the packing in each column using a distributing trough with slots in the sides to cause the reflux to fall onto the packing in two concentric and practically continuous circles.

Some of the advantages claimed for this new installation by Gesellschaft für Teerverwertung are:

1. A heat consumption below all presently known values.
2. An increased yield of oil (about 3-5%)
3. Control of all corrosion influences.
4. Avoidance of coke formation in the heating tubes.

HARD PITCH MANUFACTURE

During recent years, the use of hard pitch for pitch coke and plastic compounds created a demand for hard pitch in Germany which did not exist before to any considerable extent. To meet this demand several of the German tar plants installed special equipment for the purpose. Others, particularly Rütgerswerke and Pluto-Wilhelm, made the hard pitch in the stills ordinarily used for the production of briquette pitch.

Gesellschaft für Teerverwertung, Duisburg - Meiderich. At Castrop-Rauxel, Gesellschaft für Teerverwertung installed seven, vertical, 50 ton stills for hard pitch manufacture. They were gas heated, operated under vacuum and provided with pipes running to the bottom of each still for the introduction of air. Briquette pitch made in other stills was transferred to the hard pitch stills where distillation was continued under vacuum and with agitation by air drawn into the stills by vacuum. When the desired softening point of 150°C was reached, at which time the temperature of the pitch was 380-390°C, the pitch was dropped into transfer tanks. From the latter it was transferred by suction into insulated tank cars which transported it in liquid form to the pitch coke plant.

Ewald-König Ludwig. Bergbau A.G., Ewald-König Ludwig installed two, 3-ton, vertical, gas fired stills especially for hard pitch manufacture. Also an older, 50 ton still was sometimes

used for the same purpose. Briquette pitch made in horizontal stills was transferred to the hard pitch stills and blown with combustion gases heated by a direct flame. The combustion gases were introduced into the still under 4-5 atmospheres pressure through a distributing pipe. On occasions tar was distilled to briquette pitch with vacuum in the 50 ton vertical still and then the distillation was carried to hard pitch in the same still in the manner described above.

Gesellschaft für Teerverwertung, Bochum-Gerthe. For several years Gesellschaft für Teerverwertung at Bochum-Gerthe has used a method of hard pitch manufacture similar to the one described above. Hot combustion gases entered a vertical combustion chamber into which gas was introduced without additional air. Combustion of the gas with the oxygen contained in the combustion gas rendered the latter oxygen-free and at the same time raised its temperature to 600°C. The hot combustion gases were then drawn by suction through hot briquette pitch contained in two, 50 ton stills until the temperature of the pitch was about 380°C and softening point about 150°C. The daily capacity of each still was about 25 tons of hard pitch.

Rütgerswerke A.G., Castrop-Rauxel. At Rauxel, Rütgerswerke A.G. made hard pitch from briquette pitch in the same stills in which the briquette pitch was produced. Continued heating with vacuum and steam but without air blowing was employed. However, Rütgerswerke has a special installation at this plant for producing extra hard pitch used for making Plastix. The installation consists of an oxidizing still of 100 tons capacity, heated with gas by large ring burners located under the concave bottom of the still. Both air and steam can be introduced into the still through distributors provided for that purpose. Vapors from the still are condensed by a water cooled condenser.

A charge of 70 to 80 tons of molten briquette pitch is transferred to this equipment from the tar stills. It is then heated by the ring burners to 290-300°C. Air blowing is started and some steam is admitted at the same time. The quantity of air used for blowing the pitch is about 400 to 600 cubic meters per hour. It not only serves to oxidize the briquette pitch but also provides intense agitation of the entire contents of the still. Throughout the blowing period the temperature is kept at 350°C or above by means of the ring burners. The desired softening point of 180° to 200°C is reached in about 10-14 hours. The amount of distillate removed is about 5 to 10 percent. To make Plastix, the purpose of which is explained in a later portion of this Summary, the high softening point pitch is fluxed with filtered, high boiling tar oils to the desired final softening point, usually between 38° and 60°C.

Chemische Betriebe Pluto - Wilhelm. This Company made hard pitch for use in the manufacture of Plastix but did not employ special equipment for the purpose. Distillation was carried all the way to hard pitch in vertical stills with heat and vacuum only.

PITCH COOLING EQUIPMENT

Due primarily to the production of high softening point pitch in Germany for the manufacture of pitch coke, considerable attention has been given during the past few years to the development of improved methods of cooling pitch. Special equipment was installed at some of the tar plants for this purpose

Prior to 1938 very little hard pitch was made in Germany and the briquette pitch, the principal residual product of the tar industry, was cooled in open bays, in stationary steel pans usually mounted above railroad tracks, or in split molds. In at least two plants steel pans were used which could be tilted to dump the contents into cars or car loading equipment. All of these methods were intermittent and the tar distillers wished to use continuous methods - especially for the cooling of high softening point pitch. Different methods that were developed for this purpose are described in the following paragraphs.

Gelsenkirchen Mining Co. Carolinenglück Plant. One of the most interesting methods of cooling pitch was developed at the Carolinenglück plant of the Gelsenkirchen mining company. The equipment used in this process consisted of a horizontal tank, with valve actuated openings on the bottom, mounted above an open rectangular tank with a hopper bottom. A screw conveyor was mounted in a trough in the lower portion of the hopper bottom of the rectangular tank. A bucket elevator with perforated buckets was mounted in an inclined position on one end of the rectangular tank. This equipment was used in the following manner: Pitch having a softening point above 160°C was cooled in an overhead tank until its temperature was below 300°C. It was then run by gravity into the horizontal tank equipped with valve actuated openings and by means of the latter was dropped in small streams into water contained in the rectangular tank. The streams of pitch solidified while dropping through the water and broke into small rod-like pieces when they reached the screw conveyor. The pieces of solid pitch were then moved to the end of the tank by the screw conveyor and delivered into the bucket elevator. By means of the latter they were carried above the level of the water in the tank and discharged into a railway car. This method was

said by the inventor Mr. Georg. Speckhardt of Carolinenglück to be satisfactory for the cooling of brrquette pitch as well as hard pitch. However, when cooling briquette pitch the temperature before entering the water should be not higher than 150°C. German patent No. 743678 covering this process was issued to Mr. Speckhardt on May 12th, 1943.

Bergbau A.G. Ewald - König Ludwig. Another interesting pitch cooling device was installed at the Ewald-König Ludwig plant of Bergbau A.G. at Herten near Recklinghausen. It consisted of a rotating steel drum or cylinder about twenty feet long and two feet in diameter. The surface of the drum was divided into shallow troughs about four inches wide and one half inch deep by narrow ribs spaced about four inches apart. On one side of the drum, a short distance below the top, a water distributing manifold sprayed water onto the surface between the ribs which comprised the side walls of the shallow troughs. A pitch distributing manifold flowed liquid, high softening point pitch onto the films of water in the troughs. The films of pitch were cooled by the underlying films of water and by overlying air as the rotating drum carried the pitch films over the top and down the other side. While the pitch films were moving downward with the rotation of the cylinder, scrapers removed them from the drum and discharged them into sloping metal troughs in the bottoms of which water was running. The pitch films, which were plastic when leaving the drum, were further cooled by the water in the sloping troughs and conveyed by it to a grating made of steel rods, set close together, which separated the water from the solidified pitch. The water fell through the grating into a tank from which it was returned to the drum and the solidified pitch fell onto a conveyor which transported it to a car. The pitch arrived at the car in the form of small, thin plates. This installation was said to have had a capacity of 20 tons of high softening point pitch per hour. So far it has been used only for the cooling of high softening point pitch but tests are said to have indicated that it could be used for briquette pitch as well by making only minor changes.

G.f.T., Carolinenglück and Ruhröl G.m.b.H. Endless, inclined conveyors were used by Gesellschaft für Teerverwertung at Rauxel and Gerthe, and by the Carolinenglück tar distilling plant, for cooling high softening point pitch. Also the same type of device was used by Ruhröl A.G. for cooling a pitch residue (softening point 200°C) from the distillation of filtered coal extract. These inclined conveyors were made of overlapping steel plates. The liquid pitch was flowed onto the conveyor at its lower end and solidified, with or without the aid of water sprays, as the conveyor moved to the upper end of the incline. There the pitch discharged, in the form of thin plates, into a railway car. The conveyor then returned to the lower end of the incline .

In the case of Ruhröl installation, the steel conveyor passed through water to completely cool the conveyor plates before more of the molten pitch was applied.

Two conveyors of this type were used by Gesellschaft für Teerverwertung at Rauxel. The capacity of one was 50 tons (metric) per day and the capacity of the other was 200 tons per day. At the Gerthe plant of the same company the capacity of the inclined conveyor was 50 tons per day. Pitch coolers of this type were not satisfactory for the cooling of briquette pitch and when used on high softening point pitch they were said to require excessive maintenance.

Rütgerswerke A.G., Castrop-Rauxel and Gutehoffnungshütte. Rütgerswerke A.G. at Rauxel tried various methods of cooling pitch. One method, used only for the cooling of high softening point pitch, consisted in flowing the liquid pitch in thin streams into water flowing in inclined shallow troughs. The streams of pitch were solidified into rods by the water and carried to the lower ends of the troughs where they were separated from the water by a grating made of iron bars set close together. From the grating the rods of pitch dropped into a railway car and the water dropped through the grating into a tank from which it was returned to the upper ends of the inclined troughs. The capacity of the Rauxel installation was said to be 170 to 240 tons of pitch per day.

Another installation of the same type was used at the Gutehoffnungshütte tar plant. At this installation the high softening point pitch to be cooled was pre-cooled to about 250°C. It then entered a horizontal manifold 80 mm in diameter and emerged from the bottom of the manifold through 25 nipples with 10-12 mm openings.

The streams of pitch dropped from the nipples onto water flowing in 25 troughs each 7 m long and about 100 m.m. wide. The temperature of the water was about 40°C. The water and pitch were separated at the ends of the troughs by a grating which discharged the rods of pitch into a railway car and the water into a recirculating tank. The capacity of this installation was said to be about 100 tons per day of hard pitch. The quantity of water recirculated was 25 to 30 cubic meters per hour.

Another system of pitch cooling was being developed by Rütgerswerke at Rauxel, especially for the cooling of briquette pitch. Insufficient tests were made to establish its capacity or suitability for this purpose. In this system molten briquette pitch flowed from a wide, flat nozzle onto the surface of water flowing in a slightly inclined trough 60 meters long and about

25 centimeters wide. The thin ribbon of pitch leaving the end of the nozzle floated on the surface of the water for several meters. It was cooled by the water from below and air from above to the point where no water would be occluded when the pitch became submerged. On reaching the end of the 60 meter inclined trough the pitch and water were separated by an inclined grating which discharged the pitch into a railway car and the water into a recirculating tank.

The two methods of cooling pitch, described above, were patented in Germany by Rütgerswerke A.G. in 1940 and 1943 respectively.

ANTHRACENE RECOVERY EQUIPMENT

During the war most of the German tar plants recovered a product called "anthracene rückstand" or anthracene residue by the crystallization and filtration of anthracene oil. In the past it has been customary to cool the anthracene oil in shallow pans with occasional stirring by hand and then filter the crystalline slurry through shallow vacuum filter pans. The anthracene residue was removed from the vacuum pans by hand. In some plants centrifuges were used in place of or in addition to the vacuum filters and sometimes plate and frame filter presses were used. In each of these cases the anthracene residue when shipped to carbon black factories contained appreciable quantities of anthracene oil. Because the latter was in great demand for hydrogenation, fuel oil and fluxing purposes and because it had a higher sales value than the anthracene residue, attempts were made to lower the oil content of the anthracene residue to a minimum. Special equipment for this purpose was installed by Gesellschaft für Teerverwertung at Meiderich in the following manner:

Gesellschaft für Teerverwertung, Meiderich. Twenty water cooled, vertical crystallizers are mounted above five, vertical basket centrifuges arranged to discharge the anthracene residue onto conveyors which transfer it directly into open cars.

Each crystallizer is charged with 12 cubic meters of anthracene oil at 80°C. Cooling is then effected by water, from the canal, circulated through jackets surrounding each crystallizer. The oil is agitated continuously by a stirrer having vertical paddles close to the side walls of the crystallizers. The agitators are driven by overhead motors. Cooling to approximately 15°C requires about one and one half days per charge. The crystalline slurry is dropped by gravity into a centrifuge and the cake is whizzed for five to fifteen minutes depending upon the quality of the anthracene oil charged to the crystallizer. The baskets in the centrifuges are two meters in diameter. They are made of phosphor bronze. Normally the capacity of each centrifuge is about 80 tons of slurry per 24 hours. Anthracene residue produced in this manner contains only 2-3% of anthracene oil. The anthracene content is 20-25%. The centrifuged oil remains crystal free at 0°C.

TAR ACID EXTRACTING EQUIPMENT

Only a few of the German tar plants are equipped for tar acid refining. During the war those plants received the crude tar acids from all of the others in the form of tar acid oils, crude tar acids or phenolates and processed them to recover refined phenol and cresols. At most of the tar plants where tar acids were extracted simple, batch extracting equipment was used. However at the Ludwigshafen plant of Dr. F. Raschig G.m.b.H. counter current extraction in a tower packed with Raschig rings, started several years before, was continued and a new method was developed at the Carolinenglück plant of the Gelsenkirchen Mining Company.

Carolinenglück. The method developed by Carolinenglück, patented in 1943 by Georg Speckhardt, involved the use of two cone bottom tanks of 20 m.m capacity each with pipes running to the bottoms of the cones and terminating in sprinkler heads with holes not larger than 10 m.m. To use this equipment each cone bottom tank was partially filled with 20% caustic soda solution. The tar acid oil was then pumped slowly into one of the tanks through the distributor, located near the bottom of the cone, and the oil, after bubbling up through the caustic soda solution, flowed out of the tank and was pumped into the second tank where the process was repeated. When the caustic solution in the first tank became saturated with tar acids the process was stopped, the phenolate was withdrawn from the tank and replaced by fresh caustic soda solution. The flow of the oil was then reversed, passing first through the tank containing partially saturated caustic and then through the tank containing fresh caustic solution. If preferred, one cone bottom tank instead of two may be used for carrying out this process.

According to Mr. Speckhardt the use of the above described method of removing phenols from tar acid oils tends to avoid emulsion difficulties which are sometimes uncountered when counter current flow of both caustic and oil is employed.

NEW OR INCREASED USES OF PITCHES AND TAR OILS

ELECTRODE COKE MANUFACTURE

Lothringen-Bergbau Aktiengesellschaft. One of Germany's greatest war needs was pure carbon for electrodes used in the production of aluminium. As early as 1936 a plant had been installed at Bochum-Lothringen by Lothringen Bergbau Aktiengesellschaft to make electrode coke from hard pitch (150°C K.S.). The plant consisted of 65 refractory, gas heated, sole flue ovens, 1.5 m wide by 10 m long, a mechanical pusher, a quenching and cooling platform, level with the oven floors, and three vertical stills for the redistillation of pitch distillate.

The operation of this plant was carried out as follows: 14 tons of crushed or flaked hard pitch was charged into an oven from the top and the pitch was distilled by heat from sole flues supplemented by combustion inside the ovens, of gas from the pitch coke battery during the latter part of the coking cycle. It was necessary to supplement the heat from the sole flues in this manner in order to reduce the volatile matter of the coke below 1.0 percent. At the end of the coking cycle the luted doors were raised and the coke was pushed onto the cooling and quenching platform where it was quenched with a minimum amount of water, applied by hand. It was then loaded by hand into cars.

Distillate from the ovens, amounting to about 15% of the charge, was redistilled to briquette pitch in vertical stills with vacuum and gas heating. The briquette pitch was then transferred to another vertical still where it was blown with hot inert gases until a softening point of 150°C was reached. The hard pitch so made was then added to hard pitch from the tar plants and charged into the pitch coke ovens. The distillate produced during the blowing operation went to hydrogenation plants. The inert gas used for blowing the pitch distillate to hard pitch was made by burning a small amount of coke oven gas in hot, stack gases from the pitch coke plant. This was done in a small vertical combustion chamber located beside the blowing stills.

The capacity of the Lothringen pitch coke plant was about 200 tons of pitch coke per day. It operated throughout the war and suffered only minor damages from air raids.

Verkaufsvereinigung für Teererzeugnisse, Rauxel. In 1939 a new pitch coke plant was built to supplement the output of

The Lothringen plant. The new plant was erected for Verkaufsvereinigung für Teererzeugnisse at Castrop-Rauxel near the tar distilling plant of Gesellschaft für Teerverwertung. The latter concern operated the pitch coke plant and supplied much of the hard pitch used in its operation. The hard pitch was made by blowing briquette pitch with air in the manner previously described.

The Rauxel pitch coke plant consisted of 20 silica lined, Koppers, regenerative by product ovens separated into four blocks of five ovens each by concrete pinion walls. The ovens were 11,800 m.m long, 3,000 m.m. high and 450 m.m. wide (average width). The ovens were charged with liquid, high softening point pitch, (150°C) from 6 gas heated, vertical charging tanks with internal circulating pumps. One charging tank was used for each block of five ovens and two were held as spares. The liquid pitch was circulated by the pumps in the charging tanks, through pipe loops running over the ovens and back to the charging tanks. To charge an oven, pitch flowed from the circulating loop through a nozzle inserted through one of the charging hole covers.

The charging of an oven with 9 (metric) tons of pitch required 3 hours. During a part of this charging period a mixture of preheated oven gas and superheated steam was introduced into the top of the oven to minimize cracking of the pitch gas and vapors. The overall oven time for one charge of pitch was about 22 hours. The coke was pushed from the oven into a quencher car and was discharged from the latter onto a loading wharf. To avoid the use of an excessive amount of water in the quenching operation, a cover on the quencher car was lowered into position after the necessary amount of water had been applied to the coke. Quenching was then effected principally by the steam formed by the evaporation of the quenching water.

The vapors from the ovens passed through electrical precipitators, indirect coolers and direct coolers to remove the pitch distillate. The non-condensable gas was pumped to gas holders. The pitch distillate was shipped to Hamburg for the manufacture of a road binder (Ebano Spezialbindemittel) and the gas was used for the heating and purging of the ovens, for prewarming the oven doors, heating the charging tanks and for the generation of steam for the pitch coke plant.

In 1943 a second battery of 20 ovens was installed exactly like and in line with the first battery. Operation of the new battery was started in November of that year. The primary purpose of this second battery was not to double the capacity of the plant but to insure uninterrupted operation at the full capacity of one battery if part or all of the capacity of either battery should be lost for any reason.

During the period between December 1939 and January 1st, 1945, this plant produced 342,000 tons of pitch coke. The pitch coke plant suffered considerable damage by bombing. One block of five ovens received a direct hit and during the last month of the war the equipment was heavily damaged by artillery.

<u>Ruhröl G.m.b.H. Bottrop - Welheim</u>. So great was Germany's need for pure carbon for electrode manufacture that other sources, in addition to the two pitch coke plants described above, were sought. At the Bottrop-Welheim plant of Ruhröl G.m.b.H. a process was employed which produced electrode coke having an unusually low ash content (0.10 to 0.15 %). It commanded a somewhat higher price than the cokes from other sources on this account.

The process discussed at length by Lowry and Rose in C.I.O.S. Report Item No. 30 File No. XXXI-27 consisted in (1) digesting bituminous coal at about 400°C under 100-150 atmospheres pressure in middle oil from the liquid phase hydrogenation of a 70/30 mixture of briquette pitch and coal tar oil, (2) filtering the digestion product through special ceramic filters at about 150°C and under 4 to 6 atmospheres pressure to remove ash and carbonaceous material not dissolved by the extracting medium, (3) distillation of the filtrate to obtain the coal extract (softening point 210-220°C) and to obtain a distillate for gas phase hydrogenation and reuse as extracting medium, (4) cooling of the coal extract on an endless, steel conveyor and (5) carbonization of the solid coal extract in by product coke ovens to obtain low ash coke of suitable quality for carbon electrode manufacture.

<u>Zeche Carl Alexander, Baesweiler, near Alsdorf</u>. Another source of material for the production of carbon electrodes was the Carl Alexander plant at Baesweiler where specially selected bituminous coking coal of low ash content, after screening and preflotation, was subjected to four successive flotations using beechwood tar oil as the flotation reagent. The final froth from the flotation process went to carbon lined, concrete tanks where it was extracted with a solution containing 100 parts water,

2 parts hydrochloric acid and 1 part hydrofluoric acid. The ratio of acid solution to coal in the extraction process was 2 : 1. The ash content of the coal was reduced in this manner to 0.5%. After extracted coal had been recovered from the acid solution by filtration, washed with water, again filtered, and dried it was carbonized in by product coke ovens without the admixture of pitch or other binding materials.

The amount of clean coal made at this plant for electrode coke production was about 6000 tons per month. The process has been described in greater detail in FIAT Final Report No. 634, page 42 by Fraser and Driessen.

Mannesmannrohrenwerke, Zeche Königen Elizabeth, Essen. Another coal cleaning process for making raw material suitable for carbon electrode coke manufacture was under development at the Königin Elisabeth plant of Mannesmannrohrenwerke A.G. near Essen. It also has been described by Fraser and Driessen in FIAT Final Report No. 634, page 48. This process consisted in the picking, crushing and dedusting of bituminous coal from the Finefrau seam followed by laminar separation (7½ tons per hour) in a Vogel-Laminarström washer using a suspension of magnetite in water as the separating medium. The cleanest coal from this operation (5-6 tons per hour) contained about 1.2% ash. After rinsing on a horizontal Schieferstein, resonance screen and recovery of the suspension medium by magnetic separation, the coal was milled in a ball mill in closed circuit with a rake classifier and the fines from this operation went to flotation cells, the flotation froth was broken and dewatered and the "purest coal" was shipped to coke plants where it was mixed with 30% hard pitch (150°C) and carbonized in by product coke ovens. The ash content of the "purest coal" was about 0.5-0.7%.

Another system of cleaning coal from the Finefrau seam for use in carbon electrode manufacture was under development at Königin Elisabeth. It involved the use of a Büttner dryer and electrostatic separation. The equipment was not ready for trial before the war ended.

SPECIAL PITCH COMPOUNDS

Interest in special pitch compounds of the type called Plastix increased somewhat during the war. This was due in part to the almost complete lack of asphalt (bitumen) for those purposes for which it had previously been employed such as paving, roofing, flooring, waterproofing, insulating and pipe line protection.

Plastix were made by fluxing coal tar pitches having softening points above 100°C (K.S.) with high boiling distillates or combinations of distillates such as filtered anthracene oil, oils recovered in distilling briquette pitch to hard pitch and oil of the type used for the recovery of light oils at coke plants (wash oil). These hard pitch- heavy oil mixture have better temperature-viscosity relationships than straight distilled coal tar pitches and are, therefore, better suited to those uses which require resistence to flow at elevated atmospheric temperatures or less brittleness at low temperatures. This characteristic is indicated, in German practice, by the spread between the softening point (Kramer-Sarnow) and the breaking point as determined by the Fraas test. According to one manufacturer of Plastix the spread between softening point and breaking point should be at least 40°C.

Rutgerswerke A.G. - Rauxel. The method used by Rütgerswerke A.G. for producing Plastix has been mentioned above under hard pitch production. Briefly it consists in charging 70-80 tons of soft pitch into the special Plastix making equipment, heating the pitch to 290-300°C, then blowing with approximately 400-600cu.m. of air per hour and a small amount of steam with continued heating until the desired softening point of 180-200°C is reached. At that time the temperature of the pitch should be at least 350°C. The time required for the blowing operation is about 10-14 hours. The high softening point pitch is fluxed to the desired final softening point, generally between 38 and 60°C with filtered anthracene oil or, preferably, with a mixture of oils containing about 70% of anthracene oil having a distilling range of 350-450°C and free from crystalline solids and 30% of wash oil with a distilling range of 250-320°C, also free from crystalline material. For certain grades of Plastix such a mixture of oils and the high softening point pitch are mixed in approximately equal quantities.

Pluto-Wilhelm, Wanne-Eickel. To a considerable extent Chemical Works Pluto-Wilhelm of the Gelsenkirchen Mining Company specializes in the making of special pitch compounds of the type called Plastix. Coal tar is distilled, with vacuum, to softening points above 100°C. The hard pitch is fluxed with a mixture of heavy anthracene oil distilling about 270°C and the fraction of oil recovered from tar on distilling briquette pitch to hard pitch. Various proportions of pitch and oil mixtures are used for different purposes. A base containing 56% hard pitch and 44% of the oil mixture is used in making filled compounds in which the filler is stone dust, talc, asbestos fines, fly ash or slag wool. The same base also is fluxed with volatile solvents for cold application purposes.

Gesellschaft für Teerverwertung, Rauxel. At Rauxel, Gesellschaft für Teererzeugnisse made Plastix by fluxing hard pitch with hard pitch distillate in a 36 cubic meter, steam heated tank using air for agitation.

Some other German tar plants in addition to those mentioned above, make pitch compounds of the Plastix type for roofing, waterproofing, pipe coating, joint filling and other purposes for which pitches having improved thermal characteristics are desired.

ROAD TARS

The construction of new roads in Germany was almost completely abandoned during the war. Such road tars as were made principally for maintenance purposes, were mostly of the types developed and standardized in pre-war years. However, a special type of road tar was made by one company in substantial quantities as the result of circumstences brought about by war conditions.

Ebano Asphalt Werke Aktiengesellschaft. Ebano Asphalt-Werke Aktiengesellschaft at Hamburg-Harburg, when it could no longer supply Asphalt for paving purposes, was asked by the German Government to make a compound suitable for such use from stocks of briquette pitch, hard pitch distillate and pitch coke distillate that were available at the time. The hard pitch distillate was the oil produced in distilling briquette pitch to hard pitch and the pitch coke distillate came from the Rauxel pitch coke plant.

A mixture, called "Ebano Spezialbindemittel" was made which, at first contained no asphalt, Later asphalt from oil distillation in Roumania was added when supplies of that material became available. Following are the percentage compositions of typical mixtures prepared in 1941, 42, 43 and 44. The quantities made are also given in the table.

	1941	1942	1943	1944
Hard pitch distillate	35.1	36.2	10.3	26.5
Pitch coke distillate	32.2	39.3	29.4	11.9
Soft pitch, 40-50°	16.6	4.5	16.1	17.5
Briquette pitch, 65-70°	7.9	9.5	14.2	17.5
Low temperature tar from Silesia	4.5	7.4	2.6	-
Tar acid free tar oil	3.7	3.1	6.6	-
Roumanian asphalt	-	-	18.3	15.5
Crude coal tar	-	-	-	3.1
Slopol	-	-	2.5	-
Anthracene oil	-	-	-	8.0
Tons produced:	52,256	59,931	56,080	27,054

The equipment used for making Ebano Spezialbindemittel consisted of two tanks of 1000 tons capacity each, connected through a pump and equipped with air agitating coils. After one of the tanks had been filled with pitch or a pitch-asphalt mixture and the other with the mixture ofoils and tars the contents of the two tanks were mixed by circulation with the pump and by air agitation.

During 1943 and 1944 an emulsion was made from Spezialbindemittel by mixing 35% of it with 50-55% water and 10-15% of a mixture containing 1/3 clay similar to bentonite and 2/3 common clay in horizontal, mechanical mixers. Camouflage paint was made from the emulsion by mixing 40% emulsion with 20-30% mineral pigment and 30-40% water. The production of Spezialbindemittel ended abruptly in 1944 when the plant of Ebano Asphalt Werke was heavily damaged by air raids. The equipment for making Spezialbindemittel was completely destroyed.

CARBON BLACK

The much publicized carbon black industry of Germany which produced blacks for the rubber industry during the war was, to a large extent, made possible by the fact that large quantities of crystal free, high boiling tar oils were needed for fuel purposes and smaller quantities for the fluxing of the special compounds such as Plastix and Spezialbindemittel that have been discussed above.

The usual practice in German tar plants, when distilling coal tar, is to collect anthracene oil as a separate fraction or in two separate fractions, light and heavy. On cooling to atmospheric temperature a mixture of crystalline solids settles out. When the oil is to be used as fuel by the Navy or others, especially in combination with other liquid fuels, such separation of solids is troublesome. The crystalline solids also make the oil less suitable for fluxing pitch compounds and road tars so most German tar plants crystallize the anthracene oil and filter or centrifuge it to remove the mixture of solids which they call "anthracene-rückstand" or anthracene residue. The equipment used by Gesellschaft für Teerverwertung for this purpose has been described.

Refined anthracene can be obtained from anthracene residue but the market for refined anthracene in Germany was not large enough to absorb all of the residue. Having no other outlets for it the tar distillers sold it to the carbon black plants which considered it to be the most suitable raw material for their purpose chiefly on account of its comparatively low price. Anthracene oil could be used interchangeably with anthracene residue at the carbon black plants but cost 12.0 RM per 100 kilos as compared with 5.4 RM for anthracene residue. Naphthalene, in addition to costing 9.0 RM for the 76° grade and 11.5 RM per 100 kilos for the hot pressed grade (78°) required higher pressure steam (20-25 atmospheres) than anthracene residue and also required different combustion conditions. Because of these two requirements, naphthalene could not be used at the largest carbon black plant, located at Dortmund, but it was used at the original and smaller plant at Kalscheuren near Cologne.

The process of making carbon black from coal tar hydrocarbons was developed before the war by Deutsche Gold und Silber Scheide Anstalt at Frankfurt/Main. Plants were installed at Kalscheuren, near Cologne, at Dortmund-Ruhr and at Gleiwitz in Silesia. At the Dortmund plant (Deutsche Gasrusswerke G.m.b.H.) maximum production was reached in 1942. The plant was used to the extent of 90% of its capacity in that year and produced 14,063 tons of carbon black.

Deutsche Gasrusswerke (G.m.b.H.). The process, as operated at Dortmund was as follows: Anthracene residue, received from tar distillation plants in open cars, was unloaded into a storage bin from which it was charged into steam heated melting kettles by means of a clam shell bucket. The bucket could be moved to any part of the bin and to the feed chute of any melting kettle. The molten anthracene residue was transferred to vertical storage tanks from which it flowed to vaporizers. Each vaporizing unit was equipped with a device for metering and feeding the proper quantity of molten anthracene residue into the vaporizer. Vaporization was effected in the presence of preheated coke oven gas by steam coils. The mixture of hydrocarbon vapors and coke oven gas passed through gas heated pipes to the carbon black machines. In the latter the vapor-gas mixture was burned with a smoky flame by thirty burner tips located in a row below a steel cylinder through which hot water was circulated. The cylinder was 540 m.m. in diameter and 5000 m.m. long. Carbon black deposited on the rotating cylinder (1 r.p.m.) was removed by scrapers and dropped into a screw conveyor which transported it to other conveyors running to storage bins. The combustion gases leaving the machine passed through ducts to woolen filter bags which recovered the carbon black that had not deposited on the rotating cylinder. The black from the cylinder was classed as first quality and that from the filter bags as second quality.

LUBRICATING OILS AND GREASES

Pluto-Wilhelm, Wanne-Eickel. High boiling tar oils similar to those used by Chemical Works Pluto-Wilhelm for the making of Plastix also were used by that concern for making lubricating oils and greases. The Pluto-Wilhelm plant has a large and well-equipped department for lubricating oil and grease manufacture. Before the war petroleum products were purchased for use in the various formulations. During the war, when petroleum products were less available, tar oils were used in their place in some of the lubricating materials. Medium heavy anthracene oil 270° to 300°C and heavy anthracene oil 300° to 360-400°C were used directly in greases. For lubricating oils a mixture of medium heavy and heavy anthracene oil was re-distilled to remove oils distilling below 300°C and the residue from the distillation was crystallized and vacuum-filtered to remove any remaining portion of the "anthrasen rüchstand." Three kinds of tar-grease oil were prepared:

Tar Grease Oil	I	- 1.2 to 1.6	degrees Engler at 50°C
" " "	II	- 1.6 to 2.2	" " " "
" " "	III	- 2.75 to 3.2	" " " "

Lubricants were made by dissolving vegetable or animal fats or fatty acids in tar oils or mixtures of tar oils and mineral oils in steam heated kettles. The solution so prepared was then boiled with milk of lime to form a soap which was diluted with mineral oils or tar oils and, if necessary, other raw materials were added to obtain the desired consistency. The lubricant was then run into cooling tanks where it was cooled with stirring until the proper structure was reached, after which it was filled into containers.

To make emulsifiable boring, cutting and cooling oils, procedures similar to the above were used but tar oil sulfonates or emulsifiable waxes were added to the other ingredients.

Puttys were prepared by mixing chalk or other mineral substances with linseed oil or drying tar oils made from benzol refining-still residues. Also puttys were made with combinations of mineral substances with drying oils and resin pitches or tar pitches. A kneading mill was used for the putty manufacture.

Lubricating oils and greases, made as described above, were used principally in heavy duty applications such as the lubrication of elevator guide rails in mine shafts and for such purposes they were considered to be satisfactory.

Rheinpreussen, Moers-Homburg. Lubricating oils containing naphthalene, instead of tar oils, were manufactured by Rheinpreussen at Moers-Homberg. The process used at that plant, as described by Atwell and Schroeder in CIOS Report Item No. 30, File No. XXIV-9 was as follows: "Fischer Tropsch middle oil, boiling range 250-350°C, is chlorinated at 80-100°C to the extent of 20-25% chlorine by weight. Five volumes of chlorinated oil are reacted with two volumes of naphthalene at 70-100°C in the presence of eight volumes of Fischer-Tropsch benzin and a small amount of $AlCl_3$, as catalyst. The sludge is withdrawn, the oil is neutralized with lime and bleaching earth, filtered and stripped free of benzine. Subsequent vacuum distillation yields middle oil and turbine oil overhead, and cylinder stock as bottoms. The fractions are used as such and are also blended to make motor oils."

According to Atwell and Schroeder "all products were stated to have a high viscosity index and great resistance to oxidation but tests on captured samples do not support these claims." When interrogated concerning lubricating oils made in this manner with Fischer Tropsch oil and naphthalene, Dr. Martin, Director of Ruhrchemie at Sterkrade-Holten, expressed the opinion that they were inferior from the standpoint of thermal characteristics to lubricating oils made from Fischer-Tropsch oils alone.

HYDROGENATION OF PITCH AND TAR OILS

In 1943 the German tar plants supplied to hydrogenation plants 215,073 tons of pitch; 171,389 tons of heavy tar oils; 19,520 tons of hard pitch distillate and 2,897 tons of pitch coke distillate. Altogether, the quantity of pitch and oils furnished for this purpose was 408,859 tons or approximately 89,950,000 U.S. gallons.

Ruhröl G.m.b.H. The tar oils went to various hydrogenation plants but a large proportion of it and substantially all of the pitch went to the one hydrogenation plant at Bottrop-Welheim operated by Ruhröl G.m.b.H. That plant, by the early part of 1943, could hydrogenate 263,000 tons per year of a mixture containing 70% briquette pitch and 30% tar oils. The products from that quantity of mixture were 120,000 tons of heating oil, suitable for use by the navy; 60,000 tons of aviation gasoline and 18,300 tons of fuel gas (propane and butane).

The navy fuel oil was claimed to be completely free of asphalts and, because of its aromatic character, to have good asphalt mixing or dissolving capacity when used with inferior fuel oils. The aviation gasoline had an octane rating of 92 and was said to perform well in supercharged motors.

The hydrogenation of the pitch-tar oil mixture at Welheim was carried out in two stages in each of which the pressure was 700 atmospheres. Liquid phase hydrogenation was used in the first stage to produce the navy fuel oil and gas phase hydrogenation yielded the aviation gasoline in the second stage. Fuel gas came from both stages of the process. A complete description of the operation at Welheim has been given by C. Cockram in CIOS Report Item No. 30, File No. XXX-104.

The operation of the Welheim plant was stopped by an air raid in July, 1944. After three months of reconstruction it was ready to start operating again but was shut down completely by five air raids between October 30th and November 30, 1944.

COAL BRIQUETTING

The increased use of pitch for heating, hydrogenation and carbon electrode manufacture during the years between 1940 and 1944 caused a corresponding decrease to take place in the use of pitch for the briquetting of coal fines. This change is indicated by the following table in which the percentage distribution of high temperature coal tar pitch in 1943 is contrasted with the percentage distribution in 1940.

Percentage Distribution of Coal Tar Pitch by Uses

Uses	1940	1943
Briquettes	47.8	34.4
Carbon electrodes	15.6	20.9
Hydrogenation	5.9	17.2
Roofing felt	11.4	10.3
Road Building	7.7	2.8
Heating and Carburetion	3.8	5.3
Other uses	7.8	9.1

Ruhröl G.m.b.H. No important changes were made in methods for producing or using briquette pitch with one exception which resulted from the hydrogenation of pitch and tar oils at the Welheim plant of Ruhröl A.G. Sludge amounting to about 15% of the pitch-tar oil feed was withdrawn from the liquid phase portion of the system and was transferred to the tar distilling plant of the MathiasStinnes organization where it was added to coke oven tar in the proportion of 140 tons of sludge to 100 tons of tar. The mixture was distilled to briquette pitch. The yield of briquette pitch from the sludge component of the mixture was about ninety percent.

Investigational work was carried out, especially by the Mathias-Stinnes organization to determine to what extent, if at all, the quality of the briquette pitch was altered by the admixture of hydrogenation sludge to the tar. Tests showed that the briquette pitch, so made, was slightly inferior to normal briquette pitch but complied with standard specifications and was acceptable to the briquetting industry.

The amount of hydrogenation sludge contained in briquette pitch in 1943 was approximately 17,000 tons or about 4% of the total briquette pitch used in that year. About one-half as much hydrogenation sludge was used in this manner in 1942 and substantial none in 1941. At that time efforts were still being made, without appreciable success, to operate the Welheim hydrogenation plant on coal extract produced by the extraction of coal with a mixture of tetraline and cresylic acid.

ROOFING AND WATERPROOFING PRODUCTS

No important changes were made during the war in methods for producing tar-saturated and coated felt for roofing and water-proofing purposes except possibly for an increased use of coatings made by procedures like those employed for making Plastix, i.e., by the fluxing of pitches with heavy oils followed by the addition of mineral filters.

In the German roofing felt plants the dry felt is first impregnated with a coal tar saturant and then is coated with a soft pitch or Plastix type of compound to which sand is applied as an antistick and weather protective coating. Felts coated with the Plastix type of coating have greater flexibility at low temperature than those coated with ordinary coal tar pitches.

NEW OR INCREASED USES OF CHEMICALS FROM HIGH TEMPERATURE COAL TAR

It appears that little or no research was carried on after 1938, in laboratories associated either directly or indirectly with the German high temperature coal tar distilling industry, with a view to making new or improved products from high temperature coal tar. Procedures for the recovery of the various chemicals and products furnished by the tar distillers after 1938 had, for the most part, been worked out in previous years. Also little or no research on high temperature coal tar or products derived therefrom appears to have been carried on by research institutions which might, ordinarily, be expected to include such work in their research programs.

Undoubtedly some research involving the use of chemicals derived from high temperature coal tar was carried out in the laboratories of major industrial concerns but, with the exception of pyrene, carbazole, symmetrical xylenol and diphenylene oxide, such research does not appear to have reached the stage where commercial quantities of unusual coal tar chemicals were required.

On the other hand, large quantities of the usual coal tar chemicals were produced at the tar plants.

Naphthalene Uses. The total quantity of naphthalene sold in 1943 was about 74,000 tons (metric). It was distributed approximately as follows: chemical uses by I.G. Farbenindustrie, 30,000 tons; hydrogenation to tetralin and decalin by Deutsche Hydrierung A.G., 13,000 tons; chemical uses by others, 4,073 tons; carbon black, 15,600 tons and lamp black, 2,400 tons; insecticides 4,600 tons; preservatives, 2,300 tons; lubricants (Rheinpreussen), 1,700 tons; and miscellaneous, 400 tons, including plant protection materials, insulating brick manufacture, and Diesel fuel.

The chemical uses by I.G. Farbenindustrie included the manufacture of beta naphthol and other dye intermediates, phthalic anhydride, plasticizers, tanning agents, preservatives, wetting agents and an emulsifier for Buna rubber. Of these uses those which were comparatively new in Germany were phthalic anhydride manufacture and the production of the emulsifier for Buna (Nekal B.x or Emulgator 1000).

The manufacture of phthalic anhydride by I.G. Farbenindustrie at Ludwigshafen and Schkopau and the construction of a plant for its production at Uerdingen has been discussed at length in the following CIOS reports:

Ludwigshafen, Item No. 22, File No. XXVII - 85
Schkopau " " " XXVIII- 29
Uerdingen " " " XXVII - 80

Phthalic anhydride was made in the same manner at Ludwigshafen and Schkopau. The capacities of these two plants were approximately 600-700 tons and 500-600 tons of phthalic anhydride per month respectively. The capacity of the Uerdingen plant was expected to be about 300-400 tons per month. Its equipment was approximately the same as that used in the other two plants. The process at all plants consisted in oxidizing naphthalene with air in the presence of a vanadium pentoxide catalyst containing potassium sulfate. Salt cooled converters were used.

Nekal B.X. mentioned above was first made in Ludwigshafen in 1917 but new plants for its production were installed during the war at Schkopau, Hüls and Auschwitz. At Schkopau it was called Emulgator 1000. The capacity of the Schkopau plant alone, was about 300 tons per month. This chemical is the sodium salt of dibutyl naphthalene sulfonic acid. It was said to be the best emulsifier in Germany for use in the manufacture of Buna rubber.

The use of 13,000 tons of naphthalene in 1943 by Dehydag (Deutsche Hydrierung A.G.) for the production of tetralin and decalin was mentioned above. Those chemicals, in turn, were used by the Wehrmacht (for the Navy, Army and Air Forces), by the railroads for heating and lighting, for the extraction of naphthalene from gas, and in lacquer manufacture.

Phenol and Cresol Uses. The recovery of phenol and cresols from high temperature tar and from the ammonia liquors from coke plants amounted to about 13,000 tons in 1943. They were used for making resins, dyestuffs, plasticizers, pharmaceuticals and for other well known purposes.

One extensive use for phenol consisted in the manufacture of the so-called Igamids involving, as intermediate steps, the hydrogenation of phenol to cyclohexanol, dehydrogenation of cyclohexanol to cyclohexanone, conversion of cyclohexanone into the oxime with a solution of hydroxylamine sulfate, reaction of the oxime with sulfuric acid followed by neutralization with ammonia to obtain caprolactam and polymerization of the latter to the Igamids. The various steps mentioned above have been described in greater detail in CIOS Report Item No. 22, File No. XXVI-53 by Hasche and Boundy.

A minor use for phenol was the manufacture of Koresin although at Ludwigshafen the production of this chemical amounted to 120-140 tons per month. The process, described in CIOS report Item No. 22, File No. XXIX-62, consisted in reacting isobutylene with phenol, using $AlCl_3$ as catalyst, to obtain para tertiary butyl phenol. The latter was then reacted with acetylene, under pressure at 230°C using zinc naphthenate as catalyst. The product (Koresin) was used as a tackifier for Buna.

Pyridine Bases. Uses for pyridine bases in Germany were not numerous. The total quantity distributed in 1943 was about 950 tons of which about 190 tons were used in the dying of fabric and in wetting agents, 350 tons were used by I.G. Farbenindustrie, in part for refining anthracene and carbazole, 260 tons were employed for denaturing alcohol and 130 tons for other purposes.

Some investigational work was done by I.G. Farbenindustrie at Leverkusen on the production of pyridine from higher boiling bases. Although the yield was only about 50% of theoretical it was planned to build a larger pilot plant. The war caused this plan to be abandoned. Vapors of the mixed bases, with hydrogen and a small amount of H_2S were passed through a nickel-chrome steel tube at about 860°C.

Pyrene. In 1943 Rütgerswerke A.G. furnished about 120 tons of pyrene to I.G. Farbenindustrie for the manufacture of dyestuffs. The pyrene was produced by fractionation and crystallization from high boiling coal tar oil.

Documents indicate that I.G. Farbenindustrie was interested in the use of pyrene for dyestuff manufacture before the war and carried on some research with a view to producing it synthetically. Apparently this development was not successful or was not carried to completion but some interest in the use of pyrene from tar continued throughout the war as indicated by the purchases mentioned above.

Carbazole. In 1943 Rütgerswerke A.G. also furnished 127 tons of carbazole to I.G. Farbenindustrie. It was recovered from a crude carbazole fraction by crystallizations from solvents followed by sublimation. The carbazole was used for the manufacture of 1,3,6,8 tetranitro carbazole. The latter was used in the production of an insecticide which, according to CIOS Report Item No. 24, File No. XXVII-4, was used in vineyards in place of arsenical insecticides, against the vine moth.

Nirosan contained 25% of 1,3,6,8 tetranitro carbazole, 10% of sulfite pulp 0.25% wetting agent (Igepal) and kaolin or chalk. Carbazole for this purpose and for the manufacture of dyes was of considerable interest to I. G. Farbenindustrie. According to CIOS Report Item No. 22, File No. XXVI - 11 research was conducted at Höchst on methods for its synthesis. The report states that "the synthesis of carbazole did not look promising and no further work was contemplated".

Insecticides from Symmetrical Xylenol. Symmetrical xylenol, chlorinated by Rütgerswerke A. G. at Niederau and by Dr. F. Raschig G.m.b.H. at Ludwigshafen was supplied to manufacturers of disinfectants. 150 tons of symmetrical xylenol was produced by Rütgerswerke A. G. at Rauxel in 1943 for chlorination at Niederau and subsequent shipment to insecticide producers in Hamburg. One disinfectant of this type, made by Schulke and Mayr, Hamburg, was called "Grotan".

Diphenylene Oxide. Rütgerswerke sold 144 tons of diphenylene oxide through brokers in 1943 but the manner in which this product was used is not definitely known. It has been stated that it was used as a base for scents and for dyestuffs.

Acenaphthene and Chrysene. 263 tons of acenaphtheno were supplied by Rütgerswerke to CIBA for dye manufacture and 8 tons of Chrysene went to I. G. Farbenindustrie for the same purpose.

Methyl Naphthalenes. Some alpha methyl naphthalene was used as cetane reference fuel and about 5-10 tons of beta methyl naphthene went into the manufacture of plant growth promotion chemicals.

Miscellaneous Chemicals. In addition to the above mentioned chemicals many others were supplied, principally by Gesellschaft für Teerverwertung but the quantities were small and they were used mainly for research purposes.

PART II

Reports of visits to organizations interested directly, or indirectly, in the processing of high temperature coal tar and use of products derived therefrom. Producers and users of high temperature coal tar products, selling organizations, engineering companies, equipment manufacturers and research organizations were visited.

GESELLSCHAFT FÜR TEERVERWERTUNG
DUISBURG-MEIDERICH

The tar distilling plant of Gesellschaft für Teerverwertung at Duisburg-Meiderich was visited on September 18th, 1945. The persons interviewed were Dir. E. Moehrle, Dr. Seidler, Dr. Dorfler and Paul Von den Hövel. On other visits made to the same plant at later dates Dr. W. Fischer and Dr. O. Kruber were interviewed, in addition to the above mentioned persons.

This plant was heavily damaged during the latter part of the war. It was about 40% destroyed. Two separate tar distilling units, anthracene buildings, two naphthalene buildings, the benzol plant and storage tanks suffered the most damage.

Light oils, tar acid oils, coumarone bearing oils and residues and other crude materials are received from other plants of the same organization and from other companies. They are processed at Meiderich to make refined products. The scope of its operations may be judged by the following list of products made in 1938 and 1944 by Gesellschaft für Teerverwertung.

Products (Tons per month)	1938	1944
Briquette pitch	11,303	9,134
Prepared tars and road tars	2,776	3,256
Fuel oil	2,264	4,403
Motor fuel oil	300	18
Creosote oil	2,521	-
Wash oil	753	890
Hydrogenation oil	1,740	1,838
Benzene	15	12
Toluene	158	115
Xylene	15	38
Motor benzole	267	184
90 Benzole	11	53
Solvent naphtha	129	99
Heavy naphtha	192	108
Coumarone resins	113	98
Phenol	212	369
Cresols	441	535
Xylenols	44	19
Anthracene residue	862	478
Naphthalene, pure	304	319
Naphthalene, hot pressed	303	369
Naphthalene, washed	827	727
Pyridine bases	26	24
Ammonium sulphate	39	23
Roofing felt (in rolls)	7,360	3,680
Acenaphthene	4	-
Special road tar	398	162
Special tar products	167	291
Refrigerated tar oils	265	261
Flaming oil	6	186

No pitch harder than briquette pitch was made at this plant and no special pitch compounds of the type called Plastix were made.

Changes in processes or equipment made in the last ten years were as follows: A new continuous tar still, improved anthracene cooling equipment, continuous crude phenol dehydration, new tar acid refining stills, and new oil redistillation stills.

CONTINUOUS TAR STILL

Probably the most important innovation at this plant is the continuous tar still. It is similar in principle to other continuous tar stills employing pipe heaters and multiple columns but differs from them chiefly in the arrangement of the burners in the pipe heater, and the use of high vacuum in the flash chamber and fractionating columns.

Following are some of the more important details concerning the different parts of the installation:

Pipe Heater. The pipe heater of the G.f.T. still contains 190 steel tubes, 79 m.m. I.D. and 89 m.m. O.D. The total heating surface is 349 m^3. 104 of the tubes in 9 rows are contained in the convection bank. Their heating surface is 191 m^2. 66 of the tubes with a heating surface of 121 m^2 are located in two rows on the ceiling of the furnace and 20 tubes, in two rows, with 32 m^2 heating surface are located on the upper part of the side wall opposite the convection tubes.

The return bends of the pipe heater are made of a special cast iron called "D.E.W. Sonderguss." Formerly it was called "Emmelguss." It is said to contain 3.2% carbon, 1.3 - 1.5% silicon, 0.5 - 0.6% manganese, 0.2 - 0.5% phosphorous and 0.05 - 0.08% sulfur.

The four gas burners for the pipe heater are located in the floor of the furnace instead of in the usual position at one side. A room below the pipe heater contains the connections to the burners, the valves, meters and other equipment required for its control. Over each burner, inside the combustion chamber, is an "umbrella" made of fire brick with openings located in such manner as to give uniform distribution of the combustion gases within the furnace. By the vertical arrangement of the burners and the use of the "umbrellas" the combustion gases are spread uniformly over the entire cross section of the combustion chamber and the heat distribution is said to be more even than that of other pipe heater installations.

Pressure Reducing Cock. While the tar is passing through the tubes and return bends in the pipe heater it is kept under pressure by means of a cast iron cock located just ahead of the flash chamber or "Teerkolonne." The location of the cock or "Entspannungsventil" is shown in figure 1, which shows all of the equipment in flow line arrangement. (Figures 2 and 3 show the arrangement of the equipment in the plant.) The opening in the cock is 36 mm diameter. The inside diameter of the pipe from the furnace to the cock is 79 mm. By constricting the flow of the tar in this manner the pressure just ahead of the cock is maintained at two to three atmospheres when the pressure at the tar feed pump is 6 to 7 atmospheres. The pressure after the cock and within the flash chamber is 80 mm Hg absolute.

Flash Chamber. The flash chamber, 2.8 m. diameter and 8.0 m. high, is constructed of cast iron. The tar from the pipe still enters tangentially at a point about 2 meters from the bottom. The pitch flows downwardly over cast iron baffles and the vapors pass upwardly through a compartment containing cast iron baffles which serve as entrainment catchers. In the upper compartment of the flash chamber are steel Raschig rings. These Raschig rings serve a dual purpose. They prevent the carry over of entrained pitch in the vapors entering the first fractionating column and, by reacting with the corrosive elements in the vapors released in the flash chamber, they prevent corrosion of the columns and condensers through which the vapors pass after leaving the flash chamber. This permits all of the apparatus after the flash chamber to be made of ordinary steel. After about eight months of operation the Raschig rings are removed from the flash chamber. Those which are severaly corroded are replaced and the ramainder are cleaned and returned to the flash chamber.

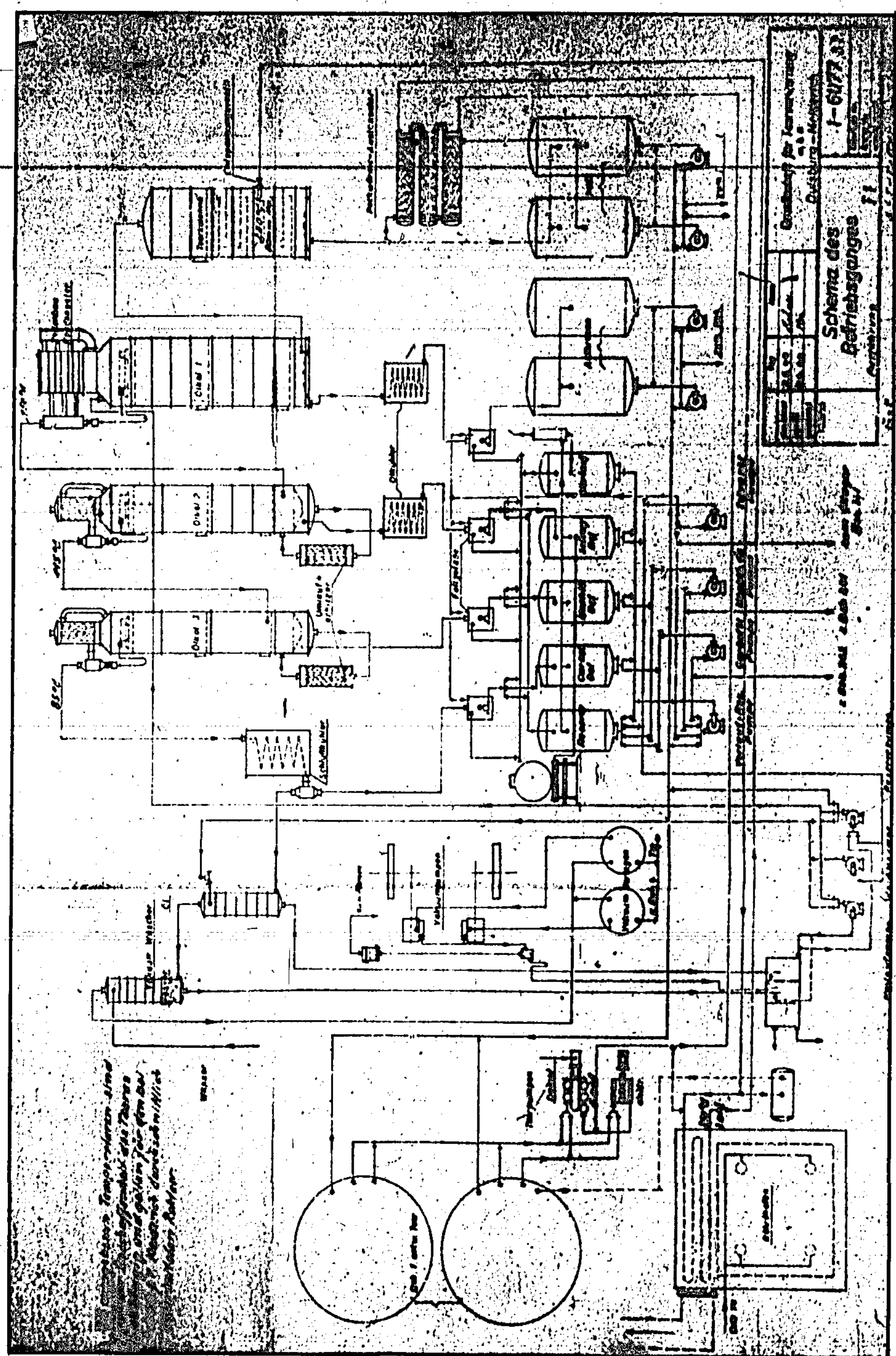


Fig. 1 - FLOW LINE ARRANGEMENT

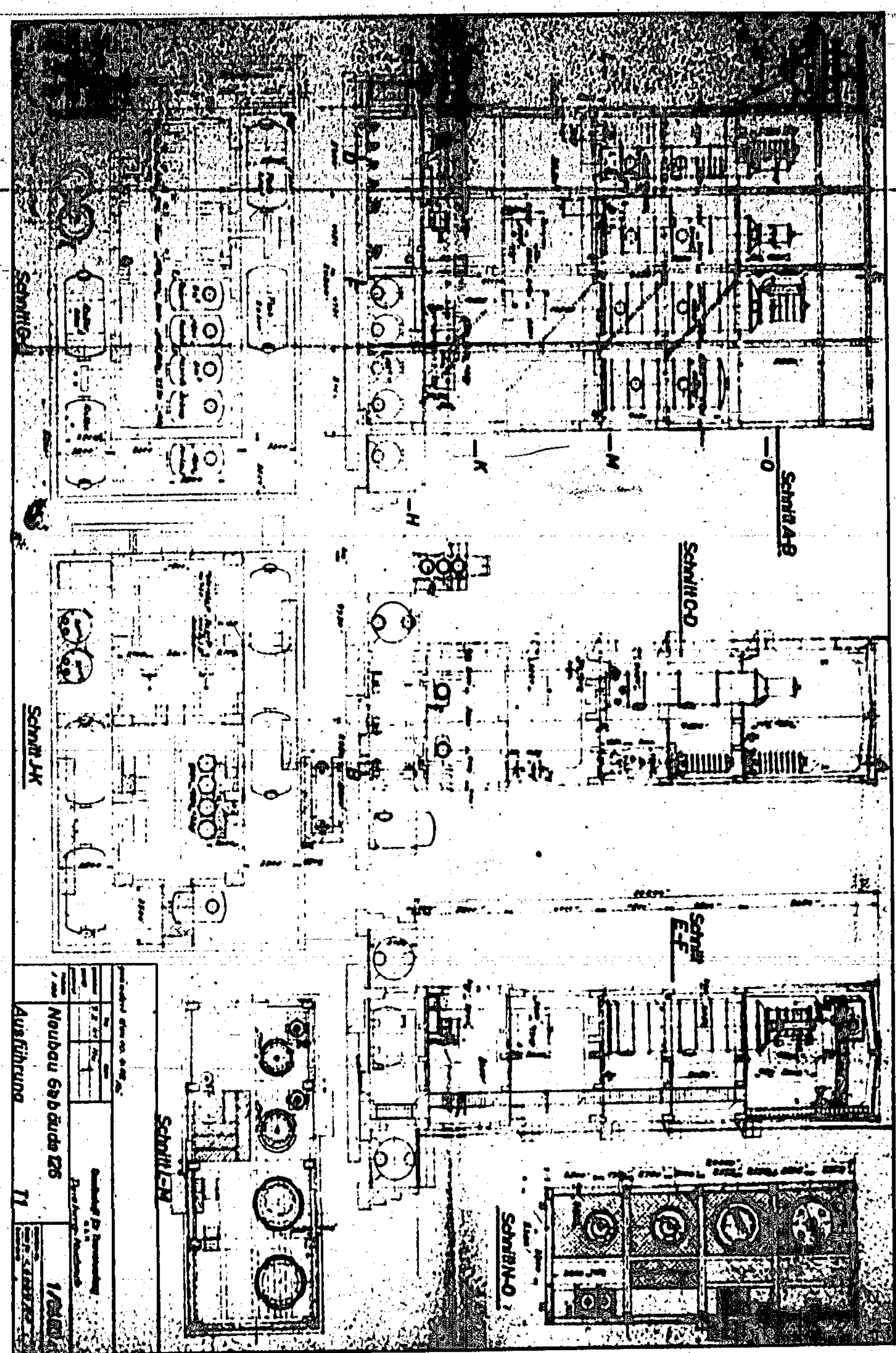


Fig. 2 - EQUIPMENT ARRANGEMENT

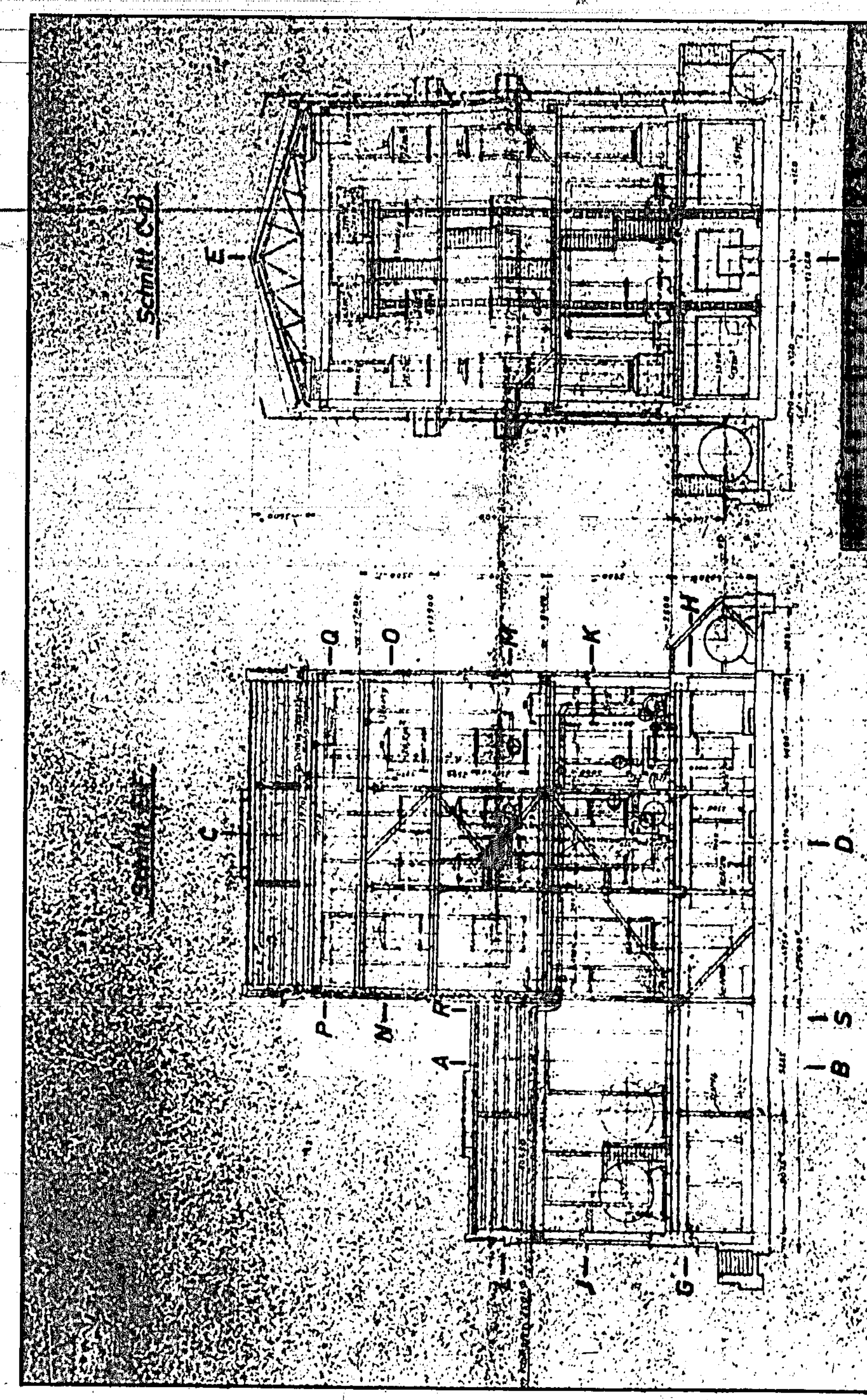


Fig. 3 - EQUIPMENT ARRANGEMENT

Fractionating Columns. The three steel fractionating columns, each 10 meters high, have the following diameters: No. 1, 2.8 m.; No. 2, 2.0 m., No. 3, 1.6 m. Column No. 1 is packed with 50 m.m., steel, Raschig rings. Columns No. 2 and 3 are packed with 35 m.m. Raschig rings.

In each column the packing is contained in two compartments which can be emptied separately. The dephlegmator on Column No. 1 is of a cellular type with 22 m^2 surface area. Those on Columns 2 and 3 have vertical tubes with 20 m^2 surface area each. Reflux is returned by gravity from each dephlegmator onto the packing within the column by means of a circular trough with vertical slots on the inner and outer side walls. There are 24 slots in the inner wall of the trough and 48 slots on the outer wall. The slots are 125 m.m. deep and only a few mm wide. Welded to the bottom of the trough is a curved ring which forms two sloping surfaces over which the reflux liquid from the slots flows onto the surface of the packing. The liquid drops onto the packing in two substantially continuous rings. The diameter of the inner ring is 1120 mm and of the outer ring 2230 m.m. in column No. 1. The inside diameter of this column is 2825 m.m.

Vacuum System. The vapors leaving column No. 3 pass through a water cooled, coil condenser with 43.7 m^2 surface area and then through two bubble tray columns each 1 m. diameter by 3 m. high. A part of the wash oil fraction is used as a scrubbing medium in the first column and water in the second. After leaving the second scrubber, the non-condensable gases pass to either of two vacuum pumps of 2850 m^3 capacity/hr. each.

Reheaters. Figure 1 shows columns 2 and 3 equipped with heaters that were intended to be used as reboilers to strip low boiling oils from the heavier oils leaving the bottoms of the columns. However, it was found to be unnecessary to supply any heat other than that furnished to the tar in the pipe heater.

Oil Coolers. The oils from the bottoms of column 1 and column 2 pass through water cooled coils having 13 m^2 cooling surface each before entering the receiving tanks.

Receivers. All of the condensates flow by means of barometric legs to seal pots from which they discharge by gravity into five steam heated receiving tanks of 8 m^3 capacity and two of 25 m^3 capacity. The former are used for forerunnings, carbolic oil, naphthalene oil, wash oil and reserve and the latter for anthracene oil.

Pitch from the flash chamber flows by gravity through a pitch-to-tar heat exchanger to either of two receivers of 25 m^3 capacity each.

Pumps. The plant is equipped with the following pumps:

1 electric, 3 piston pump, 12 m^3/hr 15 atm. (tar feed).
1 steam, single piston pump, 10-15 m^3/hr. 15 atm. (tar feed).
6 circulating pumps, rotary, 2 m^3/hr. 2 atm.
2 pitch pumps, electric, special rotary, 2 m^3/hr. 2 atm.

Temperatures, degrees C. Feed 100°; pipe still outlet, 320°; flash chamber 280°; overheads of columns 150° - 115° - 95°; pitch 260°.

Reflux ratio for each column. 1.0 - 2.0.

Consumption per ton of tar. Gas, 30 m^3; steam 30 kg; electricity 4 k.w.h., water 1.5 m^3.

Products. Pitch 68° C K.S. 55% (of dehydrated tar); anthracene oil, 300-400°C, 23%; wash oil, 240-285°C, 9% (free of crystalline solids); naphthalene oil, 220-350°C, 6% (free of phenol); carbolic oil, 190-218°C, 6%.

Personnel Required for Operation. Two men per shift.

Operating Time Between Cleaning Periods. Pipe still, one year; columns - flash chamber 6-8 months; 1,2 & 3 two to three years; condensers, coolers, etc., two to three years.

Claimed advantages. The following claims are made for this installation by Gesellschaft für Teerverwertung (stetige Destillation des Nielstickstoffgemisches Steinkohlenteer, W. Fischer, Zeitschrift V.D.I. - Beiheft "Verfahrentechnik" Nr. 1, 1944, pages 13 to 29.)

1. "A heat consumption below all presently known values."
2. "An increased yield of oil" (about 3-5%).
3. "Control of all corrosion influences."
4. "Avoidance of coke formation in the heating tubes."

According to the owners this still cannot be used to make hard pitches (above 100°C softening point). If this is desired the briquette pitch made in the continuous still will be transferred to other equipment for further distillation.

ANTHRACENE RESIDUE RECOVERY

An improved system of removing the so-called "anthrazen rückstand" or anthracene residue is used at this plant. Twenty water cooled vertical crystallizers are mounted above five vertical basket centrifuges arranged for mechanical unloading. Each crystallizer is 4630 m.m. high by 2000 m.m. diameter. An outer shell 2160 m.m. in diameter, open at the top, comprises a water jacket. The agitator driven from above by a 3.7 k.w. motor, moves the oil away from the side walls and conical bottom. It is provided with a screw on the lower end of its shaft which assists in the discharge of the crystalline slurry through the 150 m.m. opening in the bottom of the crystallizer.

The five centrifuges, made by C.G. Haubold A.G. at Chemmitz, have phosphor bronze baskets two meters in diameter. Their operating speed is 600 R.P.M. They are equipped with manually controlled mechanical cutters for unloading and discharge their contents onto conveyors which transfer the anthracene residue directly into open cars.

The quantity of anthracene oil charged into each crystallizer is 12 cubic meters. The temperature of the oil at time of charging is 80°C and when ready for centrifuging it is 20-29°C. The cooling of one charge requires from one to one and one half days. After it has cooled sufficiently the crystalline slurry is fed by gravity from a crystallizer to one of the centrifuges and the cake is whizzed for five to fifteen minutes depending upon the quality of the anthracene oil charged to the crystallizer. Normally the capacity of each centrifuge is about 80 tons of slurry per 24 hours. Anthracene residue produced in this manner contains only 2-3% of anthracene oil. The anthracene content is 20-25%. The centrifuged oil remains crystal free at 0°C.

CRUDE PHENOL DEHYDRATION

The comparatively new equipment at Meiderich for the dehydration of crude phenol consists of two, vertical, gas fired, 10 cubic meter stills equipped with columns, 1 x 5 meters, packed with iron, Raschig rings. The capacity of each still is about 20 tons of crude phenol per day (15-20% water). The temperature of operation is 160°C and the gas consumption is 120-140 tons per hour or about 60 cubic meters per ton of crude phenol.

TAR ACID REFINING STILLS

The tar acid refining stills most recently installed at this plant are 50 ton, vertical stills fired by gas from below and equipped with columns, 1.5 by 10.5 meters, packed with 25 x 25 m.m. Raschig rings.

OIL REDISTILLING STILLS

Between 1935 and 1940, four gas fired 17 ton vertical stills with packed columns, 1 meter diameter by 5 meters high were installed for the redistillation of oil. In 1940 four additional stills, of 50 tons capacity with 1.5 x 10 m. packed columns were installed. The packing rings are iron and 35 x 35 m.m.

SPECIAL CHEMICALS

For several years this plant has produced small quantities of special chemicals in laboratory and pilot plant equipment for sale to universities, research laboratories, etc. This was continued to some extent during the war. In the following table are given the approximate yearly quantities of such chemicals sold by Gesellschaft für Teerverwertung.

APPROXIMATE YEARLY PRODUCTION OF SPECIAL CHEMICALS MADE BY GESELLSCHAFT FÜR TEERVERWERTUNG, DUISBURG-MEIDERICH.

Acenaphthene, pure	100 kg
" , tech.	150 tons
Acridine, pure	50-100 kg
Anthracene, pure	50 kg
Quinaldine, pure	10 kg
Quinoline, pure	500 kg
" , tech.	60-80 tons
Chrysene, pure	20 kg
Collidine (2,4,6), pure	25 kg
Dicyclopentadiene, pure	200 kg
" , tech.	5-10 tons
Dimethyl naphthalene, pure, 1,6; 2,6; 2,7; 2,3; each	20-40 kg
2,4 dimethyl pyridine, pure	a few kg
2,6 " " "	" " "
0,0' dioxy diphenyl, pure	abt. 20 kg
" " " , tech.	200 kg
Diphenyl, pure	100-150 kg
" , tech.	500-600 kg
Diphenylene oxide, pure	100 kg
" " , tech.	200 kg
Fluoranthene, pure	a few kg
Fluorene, pure	50-100 kg
Indene, pure	50 kg
Indole, pure	220 kg
Isoquinoline, pure	a few kg
Lepidine, pure	5 kg
Methyl indole, (2),(3),& (7) each	10 kg
1, methyl naphthalene, pure	200 kg
1, " " , tech.	10-50 tons
2, " " , pure	500-1000 kg
2, " " , tech.	5-10 tons
Ortho oxydiphenyl, pure	10 kg
Phenanthrene, pure	50-100 kg
" , tech.	50-100 kg
Alpha picoline, pure	abt. 5 tons
Pseudocumol	50-60 tons
Pyrene, pure	20 kg
Xylenols	
1,2,4, pure	50 kg
1,2,4, tech.	500 kg
1,3,4, pure	5-10 kg
1,3,4, tech.	1500 kg
1,3,5, pure	5-10 kg
1,3,5, tech.	60 tons
1,4,5, pure	5-10 kg
Meta xylene, pure	50 kg
Ortho xylene, pure	50 kg

GESELLSCHAFT FÜR TEERVERWERTUNG CASTROP-RAUXEL

The plant of Gesellschaft für Teerverwertung at Castrop-Rauxel was visited on September 17th and on two occasions thereafter. The persons interviewed were Dr. E. Moehrle, Managing Director of Gesellschaft für Teerverwertung and Dr. Klemm, Director of the Rauxel plant.

This plant was about 50% destroyed by bombing and artillery. When visited it was distilling only about 200 tons of tar per day. (Previously, when operating at full capacity, it had distilled about 1500 tons per day). The chief needs of the plant to run at higher capacity were more fuel gas and more crude tar. The stock of crude tar on hand at all of the coke plants in the British Zone amounted to about 50,000 tons and current production was about 350 tons per day.

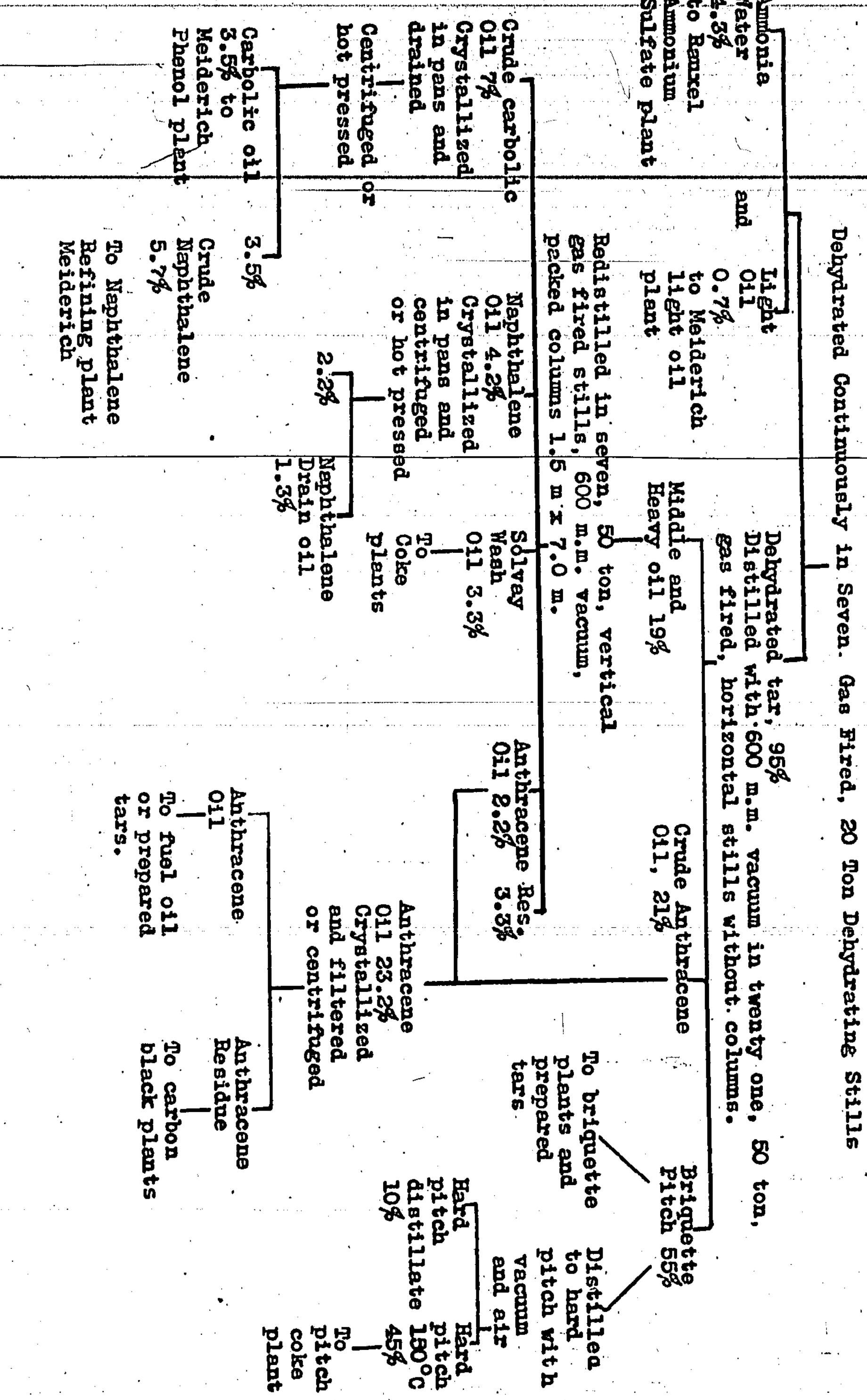
The stocks of products on hand were as follows, (approximately): Oil, in process, 3500 tons; briquette pitch, 39,000 tons; road tar, 60 tons; creosote oil, 600 tons; hydrogenation oil, 500 tons; anthracene residue, 35 tons; hot pressed naphthalene, 40 tons; pitch coke, (contaminated) 300 tons.

The accompanying flow diagram shows the methods of operation and equipment normally employed at this plant. The average monthly productions of the plant in 1938 and 1944 are given in the following table:

Average Monthly Production, Metric Tons

Product	1938	1944
Briquette pitch	7,546	7,437
Hard pitch (150°C) for coking	4,427	9,023
Prepared tars and road tars	3,697	2,179
Fuel oil	7,194	8,526
Motor fuel oil	62	48
Hydrogenation oil	703	1,353
Creosote oil	1,230	1,142
Wash oil	970	1,195
Anthracene 40%	212	135
Anthracene residue	588	870
Acenaphthene	5	-
Carbolic oil filtered	1,330	1,196
Hot pressed naphthalene	594	893
Crude naphthalene		146
Flotation oil		18
Special tar products		516

CRUDE, HIGH TEMPERATURE, COAL TAR



Gesellschaft für Teerverwertung
Castrop-Rauxel

New Processes and Products. The only changes of any consequence that were made at this plant in recent years were as follows:

Hard Pitch Manufacture. In order to supply the new pitch coke plant of Verkaufsvereingung für Teererzeugnisse at Rauxel with hard pitch (150°C), seven vertical, 30 ton stills were installed. Each still was heated by gas and operated under vacuum (about 600 m.m.) and agitated with air. The stills were charged with briquette pitch transferred directly from the tar stills and during distillation of the briquette pitch to hard pitch, air was drawn through the pitch by the vacuum in the still. Distillation and air blowing was continued until the desired softening point was reached at which time the temperature in the still was 380-390°C. The high softening point pitch was then dropped by gravity into transfer tanks from which it was loaded into tank cars by suction applied to the tank car. Transfer to the tank car by air pressure was tried at first but the transfer lines became hot and because of the danger of explosion this method was discontinued. No difficulty was experienced in the use of the suction method of transfer.

Special Pitch Compounds. To supply a recent demand for pitch compounds having better temperature susceptability characteristics than those of straight-run pitches, such compounds were made at Rauxel by fluxing hard pitch, made in the manner described above, with the distillate recovered from the same process i.e. the distillation of briquette pitch to hard pitch. The mixing of hard pitch and hard pitch distillate was done in a 36 cubic meter, steam heated tank equipped for agitation with air.

Anthracene Cooling and Recovery. The usual method of recovering anthracene and anthrocene residue from anthracene oil has been to cool the oil in pans, with manual stirring at intervals, then filter the slurry through vacuum filter pans and centrifuge the filter cake. However, during the war an improved process was installed which consisted in cooling the anthracene oil in vertical water cooled crystallizers with agitators and then centrifuging the crystalline slurry through Haubold vertical centrifuges. The anthracene residue produced in this manner contained less anthracene oil than that made in the usual manner. This method is described in greater detail in a report covering the Duisburg-Meiderich plant of this same organization.

GESELLSCHAFT FÜR TEERVERWERTUNG
BOCHUM-GERTHE

On September 20, 1945, the Bochum-Gerthe tar distilling plant of Gesellschaft für Teerverwertung was visited to determine what changes, if any, had been made in manufacturing procedures or equipment in recent years. It was found that no important changes had been made.

This plant suffered very little damage by air raids or artillery. Damage was confined principally to the buildings (no equipment) of the anthracene and naphthalene hot press plants. Normal operations of the plant were as follows:

Tar Dehydration. About 120 tons of crude tar per day was dehydrated continuously in one vertical, 30-ton gas-fired still. Water and light oil amounted to about 5.0%. The light oil (about 0.4%) was shipped to Meiderich for refining.

Tar Distillation. The ehydrated tar was distilled in eight, 30-ton vertical, gas-fired stills, with 400 mm. vacuum. Temperature at finish was about 360°C. The fractions taken were: middle oil, 200-250°, 5%; naphthalene oil, 5%; heavy oil, 250-300°, 8%; anthracene oil, 300-350°C, 25%; briquette pitch, 54%.

Tar Acid Extraction. The middle oil was extracted with 18% caustic to remove tar acids, and the phenolate was sent to Meiderich. The neutral oil was combined with the heavy oil fraction (see above) and shipped to coke plants as wash oil.

The naphthalene fraction was cooled in pans, drained and hot pressed. The hot-pressed naphthalene was refined by washing at 90-95°C with caustic soda, sulfuric acid, formaldehyde (if available) and water. The washed naphthalene was redistilled.

Anthracene Residue Recovery. The anthracene oil fraction was cooled in pans and filter-pressed to remove anthracene residue 4-5%, containing about 30% of oil. It was shipped to carbon black plants. The filtered anthracene oil went partly to road tars but principally to fuel oil.

Manufacture of Hard Pitch. The briquette pitch was transferred by suction to two vertical hard pitch stills, fired by gas where it was distilled to hard pitch (150°C) by hot flue gases (600°C), heated and made oxygen free by direct combustion of coke oven gas in the flue gas using the oxygen contained in

the flue gas to support the combustion. The temperature in the still at the end of distillation to hard pitch was about 380°C.

Hard Pitch Cooling. The hard pitch was cooled by means of an endless steel conveyor having a capacity of 50 tons per day. The pitch was transferred from the hard pitch stills to two overhead feed tanks from which it was discharged by gravity onto the steel conveyor. The latter was made up of overlapping steel plates mounted on rollers. From the upper end of the steel conveyor the pitch discharged into a car in the form of thin plates.

The plant was not operating at the time of the visit for lack of tar. Stocks of material on hand were 400 tons anthracene residue, 50 tons crude naphthalene and 600 tons of hot pressed naphthalene.

RÜTGERSWERKE A.G.
CASTROP - RAUXEL

The Castrop-Rauxel plant of Rütgerswerke was visited on September 17th, 1945 and on two subsequent occasions. The manufacturing processes and products of this company were discussed with Dr. Carl Fehr, Dr. Geller, Dr. Carl Bellwinkel and Dr. Paul Schurmann. The plant was inspected with some of these men. The quantity of tar processed in this plant in 1943 was 172,247 tons (metric).

About forty percent of the equipment at Rauxel was destroyed during the war. One lime kiln in the tar acid plant, a crude naphthalene plant having a capacity of 700-800 tons per month, a naphthalene refining plant of 800 tons capacity, a crude anthracene plant of 120-140 tons per month capacity, pure anthracene equipment (30-35 tons per month), pure carbazole equipment and carbazole sublimation equipment (20-25 tons), and equipment capable of making 40-50 tons per month of acenaphthene, fluorene and diphenylene oxide was destroyed. Also the road tar making and handling capacity was decreased 50-80%. The light oil plant was also put out of commission. Very little damage was done to the batch tar stills and oil redistilling stills with columns.

This plant distills dehydrated tar in vertical, gas fired, 60 ton batch stills. Mounted directly above the stills are cast iron fractionating columns, 2 meters diameter x 10 meters high. Each column has 40 bubble trays and at the top is mounted a vertical tube, water cooled dephlegmator. The stills are operated under about 650 mm vacuum. The following primary fractions are taken:

Carbolic oil fraction	3.0%
Naphthalene I (about 70°C)	6.0
" II (10-15°C)	6.0
Methyl naphthalene fraction	3.0
Acenaphthene fraction	3.0
Fluorene fraction	4.0
Phenanthrene fraction	3.0
Anthracene fraction (12-18% Anth.)	10.0
Carbazole fraction	8.0
Briquette pitch 70-80°C	50.0

Continuation of the distillation, with vacuum and some steam yields the following products:

Pyrene fraction (10-18% Pyrene)	4.0%
Chrysene fraction	4.0
Hard pitch (150°C)	40.0

The accompanying table lists the products made at this plant during the first ten months of 1944.

The newer developments at this plant that are worthy of note are, the manufacture of Plastix, in special equipment, new apparatus for the cooling of pitch and increased productions of carbazole, acenaphthene, diphenylene oxide, pyrene and symmetrical xylenol. The carbazole was employed by I. G. Farbenindustrie for the manufacture of tetranitro carbazole used in a grape arbor insecticide. The acenaphthene and pyrene were used in making dyestuffs. The use of the diphenylene oxide is not definitely known but it was said to have been used in scents. The symmetrical xylenol was shipped to Rütgerswerke at Niederau where it was chlorinated for use in disinfectants.

<u>Plastix Manufacture</u>. For the manufacture of Plastix a special plant was installed. It consists of a tank of 100 tons capacity, heated by gas with ring burners, provided with distributing pipes for the introduction of air. A vapor off-take connects the tank with a water cooled condenser. Following is a translation of a description of the process received at the Rauxel plant:

> The plastix oxidizing plant is charged with 70-80 tons of soft pitch with a melting point of 40-50°C. The capacity of the oxidizing tank is approximately 100 tons but there must be some empty space in order to avoid foaming over into the distilling tube. In addition to soft pitch, naturally, other grades of any softening point may be used.
>
> The batch must be heated without interruption. When the temperature reaches 290-300°C oxidizing should be started. Oxidation should normally be finished after 10-14 hours. Approximately 400-600 cu.m. of air should be used per hour, regulating the pressure so as to ensure thorough mixing of the mass.
>
> Oxidizing may be intensified by small addition of steam. The oxidizing temperature must be raised to at least 350° and maintained at this level throughout the whole of the oxidizing operation. The use of heat is absolutely essential to achieve good results. Consumption of oxygen is controlled by analyzing the waste gas. Owing to the blowing with air, and a little steam, roughly 5-10% distills off.

When the mass has a softening point of 180-200°C the process is finished. Subsequently, the softening point is adjusted (in the same vessel) to the desired level by adding heavy tar oils preferably free from residue. The quantity of oil required depends on the desired softening point which generally is between 38 and 60°, although it is possible to keep the softening point at a higher level. The interval between the breaking point of the product and the softening point must be at least 40°C.

In addition to the above it was stated that the best combination of oils for Plastix consists of 70% anthracene oil II, 350-450°C free from crystalline substances and 30% of wash oil fraction, 250-320°C. The mixture should be free of crystalline substances. Approximately 50 percent of the above oil mixture is used to flux the hard pitch (180-200°C) produced in the manner described above.

Another method for producing hard pitch in addition to the method for making Plastix, was tried by Rütgerswerke several years ago. It was described in a British patent No. 499,924 applied for on March 22, 1938. By this method briquette pitch was heated to 300-350°C and blown with steam heated to about 400-500°C. The steam was superheated electrically. This process was tried on an experimental scale but was dropped because of the war. It was mentioned that the heat consumption of this process was high but no attempt was made to conserve heat. The steam consumption was about 1½ tons per ton of pitch. The pitch produced by the process was granulated with water.

Pitch Cooling. Considerable work has been done at this plant in recent years with a view to developing improved methods for cooling hard pitch and briquette pitch. The following method was used commercially at the Rauxel plant and also at the tar distilling plant of Gutehoffnungshütte. It was especially suitable for cooling hard pitch. Liquid, high softening point pitch from an overhead feed tank flowed from small holes in the bottom of a horizontal pipe or manifold. The thin steams of molten pitch dropped onto water flowing in shallow, inclined troughs. The steams of pitch floated down the troughs with the water and cooled sufficiently before reaching the end to become semi-solid. They then were separated from the water by an inclined grating made of iron bars spaced close together. While passing over the grating and dropping into a car the moisture on the surface of the rods of pitch evaporated and the rods cooled sufficiently to break into small pieces.

The other method tried at Rauxel but not developed to the point of commercial use was intended primarily for the cooling of briquette pitch. The molten pitch flowed in a thin band onto water flowing down a shallow trough about 50 meters long. The nozzle by which the pitch was flowed onto the water was so placed that the pitch film remained on the surface for several meters. This arrangement allowed the pitch film to cool by air on the top while it was being cooled by water from below. It was claimed that by this arrangement the pitch became sufficiently solid so that water did not enter it when it became submerged in the water. A steel rod grating at the end of the trough separated the solid pitch from the water and discharged the granulated pitch into a car.

The water was recirculated.

The two methods of pitch cooling mentioned above have been patented in Germany by Rütgerswerke A.G. The patent numbers and dates are as follows:

No. 688,505 - published Feb. 1st, 1940.
No. 740,055 " Aug. 26th, 1943.

SHIPMENTS 1944

	in kg		in kg
Säureharz	46.873	Flotationsöl	128.586
neutrales Leichtöl	41.52o	Anthracen 4o%ig	684.17o
Weichpech	2.o17.292	Brandmasse	7.195
Pech	37.327.533	Anthracen 7o%ig	15.o15
Stahlwerksteer	1.864.193	Testbasen a.T.	2o
Kabelteer	249.57o	Pyridin n.T.	131.517
präp. Teer	1.o58.593	Reinpyridin	21.95o
Strassenteer o.B.	391.641	Chinolin techn.	13.463
Kaltteer	16o.33o	hochs. Basen	2o
Lithosot	1.231.341	schwefels. Ammoniak	357.792
Eisenlack	697.422	Pyridinrückstd. II	62.832
Isolieranstrichmittel	313.936	Reinanthracen	113.223
ger. 9oer Benzol	16.548	Anthracensublimat	16o
Motorenbenzol	1.o57.6o2	Imprägnieröl	146.o7o
ger.Toluol	177.252	Heizöl	3.693.oo6
ger.Lös.Bzl. I	1.688	Marineheizöl	27.569.8o1
Reinbenzol	11	Solvaywaschöl	6.867.641
Pseudokumol	1	Motorentreiböl	158.994
Reinxylol	567.587	Hydrieröl "M" ./.	5.458
Neutralöl III	325.865	Hydrieröl I	11.o96.885
Neutralöl IV	46.737	Hydrieröl V	8.139.o72
Kumaronharz	1.167.93o	Marineheizöl m. Pechzus	8.566.462
Benzoatlauge	179.49o	Neutroöl	5o.651
Phenolpech	218.582	Xylenol 1-2-4	5.781
Phenol/Kresol-Gemisch	277.51o	Xylenol 1-3-5	1oo.327
Phenol	2.766.519	Xylenol 1-4-2	16
Phenol fl.	128.945	Hartpech	1.392.6o4
Kresol DAB IV	2.152.8o1	Plättchenpech	27.265.885
Kresol DAB VI	1.373.o7o	Hartpech fl.	259.79o
Orthokresol rein	273.o26	Plastix	485.355
Metaparakresol rein	78.421	Kumaronharzrückstd.	1o4.87o
Amerikakresol	1.o42.921	Ortho-Oxydiphenyl	9.21o
Rohbrenzkatechin	28.44o	Para- "	35
Orthokresol techn.	79.74o	Spezialteeröl	1.225
Rohnaftalin	1o7.6oo	Fluoren techn.	14.2o6
Warmpressgut	2.137.83o	Fluoren techn.rein	3.485
Reinnaftalin	3.5o4.832	Phenanthren geschl.	1o1
Anthracenöl filtr.	1.o67.152	Phenanthren umgearb.	1oo
Karbolineum	3.485	Acenaphthen	229.384
Teerfettöl I roh	45o.24o	Inden	13
Teerfettöl Ess	5o	Diphenylenoxyd	235.911
Teerfettöl II	175.937	Kohlenwasserstoffmisch.	9.982
Teerfettöl III	143.443	alpha-beta-Methylnapht.	22.683
Teerfettöl IV	18.3o9	alpha-Methylnaftalin	3.751
Naftalinwaschöl	374.4o6		

beta-Methylnaftalin	315
Diphenyl techn.rein	891
Karbazol 85%ig	59.676
Fluoranthen	3o1
Pyren	25.242
Chrysen	23
Rohäthylenclorid	123.68o
Teeröl " Rütgers "	3o6
ger. Lös.Bzl. II	1.o43.638
Reintoluol	223.915
Spezialtränkmasse	981.o4o
roh. Lös. Bzl. I	16o
Amerikakresol roh	5o.o1o
Pechkoksöl	3o
	165.817.222

Die Production des Jahres 1944 erstreckt sich nur auf 1o Monate. Vom 6.Nov. 1944 bis etwa Ende des Jahres 1944 stand die Fabrik still infolge Bombenangriff am 6.Nov.

RÜTGERSWERKE AKTIENGESELLSCHAFT, BERLIN

Rütgerswerke Aktiengesellschaft, Berlin W-35, Lützowstrasse 33-36, was visited on October 31, 1945. The persons interviewed were Directors Fabian, Müller, and Kahl and Professor Dr. Mallison. General information concerning the Rütgerswerke tar plants in eastern Germany was requested. The following information was obtained:

Erkner Plant (near Berlin). This plant is operating but at only about ten percent of its capacity, because of insufficient tar. The capacity of the plant is about 100,000 tons. The amount of tar distilled in 1943 was about 60,000 tons. The plant was not badly damaged during the war, and, except for the naphthalene cooling and hot pressing departments, could be operated at full capacity if sufficient tar were available.

The equipment and operating procedures at Erkner are like those used at Rauxel by this same company. There are eight, forty-ton vertical batch stills, mounted directly below cast iron columns for tar distillation; two continuous stills for tar dehydration (one damaged); two, one-hundred ton and six, twenty-ton stills for benzol refining; a complete tar acid plant (capacity about 10,000 tons per year), containing five, crude acid stills, and one pure phenol still; a naphthalene cooling building and hot press (damaged); a cooling house for pure naphthalene (damaged), and plastix making equipment (damaged).

In the way of new developments at Erkner the following processes were mentioned:

A method of making hard pitch (200°C) from briquette pitch was tried which consisted in passing briquette pitch heated to about 300-350°C and steam heated to about 400-450°C by means of an electric heater through a pipe into a flash chamber. The hard pitch coming from the flash chamber was granulated with water. According to Dr. Geller who was interviewed later in regard to this process, the steam consumption was about 1.5 tons per ton of pitch. The heat consumption was said to be high, but might be improved by means of heat economizers. This process was tried on an experimental scale during the war, but had to be dropped before development was completed.

In making plastix, it was said that the important thing is to use a proper combination of high and low boiling tar oils for fluxing hard (200°C) pitch and the quantity of oil mixture used must be just enoughto "swell" the pitch and not dissolve it. The proportions used by Rütgerswerke were said to be about 30% oil and 70% pitch. The oil mixture contained oil distilling 380-450°C and oil distilling about 300°C.

In the benzol plant at Erkner a method of removing thiophenes from benzol, to be used for synthetic phenol production, was used which consisted in giving the benzol a preliminary wash with 66° sulfuric acid, then adding solvent npahtha II, containing indene and washing again with 66° sulfuric acid. Agitation was continued for two to four hours. For benzene containing 0.2 to 0.4% thiophene, the amounts of acid and solvent naphtha II used were about 14-18% by weight and 3% by weight respectively. For a thiophene content of 0.05-0.15%, the percentages of acid and solvent were 6-12% and 1.5% respectively. Following the acid treatment the benzol was washed with water and soda.

Breslau. The Rütgerswerke plant at Breslau was said to have been almost completely destroyed during the war.

Niederau. The Rütgerswerke plant at Niederau (near Dresden) whose capacity was about 48,000 tons per year is believed to have been dismantled and prepared for shipment to Russia.

CHEMISCHE FABRIK WEYL A.G., MANNHEIM

Chemische Fabrik Weyl A.G. at Mannheim-Waldhof was visited on December 1st and December 4th. The persons interviewed were Dr. Charles F. Lang (a director of Rütgerswerke and at present, manager of the Mannheim plant) and Dr. Friederich Cröy, Director of Chemische Fabrik Weyl A.G.

This is the largest tar plant in the American zone of occupation. In 1943 the amount of tar processed was approximately 73,000 tons. Crude tar supplies came from coke plants in the Saar and from gas plants (about 50) in Hessen, Würtemberg and Baden.

About fifteen percent of the equipment in this plant was damaged by air raids. The departments for cooling and hot-pressing naphthalene and for the production of fruit tree carbolineum were destroyed. Some of the other departments were slightly damaged.

Except for the stills and pitch cooling pans, the equipment used at this plant is similar to that used in most of the German tar plants. The six stills, two of which are used for dehydrating and four for distilling tar, are heated by coal with mechanical stokers. Each still has a capacity of 62 tons and is constructed like a horizontal tubular boiler. 76 tubes, each 7058 mm. long, 121 mm. O.D., and 108 mm. I.D. connect the two ends of the stills. The ends of the tubes extend through the end plates and are provided with a bead or rim which keeps them from pulling away from the end plates when the stills are heated. Combustion gases pass through the tubes and they are surrounded by the tar to be dehydrated or distilled. The two stills used for dehydration are not equipped with columns. The four tar-distilling stills have cast iron bell-and-tray columns 15 meters high and 120 cm. in diameter. Each column has 48 trays and is provided with a water-cooled dephlegmator containing 17 vertical tubes through which the vapors pass. The columns are so heated that reflux from them returns directly by gravity, to the stills. The stills were originally installed by Weyl and are unlike those used by Rütgerswerke in its other plants but the columns were installed by Rütgerswerke and follow their standard design.

Pitch cooling is carried out by equipment developed by Weyl which operates satisfactorily but, according to the directors of the plant, is unnecessarily expensive. Eighteen shallow steel pans, each capable of holding one ton of pitch, are mounted in cascade arrangement above a railroad track and above a moveable hopper which can receive the pitch by gravity from any pan and drop it into a railroad car. Molten briquette pitch from a tank mounted above the topmost pan flows into the latter and

overflows into each of the other pans until all eighteen are full. After cooling for a day the pans are inverted by rotating them around trunnions mounted at each end. The solid pitch drops from each pan into the moveable hopper which conveys it to the car. The pitch-cooling building contains four units of this kind, or 72 pans divided into four groups of eighteen each.

Following the destruction of the naphthalene hot presses, this plant started making 78° naphthalene by the redistillation of crystallized and drained naphthalene. A steam-heated vacuum still with columns is used for this purpose.

The various products made at this plant and their respective yields were as follows in 1943:

Water 5%

Light Oil 2% - fractionated in benzol plant to produce crude benzol, toluol, xylol, and solvent naphtha. The first three were washed and redistilled to make pure light oil products and the solvent naphtha was shipped to Rütgerswerke at Rauxel.

Carbolic Oil 3% - shipped to Rauxel with the solvent naphtha.

Naphthalene Oil I 7% - cooled in pans, drained and hot pressed to make 79.0-79.5° naphthalene (or 78.0 to 78.5° a few years ago for American shipments). The oil from the hot presses was sent with the carbolic oil and solvent naphtha to Rauxel. The 79° naphthalene was washed and redistilled to make refined naphthalene. Present practice since the hot presses were destroyed is to crystallize this fraction, drain, centrifuge, wash and redistill. The freezing point of the naphthalene is about 79.6°.

Wash Oil 7% - shipped to coke plants for recovery of light oil from the gas.

Anthracene Oil I 4% - shipped principally for fuel oil or hydrogenation. Small part for wood preservation (carbolineum).

Anthracene Oil II 7% - a part of this oil was crystallized in pans and filtered through vacuum filter pans to obtain anthracene residue for shipment to carbon black plants. The filtrate went to fuel oil, naphthalene wash oil (used at coke plants) or carbolineum. Another part of Anthracene Oil II was mixed with wash oil or naphthalene oil, cooled in a water-cooled tank with agitation and centrifuged to make 40% anthracene. The centrifuge oil went to fuel oil.

Column Oil 2% - this oil was drained from the column after completion of distillation and before filling the column with tar for the next distillation. The oil went to fuel or hydrogenation.

Briquette Pitch 54% - cooled in one-ton pans and shipped to briquetting plants.

When visited, this plant had received some crude tar from the Ruhr but was receiving practically no tar from its regular sources of supply. Stocks on hand consisted of 7,000 tons of briquette pitch, about 300 tons fuel oil, 500 tons carbolic oil, and 300 tons of naphthalene.

GELSENKIRCHENER BERGWERK AKT. GES. GRUPPE BOCHUM

ZECHE CAROLINENGLÜCK, BOCHUM

This plant was visited on September 20 with Dr. Geiselbrecht. The persons interviewed were works director Dipl. Ing. George Speckhardt and Superintendent Voigt. They also conducted the trip through the plant and explained the various operations.

No part of the plant was working at the time of this visit because permission to operate had not been granted. Some departments will require rebuilding before operation can be started but others are ready for immediate use. The situation with respect to bomb damage is as follows:

Tar Stills: Can be run at full capacity, i.e., 600 tons per day.

Oil Redistilling Stills: Can be run at full capacity.

Naphthalene Washing Equipment: Can be repaired.

Naphthalene Refining Equipment: Can be used.

Phenol Extraction Equipment: Can be repaired.

Naphthalene Pans and Hot Presses: Totally destroyed.

Anthracene Recovery Equipment: Totally destroyed.

Naphthalene Sublimation House: Totally destroyed.

The light oil plant, although severly damaged, can be placed in operation soon because the equipment is in good condition. Most of the damage suffered by this plant was confined to the building.

When running at full capacity this plant received 600 tons of tar per day from eight sources, namely: Carolinenglück, Bruchstrasse, Dannenbaum, Nordstern, Alma, Fried. Wilhelm Hütte, Hoerderverein, and Tremonia. The tar was received by tank cars and unloaded by dropping it into troughs beside the track which run to tar storage tanks.

Dehydration. Dehydration of the crude tar was carried out in one 50-ton vertical pot type still, running continuously without column. The water and light oil together amounted to about 5%.

Distillation. Seven stills were used for distilling the crude tar. They are divided into two groups. Three 50-ton vertical pot style stills without columns are in one group, and four 50-ton horizontal stills, provided with two fractionating columns, are in another group.

The horizontal stills have two large heating tubes (as at Gutehoffnungshütte). They are so connected with the columns that any one of the four stills can be run with either of the columns. Each of the columns is 2 meters in diameter and 19 meters high. There are 19 sections and 38 trays, the tray spacing being 500 mm. They are provided with water-cooled dephlegmators, condensers, and barometric legs for the condensates. The columns are not enclosed. Both the vertical and horizontal stills operate under about 650 mm. vacuum.

The products from the vertical stills were:

Water and Light Oil, 5% - The light oil (about 2%) went to the benzol plant.

Crude Wash Oil, 15% - Redistilled in 4 vertical stills with small bell and tray columns (not enclosed) to make:

Carbolic oil shipped to Meiderich for tar acid extraction.

Naphthalene oil washed with 20% NaOH to recover tar acids for shipment to Meiderich. The neutral oil went to wash oil or fuel oil.

Wash Oil - shipped to coke plants for light oil recovery.

Residue - returned to crude tar.

Anthracene Fraction, 17% - Crystallized in pans, drained, filtered (with vacuum filter pans), and centrifuged to obtain anthracene residue (20% anth.) for carbon black or refining to 40% anthracene and anthracene oil (to road tars or other prepared tars or fuel).

Heavy Oil, 8% - Hydrogenated or mixed with bitumen for roads.

Hard Pitch, 50% - Shipped to pitch coke plant at Lothringen.

From the horizontal stills with columns, the following products were obtained:

Carbolic Oil, 4% - Shipped to Meiderich.

Naphthalene Fraction, 8-9%, 65° freezing point, washed for tar acids (phenolate to Meiderich), crystallized in pans, drained and hot pressed to make 78° + naphthalene and wash oil.

Wash Oil, 8% - Used directly for light oil scrubbing.

Anthracene Oil, 20-22% - Cooled in pans, filtered and centrifuged.

Briquette Pitch - Shipped to coal briquetting plants.

The most interesting points observed at this plant were the following:

Oil Redistilling Stills. (4 horizontal stills equipped with 2 effective, 38 plate, fractionating columns) This installation (described above) was said to have been built by Carl Still of Recklinghausen.

Tar Acid Oil Extraction. Two vertical, cone bottom tanks which previously had been employed as benzol washers were being used as extractors for the removal of tar acids from tar acid oil. Each tank is filled about one-half to two-thirs full with 20% caustic soda solution. Then the tar acid oil is pumped continuously into the tank through a pipe that extends almost to the bottom of the cone. The oil rises through the caustic soda solution and overflows near the top of the tank, into a run-down tank. The tar acids are extracted from the oil as it rises through the caustic soda. Little or no emulsification takes place because agitation is not employed. When the caustic becomes saturated with tar acids, it is replaced with fresh caustic and the cresylate is sent to Meiderich. The process was developed by the director of the plant, Dir. Ing. George Speckhardt. He provided a copy of his patent application in which the process is described.

Pitch Cooling. Two methods of cooling pitch have been used at this plant. One is an inclined conveyor with overlapping plates. The molten pitch is fed onto the lower end of the conveyor and plates of solid pitch are discharged from the upper end into a railway car. According to Mr. Speckhardt this type of cooler is suitable only for hard pitch (for briquette pitch its capacity is low) and maintenance costs are considerable. The method which Mr. Speckhardt favors, for either briquette or hard pitch, uses an elevated tank for precooling thepitch, a feed drum with several valves and small tubes on the bottom, a rectangular water tank with the bottom portion arranged with sloping sides, and a screw conveyor and perforated bucket elevator discharging into railway cars. To operate this device the liquid pitch to be cooled is pumped to the overhead precooling tank where it is cooled atmospherically to below 300°C (about 230-235°) for hard pitch, and below 150° (about 120°C) for briquette pitch. It is then run into the feed tank and the valves are opened to permit the pitch to run through the bottom openings into the water. The streams of pitch congeal in the water and drop to the bottom of the water tank where they are moved by the screw conveyor to the end of the tank. They are then transported by the inclined perforated bucket elevator to a chute which delivers them, in granulated form, into an open railway car. This arrangement for the cooling of pitch is described in German Patent No. 743678 issued to George Speckhardt, works director of the Carolinenglück plant.

The benzol plant building at this plant had been heavily damaged by air raids but the equipment was not appreciably affected and could be placed in operation again in a rather short time.

Stocks of raw and finished materials on hand at this tar plant were not large. The quantity of crude tar in sorage was only 400 tons.

GELSENKIRCHENER BERGWERKS A.G.
CHEMISCHE BETRIEBE PLUTO-WILHELM, WANNE-EICKEL

The date of the visit to this plant was September 15th, 1945. Works Manager Mr. Slumberger was interviewed.

The plant is divided into the following departments with separate buildings for each. 1. Tar distillation, 2. Cements and coatings, 3. Bituminous paints, 4. Lubricating oils and greases, 5. Reclaiming of used lubricating oils.

The tar distilling plant is small and of the type most frequently used in Germany. It processed approximately 2300 tons of crude tar per month and 400 tons of benzol wash oil and coumarone residues. There were 8, vertical gas fired stills of twenty tons capacity each. Some of the stills and all of the condensers and receivers were damaged or destroyed by bombs. One still with a temporary condenser was operating at the time of the visit. The stills were operated under vacuum to briquette pitch and also to hard pitch, (100°C +). The following fractions were collected from dehydrated tar: (Dehydration continuous in a separate still).

(1)	Water and light oil	0.5%
(2)	Middle Oil 200-235°	10-12%
(3)	Heavy Oil 235-250°	3-5 %
(4)	Anthracene Oil (light) 250-270°	8-10%
(5)	Anthracene Oil (med. heavy) 270-300°	5 %
(6)	Anthracene Oil (heavy) 300°- (360-400)	3-5%
(7)	Pitch 100°-150° K.S.	45-50%

The fractions were handled as follows:

(1) Light oil used in bituminous paints.

(2) Middle oil cooled in pans and drained, 55-60°.
Naphthalene centrifuged or sent to carbon black plants or fuel oil.
Drain oil redistilled to make wash oil, coal flotation oil or oil for shipment to tar acid plants.

(3) Heavy oil cooled in pans. Naphthalene added to that from (2). Drail oil to wash oil or carbolineum.

(4) Cooled in pans, filter pressed; oil used as flux for road tars, steel works tar, refined tar or as fuel. Anthracene residue, (2.0-3.5%) shipped to carbon black plants.

(5) Cooled in pans, filter pressed: Anthracene residue added to that from (4); oil redistilled to make light and heavy fluxes for special purposes.
(6) Used in making lubricating oils after cooling, filtering and redistilling. Also used for flux for roofing and waterproofing compounds.
(7) Hard pitch, shipped to pitch coke plants.

This tar plant specializes in the making of compounds of the type called Plastix in Germany. Hard pitch is fluxed with heavy anthracene oil or a mixture of the latter with hard pitch distillate. Such mixtures have improved temperature characteristics and are used in roofing, waterproofing, coating and paint compounds.

The plant for making cements and coatings is equipped with three horizontal, heavy duty mixers and two smaller, pony mixers for making filled compounds. Soft pitch is mixed with various fillers and in different amounts to make hot application compounds for use on roofs, foundations, tunnels, cables, pavements etc.

The factory for making bituminous paints has five steam jacketed, agitated, vertical mixing tanks with reflux condensers. Coal tar pitches and petroleum asphalts are fluxed with solvents to make metal paints of all kinds.

The lubricating oil and grease factory is well equipped with mixers, coolers and kneaders for making various kinds of lubricants and greases. During the war heavy anthracene oil was used as a substitute for petroleum oil in the formulation of lubricants, greases and puttys. The products were used for heavy duty purposes for which they were considered to be satisfactory.

The plant for reclaiming used lubricating oil settled, dehydrated and centrifuged such oils and returned them to the plants from which they were received.

TAR DISTILLATION PLANT

The König-Ludwig tar distilling plant, near Recklinghausen, was visited by the writer, Mr. Hunter, (civilian officer, Military Government, Gelsenkircken), and Dr. Geiselbrecht on September 27.

The plant officials who conducted the inspection were Westmann (Director of coke plant and tar plant), Flottmann, Assistant Director of coke plant and tar plant, Ahnen, Assistant Superintendent of coke plant, and Klussmeier, foreman of tar plant.

The plant had not been damaged by bombs or artillery fire and operations could be resumed immediately if permission to reopen the plant were received.

Operations were stopped completely on March 21, 1945. Since that time tar has been received at the rate of about 300 tons per month from König Ludwig mine 4/5 which is carbonizing 300 tons of coal per day. This is one-half of the plant's carbonizing capacity. The amount of coal mined per day at this plant is now 3000 tons. Ordinarily it would be 6000 tons but destruction of one of the two hoisting drums cut the production in half.

The tar plant has on hand at this time about 2000 tons of crude tar, 385 tons of anthracene residue, 210 tons hot-pressed naphthalene, 5000 tons briquette pitch, 200 tons hard pitch, 272 tons crude naphthalene, 25 tons naphthalene for naphthalene motors, 68 tons of 40% anthracene, and various grades of oil about 1500 tons.

The number of people employed during the war was 81. No operators are employed at this time, while the plant is shut down. All of the equipment in this plant is well arranged and in good condition.

TAR RECEIVING

Tar arrives at the plant in tank cars which are switched into a large building with two tracks running lengthwise. The building is long enough to accommodate about ten cars on each tract. Between the two tracks in a steel trough into which the tar from cars on either track can be discharged by gravity. The trough delivers the tar to a sump from which it is pumped to storage. It was explained that this tar receiving building was provided to take care of shipments of brown coal tar which König-Ludwig expected to handle because another plant which had been distilling this tar had been destroyed. Brown coal tar becomes more viscous than bituminous coal tar on cooling and the building was provided to keep the tar warm.

TAR DEHYDRATION

Two 50 ton vertical, gas fired stills are used for continuous dehydration of the tar. The crude tar is pumped through a vapor-to-tar heat exchanger or condenser and to an overhead feed tank from which it flows to the two stills. The dehydrated tar flows continuously to receivers from which it is pumped to storage and the vapors pass through tar cooled and water cooled condensers. The combined condensate is decanted continuously to separate light oil (about 1%) and water. The light oil is sent to the benzol plant for refining.

TAR DISTILLATION

The dehydrated tar is distilled to briquette pitch in four gas-fired horizontal stills of 50 tons capacity each. They are constructed like those used by Carolinenglück and Gutehofnungshütte. The heat passes first through two tubes, then back around the lower half of the still and returns to the stack through a horizontal flue located about two feet below the bottom of the still. The stills are not equipped with columns. The only fractions taken to briquette pitch are "creosote", about 24% and anthracene oil, about 18%.

PITCH DISTILLATION

In addition to briquette pitch this plant makes hard pitch, about 150°C K & S, for electrode coke production. The briquette pitch to be run to hard pitch is transferred from the four horizontal stills described above to three, gas-fired, vertical stills, one of 50 tons capacity and the other two of 30 tons capacity each. The 50 ton still is sometimes employed to distill dehydrated tar all the way to hard pitch. The distillation to briquette pitch, either in the horizontal stills or in the 50 ton vertical still is carried out with vacuum and no agitation,

but the distillation from briquette pitch to hard pitch in the three vertical stills is effected by blowing the pitch with air. Some heating is done at the same time. The air with which the pitch is agitated is introduced under about 4-5 atmospheres. The amount of air used was said to be small but the quantity was not stated. The time required to go from briquette pitch to hard pitch is about 4-5 hours.

PITCH COOLING

Cooling of briquette pitch is carried out entirely in steel pans. The pan building has 20 pans of seventy tons capacity each, mounted on a single level above railroad tracks. An arched roof with central ventilator extends over all the pans. Pitch is discharged from the pans into the cars below through narrow chutes arranged like funnels. Fifteen of these pans were used ordinarily for briquette pitch. The other five were used for cooling the hard pitch that was shipped to the pitch coke plant at Lothringen.

Another method of cooling was used for the hard pitch that was added to special low ash coal (30% hard pitch and 70% special coal) for the production of low ash coke in by-product coke ovens. The method of cooling was as follows:

A steel drum, about 15 feet long and 30 inches in diameter, revolves about 30 times per minute in a horizontal position below a liquid, hard pitch distributing manifold and a water distributing manifold. The water falls on the drum a short distance ahead of the liquid pitch, so the latter actually falls onto a film of water. The surface of the drum is divided into channels about four inches wide by means of rings about ½" high and ½" wide. A doctor blade, pressed against the bottom of each channel, removes the thin film of pitch that has been solidified by the thin film of water on which it was deposited; and the flake pitch and water fall from the blades onto water flowing in shallow troughs. The troughs discharge onto a grating made of steel bars placed close enough together to keep the pitch flakes from falling through, but with sufficient clearance to drain the water into a tank below from which it flows through a coke bed to another tank. From there it is pumped back to the chilling roll. The flake pitch slides down the grating and onto a belt conveyor which transports it to a car. A fume hood mounted above the roll conducts objectionable vapors away from the equipment. This chilling device was said to have a capacity of 20 tons per hour when operating on hard pitch. This appears to be a practical pitch cooling device of large capacity.

OIL REDISTILLATION

As stated above, the oil fractions taken when making briquette pitch are "creosote" 24% and anthracene oil, 18%. The creosote is redistilled to make light oil 0.5%, carbolic oil 3.0%, naphthalene oil 10.0%, wash oil 5%, and residue 5% (all percentages on the basis of dehyd. tar). The oil redistillation is carried out in six, 40 ton vertical, gas heated stills equipped with fractionating columns. Each column is 1.2 m. in diameter x 7 m. high (overall) and provided with 12 trays and bubble caps. The top of each column is a four plate dephlegmator. Reflux is controlled by regulation of the cooling water to the dephlegmators.

NAPHTHALENE

The naphthalene cooling building has horizontal drain oil tanks on the first floor, anthracene cooling pans on the second floor and naphthalene cooling pans on the third and fourth floors. Each naphthalene floor has ten pans about 15' x 15' x 4' arranged in two rows of five each. Between the pans on the first naphthalene floor, (No. 3) is a screw conveyor into which the naphthalene may be shoveled from the pans on that floor. Between the two rows of pans on the fourth floor are chutes which discharge into the conveyor on the third floor. The anthracene pans are drained by gravity to tanks on the first floor. By means of a conveyor, the naphthalene from the pans (about 69°C) is transported to the hot press building. It goes into hoppers above two hot presses, drops into their mixers where it is mixed at a temperature of about 30°C (said to be no admixture of molten naphthalene or oil at this plant) and pressed at that temperature.

ANTHRACENE

The anthracene oil from the primary distillation to briquette pitch (without redistillation) is crystallized in the pans mentioned above and in another anthracene cooling house with pans. After crystallation, it is pumped to vacuum pan filters (nütchen) mounted above filrate receiving tanks. The vacuum filters are rectangular pans about 10' x 15' and are not equipped with agitators. The anthracene residue, when removed from the filters, is conveyed to a covered storage outside the filter pan building.

To make 40% anthracene, the anthracene made as described above is dissolved in an equal quantity of wash oil, in a horizontal. steam heated tank, recrystallized, and centrifuged continuously with 2 Hauboldt centrifuges. The amount of 40% anthracene made is not large. The centrifuges and dissolving equipment are located in the same building as the vacuum filter pans.

NAPHTHALENE BRIQUETTES

In a separate building is housed the equipment used for making naphthalene briquettes for stationary engines. Drained naphthalene from the pans mentioned previously is recrystallized in elevated pans, drained, crushed and molded into blocks about 3"x3"x2". The two presses used for this purpose were made by Fritz Kalian, Berlin-Lichtenberg, Herzbergstr 102-104. They operate in the same manner as ball presses.

They were two or three ball presses in this same building and also equipment (not used) that was intended for the continuous extraction of tar acids from brown coal tar oil.

SPECIAL PRODUCTS

A separate building housed mixing and barrelling equipment for heavy greases. A petroleum lubricant was compounded with soap to make heavy greases for use by the König-Ludwig mines. No tar oils were used in these products.

This plant did not make special pitch compounds like plastix.

Gutehoffnungshütte, Zeche Sterkrade near Oberhausen-Sterkrade.

The Sterkrade plant of Gutehoffnungshütte, was visited on September 19th, 1945. The persons interviewed were Kokerei Director Dr. Kleingrotthaus and Kokerei Betriebsführer Ludwig Koerdt.

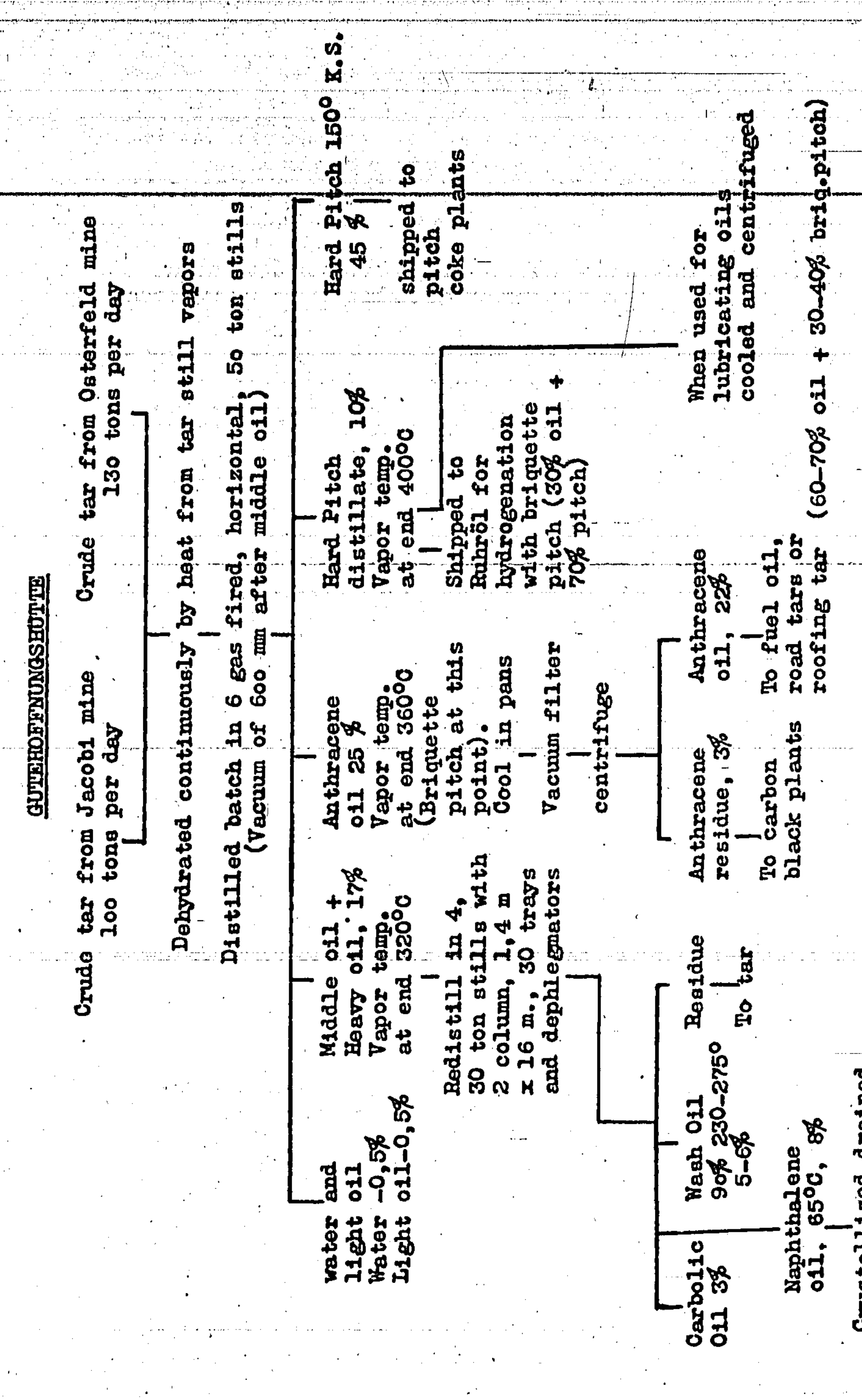
The plant had been damaged by bombs and artillery to the following extent : a naphthalene cooling and hot press building was destroyed by incendiary bombs and one oil redistilling still was damaged by artillery.

Stocks of material on hand were 400 tons of crude tar, about 3500 tons of pitch and about 400 tons of tar oils. Tar receipts from the Jacobi mine amounted to about 50 tons per day.

Crude tar distilled by this plant during the period 1941 - 43 amounted to about 73,000 tons per year. Products made were heavy solvent, carbolic oil, crude naphthalene, wash oil, anthracene residue, anthracene oil, hard pitch distillate, briquette pitch and hard pitch. The operating procedure is illustrated by the accompanying flow diagram.

Briquette pitch is cooled in pans but the hard pitch is cooled in a special device in which the molten pitch, at 250°C, flows from 25 small tubes, about 10 m.m in diameter onto the surface of water flowing in small semi circular troughs about 10 cm wide and 7 m long. The streams of pitch, congealed by the water, flow with the latter to the ends of the troughs where they are separated from the water by a grating. They fall into a car which transports them to pitch coke plants. The cooling water drops into a tank and is recirculated to the troughs. The temperature of the water is about 40°C. The quantity of water used per hour is about 20 to 30 cubic meters. The amount of pitch cooled per hour is about 5 to 8 tons.

For the making of blended mixtures such as road tars and special pitch compounds, two horizontal tanks equipped with mechanical agitators are employed.



TEERDESTILLATION MATHIAS STINNES, ESSEN-KARNAP

Teerdestillation Mathias Stinnes 1/2/3, Essen-Karnap, was investigated on September 21, 1945. The persons interviewed were Bergwerk's Direktor Dr. Broche, Betriebsführer Halfmann and Betrieb's Direktor Rummel.

War damage to this plant was very light and all departments could be operated. The plant was operating at partial capacity when visited. (50 tons per day)

This well arranged and well operated tar plant distilled about 38,000 tons of crude tar per year during the period 1941-43, but in addition it distilled a residue coming from the hydrogenation plant of Ruhrol A.G. Bottrop, an associated company. The quantities of crude tar and hydrogenation residue distilled per day averaged about 100 tons and 140 tons respectively during the war years.

If distilled by itself, the hydrogenation residue would yield about 10% heavy oil and 90% briquette pitch but actually the hydrogenation residue was mixed with crude tar in the above proportions, so the heavy oil from the hydrogenation residue was combined with that from the tar and the briquette pitch was a mixture of pitches from the two sources.

Because the briquette pitch was of mixed origin, this company carried on considerable investigational work to determine and establish its value for coal briquettes. It approximated, but did not quite equal in binding capacity briquette pitch from tar alone.

The operations at this tar plant are carried out in the following equipment:

Dehydration: Tar is dehydrated by using it to condense the vapors from the tar stills.

Distillation: Six horizontal, 50-ton stills heated by gas through two large tubes in each still with return of combustion gases beneath the still before passing to stack.

Oil Redistillation: Two vertical, 25-ton stills, equipped with 800 mm diameter, bell and tray columns, and two vertical, 50-ton stills with 25 tray columns, 1200 mm diameter and 12 m. high.

Pitch Cooling: Steel pans mounted above railroad tracks.

Naphthalene Recovery: Cooling pans, centrifuge and hot presses.

Tar Acid Extraction: Batch extraction in tank with mechanical agitator.

The following are the operations and yields of products at this plant when crude tar alone is worked up. When the mixture of

tar and hydrogenation residue was processed the yields of pitch and anthracene oil were increased and the yields of all other products were decreased.

Tar Dehydration

Water. 4.0%
Light Oil. 0.5%

Tar Distillation

Middle Oil 25%, redistilled Light oil. . . 0.5%
Naphthalene oil 15%, cooled in pans,
  drained and centrifuged or hot pressed crude naphthalene. 8.0%
Naphthalene drain oil 7%, extracted
  with crude caustic soda
  Sodium phenolate (as phenols) 0.5%
  Neutral oil 6.5%
Wash oil 6.0%
Anthracene oil 20%   filter pressed:
  Crude anthracene. 5.0%
  Filtered anthracene oil 19.0%
Briquette Pitch, 70° softening point,K.S..50.0%

HANSEATISCHE TEERPRODUKTE FABRIK, HALTERMANN & CO.
HAMBURG, GERMANY

An inspection of the above named plant on October 10, 1945, was conducted by Mr. Johann Haltermann, owner, and Dr. Alfred Glawe, Director.

It is an old tar-distilling plant but it is the largest and, therefore, the most important plant of its kind in northern Germany. It distilled about 30,000 to 35,000 tons of tar per year during the war. The tar was received from city gas works and also from by-product coke oven plants.

In addition to the tar distillation plant this company has a benzol refining plant in the same location. Bomb damage during the various air raids was large. Mr. Haltermann and Dr. Glawe estimated that about 65% of the plant had been damaged by bombs. The naphthalene refining plant was totally destroyed; about 45 storage tanks out of more than 60 were destroyed or badly damaged; the settings of the tar stills were broken and are now being rebuilt and considerable damage was suffered by several of the buildings.

At the time of this visit one tar still was operating for the production of briquette pitch and various oil fractions.

A new 50-ton oil redistillation still and column is being installed and preparations have been made for the installation of a continuous dehydrating still.

Products:

The products made in the plant were as follows:

Light oil products - benzol, toluol, xylol, solvent naphtha; naphthalene products - hot pressed naphthalene, refined naphthalene, balls, flakes and naphthalene oil for insecticides; anthracene residue for carbon black manufacture; anthracene oil for fuel, road tars and refined pitches; crude, sprung tar acids, shipped to Meiderich and Rauxel; wash oil of regular grade and special wash oil filtered at 0°C (for use at coke plants in naphthalene scrubbers); briquette pitch; road tars and roof cement.

Manufacturing Procedures and Equipment:

The crude tar was dehydrated and distilled in two 25-ton vertical batch stills and two 40-ton vertical batch stills. The

stills were operated under approximately 600 mm vacuum. The primary fractions were: Water 5.0% and light oil 0.2%; middle oil 10%, anthracene oil 20%, and briquette pitch 55% (all yields approximate). The light oil was worked up in the benzol refining plant together with light oil brought in from coke oven plants (about 6000 tons per year). Steam heated stills equipped with bell and tray columns were used for this purpose. One still, used especially for the fractionation of toluene, has two columns in series. It was said to be very satisfactory. The fractionation and washing operations as carried out in this plant, were like those of other plants of the same kind. Disposal of the benzol sludge resulting from the sulphuric washing of various light oils was carried out as follows: 2 parts of sludge were mixed with one part light oil (boiling range 200-250°C) and, while mixing, additional water was added. The mix was then allowed to settle, after which the dilute sulphuric acid was drained to the sewer and the oil layer was washed with a solution of sodium carbonate. After settling again, the sodium carbonate solution was drained to the sewer and the oil layer was pumped to crude tar storage.

The middle oil resulting from the primary distillation was redistilled in a 50-ton vertical still upon which was superimposed a 40-tray column, two meters in diameter. There are 42 bells, 200 mm in diameter, on each tray. The still was operated under 65-70 cm vacuum. The fractions collected were:

Crude benzol - sent to light oil plant.

Carbolic oil - washed with NaOH and then redistilled for additional benzol and solvents in the light oil plant. Phenolate sprung with H_2SO_4. Sodium sulphate to sewer and the crude phenols to Rutgerswerke at Rauxel.

Naphthalene oil - cooled in pans and drained, hot pressed, washed (in 2 lead-lined washers), redistilled and made into balls, flakes, etc. The naphthalene oil was sold to makers of creolin (Pierson) and insecticides (Altenlander Pflanzenschutzmittel Fabrik and Staedter, Holland and Stade). The naphthalene washing was carried out with 6 successive washes with 66°Be H_2SO_4, two washes with 10% NaOH, two water washes and a final wash with 10% NaOH. Washing was followed by a final redistillation in a steam heated still, under vacuum and fractionated with a bell and tray column. The naphthalene sludge was disposed of in the following manner - all of the washes (H_2SO_4, NaOH and H_2O) from the washing operation described above were collected together in an acid-proof, brick-lined, rectangular concrete tank. More water was added (about 10 parts water to one of sludge). After settling, the supernatant aqueous portion was run to the sewer (Dr. Glawe said it was practically neutral) and the mixture of naphthalene and resins was shovelled from the tank and burned under the boilers.

Wash oil - A part was sent directly to coke plants for light oil recovery and the remainder was refrigerated to 0°C, filter pressed and shipped to coke plants for scrubbing naphthalene from the gas.

Residue from oil redistillation - added to filtered anthracene oil. Anthracene oil from the primary distillation was cooled in pans, filtered in vacuum filters and used in road tars or for making prepared tars. The anthracene residue, filtered from the anthracene oil, was sold to the carbon black plants.

A special roof cement was made for one customer. The composition of this material was as follows:

Coal tar pitch, 200°C K&S -	53-79%
Heavy benzol, 160-200° -	6-7 %
Asbestos fiber)	
Kieselguhr)	15-40%

The composition of the cement was varied to suit climatic conditions. It was used as a heavy roof-coating material.

In addition to the equipment discussed above for the manufacture of tar products, this company has a separate department for redistilling a light petroleum distillate, imported from America. From it are made gasoline and various solvents for the

rubber, paint, chemical, vegetable oil and other industries. Steam heated stills provided with effective fractionating columns are used for this purpose. The columns are bell and tray with pump-back reflux. (All other columns in the plant use dephlegmators.)

Mr. Haltermann commented on the fact that the wooden roofs on the benzol refining and benzine refining buildings had proven to be superior to steel roofs during the air raids. They burned and fell on the stills but they did not collapse the walls (as steel roofs did in other cases) and the stills and receivers (although full of flammable materials) did not ignite because they were heavily insulated.

AKTIENGESELLSCHAFT JOHANNES JESERICH, HAMBURG, GERMANY

TAR DISTILLATION AND MANUFACTURE OF BITUMINOUS AND RESINOUS PAINTS AND COATINGS

The plant of Aktiengesellschaft Johannes Jeserich, Zweiniederlassung, Hamburg, Germany, was visited on October 13, 1945, in order to find out what products are made by this company from coal tar.

The only person interviewed was Johannes Jeserich, the owner of the plant, who also took the writer through the plant and explained its operations.

The plant had been heavily damaged by one bombing attack of about ten minutes duration. The roofing felt plant, warehouse and other buildings were completely destroyed. The tar receiving equipment, tar storage tanks and tar stills were not damaged appreciably and are in operation. Also the laboratory building (used also as an office building), the plant formerly used for distilling wood distillates and a small varnish cooking plant were largely undamaged and are still usable.

There were practically no stocks of raw materials or finished materials on hand at this plant and Mr. Jeserich was anxious to obtain more raw materials so that he might increase those operations which can be carried out with the remaining equipment.

Crude coke oven tar is distilled in five vertical stills of ten-tons capacity each. One still is provided with a mechanical agitator, vapor vent to the atmosphere and a manhole on top. This still is used for making paints and mixtures as well as for tar distillation. The maximum capacity of this tar distillation plant is 100 tons per week. Approximately two days are required for the dehydration and distillation of each batchof tar.

The primary products of the distillation are as follows:

Water, average about 5%, but recent shipments have contained as much as 30%

Heavy benzol, about 2%. It is used with additional heavy benzol purchased from others to make paints.

Middle Oil, 16%-18% } The middle oil and heavy oil are combined
Heavy Oil, 10%-12% } with pitch to make saturating tar for the roofing paper.

Briquette pitch (55°-60° K & S) 60%-65%, used for making paints, prepared tars, saturating tars, etc.

The names of the more important coal tar products of this firm and their approximate compositions are as follows:

Beton-Lubrose-Schwarz

60% pitch (55°-62° K & S) + 40% heavy benzol. Used for the protection of concrete structures against industrial waters, moist earth, etc. Applied cold.

Heiss-Lubroze, Schwarz and Rotbraun

95% pitch (55° K & S) and 5% asbestine. For hot application to concrete structures, foundations, walls, etc.

Carbolineum, Nussbraun

A mixture of middle and heavy coal tar oils. For brush or spray application to wood for protection against fungus.

Jeserit-Carbolineum, Farbig

An emulsion containing water, pigment, drying oils, and a small amount of carbolineum. For combined painting and fungus protection of wood.

Steinkohlenteer, Präpariert, Strassenteer

Partially distilled tars or combination of pitch, heavy oil, and middle oil. Used for the impregnation of roofing felt, construction and maintenance of roads, etc.

Klebemasse

Pitch cement and coating for use with the roofing paper.

Some of the products made from petroleum bitumen (asphalt) are:

Beton-Lubrose 714 b

55% bitumen (70°-80°C K & S) + 45% benzine. Used for drinking water containers, reservoirs, tanks, etc.

Lubroplast A 111, Schwarz

#1, 55% bitumen (45°-50° K & S) + 35% benzine + 10% asbestine
#2, 55% " (45°-50° K & S) + 45% benzine

Jeserit-Bitumen-Emulsion D.R.P. 399557 Schwarz

52% Bitumen (45° K & S) + Emulsifier"

Aluminium-Bitumen-Lubrose, Silbergrau

80% Beton-Lubrose 714 b + 20% aluminium powder

Silofarbe 1089, Schwarz

50% bitumen, 90°-110° K & S. + 50% benzine

In addition to the above bituminous materials, this company made a phenolic resin paste called "copalit" for use on gaskets for pipes and machinery and also a wide variety of colored paints in which before the war wood rosin, wood oils, and linseed oil were used. During the war these were not obtainable and Albertol synthetic resins were used.

One of the principal products of the plant was roofing felt. It was saturated with tar, coated with filled pitch and covered with sand or granules.

CONCLUSION

From the standpoint of tar distillation this company offers little that is new or different from usual practice in Germany. Apparently

Note: (*) Rosin + NaOH before the war, bentonite during the war.

little investigational work has been done and no effort has been made to produce improved coal tar paints, coatings, road tars, pitches, etc. The roofing felt could not be evaluated because none was on hand and the plant had been destroyed.

BINNE AND SON
HAMBURG, GERMANY

TAR DISTILLATION AND ROOFING FELT MANUFACTURE

The plant of Binne und Sohn, Pinneberg (near Hamburg), Mühlenstrasse 60, was visited on October 13, for the purpose, primarily, of seeing a plant where the manufacture of roofing felt is the principal line of business.

The persons interviewed were Dr. Hugo Binne and Mr. Wuerner Binne. They are co-owners of the company with their father. The business is quite old, having been founded by their grandfather, but the plant is modern with respect to its equipment as compared with other plants of this type that had been visited previously.

This plant was not bombed at any time and all parts of it were in good operating condition. However, it was running at partial capacity for lack of sufficient raw materials. No stocks of finished materials were on hand and there was only enough crude tar for two days operation. The owners had been given no indication as to when more crude tar might be made available to them.

The following information was obtained from the inspection of the plant and from discussions with the Messrs. Binne.

Tar dehydrated in the tar stills is mixed with pitch made in the same stills to produce a saturant for the roofing felt. For the coating material, to which sand is applied, pitch is mixed with 30% Kieselgubr or 40% - 45% of calcium carbonate. The saturant and coating are produced as they are needed by the felt plant.

By-products of the tar distillations to make saturant and coating are light oil, sodium phenolate, and a mixture of neutral, middle oil, and heavy oil. The light oil is used for making black varnishes from coal tar pitch or petroleum bitumen, the sodium phenolate is sold to Rütgerswerke for the recovery of tar acids and the mixture of neutral oil and heavy oil is sold as carbolinum. During the war it was furnished to the Navy for use as fuel.

Before the war this plant made both asphalt and tar roofing felts but during the war (as at present) only the tar product was made because no asphalt was available for this purpose.

The equipment used for the various operations is as follows:

TAR DISTILLATION

There are two ten-ton, vertical, steam-heated flat bottom stills equipped with mechanical agitators and vapor connections to water-cooled coil condensers which discharge directly into small receivers. The stills are also connected to a vacuum pump and vacuum of about one-half atmosphere is maintained during distillation. The agitators are used only when the stills are being used as mixers for making black varnishes from pitch and light oil or from bitumen and benzine.

FELT SATURATING MACHINES

Two felt saturating machines are housed in separate buildings. Each consists of a saturating tank, a coating and sending section, a looper and a winder. Both machines were made by Otto Kinne, at Halle/Saale. Each machine has a daily capacity (24 hours) of 30,000 square meters or about 90 tons of finished roofing.

MIXERS FOR COATING COMPOUND

One of the saturating plants has a horizontal, steam-heated mixer for making the coating compound. The other plant has two horizontal, coal-fired mixers for the same purpose. Previously the steam-heated mixer was used for pitch coating manufacture and the coal-fired mixers for asphalt coating compound. The latter are now used for pitch compound making and the other plant is idle. Each mixer holds about five tons of finished coating material.

MISCELLANEOUS

The remainder of the plant consists of office, boiler room, service building, and shop. Sand (0.6 to 1.0 mm.) for coating the felt is dried in steam-heated pans.

CONCLUSION

This plant is well equipped for the making of tar saturated and coated roofing felt. The plant is in good condition but lacks an adequate supply of crude tar for the making of the saturant and the coating. The type of roofing felt made at this plant is like that made at other tar plants in Germany such as Ges. fur Teerwertung, Meiderich, and F. Raschig at Witten. The same type of product ordinarily is not made in the United States from coal tar.

From the standpoint of tar distillation (included only to take care of the requirements of the felt plant), this plant is unimportant.

NORD DEUTSCHE KOHLEN UND COKES WERKE A.G.

Hamburg

Nord Deutsche Kohlen und Cokes Werke A.G. was visited on October 13 1945. After meeting the owner and manager, Mr. Erik Blümenfeld, at his office in Chile House, Section B, Hamburg, the tar plant at Hamburg-Freihafen was inspected. Director Johannes Plückthun assisted Mr. Blümenfeld in explaining the operations of the plant.

The entire tar distillation plant is completely undamaged and ready for operation when sufficient tar is supplied or produced to keep the still operating without interruptions. Formerly, the tar came from two adjoining batteries of coke ovens belonging to this same company. One of the batteries (Koppers) was heavily damaged by bombing and will have to be rebuilt. The other battery (Otto) is useable and sufficient by-product equipment soon will be operable to permit its use if permission to start operating again is granted.

The continuous tar still in use at this plant was the first of its kind built by H. Koppers of Essen. When it was installed in 1935 it was intended to be both a pilot plant and a commercial tar-distilling unit. Mr. Blümenfeld said that it had been completely satisfactory in both respects. It had established the suitability of the design for continuous tar distillation and it had always been operated with profit to the tar company.

The capacity of the plant is only 25 tons of crude tar per day. The principal products and their approximate yields are given on the accompanying flow diagram. It also shows the arrangement of the equipment and the approximate temperatures of the tar. oil vapors, and residues in different parts of the plant.

The flow diagrams show a steam connection (dampf) to the pitch blower (verblaser). The steam line is also connected to compressed air so that either steam or air may be used for blowing the pitch. When a maximum yield of anthracene oil is wanted, steam is used. When more pitch and less anthracene oil is wanted, air is used for blowing and, by polymerization, the pitch yield is increased. The pitch yield also is increased, at the expense of heavy oil, by returning a part of the latter as it comes from the bottom of the naphthalene column to the top of the pitch column. A part of this oil leaves the bottom of the column with the pitch and is converted into pitch in the verblaser by the air-blowing operation.

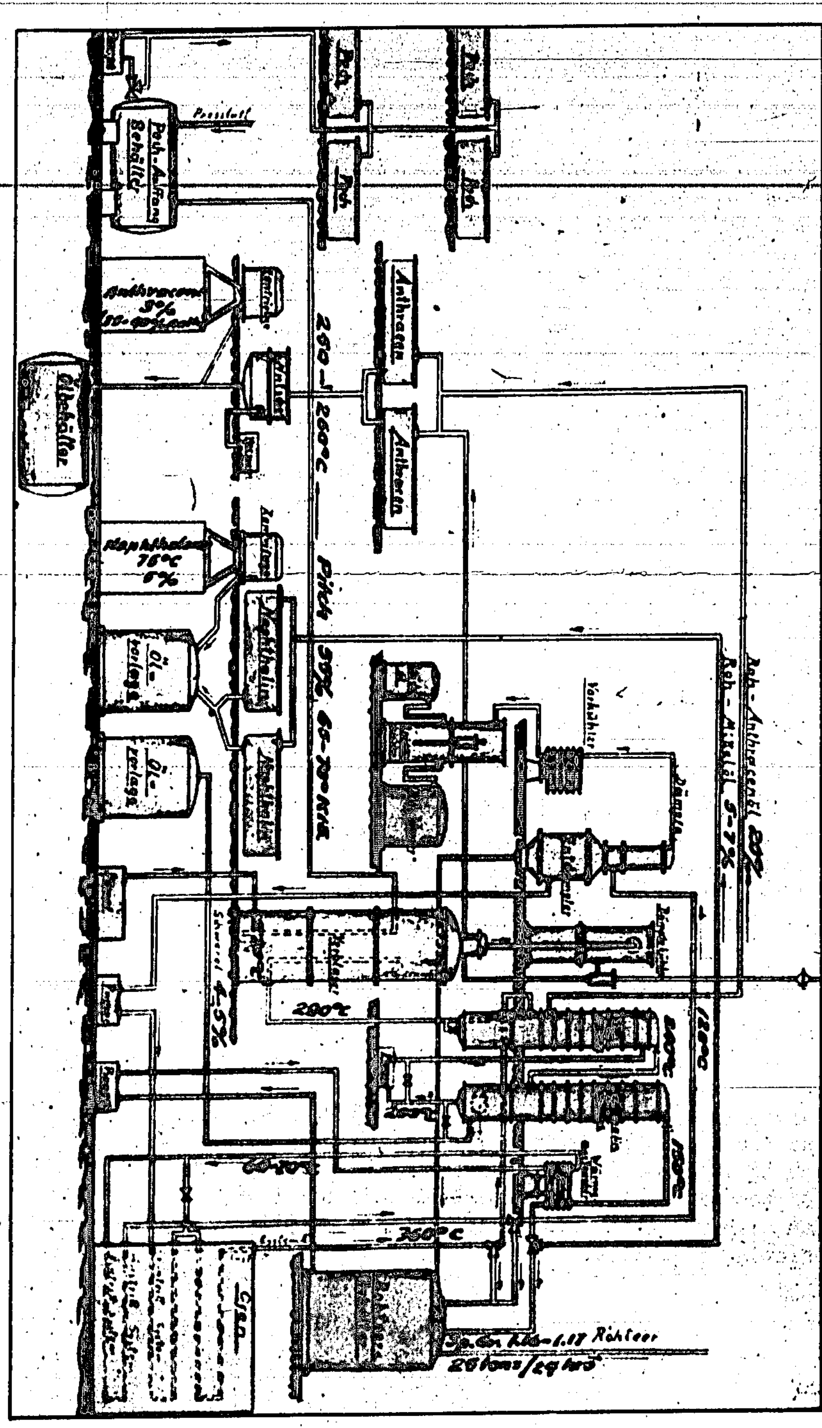


Fig. 1 - FLOW DIAGRAM

The light oil from this plant was combined with that from the coke ovens and worked up in the benzol refining plant. The middle oil was crystallized in pans to recover naphthalene, which was centrifuged and marketed as crude naphthalene. The naphthalene drain oil was sold to other tar distillers who extracted and refined the tar acids and combined the neutral oil with that from their own production. The heavy oil was used as wash oil by the coke plant and the anthracene oil, after cooling and filtering to recover anthracene residue, was sold for hydrogenation or fuel oil purposes. The anthracene residue recovered by crystallization and filtering was centrifuged before shipment to carbon black makers. Its anthracene content was said by Director Plückthun to be about 35-40%. The briquette pitch was used in the briquetting plant owned by this same company where fines from its coal-washing operations were mixed with the pitch and briquetted.

The tube still contains two sets of tubes - one for dehydration and the other for distillation. All of the tubes are made of chrome steel. In the dehydrating section, according to Director Plückthun, the life of the tubes is about four to five years. In the distillation section it is two to three years.

The pipe still is heated with gas having a heating value of 4,500 kilogram calories per cubic meter. The gas consumption is about 55 cu.m. per ton of crude tar (containing about 3-5% water, and having a specific gravity of 1.16-1.17).

Conclusion

With its combination of coal-washing plant, coke plant, tar plant, and briquette plant, this company's operations are well integrated. Incoming coal (by water) is washed. The coarse coal goes to the coke plant and the fines to the briquetting plant. Coking of the coarse coal produces coke, tar, etc. The tar is distilled to make oils and pitch and the pitch is used to briquette the fine coal coming from the coal-washing plant.

The tar plant is arranged for efficient operation and is said by the owner to have been entirely satisfactory. No unusual features were observed other than the use of steam or air in the "verblaser" to vary the pitch and anthracene oil yields.

DR. F. RASCHIG G.m.b.H., LUDWIGSHAFEN am RHEIN

The Ludwigshafen plant of Dr. F. Raschig G.m.b.H. was visited on December 6th and 7th.

The persons interviewed were: Dr. Kurt Raschig, Dr. Klaus Raschig and Dr. Thönnessen. Dr. Kurt Raschig is the tar specialist; Dr. Klaus Raschig's speciality is synthetic resins, and Dr. Thönnessen is the specialist on organic derivatives.

This plant was bombed several times and was seriously damaged. For example, the synthetic phenol plant was 95% destroyed, the naphthalene cooling house 100%, warehouse 80%, tanks 80%, synthetic resin plant 60%, power house 60%, hydrazine plant 20%, pitch building 100%, boiler house 30%, etc. The building containing the four continuous tar stills was not destroyed and all of them could be operated if sufficient tar were available.

Tar Distillation

The tar stills in use at this plant were developed by Dr. F. Raschig, the founder of the company. They have been described in the literature so no detailed description of them will be given here. Briefly, however, each still consists of three, shallow, cast iron vacuum pans mounted high enough above the ground so that all distillates and residues can be dropped to the first floor by barometric legs and withdrawn at atmospheric pressure. Each pan is about five feet wide, nine feet long and eight inches deep. Baffles within the pans cause the tar to travel back and forth while passing from one end to the other. A pipe coil lying in the bottom, between the baffles furnishes the heat for dehydration and distillation. The first two pans are heated by steam at 12 atmospheres pressure and the third pan is heated by hot water, under high pressure in a coil heated by coke. The total amount of steam used in the first two pans is about 150 kilograms per ton (metric) of tar. The third pan requires about 15 kilograms of coke per ton of tar distilled for heating the high pressure water. The first pan operates at atmospheric pressure and is not provided with a fractionating column. In it the water and light oils are removed from the tar. The temperature leaving the still is 160°C. The second and third pans operate under 600 to 700 mm. vacuum and are equipped with columns packed with Raschig rings. Naphthalene oil comes from the bottom of the column on the second pan and carbolic oil from the top. The column receiving the vapors from the third pan discharges anthracene oil from the botton and wash oil from the top. All of the condensates are discharged by barometric legs and seal pots to atmospheric receivers. Briquette pitch

from the third still flows by gravity through a barometric leg which also is a pitch-to-crude tar heat exchanger. From this heat exchanger the pitch flows to pans or split molds and the crude tar goes to the steam heated, atmospheric, dehydrating still.

One complete distilling unit of the kind described above has a capacity of about 40 tons (metric) of crude tar per day or 8800 gallons. The fuel, power and water requirements per ton of tar are as follows:

Steam - 150 kilograms
Coke - 15 kilograms
Power - 4 K.W.
Cooling water - 4 cubic meters

The Raschig plant has four units of the type described above for the distillation of coal tar. They are housed in one building which was not damaged by bombs, although an adjoining building was completely destroyed. The men directly in charge of the tar plant are Dr. Hummel and Dr. Klamath.

Pitch Cooling

Two systems of pitch cooling are used at this plant, viz., split molds and tilting pans. The split molds were filled in the usual manner on a covered platform. The roof was destroyed but the platform is still useable.

The tilting pan installation is comparatively new. In principle, it is like the one used by Gesellschaft für Teerverwertung at Rauxel. Fifteen shallow, steel pans holding about four tons of pitch each are mounted beside a railroad track high enough above the ground so that the pitch will drop directly into a car when the pans are tilted. Each pan is tilted by a telescoping hydraulic jack, which moves the pan to an angle of 40 degrees with respect to the ground. Although the inside of the pan is coated with lime before it is filled, a small hydraulic pusher is provided at the center of each pan to start the movement of the pitch if it tends to stick. It is sufficient to loosen the pitch in one place to dislodge the entire charge.

Batch Tar Still

In addition to the four continuous stills described above the Raschig plant has one fifty-ton, vertical tar still of the usual design for producing special grades of pitch or road tar without disturbing the continuous operation of the Raschig stills to briquette pitch.

Compound Mixing Equipment

Two horizontal, coke-fired mixers with mechanical agitators are used for making special pitch compounds, such as paving fillers, paints, etc. The mixers are charged by hand-operated hoists and they discharge by gravity into drums or other shipping containers.

Oil Redistillation

For redistilling contaminated tar acid-naphthalene oils received from others or for redistilling naphthalene oil from which the tar acids have been extracted, two continuous Raschig units are used in a special installation provided for this purpose. Each unit consists of one steam heated, vacuum pan with packed column. When used for the redistillation of tar acid-free naphthalene oil the latter is pumped to the vacuum pan at the rate of 1000 liters per hour. The fraction from the top of the column, boiling below 200°C, goes to the light oil plant and the fraction from the bottom of the column (200-240°C) to the naphthalene pans. The residue from the still is returned to crude tar. The condensates and residue discharge through barometric legs to atmospheric receivers.

Naphthalene Recovery and Refining

The redistilled naphthalene oil, free of tar acids and tar bases, was cooled in sixty-five pans of 10-12 cu.m. capacity each. After draining, the naphthalene was centrifuged to make 70° + crude or hot pressed to make 79° crude. Refining of crude naphthalene was carried out in the usual manner, using two lead-lined washers (5 cu.m.) operated under 600-700 m.m. vacuum and provided with 1.2mx6.0m packed columns. The naphthalene refining equipment was not damaged during the war but the naphthalene pans and hot presses were destroyed.

Light Oil Refining

The light oil refining plant contains five 10-ton steam heated stills and two 20-ton stills. All the stills are equipped with packed columns. Two large washers and one small one are located on an elevated platform outside the light oil building.

Pyridine Recovery

Pyridine bases recovered from the tar acid-free, naphthalene oil in the usual manner are distilled in two five-ton steam heated stills with packed columns, about 8 m. high and 65 cm. diameter. The grades of pyridine produced are pure pyridine, "test" pyridine (denaturing grade), and tar bases boiling above 160°C.

Tar Acid Plant

Recovery of crude tar acids from freshly distilled carbolic oil is effected continuously by counter current flow of the oil and 8-10% caustic soda solution through a vertical tower 1m x 16m filled with Raschig rings (35 m.m.). The oil feed rate is about 1400 liters per hour and caustic rate about 2000 to 2400 liters per hour.

CO_2 from vertical, batch lime kilns was used for springing crude tar acids. Batch causticizers and a batch filter were used for recausticizing the sodium carbonate. The $CaCO_3$ from the filter was used to neutralize acid wastes leaving the plant.

The crude tar acids are distilled continuously to make a rough separation of phenol and cresols. Two Raschig stills of the same size and type as those described above are used. The feed rate is 600 to 700 kilos per hour. The first still heats the tar acids to 160°C and removes the water (without column). The second still, with packed column, removes overhead a mixture containing about 80% phenol and 20% cresol. The residue from the still consists of cresols and xylenols.

The phenol fraction from above is redistilled in eight five-ton stills using steam at 12 atm. for heating and 650-720 m.m. vacuum. Columns are about 12m. high by 65 cm. diameter. They are filled with 25 m.m. Raschig rings.

For cresols ten five-ton stills are used with packed columns about 14 m. high, also one 50-ton still with a packed column about 14 m. high and 120 cm. diameter.

Residues from the cresol stills are redistilled in four 5-ton stills with 14 m. packed columns and the residues from these stills are further distilled in one horizontal, 20-ton still equipped with a mechanical agitator. The still is heated by 12 atm. steam in a jacket and a vacuum of about 600 m.m. is maintained in the still. The distillate is cooled by a water cooled condenser. The residue just fluid enough to run out of a large gate valve in one end of the still, is loaded into steel drums and finally is returned to crude tar.

Symmetrical xylenol is made by cooling the fraction from the xylenol stills in steel cans about 30" x 8" lowered into a water bath. After crystallization of the xylenol, the cans are warmed in a steam box, inverted over an inclined table and the solid xylenol is crushed and centrifuged.

Chlorination of Tar Acids

Raschig chlorinates tar acids either with chlorine or sulfuryl chloride but prefers the latter because it is more easily controlled and gives a more favorable yield of the monochlor compound. The chlorinating equipment consists of four vertical lead-lined tanks about 3 m. high and 1 m. diameter. Each tank has a working capacity of 1700 kilos. A jacket is used either for cooling or heating. The lead-lined top is removable for inspection and is vented into tile pipe which discharges fumes into a scrubber consisting of a tile-lined wooden tower filled with ceramic Raschig rings sprayed with water.

The chlorinating agent is introduced through a small pipe running to the bottom of the tank and the contents, after chlorination, are discharged through the top of the tank by air pressure. There is no outlet on the rounded bottom of the tank.

The chlorinated mixture from the above reaction tanks is transferred, by air, to three 5-ton lead-lined stills with columns 60 cm. by 12 m., heated by steam and operated under vacuum.

Parachlor metacresol is made in the following manner: Meta-para cresol is chlorinated with sulfuryl chloride. The reaction mixture contains parachlor metacresol, paracresol, metacresol and a small amount of dichlor metacresol. This mixture is fractionated to obtain cresols, parachlor metacresol and pitch. The parachlor metacresol is dissolved in xylol and crystallized in three lead or enamel-lined half round, jacketed crystallizing pans. The crystallized product is then centrifuged to obtain pure parachlor metacresol.

Dinitro-Orthocresol

This chemical is made by Raschig in the following manner: Orthocresol is sulfonated (with sulfuric acid in excess at 50-60°C) in two horizontal sulfonators equipped with mechanical agitators. The sulfonated cresylic acid is transferred to small wooden tanks lined with acid-proof brick where it is mixed with sodium nitrate and water, using a wooden paddle for stirring the mixture. A large amount of water is added for cooling and to crystallize the dinitro orthocresol.

When the crystalline product is formed, it is filtered by gravity through a canvas filter supported by a wooden grid. The wet dinitro orthocresol is shoveled into wooden containers and is shipped wet to avoid hazards involved in handling the dry product.

Dinitro paracresol is made in the same manner, by the Raschig plant, starting with paracresol. The equipment used for making these products was not damaged.

Xylene Musk

Xylene musk is made by the Raschig company in the following manner: 22 kilograms of isobutylene (made by dehydrating isobutyl alcohol at 200°C with alumina as catalyst), is reacted with 70 kilograms of metaxylene and a small amount of hydrochloric acid with aluminum chloride as catalyst at a temperature below 5°C. The impure isobutyl xylene, made in this manner, is washed with water to remove HCl and fractionated to obtain forerunnings, 50 kilograms of isobutyl xylene and a residue. The isobutyl xylene is nitrated with a nitric acid-sulfuric acid mixture and cooled on ice to obtain 85 kilograms of crude musk. The latter is dissolved in benzine, distilled with direct steam, to remove the benzene and impurities and the musk, amounting to 85 kilograms, is recrystallized twice from pure alcohol. The final yield of pure musk is 68 kilograms.

Ketone Musk

For ketone musk, 80 kilograms of isobutyl xylene is reacted with 30 kilos of acetylchloride and 25 kilos of aluminum chloride for one to two hours at 20°C. The reaction mixture is cooled on ice and the yield of ketone is 35 kilograms. The latter is nitrated with 98% nitric acid at -5°C, stirring for one hour, and the crude ketone musk is cooled on ice. It is washed with sodium carbonate, recrystallized twice from pure alcohol and centrifuged. The yield of ketone musk is 26 kilograms.

Ambrette Musk

Pure metacresol is methylated with methyl sulfonic acid. The methyl compound is butylated with isobutylene using aluminum chloride as catalyst, fractionated, nitrated and crystallized from benzine to produce ambrette musk.

Methylphenyl Ether (Anisole)

To make anisole the Raschig plant reacts methyl sulfonic acid (made from methyl alcohol and sulfuric acid) with sodium phenolate. The reaction product is methylphenyl ether or anisole.

Paracresol methylether is made in the same way, starting with paracresol.

Chlorthymol

For chlorthymol, parachlor metacresol made as described above, is fused and reacted with isopropyl chloride using $Fe\ Cl_3$ as catalyst. After several hours, the reaction product is washed with water to dissolve the $Fe\ Cl_3$. It is then fractionated in vacuum to obtain forerunnings, consisting of parachlor metacresol, chlorthymol and a residue.

Xylene musk, ketone musk, ambrette musk, phenyl ethyl ether, paracresol methylether and chlorthymol were made only in small quantities by Raschig before the war and production was negligible during the war. The equipment was all small and of pilot plant type but well constructed and arranged for convenient and efficient operation. Most of this equipment was destroyed or badly damaged by bombs.

Hydrazine

Dr. F. Raschig G.m.b.H. claims to have originated the commercial manufacture of hydrazine in Germany and has made this product for many years. The hydrazine plant was operated at full capacity until almost the end of the war when it was damaged to some extent. The process consists in reacting NaOCl (made from chlorine and caustic soda) with ammonium hydroxide to obtain the hydrazine and sodium chloride.

Synthetic Phenol

The Raschig process for the catalytic synthesis of phenol at atmospheric pressure using hydrogen chloride and atmospheric oxygen to form chlorobenzene which, on hydrolysis with steam, gave hydrogen chloride for reuse and phenol, was developed at this location. A fairly large plant for carrying out the process was operated for several years. However, operation was stopped early in the war for lack of benzene and later the plant was 95% destroyed by bombing.

Synthetic Resins

An extensive line of phenolic resins was made, including cast resins, resins for cementing, for plastic masses, for binding paper (hard board resin) for paper manufacture, for grinding wheels and water soluble plywood glues. Novolak also is produced and this company claims to be the only producer of Novolak using an alkaline catalyst. A horizontal, steel reaction kettle with mechanical agitator is used for Novolak production. The temperature used is 160°C. This information was reported to a resin specialist who will investigate the Raschig resin plant more thoroughly and obtain further information about the Novolak production.

The resin plant was partially destroyed by bombing but the Novolak department was in operation when visited. Some of the remaining resin kettles were being used for making disinfectants.

Disinfectants

Previously Raschig furnished chlorinated cresols and xylenols to disinfectant manufacturers who made high coefficient disinfectants. At present, the chlorinated tar acids cannot be furnished to the manufacturers who are in another zone of occupation and the products from those manufacturers are not available in the French zone so the Raschig company is making large quantities of disinfectants themselves. Tin cans and plastic bottles previously intended for other purposes are being used for the distribution of these disinfectants.

DR. F. RASCHIG G.M.B.H., LANGENDREER

The plant of Dr.F. Raschig G.m.b.H. at Zweigniederlassung, Langendreer (near Bochum), was visited on September 19, 1945. The manager, Dr. Heuck, was interrogated and the plant was inspected with him.

This plant is quite old and relatively unimportant both from the standpoint of production and improved manufacturing procedures. During the period 1941-1943 the amount of crude tar distilled was about 38,000 tons per year.

Crude tar is dehydrated continuously by using the crude tar to partially condense the vapors from the tar stills. The dehydrated tar is then distilled in six vertical, coal-fired, 20-ton tar stills under 600 mm. vacuum.

The primary products are light oil 0.5%, middle oil 4.0%, heavy oil 10-12%, anthracene oil 12-15%, briquette pitch 65%, or when hard pitch is made, hard pitch distillate 10% and hard pitch 55%.

The middle oil is redistilled under 600 mm. vacuum in two 30-ton vertical batch stills with 1 x 12 m packed columns to obtain carbolic oil and a residue that is added to heavy oil. The carbolic oil is crystallized in pans and centrifuged to obtain crude naphthalene and carbolic oil (1.2%). The latter is shipped to the Raschig plant at Ludwigshafen.

The heavy oil is redistilled in three 20-ton vertical batch stills with 1 x 6 m. packed columns to produce heavy carbolic oil, benzol wash oil (to coke plants), and residue (used in pitch compounds). The heavy carbolic oil is crystallized and centrifuged to obtain crude naphthalene (total 6%) and crystal-free, heavy carbolic oil. The latter is extracted with caustic and the cresylate is shipped to Raschig at Ludwigshafen. The extracted oil is mixed with anthracene oil and sold for fuel purposes.

The anthracene oil is cooled in a vertical crystallizer with agitator and centrifuged to obtain anthracene residue and anthracene oil. The former went to carbon black plants and the latter to fuel oil.

The briquette pitch is cooled in bays or split molds and is shipped to briquette plants.

The hard pitch distillate is shipped to hydrogenation plants and the hard pitch to pitch coke plants.

Some of the coatings and compounds made at this plant were produced as follows:

Felt saturant:	Distilled tar, about 20° K.S. for summer and 10° K.S. for winter.
Felt coating:	80% distilled tar, 25° K.S. and 20% stone dust filler.
Roofing pitch:	(Klebemasse), soft pitch +0-+5°C.
Clay tube coating mass:	60° K.S. pitch 50-52% and 48-50% lime stone dust filler.
Black paint for iron:	60%, 60° pitch and 40% heavy benzol.
Colored carbolineum 91%:	(Anthracene oil 60% and water 40%), sulfite lye 3%. and colored pigment 6%.

TEERDESTILLATION STUTTGART G.m.b.H.

Teerdestillation Stuttgart G.m.b.H., Stuttgart-Geisburg im Kienle #16, was visited on November 29. The tar plant is located on the same property as the gas plant and works up the tar that is produced there. The amount of crude tar processed in 1943 was about 15,000 tons.

The manager of the plant is Mr. Hall, but he was not at the plant at the time of this visit. The only persons interviewed were Miss Stenipfle, acting chemist, and two of the plant foremen. Miss Stenipfle said that the two men who had been responsible for running the tar plant and laboratory had been removed because they had been members of the Nazi party, and she was not well acquainted with the operations or control of the plant.

The tar plant was not running because of lack of tar. The gas plant had been severely bombed and the coke ovens were damaged to such an extent that only a few were back in operation when the tar plant was visited. The production of tar at that time was only three to four tons per week.

The equipment of the plant consists of three stills, three mixing tanks for road tars, one tar acid extracting tank, and a pitch cooling shed. One of the tar stills is of the continuous Koppers type. A gas heated pipe still and column arrangement similar to that of Nord Deutsche Kohlen und Cokes Werke at Hamburg is employed. This unit runs to briquette pitch and its capacity is about 18 tons of crude tar per day. The other two units make only road tars. Their capacity is about 18 tons and 25 tons of crude tar per day respectively. Each of these distilling units consists of a steam heated still with a vapor to tar heat exchanger on top which partially cools the vapors leaving the still and at the same time dehydrates the crude tar entering the still. In each type of installation the light oil from the dehydration of the tar is recovered separately and the tar acid and naphthalene oils are combined. Wash oil and anthracene oil fractions also are recovered from the pipe still unit running to briquette pitch, but the other stills do not make these fractions because they run only to road tars.

The mixing tanks are equipped with mechanical agitators. They were used for making road tars containing pitch and heavy oil or for tar-bitumen road mixtures.

Tar acids are extracted from the tar acid naphthalene oil in a vertical tank equipped with an agitator. Pitch is pumped to a loading station where, by means of troughs, it is run into split molds.

Conclusion. From the standpoint of improved processes or equipment this plant is unimportant.

Ebano Asphalt-Werke Aktiengesellschaft

The plant of Ebano Asphalt-Werke Aktiengesellschaft at Hamburg-Harbourg, Moorbourgerstrasse 16, was visited on October 12th with Dr. Walter Becker and Hans Petersen.

The purpose of this visit was to obtain information about a special road binding material (Spezialbindemittel) which this company was said to have made during the war from coal tar pitch and tar oils.

The persons interviewed at this target were: Dr. Walter Becker, Hans Petersen, and Joseph Schmid. The following information was obtained. Before the war there were large stocks of briquette pitch in Germany and Ebano and others considered the possibility of using the briquette pitch with or in place of petroleum bitumen. Laboratory tests were made using 50/50 mixtures of briquette pitch and bitumen from Mexican petroleum but the results were not satisfactory. When supplies of bitumen for road purposes became less available and the need for substitute materials increased, Ebano found that a distillate obtained by distilling pitch coke distillate to coke with superheated steam and vacuum could be mixed 50/50 with bitumen and, after blowing with air, this mixture appeared to be stable. The pitch coke distillate for this purpose came from the pitch coke plant at Castrop-Rauxel.

Preparation of a road material in the manner described above was considered but because of the work involved and the increasing shortage of bitumen, the General Inspector ordered that road binders should be made containing only coal tar materials. According to Dr. Becker, the tar distilling companies were asked first to make road binders using pitch and the pitch coke distillate, but they did not care to take the responsibility for such mixtures so the request was referred to Ebano. They accepted the request reluctantly because prior to that time they had made only asphaltic road materials. Also they considered it to be uneconomical to move so much raw material to Hamburg from the Ruhr. However, they proceeded to make a mixture of briquette pitch, pitch coke oil, hard pitch distillate and neutral coal tar oil (carbolic oil from which the tar acids had been extracted). The hard pitch distillate was the oil obtained at the tar plants in listilling from briquette pitch (75°C K & S) to hard pitch 150° K & S. The neutral oil was used in this mixture, during the earlier part of the war, as a solubilizing agent.

Later it became unavailable and anthracene oil was used in its place.

Dr. Hoepfner (an authority on bituminous road materials) expressed the opinion that this special binder was an ideal road material. At First, the road engineers did not like it because it had to be heated to higher temperature (140°C) than the grades of tar which they usually employed (110°C). Later, according to Dr.Becker they liked to use the new binder although its viscosity was further increased and higher applimation temperature were needed due to a lack of suitable fluxing oils.

In 1942, large supplies of Roumanian bitumen became available for use in the road materials so the General Inspector ordered that this Roumanian bitumen be added to the "Spezialbindemittel". In addition to using available raw material, it was believed that it might make the road tar less " grainy" or crystalline and further increase its binding capacity. Dr. Becker is of the opinion that there may have been some improvement along these lines. The proportion of bitumen used in the mixture was 20%. The Roumanian bitumen had a penetration of 100-200 at 25° (100 grams, 5 seconds), R.& B softening point 40-50°C and contained 2-3% paraffins.

Still later in the war some low temperature tar from bituminous coal became available from Upper Silesia so 4-5% of this tar was added to the mixture in making the special binder.

The equipment used for making this product consisted only of tanks and a pump. Two tanks of 7000 tons capacity each, equipped with air agitating coils, were connected through a pump. After one of the tanks had been filled with pitch or a pitch-bitumen mixture and the other with the mixture of oils, or mixture of oils and tar, the contents of the two tanks were mixed together by means of the pump and air agitaton. Thetanks and pump used for this purpose were totally destroyed by the air raids. More detailed information about this product is contained in a statement "Herstellung und Verwendung des Spezialbindemittel der Ebana Asphalt-Werke Aktiengesellschaft" furnished by Dr.Becker. Also Dr.Becker furnished copies of the following documents :

Four laboratory analysis reports dated August 4, 1941, March 31, 1942, Novemver 8, 1943, and February 7, 1944, covering "Spezialbindemittel".

Herstellung von Spezialbindemittel.

Spezialbindemittel - Erzeugung in den Jahren 194o, 1941, 1942, 1943 and 1944.

Emulsion Paint

During 1943 and 1944, Ebano made an emulsion containing the following materials :

35% " Spezialbindemittel" without bitumen
10-15% of a mixture containing 1/3 clay similar to bentonite and 2/3 common clay
50-55% water

Three horizontal mechanical mixers mounted above storage tanks were used for making this emulsion. The clay was mixed first with a small amount of water to make a stiff paste. A small amount of caustic soda (from 0.5 to 0.8% depending upon the alkalinity of the clay) was added next and also the "Spezialbindemittel". The remainder of the water was then added and after mixing thoroughly the emulsion was dropped into the storage tank.

Camouflage paint for use on buildings, airport runways, roads, etc. was made from the emulsion by mixing it with mineral colors and water. The proportions were approximately 40% emulsion, 20-3o% mineral pigment and 30-4o% water. Some of the pigments used were iron oxide, yellow ochre, carbon black and a green pigment made by coating finely divided barium sulfate or calcium carbonate with a green aniline dye supplied by I.G. Farbenindustrie.

According to Dr. Becker, for paints of this kind it is necessary that the emulsified particles be fairly large but not too large. 30 to 40 microus is preferred. Such an emulsion, even though prepared entirely from coal tar materials, does not mask the color of the pigment like a similar emulsion having particle size of 3-5 microus.

The quantity of camouflage paint of this kind that was made in 1943 and 1944, except in the winter months, average about 50 tons per day.

Plant Inspection

Following the discussion reported above, the plant was visited. Practically all tanks were destroyed by air raids and also almost all of the buildings and equipment except the three continuous petroleum stills. They were damaged somewhat but can be repaired without much difficulty. The laboratory building was damaged but now is used largely as an office building in place of the regular office building which was totally destroyed. This plant was undamaged during the first three quarters of the war, but was almost completely destroyed during the last quarter.

EMSCHERGENOSSENSCHAFT, ESSEN

Emschergenossenschaft, Essen, Germany, Kronprinzen Str., 24, was visited on November 23, 1945. The persons interviewed were Regierungsbaumeister Wiegmann and Dr. Schreiber.

"The Emschergenossenschaft was founded as a cooperative society legalized in 1904 by special law for the purpose of draining the entire Emscher district. This was done by regulating the natural and artificial draining ditches by building pumping stations and by purifying the sewage. All towns and rural districts of the Emscher area, which direct all or part of their sewage to the Emscher river or its tributaries have to be members of this cooperation."

One of the activities of Emschergenossenschaft is the extraction of phenols from the waste ammonia liquors from by product coke plants in the Emscher Valley. Twenty-four dephenolyzing plants were installed to recover phenolates that are sold by Emschergenossenschaft to Gesellschaft für Teervertung and Rütgerswerke, (60% to the former and 40% to the latter). The total quantity of phenol and cresols recovered from this source by the two tar distilling companies is about 5000 tons per year.

All of the dephenolyzing plants operate on the same principle although no standard design has been used for their construction. They were built by Koppers, Still, Kogag and Otto.

The ammonia liquor to be dephenolized is washed continuously with benzol to extract the phenols from the ammonia liquor and the benzol is then washed continuously with caustic soda to extract the phenols from the benzol. The phenolate is shipped to the two tar distilling companies mentioned above and the benzol is returned for the extraction of more ammonia liquor.

Ammonia liquor containing, for example, 4.13 grams per liter is reduced by this method to 0.33 grams per liter and liquor containing 1.05 grams per liter is reduced to 0.09 grams per liter before they discharge into the Emscher River.

LOTHRINGEN BERGBAU AKTIENGESELLSCHAFT, BOCHUM-LOTHRINGEN.

The pitch coke plant of Lothringen Bergbau Aktiengesellschaft is located near the Bochum-Gerthe tar plant of Gesellschaft für Teerverwertung. It was visited on September 17th.

The ovens at this plant were not damaged by the air attacks but piping was about fifty percent destroyed. Also some damage was done to the roof over the quenching and loading platform.

The plant consists of 65 horizontal, fire brick lined, sole flue ovens, 1,5 meters wide by 10 meters long. Because heating through the sole flues alone was insufficient to calcine the pitch coke to less than 1,0% volatile matter, some gas was burned within the ovens themselves during the calcining period.

The ovens were charged with 150° pitch, in crushed or flake form, through charging holes on the tops of the ovens. The quantity of pitch charged was 14 tons. The coke was pushed from the oven by a ram onto a quenching and loading platform level with the floors of the ovens. Quenching water was applied by hand. The quenched coke was loaded into cars directly from the quenching platform.

The pitch distillate, amounting to approximately fifteen percent of the pitch charged into the ovens, was distilled, in a special installation for the purpose, to hard pitch. The latter was charged into the ovens with hard pitch from the tar plants. The distillation of the pitch distillate to hard pitch was carried out with oxygen free combustion gas. A small amount of coke oven gas was burned in stack gases from the ovens. Combustion of the coke oven gas was supported by the oxygen in the stack gas. By this method the stack gas was made oxygen free and at the same time heated to about 600°C. The oxygen free stack gas then bubbled through the pitch distillate which previously had been topped under about 400 m.m. vacuum to remove light distillate for sale as fuel oil. The heavy oil obtained by distilling the topped distillate to hard pitch was shipped to hydrogenation plants.

The capacity of this pitch coke plant was about 200 tons of coke per day. It was operated at full capacity throughout the war

VERKAUFSVEREINIGUNG FÜR TEERERZEUGNISSE, CASTROP-RAUXEL

The pitch coke plant at Castrop-Rauxel was visited on September 17th and again at a later date. The persons interviewed on the first visit were Dr. Moehrle, Director of Gesellschaft für Teerverwertung and Dr. Klemm, Manager of the Castrop-Rauxel tar plant of the same concern. On the later visit Mr. Zumbusch, superintendent of the pitch coke plant was interviewed.

The pitch coke plant suffered considerable damage by bombing and from artillery. One block of five ovens was destroyed by a bomb and much of the piping and steel work was severely damaged.

The plant consists of forty Koppers, silica lined, regenerative, by product ovens built in two batteries of twenty ovens each. Each battery consists of four blocks of ovens with five ovens in a block. The blocks are separated by pinion walls so that any group or block of five ovens can be operated or shut down independently of the other blocks.

The first battery of twenty ovens was built in 1939. The second battery was built in 1943. It was placed in operation in November of that year.

The dimensions of the ovens are as follows: overall length, 11,800 m.m.; length between doors, 11,000 m.m.; height, 3000 m.m.; average width, 450 m.m. The twenty ovens in a battery are connected through standpipes to a common collecting main. The two collecting mains from the two batteries are, in turn, connected to two electrical precipitators, two indirect coolers, two direct coolers and two exhausters. Decanting tanks separate the flushing liquor from the pitch distillate condensed in the collecting main, electrical precipitators and coolers.

For charging the ovens with liquid, high softening point pitch (150°C) there were six vertical, gas heated tanks equipped with internal pumps. Each pump was connected to pipe loops that extended over the oven blocks. The molten pitch in a charging tank was kept in constant circulation through a loop by means of the pump. A float mechanism indicated the level of the pitch in the charging tank at any given time. The charging tank was filled with liquid pitch from an insulated tank car which transported it to the pitch coke plant from the tar plant.

To charge an oven, a valve on one of the pitch circulating loops was opened and pitch flowed into the oven through a pipe inserted through one of the charging hole covers. The introduction of the pitch was so regulated that nine tons entered the oven during a period of three hours. Also the rate of introduction was decreased during the three hour period according to a fixed schedule. During the first part of the charging period a mixture of superheated steam and coke oven gas was introduced into the oven through another charging hole cover to minimize the cracking of vapors within the oven.

After 21-22 hours the coke was pushed from the oven into a quenching car. A minimum amount of quenching water was used by covering the car, after the application of the water, with a steel cover provided for that purpose. The quenched coke had a water content of about one percent. After quenching, the coke was dropped onto a wharf and loaded into cars for shipment to the electrode factories.

Following are the data collecting during a 13 day test conducted in 1940. The pitch charged during this period had a softening point of 155°C, ash content of 0.3% and specific gravity of 1,293.

	13 Days	Per Day	Percent
Pitch charged	3159 tons	243 tons	100
Pitch coke made	2208 tons	165.9 "	69.9
Pitch distillate	650 tons	50 "	20.6
Gas (heating val min. 3362 K cal. max. 3874 K cal.)	785.000 Nm^3	60.400 Nm^3	7.0
Loss	79 tons	6 tons	2.5

The analyses of the products made during this test period were as follows:

Pitch coke		Pitch Distillate		Gas			
water	- 0.8%	water	- 2.0%	CO	- 0.9%	H_2S	- 3.09/m^3
ash	- 0.4%	ash	- 0.025%	CnHn	- 1.0%	HCN	- 0.59/m^3
V.M.	- 0.4%	coke	- 18.5.%	O_2	- 0.5%	NH_3	- 2.39/m^3
Pure coke	-98.4%	S.P.	- 40°C K.S.	CO	- 3.5%	Naphthalene	-23.09/m^3
		sp.gr.	- 1.262	H_2	- 11.8%	Benzene	- 13.09/m^3
				CH_4	- 13.0%	*sp.gr.	- 0.877
				N_2	- 3.3%		

The amount of gas produced by the ovens during the 13 day test was not sufficient for the entire needs of the plant, (heating the ovens, boiler house, pitch charging tanks and door prewarming), and 193,640 Nm^3 of coke oven gas was used in addition. Its minimum heating value was 4099 K cal. The mixture of heating gases, having a minimum heating value of 3380 and maximum of 3895 K cal. was distributed in the following manner:- heating the battery, 36.5%; boiler house, 45.5%; pitch feed tanks, 14.5% and door prewarmer, 3.5%.

Following are the tonnages of pitch coke produced by this plant during the period Dec. 1939 to Jan. 1st 1945:

December	1939 -	522 tons	
Year	1940 -	53,518 tons	
"	1941 -	64,007 "	
"	1942 -	69,375 "	
"	1943 -	76,776 "	(new battery started in Nov.)
"	1944 -	77,724 "	
	Total-	341,922 tons	

MANNESMANNRÖHRENWERKE KÖNIGIN ELISABETH MINE, ESSEN

At this target, on November 23rd, the plants for briquetting bituminous coal fines and for producing pure coal for electrode coke were inspected.

The briquetting plant was operating at partial capacity when visited. It was using a stock of washed coal that was on hand but no more was being produced. The plant had not been seriously damaged and could operate at capacity if sufficient coal were available. The capacity of the plant is about 400 tons a day (one shift). Briquette pitch is crushed to about 3/4 inch by an edge runner with perforated bottom. It is then elevated to a bin above a crusher, crushed in a hammer mill to 0-2 m.m. and added continuously to crushed, washed coal (0-10 m.m.) in the proportion of 6-7% pitch to 93-94% coal. The mixture is heated, indirectly, in large, shallow pans with revolving agitators. The coal-pitch mixture enters the pan at the center and is moved outwardly by rabble arms. It is then conveyed by a screw conveyor to vertical mixers where steam is added in small amount. The mixture then drops into presses where 3 kilogram, rectangular briquettes are made. The pressure is about 150 atmospheres. The briquettes are transported to cars by conveyors but are loaded into the cars by hand.

The plant for producing pure coal for electrode coke production was not operating for lack of raw material. It had not been damaged by air attacks. The process used in this plant involve (1) Removal of "Finefrau" seam coal, containing about 12% ash, from the Emil shaft, (about 930 m. deep); Removal of shale on a picking belt; (3) Crushing to below 8 m.m. and removal of fine dust; (4) Screening to remove coarse dust, - 1 m.m.; (5) Laminar separation, followed by spraying, to separate refuse, briquetting coal and the product which is floated to obtain pure coal containing about 0.5-0.7% ash. The yield of pure coal is about 27%. Apparatus was being installed for the electrostatic cleaning of the 0-1 m.m. dust from the first screening operation. The process was to include drying in a Büttner turbo dryer, sifting in a vibro filter to about 0.1 m.m., electrostatic separation and flotation. This part of the plant was not completed. The older part of the plant, described above, produced pure coal that was mixed with 30% coal tar pitch and carbonized in by product ovens, at the König Ludwig coke plant.

HEINRICH KOPPERS G.m.b.H., ESSEN

This firm was interrogated on September 22nd, 1945, concerning tar plant, pitch coke and low temperature carbonization equipment built in Germany and elsewhere. At a later meeting the dephenolizing of waste waters was discussed.

This firm has built numerous continuous tar distilling plants in England and Europe. The original and smallest plant is owned and operated by Nord Deutsche Kohlen und Cokes Werke A.G., Hamburg. The largest plant is owned by Kommanditgesellschaft Julius Rütgers, Mährisch-Ostrau. The Heinrich Koppers firm now has an agreement with Gesellschaft für Teerverwertung, which will permit it to build continuous tar stills of the G.f.T. type as well as its own.

The arrangement of the continuous tar still built by Koppers for Nord Deutsche Kohlen und Cokes Werke is illustrated by a flow-diagram included in the target report on that company. A diagram showing the arrangement of the Mährisch Ostrau installation accompanies this target report. The continuous still of Gesellschaft für Teerverwertung is described in the report covering a visit to that company's plant at Duisburg-Meiderich.

The pitch coke plant at Castrop-Rauxel owned by Verkaufsvereinigung für Teererzeugnisse and operated by Gesellschaft für Teerverwertung was built by this company. The construction and operation of the pitch coke plant at Rauxel is discussed in the target report on Verkaufsvereinigung für Teererzeugnisse, Rauxel.

During the war Koppers, Otto and Didier Werke collaborated in the development of a vertical, refractory oven for the low temperature carbonization of non-caking coal. The Koppers firm was authorized to construct a large plant for this purpose but construction was not completed before the war ended. Information concerning this installation will be included with other information about low temperature carbonization in a separate report.

The Heinrich Koppers organization has installed equipment at tar plants for the recovery of naphthalene, anthracene and other coal tar products.

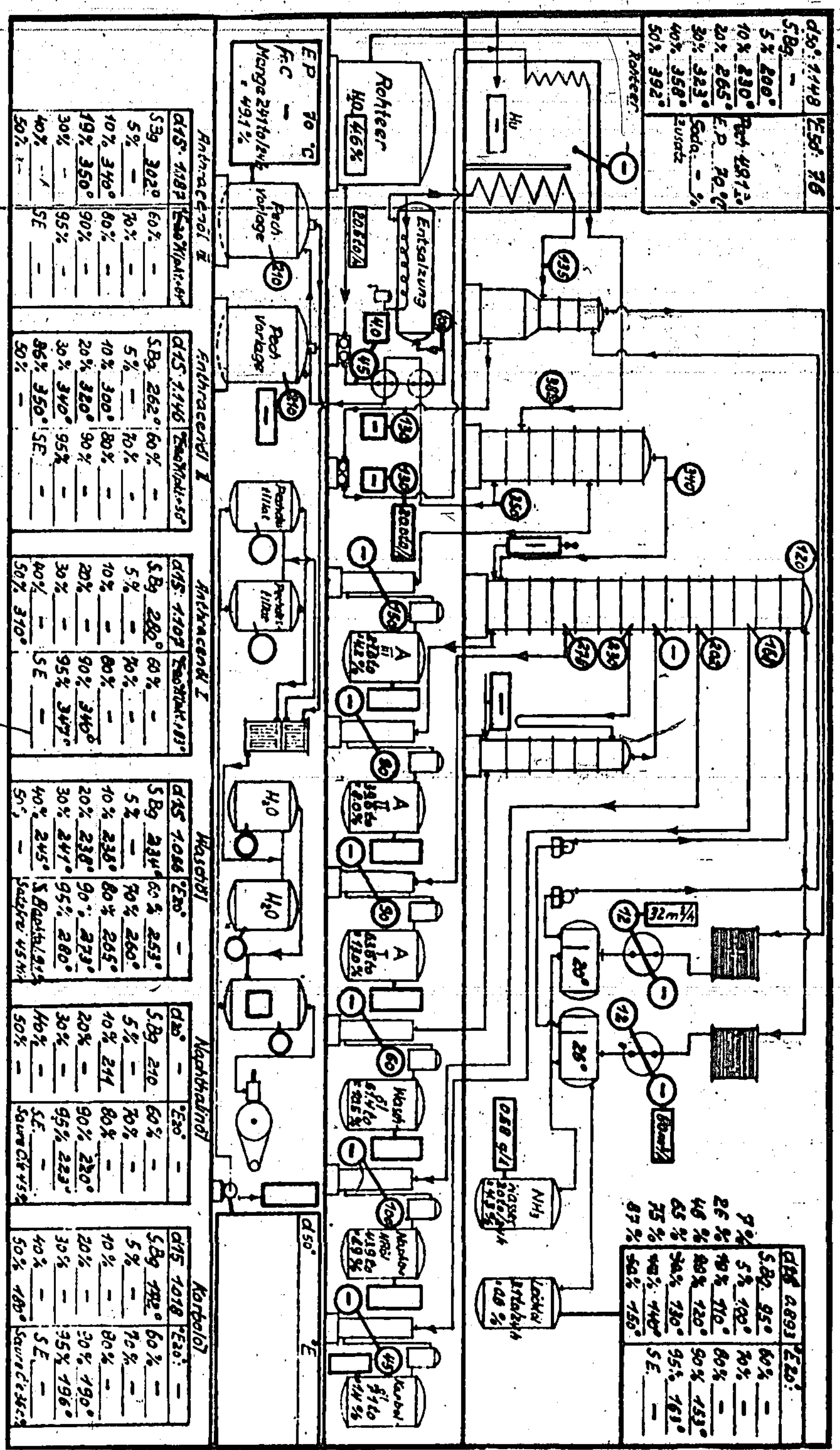


Fig. 1 - MÄHRISCH OSTRAU INSTALLATION

DR. C. OTTO & COMPANY, G.m.b.H.

At the office and plant of Dr. C. Otto and Company, Dalhausen, visited on October 16, 1945, the following persons were interviewed: Dr. Stuhlmann, Dr. Brensing, Mr. Schoen, and Mr. Boskamp.

The following information was obtained: This company has carried on a considerable amount of investigational work on low temperature carbonization of bituminous coal. A pilot plant was built in which, at first, it was tried to carbonize the coal by sensible heat alone. This attempt was not successful and a small coke oven with solvay type, horizontal, heating flues was erected and inside the oven were placed, in vertical position, steel members resembling I beams. When they were in place the flanges of the I beams were close to the walls of the oven and the webs extended crosswise between the walls. The coal was filled into the spaces between the I beams. Heat was transmitted to the coal through the side walls of the oven and also by the steel plates, or webs, of the I beams. At the end of the carbonizing period the low temperature coke was dropped from the bottom of the oven into a car. The oven was provided with a regenerator which was directly in line with and on the same level as the coke oven. The coke produced in this oven was of good quality but the steel heating members warped badly and made it difficult to discharge the coke so the steel members were removed and tests were made with heat supplied only through the side walls. A plant designed on this basis was projected but was not built.

The Otto Company has not investigated low temperature tar. Dr. Stuhlmann mentioned the fact that a large amount of work had been done on the miscibility of low temperature tar by Krupp under Director Müller and Dr. Demann. The Otto Company also has not built tar plants for the distillation of low temperature tar.

This company has built batch stills for companies that process high temperature tar. In one installation in Italy, batch stills, with bell and tray columns mounted directly above the stills, were installed. In this case high vacuum was used during the distillation of the tar. In principle, this installation was the same as those which Rütgerswerke uses in its various plants.

During the war this company shipped a continuous tar distilling plant to the Japanese in Manchukuo. Diffic lties were encountered in getting the various parts to the site near M kden but from telegrams received in 1943 the Otto Company assumes that operations were started.

Recently, a plant similar to the one sl pped to Manchukuo was projected for Switzerland but work on the project was stopped and has not been resumed. A description of this plant with a flow diagram attached and dated December 16, 1943, accompanies this report. The description also covers the Manchukuo plant in most respects. The tar still in the Manchukuo plant was provided with alloy steel tubes and cast iron fractionating columns. The Swiss still was to be built with plain steel tubes and steel columns.

An oil redistilling plant was built by the Otto Company for Mathias Stinnes and at other tar plants equipment for tar distillation, oil redistillation, naphthalene recovery and refining, and anthracene recovery has been installed.

Conclusion

The Dr. C. Otto Company has been fairly active in the building of tar stills and tar processing equipment. However, the most pretentious distillation units have been constructed for Japan and Italy. Installations built in Germany were all of the usual vertical or horizontal batch types without columns except on certain stills used for oil redistillation. A pitch coke plant similar to the one at Lothringen was projected for Japan but was not built.

Description of a continuously working tar-distillation plant with tube furnace for the manufacture of:

light oil	washing oil
carbolic oil	anthracene oil
naphthalene oil	briquette pitch

(See Fig. 1 on Page 117)

I. Dehydration

From the crude tar tanks in which the tar must be heated to a temperature that ensures its being easily pumped, the tar is forced by one of the crude tar pumps (1) through the heat exchanger (2) in which the pitch is cooled while the crude tar is preheated to about 100°C.

The preheated crude tar now passes through the pipes of the dehydration section of the tube furnace 3 where the temperature is raised to about 160°C. At this temperature the crude tar is conveyed to the vapor liberator (dehydration column) 4 where ammonia liquor and light oil leave the top of the column and the dehydrated tar sinks to the bottom. The vapor mixture of ammonia liquor and light oil which leaves the top of the vapor liberator is condensed, cooled and separated in the cast iron plate condenser 5 and in the light oil cooler 6 fitted with the decanter 7. From the decanter the ammonia liquor runs into the receiver 8 while the crude light oil is normally conveyed by means of the return pump 9 as reflux liquid to the fractionating column 10. This operation finishes the dehydration of the crude tar.

II. Distillation

The dehydrated tar as it accumulates in the sump of the vapor liberator 4 flows to one of the tar pumps 11 which forces it in succession through the convection and radiating zones of the distilling section of the tube furnace 3 while the temperature is raised to 350-360°C. The mixture of liquor and vapor so produced now enters the pitch column 12 where it is separated into pitch and oil vapors. The pitch which sinks into the sump is treated there with steam in order to vaporize enough of the oil still present in the pitch to produce briquette pitch which runs through the above-mentioned heat exchanger 2 into the alternately operated pitch receiver 13. Vapors liberated in the pitch receivers are absorbed in the fume tower 14.

The vapors liberated in the pitch column pass in succession through two fractionating columns 15 and 10 where they are separated into the individual fractions. By means of the accessory columns 16, 17, 18 and 19 effective fractionation is achieved. The oils, as they flow out of the columns, are cooled in the coolers 20, 21, 22, 23 and 24 and pass on to the receivers 25, 26, 27 for anthracene oil, 28 for naphthalene oil, 29 for carbolic oil and 30 for washing oil. A portion of the washing oil in the sump of the separating column 10 is pumped by means of one of the pumps 31 as reflux liquid to the fractionating column 15. The vapor leaving column 10 is light oil which consists partly of crude light oil from the vapor liberator 4 which was pumped by the return pump 9 as reflux liquid to column 10. The vapor from column 10 is condensed in the cast iron plate condenser 32, is cooled in the cooler 33 and separated in the separator 34. A portion of this top light oil, together with the crude light oil from the vapor liberator 4, is pumped by means of the return pump 9 as reflux liquid once more to fractionating column 10, while the balance goes into receiver 35 for production. Pumps 36-41 transfer products from the receivers to the available cooling pans (anthracene oil, naphthalene oil) or to the appropriate storage tanks 42 and 43 serve for the emptying of the furnace coils and of the plant whenever this becomes necessary.

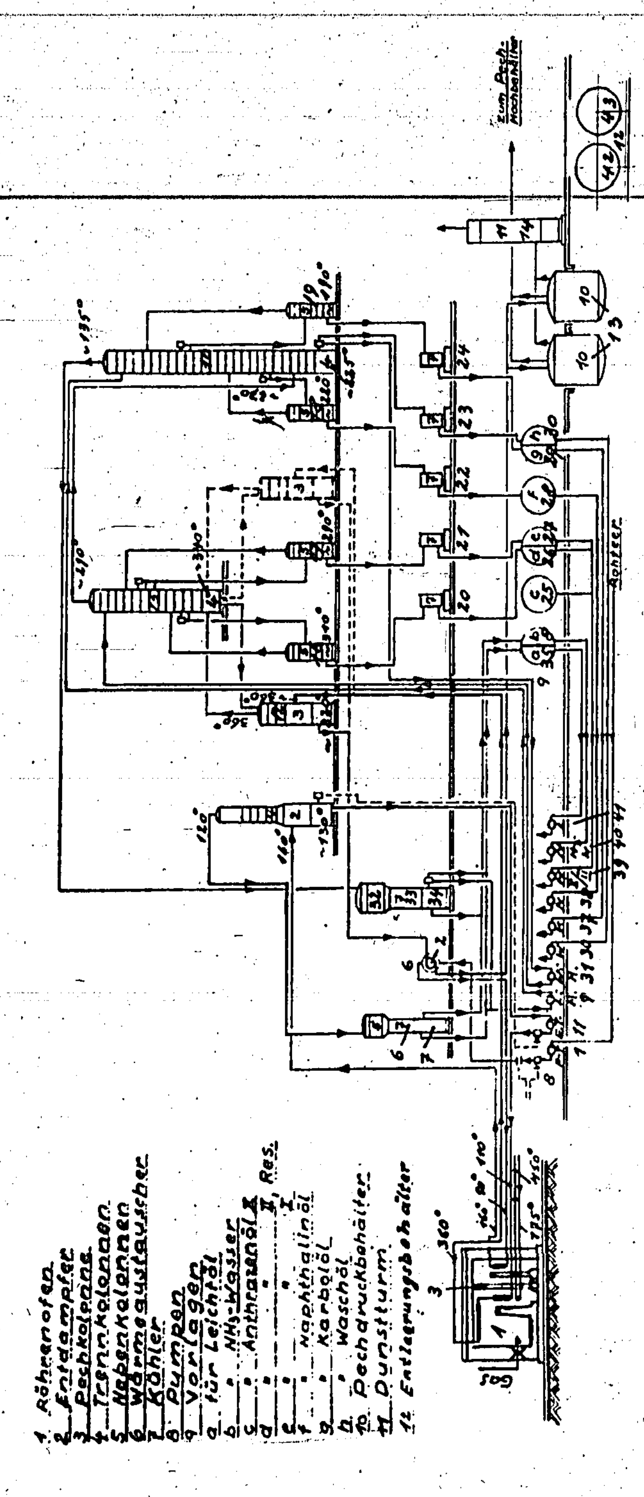


Fig. 1 - C. OTTO TAR DISTILLATION PLANT (See Pgs. 115-116)

FIRMA CARL STILL, RECKLINGHAUSEN

The Carl Still organization was visited on November 9th and again on October 15th, 1945, to discuss the types of equipment used in Germany for the processing of coal tar and coal tar products. This firm has been active in this field and has built tar stills, oil redistillation stills with columns, pitch cooling equipment, anthracene plants, naphthalene plants, etc.

A new type of installation was designed by Carl Still for the plant of Ruhröl A.G. to distill the coal extract produced there to a hard residue suitable for charging into coke ovens for pitch coke production. The coal extract was pumped through a pipe heater, fired by gas, and released into either of two gas heated, batch stills equipped with agitators arranged to clean the bottoms and side walls. At the outlet of the pipe heater an automatically operated valve maintained a pressure on the extract while it was passing through the pipe heater. The operation of the two batch stills was so scheduled that the pipe heater could operate continuously and discharge alternately into the batch stills without interruption. The residue from the batch stills discharged onto an endless conveyor made of overlapping steel plates which cooled the residue and discharged it into a car. The conveyor passed through water, after discharging the pitch, to cool the plates before coating them with more pitch.

The largest installation of tar processing equipment built during the war was constructed by this firm for use on brown coal tar. The plant was built for the Aktiengesellschaft Sächsische Werke at Espenhain near Leipzig. The capacity of the plant was 200,000 tons of brown coal tar per year.

It consisted of a continuous pipe heater unit operating atmospherically, a vacuum, continuous unit to further distill the residue from the first unit, batch stills in which the residue from the second continuous unit was further distilled to hard pitch with inert gas, pitch coke ovens into which the hard pitch was charged and an Edeleanu extraction plant to remove paraffin from a part of the distillate that went to diesel oil and fuel. The balance of the distillate went directly to fuel oil.

The Still organization built an oil redistilling unit for Carolinenglück consisting of four, 50 ton, horizontal stills connected to two, offset, cast iron, bell-and-tray columns.

DIDIER WERKE A.G.- BERLIN

On October 31st, Dr. Adolf Thau was interviewed at the office of Didier Werke A.G., Westfälische Str. 90, Berlin-Wilmersdorf. Matters pertaining to high temperature and low temperature coal tar were discussed. Subsequently, Dr. Thau submitted a brief statement, in writing concerning "Developments in the Technics of Tar Distillation in Europe."

The statement points out that vertical pot stills have been replaced to some extent in Europe by horizontal stills to obtain greater heat economy. Also there has been a trend toward the use of continuous stills.

To use the stocks of briquette pitch which, at times accumulated at tar plants and at the same time to supply the need for pure coke for carbon electrodes, pitch coke plants were erected in two locations.

Dr. Thau, concludes that except for these two developments there has been little advancement in the field of coal tar processing in Europe in recent years.

A statement concerning the low temperature carbonization of coal in Europe also was received from Dr. Thau. It is largely historical in nature. It will be covered separately with other documents on the same subject.

SCHUMACHERSCHE FABRIK

Bietigheim, Württemberg

On November 29 and 30 the factory of Schumachersche Fabrik at Bietigheim, near Stuttgart, was visited for the purpose of inspecting filters of the kind that were used by Ruhröl A. G. for filtering coal extracts. They were also used by various hydrogenation plants such as Hiedrierwerk Scholven, Gelsenkirchen-Buer; Rheinpreussen, Homberg-Mörs; Rheinischebraunkohle, Cologne; Wesseling near Cologne, and others for filtering middle oil produced by the hydrogenation of coal.

The filter rings for the pressure filters used by Ruhröl A.G. were supplied by Schumachersche Fabrik to Atlas-Werke A.G., Bremen, who made the filters. The use of these filters has been described, briefly, by this investigator in a separate report and in detail in the following CIOS Reports:

Item No. 30 File No. XXXI-27, Coal Extraction Plant of Ruhröl G.m.b.H. by H. H. Lowry and H. J. Rose June 29 and 30, 1945.

Item No. 30 File No. XXX-104, Botrop-Welheim Hydrogenation Plant by C. Cockram, August 8, 1945.

The filter rings supplied to Atlas Werke by Schumachersche for this purpose are described by the latter company as "Braungelb #50 filter rings". Their dimensions are 125 mm O.D., 81 mm I.D., and 30 mm high. When the factory was visited there were 20,000 of these filter rings on hand. Each of the Atlas filters at Botrop contained 32 candles and each candle was made up of 60 filter rings, so the number of rings required for each filter was about 1920. The rings were replaced after 5000 filtering cycles of 15 minutes each.

Schumachersche Fabrik furnished complete filters to the hydrogenation plants. Their No. 100 filter was supplied to those customers. Each filter contained one filter tube one meter long and 300 mm O.D., made up of Braugelb #50 rings of 300 mm diameter. The larger diameter rings could be used for filtration of middle oil at hydrogenation plants because the liquid to be filtered was much cleaner than the coal extract at Botrop and lower filtering and cleaning pressures were employed. A single hydrogenation plant used as many as 40 filters at one time.

When the Schumacher factory was visited it had on hand about 100 #42 filters. These filters hold one candle 420 mm long and 125 mm O.D. The candle is made by cementing individual rings together

with a phenolic resin and then turning the tube to proper diameter and length. One #42 filter complete with filter cylinder, brown yellow #50 and three reserve cylinders of the same kind were purchased for shipment to America for experimental purposes. The price of the complete filter was 195 RM and each additional cylinder cost 12.20 R.M. Cost of case and packing was 12.0 R.M. In addition to the above, two "Carbo" No. 40 filter cylinders to fit the No. 42 filter were purchased and packed for shipment to America. These filters cost 15.70 R.M. each and packing, 3.0 R.M. Alkaline liquids can be filtered with the Carbo filters, whereas the "Braungelb" filters must be used with neutral or acid liquids.

The numbers 40 and 50 refer to the grain sizes and porosities of the filter tubes. Porosities range from fine (#10) to coarse (#60). No. 50 has been found to be the most suitable porosity for middle oil from hydrogenation plants and for coal extracts of the kind produced at Botrop.

In using the #42 filter equipped with #50, "Braungelb" cylinder, the working pressure should be about 8 kgs. per sq. cm. and the highest permissible pressure is 12 kilograms per sq. cm. When cleaning the filter with air, the pressure should be about 4 kilos per sq. cm.

Manufacture of the filter rings at Bietigheim is done entirely by hand. A weighed amount of "Chamotte" is poured into a mold, subjected to high pressure with a hydraulic press, and the ring is dried atmospherically and then fired in gas-heated kilns.

The persons dealt with at this plant in purchasing the equipment were Albert vom Hofe, Helene Martin and Gottlieb Xander. Army Headquarters at Esslingen and the Military Government for Bietigheim were consulted before purchasing the equipment.

AKTIENGESELLSCHAFT DER KOHLENWERTSTOFF-VERBÄNDE BOCHUM

The offices of Aktiengesellschaft der Kohlenwertstoff-Verbände were visited on September 13, 1945, and several times thereafter. At present, the offices of this organization are located at Waldring 71, (Schillerschule) in Bochum. The central office building previously occupied in Bochum was totally destroyed by air raids, and most of the records, stored in safes in the basement, also were destroyed.

Mr. Kurt Haver, Director of Kohlenwertstoff-Verbände, with the permission of the Military Government, assisted materially in the collection of information about the German coal tar industry, arranged appointments for visits to some of the tar plants, and assigned Dr. Geiselbrecht, of his organization, to accompany the investigator to some of the targets as guide and to assist in the interrogations.

During the war, Mr. Haver's organization directed for its associated members the production and distribution of nitrogen; benzol and benzine; coal tar products; coumarone resins; phenol and methane. The individual groups which were brought together during the war to form the organization known as Kohlenwertstoff-Verbände and the products which they handled were as follows:

1. Deutsche Ammoniak-Verkaufs-Vereinigung (D.A.V.V.); nitrogen obtained from synthetic plants, coke ovens, and gas works.
2. Benzin-Benzol Verband (B.V.); Benzol and benzine.
3. Verkaufsvereinigung für Teererzeugnisse (V.F.T.); coal tar products.
4. Coumarone Verband, (C.V.); coumarone resins and residues from coke plants and tar plants.
5. Phenol Verband (P.V.); phenol from tar plants and from synthetic phenol plants.
6. Treibgas Vereinigung (T.V.); methane from gas wells for use as motor fuel. Another group of Kohlenwertstoff Verbände, "Gruppe Schwefelsäuregemeinschaft", (S.G.), purchased all supplies of sulfuric acid for the coke ovens, synthetic nitrogen plants and metal working factories belonging to its associated members.

Mr. Haver and Dr. Geiselbrecht furnished the investigator with product specifications and miscellaneous statistical information concerning German coal tar products.

PHENOL-VERBAND, BERLIN

An attempt was made on October 21, 1945, to find Dr. Westermann, Director of Phenol-Verband, at his office in Berlin, Gross Admiral von Koster-Ufer, 59. The building was badly damaged but a secretary was found in the office who thought Dr. Westermann might come there the following day. A return visit by the writer could not be arranged but, subsequently, Dr. Müller, President of Phenol-Verband, was contacted in the office of Rütgerswerke A.G. and he furnished the following information:

Phenol-Verband now has only two members in its organization in eastern Germany, namely, Rütgerswerke and I. G. Farbenindustrie at Leuna. All of the other former members are now in the Polish sector and are no longer German firms.

The I. G. plant at Leuna recovers tar acids from the water resulting from the distillation of brown coal and from oils produced by the hydrogenation of brown coal and brown coal tar. Previously, the Leuna plant produced 24,000 tons of phenol per year.

Rütgerswerke had a synthetic phenol plant at Niederau (now dismantled for shipment to Russia) and also extracted phenols and cresols from tar oils. Its production was about 2,500 tons of phenol and 4,000 tons of cresols per year. It now is about 240 tons of phenol and 480 tons of cresols per year.

The total output of phenol by the two companies was said by Director Müller to be about 60-100 tons per month at the present time.

VERKAUFSVEREINIGUNG FUR TEERERZEUGNISSE, BERLIN-WILMERSDORF

The Berlin office of Verkaufsvereinigung für Teererzeugnisse, Berlin-Wilmersdorf, Hohenzollerndamm 42a, was visited on October 30, 1945, to obtain information about the production and sale of coal tar products, particularly in the central and eastern parts of Germany.

The persons interviewed at this target were Director Weger and Director Weiss. Previously, this organization sold the products of all of the tar distillers in eastern Germany. At present, it has very little to sell because tar production is only about 5-10% of normal and because most of the tar plants which were under German control during the war are now destroyed or under Russian or Polish control or ownership. V.f.T. sells small quantities of pitches and oils from Erkner which the Russians do not want for their own use, but benzol, naphthalene, phenols, creosote and other products are not made available for sale by V.f.T.

Following is the status of each of the tar plants of eastern Germany according to Weger and Weiss:

- Rütgerswerke Erkner - in order and working at part capacity.
- Rütgerswerke Breslau - completely destroyed (beyond repair).
- Rütgerswerke Niederau - equipment about to be removed to Russia; nothing left but the buildings.
- Rütgerswerke Teerag, Wien - no information; presumed that plant exists.
- Borsig (Scala) Hindenburg - benzol and tar plant completely removed to Russia.
- Odertal, Gleiwitz - Polish but no news as to condition.
- Oberhütten - plant is working but for the Poles.
- Kattowitz - Polish; no news as to condition.
- Emmagrube, Kattowitz - Polish; no news (only made pitch and oil).
- Döbeln - German but no news (said by Kahl to be in order).
- Zwickau - German but no news (said by Kahl to be in order).
- Velten - German but no news (said by Kahl to be in order).
- Waldenburg - German (Polish according to Kahl); no news.
- Küstrin - Polish; completely destroyed by war.
- Danzig - Polish; no news (said by Kahl to be in order).
- Rütgerswerke Preussen - Russian; condition unknown.

According to Weger and Weiss, the future of the German tar industry will depend on (1) production of tar; (2) attitude of the Russians; (3) availability of materials for reconstruction.

I.G. FARBENINDUSTRIE, LUDWIGSHAFEN.

At the Ludwigshafen plant of I.G. Farbenindustrie, visited on December 5th and 6th some of the war time uses for coal tar products at that plant were investigated. The persons interrogated were Dr. Snell, Dr. Fischer, Dr. Wimmer and Dr. Johannsen.

According to Dr. Snell the principal uses for naphthalene at Ludwigshafen were: for phthalic anhydride manufacture about 550 tons per month; for beta naphthol about 500 tons, and for tanning agents, detergents and dyestuffs about 150 tons per month. The latter, i.e. tanning agents, detergents and dyestuffs were made in greater quantities at Leverkusen than at Ludwigshafen, according to Dr. Snell. Naphthalene also was used at Ludwigshafen for the manufacture of naphtylamines.

Brief visits were paid to the plants in which phthalic anhydride, maleic acid and koresin were made. These plants have been discussed in reports submitted by other investigators so no attempt was made to cover them in detail.

In the phthalic anhydride plant two units, of 80-100 tons capacity each were operating and making about 150 tons of phthalic anhydride per month. It was expected that the capacity would be doubled in about three months when two more units would be ready for operation. Originally there were seven units in this plant but three were destroyed by air attacks. The two units that were operating had been started without difficulty although the catalyst was approximately six years old. The construction and operation of this plant is discussed in CIOS Report Item No. 22, File No.XXVII-85 by Kern, Murray and Sudhoff.

Maleic acid is made at Ludwigshafen from Crotonaldehyde instead of benzene. It was explained that after 1933, "cheap benzol" from America was no longer available and it became less expensive to use crotonaldehyde. It was stated however that if cheap benzol becomes available again it may replace the crotonaldehyde.

The process used in the maleic acid plant also has been discussed in CIOS Report Item No. 22, File No. XXVII-85. The maleic acid plant was not operating when visited. It was heavily damaged by air attacks and no repairs, either to the building or equipment had been started.

Koresin was made by reacting isobutylene with phenol, using aluminium chloride as catalyst, to obtain para-tertiary-butyl phenol. The latter was then reacted with acetylene, using zinc naphthenate as catalyst, to obtain the koresin. This process has been described in CIOS Report Item No. 22, File No. XXIX-62 and the Quartermasters Technical Intelligence Report on "The German Plastics Industry." When visited, this plant was operating at partial capacity.

PHTHALIC ANHYDRIDE AND ALKYD RESIN MANUFACTURE BY I.G. FARBENINDUSTRIE, UERDINGEN, GERMANY

The plant of I.G. Farbenindustrie at Uerdingen, Germany, was visited on October 1, 1945, ~~in order to obtain information concerning the manu-~~ facture of phthalic anhydride and the use of this chemical in the making of alkyd resins and phthalates.

The persons interviewed were Dr. Dilthey, Assistant Manager, Dipl. Ing. Kutsch, Engineer in charge of the construction of the phthalic anhydride plant; Dr. Hamann, Director of Research; and Dr. Kinsky, Superintendent of the alkyd resin plant.

Dr. Dilthey stated that there are only three phthalic anhydride plants in Germany, i.e., I. G. at Oppau - Ludwigshafen, I. G. at Schkopau (near Leuna), and I. G. at Uerdingen. Construction of the Uerdingen plant was begun in 1942. When it was about 80% completed, construction was stopped because of the greater necessity for work along other lines. Construction was resumed again a short time ago by permission of the Military Government. It is expected that the plant will start production shortly after the first of the year. The plant suffered no damage from air raids except for broken windows.

The phthalic anhydride plant is housed in two buildings. One building is devoted entirely to the receiving, storage, melting and filtering of flake naphthalene made at tar plants in the Ruhr. The flake naphthalene, in paper bags, arrives in railway cars or trucks and is dumped into a pit from which it is elevated by a crane to a storage platform. It then is fed by the crane into a hopper above a crusher. After passing through the crusher the naphthalene is elevated to an overhead bin from which, by means of a screw feeding mechanism, it is filled into an automatic weigh tank suspended from a trolley. The weigh tank discharges the naphthalene into three steam-heated, melting kettles equipped with mechanical agitators. The molten naphthalene flows by gravity through three filters containing "zellwolle" to pumps by which it is transferred to six other filters (three large and three small) in the top of the building. From there it drops into steam-heated storage tanks. The equipment for carrying out the above operations is shown in flow line arrangement in drawing No. 13616.

The second building contains in one end offices, service facilities, and plant laboratories. The next section of this same building contains

eight steam-jacketed tanks which feed the molten naphthalene to the vaporizers. There are two feed tanks for each vaporizer. On the floor below the feed tanks and directly below them are the instrument control panels. On this same floor are the air-to-air heat exchangers, vapor~~izers and converters. In a lower portion of the same building adjoining~~ the portion which houses the offices and laboratories are the various air pumps required for feeding air to the converters and air to the heat exchangers. There are eight blowers for each of these purposes (two blowers of each kind per converter).

The vaporizing of the naphthalene and conversion to phthalic anhydride is carried out as follows: Molten naphthalene from the overhead feed tank flows through a rotameter into the vaporizer where it is vaporized by air preheated in the heat exchanger by hot air from the cooling section of the converter. The mixture of air and naphthalene vapors passes downwardly through the converter and to the condensing chambers. There are fourteen chambers for each converter. The first four are water-jacketed for cooling and the last ten are air-cooled. Each cooling chamber is equipped with a screw conveyor at the bottom which discharges the phthalic anhydride into a Redler conveyor running below the fourteen chambers and to the melting kettles.

The construction of the converters is as follows: 3000 tubes of 25 mm. diameter are arranged concentrically between tube sheets, around a central pipe about 24" in diameter. The catalyst is placed inside the tubes and a mixture of salts used for cooling the converter surrounds the tubes. During operation, a molten salt bath is circulated through the central tube by means of an impellor in the top of the tube which is driven by a motor mounted on top of the converter. Cooling of the salt is accomplished inside the central tube by air circulated upwardly and then downwardly through double-walled pipes which are closed at the upper end (air rises in the outer pipe to the closed end at the top, then reverses and goes down through the inner pipe). The hot air from this cooling system next circulates through the tubular heat exchanger where it preheats filtered air going to the steam-heated vaporizers and from there goes to the converters. In the vaporizers the preheated air flows over the molten naphthalene and becomes saturated with naphthalene vapor.

The cooling chambers are made of ordinary steel. They are about two feet wide by four meters long and four and five tenths meters high.

A single partition inside causes the vapors to pass downwardly and then upwardly in passing through the chambers. From the last chamber the air passes through a cyclone dust collector, exhaust fan and stack to the atmosphere.

The arrangement of the equipment discussed above, in flow line, is shown in drawing No. 13618.

Drawing 15620-0 shows the arrangement of equipment and flow of materials in the refining section of the building. The "hay" from the cooling chambers goes by means of the Redler conveyor to the four steam-jacketed melting kettles equipped with agitators. Vapors from these melters are scrubbed by water in Lurgi mechanical scrubbers. From the melting kettles the molten crude phthalic flows by gravity to storage tanks (steam-jacketed). From the latter it flows to two refining stills heated by means of hot water under 68 atmospheres pressure (equipment made by Bamag-Meguin at Giessen). The refining stills are equipped with packed columns, water-cooled condensers, overhead receivers for separating vapors from condensate and barometric legs which deliver the condensate to small jacketed tanks which feed the flaking rolls. From the latter the flaked phthalic anhydride goes to the storage bins.

Vapors from the refining still condensers pass to sublimate catchers, then through caustic soda scrubbers and to the vacuum pumps. Residues from the two large refining stills are transferred to two small hot water-heated stills for further distillation. The vapors from these stills are handled in the same manner as those from the large stills.

According to Dr. Dilthey, the naphthalene used in this plant will have a freezing point of 80°C. The catalyst will be of the vanadium pentoxide type. Enough catalyst is on hand to charge the converters at the start. This catalyst probably was made at Ludwigshafen.

According to Mr. Kutsch, it is expected that the catalyst will last at least ten years and that the converters will not need to be opened in that time. Two of the converters intended for this plant did not arrive before shipments of materials to the plant were stopped. They probably are at the Ludwigshafen plant.

OTHER PRODUCTS DERIVED FROM COAL

Following the visit to the phthalic anhydride plant with Mr. Kutsch, various products made by I.G. Farbenindustrie were discussed briefly with Dr. Dilthey and Dr. Hamann. Those products which are made from materials derived from coal were of especial interest.

Crude benzene is refined at this plant. From the pure benzene are made large quantities of aniline by nitration followed by reduction with iron. Yellow and black iron oxides are produced as by-products which were said to be excellent pigments for use in the building industries.

Toluene is chlorinated to make benzyl chloride, benzal chloride and benzene trichloride. By the use of alkalies benzyl alcohol, benzaldehyde and benzoic acid are produced. Benzoic acid also is made from toluene, using sodium bichromate, by the Bozel-Maletra process. Parachlorbenzoic acid is produced for use in the making of saccharine.

Solvent naphtha from the crude benzol refining plant is used in the resin plant.

Naphthalene will be used after the first of the year for the making of phthalic anhydride as stated above. However, phthalic anhydride produced by I.G. at Ludwigshafen has been used in the past in large amounts at Uerdingen for making alkyd resins.

ALKYD RESINS

Following the general discussion reported above, the alkyd resin plant was visited with its superintendent, Dr. Kinsky. It is all housed in one building on one floor and balcony. On the balcony are eleven stainless steel resin kettles equipped with agitators. Seven of these kettles are of 4½-tons capacity each and are heated by gas (the burner is located below the kettle in a jacket that extends up the sides almost to the top). These kettles have stainless steel coils on the inside for cooling with water. The other four kettles are of 6½-tons capacity and they are heated by steam from the boiler house at 60 atmospheres pressure. These same coils are used for cooling (with water).

No vacuum is used on the kettles but inert gas is bubbled through the reaction mixture each time a batch of resin is made. The inert gas is also used for blowing the finished resins to the zinc-coated drums in which they are shipped. The inert gas used for this purpose is usually nitrogen made by the Linde process but carbon dioxide also is used at times.

The principal raw materials used in this plant are phthalic anhydride, glycerol, hexanetriol, pentaerythritol, 3-methalol propane, 3-methalol ethane, castor oil, linseed oil, tung oil, and vorlauf fettsäure (a product derived from Fischer-Tropsch paraffin).

According to Dr. Kinsky, alkydal T, suitable for air drying and baking purposes, comprised at least 75% of the total output of this plant. This product contains the following: 55% linseed oil, 18% glycerol, and 35% phthalic anhydride. The linseed oil and glycerol are mixed together and heated at 260°C. for 5-6 hours or until the mixture is soluble in alcohol. The mixture is cooled to 150°C and the phthalic anhydride is added. The temperature is then raised to 240-250°C and the product is sampled and tested for acid value and viscosity. When proper values are reached the product is cooled to 150°C and is filled into zinc-coated drums. Alkydal "T" is made in three consistencies "TN", "TM", and "TH". Solutions of these different grades of alkydal "T" are made in benzene using 25% to 40% benzene. (The "T" comprises 60%-75% of the final solution.) The solutions are made in mixing kettles located on the ground floor below and in front of the resin kettles that stand on the balcony. The solution kettles are provided with mechanised stirrers and means for heating and cooling.

According to Dr. Kinsky, comparatively small amounts of non-drying resins are made. They contain castor oil or vorlauf fettsäure. A slow drying resin called Alkydal ST also contains vorlauf fettsäure.

In addition to the alkyd resins, this plant makes large amounts of a plasticizer called Eleol. It is a plasticizer for nitrocellulose and polyvinyl chloride. It contains three mols of vorlauf fettsäure (3200 kilos) and one mol of hexanetriol (1300 kilos). These ingredients are heated together in a resin kettle with inert gas and mechanical agitation to 150-190°C. Water distills off during this period. The reaction product is then cooled to 50-60°C and washed with sodium carbonate solution to remove excess vorlauf fettsäure, then centrifuged to separate the raw Eleol and soap solution, dried by heating in a kettle to 100°C

while agitating with inert gas, bleached with tonsil (activated carbon), filtered through a plate and frame filter press and transferred to storage tanks or drums.

The alkyd resin plant was undamaged by the war and is able to operate at full capacity if sufficient raw materials are available.

I.G. FARBENINDUSTRIE, SCHKOPAU

The primary purpose of a visit to this target on January 5th was to inspect a Phenosolvan installation for the recovery of phenols from waste waters.

Information to the effect that there was such an installation at Schkopau proved to be incorrect. However, Dr. Alfred Dierichs, who had worked with the process at Leverkusen furnished a copy of an article written by himself, and published in "Chemiker Zeitung" 1942, Volume 66, No. 27/28, page 288. This document has been microfilmed with documents on dephenolization of waste waters collected from other sources.

A list of the products manufactured at Schkopau was requested in order to determine to what extent products derived from coal tar were employed as raw materials at that plant. The accompanying list of products was obtained. The list gives the capacity of the plant for each chemical in May 1945 and also as it would be after damage caused by air attacks had been repaired.

Documents relating to the following products were received:

Palatinol A.H. (diethyl hexyl phthalate) a plasticizer made from phthalic anhydride and ethyl hexanol. Emulgator 1000 or Nekal B.X. (sodium salt of dibutylnaphthalene sulfonic acid) made by sulfonating a mixture of butanol and hot pressed naphthalene to form dibutylnaphthalene -monosulfonic acid and converting the latter to the sodium salt with caustic soda and sodium hypochlorite. It was used as an emulsifier for Buna.

Phthalic anhydride, made by the oxidation of naphthalene with air using a vanadium catalyst containing potassium sulfate. (Described in CIOS Report Item No. 22, File No. XXVIII-29).

Polystyrol E.F., made by polymerizing styrene in a weak, alkaline soap solution with potassium persulfate as catalyst. The polymer is precipitated from the latex by acidifying with formic acid. It is then filtered, washed and dried. (Described in CIOS Report, Item No. 22, File No. XXVII-6); "Diskontinuierliche Destillation B 17." This document relates to the refining of butanol-residue, palatinols and solvents using discontinuous distillation. It also contains a section on the continuous production of butylacetate from butanol and acetic acid.

Because of insufficient time it was impossible to visit the plants in which the products mentioned above were made.

Schkopau, May 23rd 1945

CAPACITY, TONS PER MONTH

	Present position	After repair of bomb damage.
Äthylbenzol	2 700	2 700
Styrol	1 600	1 600
Polystyrol	50	50
Sinterkalk	15 000	15 000
Hydrieräthylen	2 200	2 200
Essigsäure	1 200	1 200
Aceton	200	200
Hexantriol	300	300
Carbid	26 000	30 000
Acetylen	9 000	9 000
Acetaldehyd	11 000	11 000
Butylenglykol	11 000	11 000
Butadien	5 000	5 000
Buna S	4 800	6 000
Emulgator 1ooo	300	300
Zahlenbuna	270	270
Spritäthylen	600	600
Äthylenoxyd	350	700
Glykol	700	700
Chlor	3 000	4 000
Chlorwasserstoff	1 600	2 100
Natronlauge	3 400	4 300
Vinylchlorid	3 200	3 200
Igelit PCU	35o	600
Chloräthyl	120	120
Tetrachloräthan	700	700
Trichloräthylen	400	400
SS-Öl	500	500
Aluminiumchlorid	700	700
Formaldehyd	750	750
Butindiol	500	500
Butandiol	500	500
Tetrahydrofuran	300	300
Phthalsäure	270	540
Palatinol	150	150

Chemische Werke Albert, Wiesbaden-Biebrich

Alkyd and Phenolic Resins

Chemische Werke Albert, Albertstrasse, Wiesbaden-Biebrich, was visited on September 10th. The persons interviewed were Dr. Arthur Greth, Manager and Chemist for the development of synthetic resins and Dr. Stiegel, plant chemist. The chief manager of the plant is Dr. Winkler but he was not interviewed.

The purposes of this visit were as follows:

1. To find out whether this company, as reported by one investigator, makes phthalic anhydride.

2. To learn whether and how phthalic anhydride or other coal tar derivatives are used by this firm.

3. To obtain for the TIIC-Forest Products Group, at the request of D. G. Coleman, as much information as possible about the formula, manufacturing procedure and uses of Albert's glue #319-J

In answer to the first question, Dr. Greth said that his company does not make phthalic anhydride; that all phthalic in Germany is made by I.G. Farbenindustrie, the principal plant being at Ludwigshafen and another at Schkopau.

Concerning item No. 2, Dr. Greth said that his firm is a large user of phthalic anhydride and phenols. Alkyd resins of both drying and non-drying types are made. Dr. Greth mentioned that whereas he uses adipic acid in some non-drying types, one company in America (Resinous Products Co.) uses sebacic acid.

Dr. Stiegel and Dr. Greth furnished the following information about their alkyd resins:

#246 B - Linseed oil type (air drying)

Formula:

1930 kgs. linseed oil
1190 " phthalic anhydride
565 " glycerol

Procedure:

The linseed oil and phthalic anhydride are placed in the kettle together and heated to 200°C. The glycerol is then run in and the heating is continued until a temperature of 240°C is reached. Acid number and absolute viscosity in centipoises (determined by Ubbelohde capillary viscometer) are determined.

When the acid number is about 30, vacuum (about 700 mm) is applied to the kettle and held until the acid number is below 10 and the viscosity is between 35 and 40 for a 1:1 solution of the resin in toluene. The resin is then cooled to 180°C and the kettle is emptied.

Note: Other grades of this same alkyd having higher viscosities are made using the same formula but the vacuum is applied for a longer time.

#125-Z Linseed oil long oil-type alkyd (air drying)

Formula:

1800 kgs. linseed oil
480 " Phthalic anhydride
225 " glycerol

Procedure:

Same as for #246-B. Acid number of product should be below 10 and the viscosity about 20 for a 1:1 solution in toluol.

#378-B Drying type alkyd made with synthetic fatty acid

Formula:

1140 kgs phthalic anhydride
1270 " linseed oil
250 " synthetic fatty acid. Purchased from Henkel. Consists of the first fraction from the distillation of synthetic fatty acid made from Fischer Tropsch paraffin. Chain length 4 to 9. Called "Vorlauf Fettsaure" in Germany.

850 kgs hexanetriol (I.G. Farben)
170 kgs pentaerythritol

Procedure:

All of the ingredients are placed in the kettle together and the mixture is heated to 215°C. It is held at that temperature until the acid number is 35 when the vacuum is applied and held until the acid number is about 15 and the viscosity 30 - 35 centipoises for a 1:1 solution in toluene.

#358-D (formerly) #248 D air drying type made with castor oil (middle oil type)

Formula:

2240 kgs castor oil (not dehydrated)
1000 " phthalic anhydride
440 " glycerol

Procedure:

Put all ingredients in kettle together. Heat as rapidly as possible to 270°C. Apply vacuum (700 mm). Determine the viscosity. The viscosity drops to a minimum and if heating is continued at the same temperature it will rise again. The minimum is the point at which dehydration is completed. It should be about 15 centipoises (1:1 in toluene). At this point the temperature should be lowered to 240° and heating continued, at this temperature, until the viscosity is 35 centipoises and acid number below 10. Cool to 180°C and empty the kettle.

Note: When resin of this type is to be used in white enamels it is desirable to substitute soya bean oil for a part of the castor oil.

#380-D formerly #198-Z air drying - castor oil (long oil) type

Formula:

1860 kgs castor oil
615 " phthalic anhydride
260 " glycerol

Procedure:

Same as for #358-D

#251-G Baking type - Castor Oil (short oil type)

Formula:

1025 kgs castor oil
900 " phthalic anhydride
500 " glycerol

Procedure:

Same as for #358-D except final viscosity (1:1 in toluene should be 300 - 400 centipoises and acid number about 20).

The following are some of the non-drying types of alkyds made by Chemische Werke Albert:

#166-S Middle oil type - non-drying alkyd

Formula:

920 kgs phthalic anhydride
1630 " synthetic fatty acid
1020 " pentaerythritol

Procedure:

All ingredients placed in kettle together. Mixtures heated at rate of 15° per hour to 240°C, held at that temperature until acid number is 10-15 and viscosity (1:1 in toluene) 20-30 centipoises. Cooled to 180° and emptied.

#215-S Long oil type - non-drying alkyd

Formula:

2060	kgs	synthetic fatty acid
780	"	phthalic anhydride
930	"	pentaerythritol

Procedure:

Same as for #166-S except the viscosity of final product (1:1 in toluene) should be 8-12 centipoises.

#393-S Non-drying alkyd - adipic acid type

Formula:

1970	kgs	synthetic fatty acid
860	"	adipic acid
955	"	pentaerythritol

Procedure:

Same as for #166-S. Final viscosity (1:1 toluene) 80-100 centipoises and acid number 10-15.

#398-A Non-drying adipic acid type

Formula:

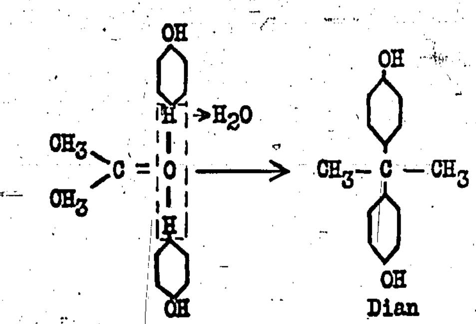
196	kgs	adipic acid
1600	"	triethylene glycol
350	"	glycerol

Procedure:

Same as for #166-S. Final viscosity (1:1 in <u>butyl acetate</u>) 80-100 centipoises and acid number about 15.

Phenolic Resins

Chemische Werke Albert makes many types of phenolic resins. For some of them it makes a special base from phenol and acetone which it calls Dian. The reaction is as follows:



Dian

For "Albertols", modified phenolic resins, diphenylol propane is condensed with formaldehyde. Albertols are described as combinations of phenolic resins with natural rosin acid (abietic acid).

Following is a typical formula for a modified (plasticized) phenolic for varnish purposes as made by Albert.

#308-V Modified (Plasticized) Phenolic

Formula:

1080	kgs	cresylic acid (35% meta and balance para and ortho)
2000	"	formaldehyde (30%)
40	"	ammonia (25%)
800	"	butanol
1250	"	tall oil ester (glycerol) (contains about 35% abietic acid and balance is fatty acid similar to linseed oil fatty acid)
850	"	xylene

Procedure:

The cresylic acid is placed in the kettle and heated to 60°C. Then a mixture of the formaldehyde and ammonia is run in with cooling. After the formaldehyde-ammonia mixture has all been added the water is distilled off at 50-55°C under vacuum (700 mm). Dilute with the butanol and add the tall oil ester. Then distill off about 50 liters of butanol and water per hour. When a viscosity (1:1 in toluene) of 140 centipoises is reached, dilute with the xylene. The final viscosity should be 20-25 centipoises and the solid content 65%.

Glue No. 319-J

Information concerning the manufacture of glue No. 319-J, requested by Mr. Coleman, was submitted to Mr. B. H. Wilcoxon in a letter dated September 10, 1945. A copy is hereto attached.

Other Products

A list of the synthetic resins and products based on resins manufactured by Chemische Werke Albert at Wiesbaden-Biebrich accompanies this report.

Manufacturing Facilities

The resin making plant on Albertstrasse, Biebrich, was heavily damaged by bombs on September 19, 1944. The Dian plant was completely destroyed, also a building which contained most of the resin kettles. A new building which was to house the new resin plant with gravity flow of materials was damaged but not destroyed. Six complete, stainless steel kettles, of 5000 liters capacity each, had been installed in the new building but, before the air raid, were moved to the Schönebeck plant near Magdeburg so they are no longer available for use by Albert.

Altogether twelve kettles were destroyed by the raid at Biebrich but four kettles of 5000 liters capacity are now operating and two 500 liter kettles are making phenol-formaldehyde resins. Also two kettles of 2000 liters capacity and some smaller kettles are available for the same purpose.

Before the bombing "Albert" was making about 1000 tons of varnish resins per month. Present production is about 250 tons per month of which phenolics amount to about 100 tons and alkyds 150 tons.

The preferred method of heating resin kettles as used by Albert is by means of hot water. Water circulates through heating coils in a furnace and then through a coil welded to the surface of the kettle. Albert has replaced most of its steam jacketed kettles with water heated kettles of this type. The builder of the hot water heating system is Samesreuther, located in Butzbach (Oberhessen) near Friedberg.

B. H. Wilcoxon
TIIC, Miscellaneous Chemicals Group

This afternoon during an interview with Dr. Arthur Greth and Dr. Stiegel of Chemische-Werke Albert, Wiesbaden-Biebrich, I obtained the following information concerning glue No. 319-J. This is one of the plastic glues mentioned in Mr. D. G. Coleman's letter addressed to you and dated September 6, 1945.

Formula for No. 319-J

153	Kgs. of	phenol
326	" "	formaldehyde (30%)
3.5	" "	33% caustic soda
9.6	" "	para toluene sulfonic acid of 50% concentration in water solution
62.5	" "	softener No. 398 A
37.5	" "	methanol

Manufacturing Procedure

To make glue No. 319-J the following procedure is followed:

The phenol, formaldehyde and caustic soda are charged together in a stainless steel, steam heated kettle provided with a mechanical agitator. The temperature of the mixture is raised to 90°C and this temperature is maintained until the mixture becomes turbid. The mixture is then cooled to 60°C and is neutralized with the para-toluene sulfonic acid solution. The mixture is then allowed to settle and the water layer is decanted. The resin is then dehydrated under approximately 700 mm. vacuum and heating, under vacuum, is continued until the viscosity (when the resin is dissolved 1:1 in ethyl alcohol) is 18-20 centipoises. (The viscosity determination is made with an instrument of the Ubelohde, capillary, type.) The resin is then diluted with the methanol and the 398A softener is added. The 398A softener is prepared as follows:

Preparation of No. 398A Softener

Formula:

195	Kgs.	adipic acid
1500	"	triethylene glycol
350	"	glycerol

Procedure: The three ingredients are mixed together in the kettle and heated at the rate of 15°C per hour to 240°C. This temperature is maintained until the acid number is about 15 and the viscosity of a 1:1 solution in butyl acetate is between 80 and 100 centipoises.

Attached is a three-page discussion (In German) of the ways in which glue No. 319-J may be used. Of particular importance is the part that just before this glue is to be used it must be mixed cold with a catalyst in the proportions of 100 parts 319-J to 10 parts of catalyst. The catalyst is prepared as follows: 50 parts of paratoluene sulfonic acid, 25 parts of methanol and 25 parts of 398 A are mixed cold to form a solution which is the catalyst.

<u>Origin of 319-J</u>

Dr. Greth said that the origin of 319-J was as follows: The glue originally used by the air ministry was Kaurit (a water soluble urea resin from I. G. Farbenindustrie). Its water and mildew resistance was not sufficiently great, especially in the tropics, so the Air Ministry asked Chemische-Werke Albert to develop a more suitable glue. 319-J was produced and commercial manufacture was just starting when the plant was bombed on September 19, 1944. According to Dr. Greth no large quantity of 319-J has been made.

Another glue used during the war was P-600 made by Dynamit at Troisdorf. It was a water soluble phenolic. Polystal, another resin glue made by I. G. was produced at Ludwigshafen. Dr. Greth said that he was not able to give the compositions of P-600 or Polystal. The writer will inquire further about Polystal and Kaurit on visits to I. G. plants.

E. O. Rhodes

Gebrauchsanweisung

CHEMISCHE WERKE ALBERT

Wiesbaden-Biebrich

Albert-Leimharz 319 J

Das Albert-Leimharz 319 J ist zur Kaltverleimung von Holz jeder Art und Bearbeitungsstufe bestimmt und kann auch zur Warm-oder Heissverleimung angewandt werden. 319 J bindet sehr rasch ab und ergibt deshalb schon innerhalb kurzer Zeit sehr feste und dauerhafte Leimverbindungen, die allen Beanspruchungen standhalten und gegen Wasser, feuchte oder trockene Wärme wie auch gegen Mikro-Organismen unempfindlich sind.

Das Albert-Leimharz 319 J ist in mehrjähriger intensiver Forschungsarbeit entwickelt worden und hat bei Amtsstellen und Grossverbrauchern auch die Prüfungen unter erschwerten Bedingungen mit vollem Erfolg bestanden.

Allgemeines

Zum Albert-Leimharz 319 J gehört der Härter H 25. Beide sind flüssig und werden zur Herstellung des gebrauchsfertigen Leimes miteinander vermischt.
Bei kühler und trockener Legerung sind Harz und Härter für sich monatelang haltbar. Eine etwaige Viskositätszunahme des Leimharzes, die nur sehr langsam erfolgt, ist ohne Einfluss auf Leimeigenschaften und Verarbeitbarkeit. Die Behälter sind nach Entnahme gut zu schliessen.

Ansetzen des Leimes

Das Ansetzen des Leimes muss in Gefässen aus Steingut, Porzellan, Glas, Emaille oder Holz erfolgen, Gefässe und Rührer aus Metall sind unbrauchbar.
Zur Herstellung des gebrauchsfertigen Leimes werden im Normalfall 100 Gewichtsteile Harz mit 10 Gewichtsteilen Härter H 25, also im Verhältnis Harz zu Härter wie 10 zu 1, gründlich unter kräftigem Rühren gemischt, wobei die ursprünglich violettrote Farbe ins Bläuliche umschlägt. Während für das Harz das Abwiegen einfacher, genauer und deshalb vorzuziehen ist, kann der Härter evtl. im Messglas abgemessen

werden; 10 Gramm H 25 entsprechen rund 8,9 Kubikzentimeter, spezifisches Gewicht = 1,13. Auf 1 kg Harz sind somit 100 Gramm oder 89 Kubikzentimeter Härter H 25 anzuwenden.

Geringe Abweichungen nach oben oder unten sind zulässig, sollten jedoch nicht ohne besonderen Grund erfolgen, da Harz und Härter genau aufeinander abgestimmt und eingestellt sind.
Nach erfolgter sorgfältiger Durchmischung ist der Leim sofort gebrauchsfertig. Er bleibt je nach Ansatz und Raumtemperatur 3 bis 5 Stunden verarbeitbar und soll nach dem Ansetzen laufend verbraucht werden. Der Leim ist verwendbar, solange ein zügiger Aufstrich möglich ist.
Die beste Arbeitstemperatur liegt zwischen 18 und 22°C. Bei weniger als 16°C bindet der Leim zu langsam ab, bei mehr als 24°C ist Einstellen der Leimtöpfe in kaltes Wasser zu empfehlen, um die Gebrauchsdauer zu verlängern. Es ist zweckmässig, lieber öfter ein kleineres Quantum Leim anzusetzen und den frischen Ansatz auf kleinere Arbeitsgefässe zu verteilen. Am besten werden stets die gleichen zuverlässigen Arbeitskräfte mit Leimansatz und -ausgabe beauftragt.

Angeben des Leimes

Der fertige Leim ist klar, dünnflüssig und lässt sich mit Pinsel, Bürste oder Spachtel auftragen. Der Auftrag erfolgt satt, aber nicht im Übermass. Stets sind die beiden zu verleimenden Flächen mit Leim anzugeben.
Ein Verdünnen des gebrauchsfertigen Leimes ist nicht erforderlich. Soll für Spezialzwecke ein ganz besonders dünnflüssiger Leim hergestellt werden, so dürfen auf 100 Gewichtsteile 319 J höchstens 5-10 Gewichtsteile Methanol oder Äthanol (Sprit) zugegeben werden, wobei zu beachten ist, dass jeder Zusatz die Abbindezeit verlängert. Die Zugabe von anderen organischen Verdünnungs-oder Lösungsmitteln ist nicht statthaft.
Bei allen Leimarbeiten, die mit verdünnten Ansätzen erfolgen, ist zweimaliger Auftrag ratsam.
Nach dem Angeben des Leimes bleiben die Leimflächen etwa 30 Minuten offen liegen. Dies Antrocknenlassen ist besonders beim Verarbeiten von Harthölzern, Sperr-und Schichtholz und beim Verleimen grösserer Flächen erforderlich. Kiefer kann u.U. ohne Antrocknen verarbeitet werden.
Grössere Leimflächen werden nach dem Antrocknenlassen zweckmässig durch Zahnspachtel, Kamm oder dergl. wieder angefrischt. Während des Antrocknens dürfen die Teile nicht in die Nähe von Öfen, Heizkörpern usw. gebracht werden, da dann ein ungleichmässiges Antrocknen erfolgt, das zu Fehlleimungen führen kann.

Pressen und Weiterverarbeiten

Nach dem Antrocknenlassen werden die Einzelteile zusammengefügt und möglichst bald in den Pressvorrichtungen, z.B. Zwingen, unter Druck

gesetzt. Es ist zu vermeiden, dass zusammengefügte Teile ohne Druck liegenbleiben. Bereits nach 3-5 Stunden Presszeit ist die Leimfestigkeit so gross geworden, dass die Bauteile wieder aus den Pressvorrichtungen entnommen werden können.
Bei allen Teilen mit Eigenspannung sowie bei grösseren Flächen und sonstigen schwierigeren Werkstücken ist die Einhaltung einer längeren Presszeit wünschenswert; die optimale Einspannzeit kann in solchen Fällen durch einen Vorversuch leicht ermittelt werden.
Die Festigkeit der Verleimungen steigt auch nach dem Ausspannen weiter an und erreicht erst nach mehreren Tagen ihren vollen Endwert. Es ist daher vorteilhaft, die verleimten Teile vor der Weiterverarbeitung einige Zeit, mindestens 12-24 Stunden, liegen zu lassen; dies gilt besonders dann, wenn die Weiterverarbeitung die verleimten Teile mechanisch stark beansprucht.

Warm-und Heissverleimungen

Das Albert-Leimharz 319 J ist auch zur Warm-und Heissverleimung gleich gut geeignet. Grundsätzlich gelten dabei für das Durchmischen, Auftragen, Antrocknen die Regeln der Kaltverleimung. Die Menge des Härters kann jedoch auf die Hälfte bis ein Viertel der zur Kaltverleimung üblichen Menge herabgesetzt werden. Bei Warm-oder Heissverleimung werden daher im allgemeinen auf 100 Gewichtsteile Leimharz nur 5 bis 2,5 Teile Härter H 25 zugesetzt.
Die Presszeit sinkt dann je nach Werkstück, Leimansatz und Heiztemperatur auf 3 Minuten bis 2 Stunden. Auch hier ist es besonders bei Serienanfertigungen zweckmässig, das Optimum an Presszeit, Heiztemperatur und Härtermenge durch einen Vorversuch festzulegen. Bei Warm-und Heissverleimung färbt sich der Leim dunkelrot, was sonst erst nach einiger Zeit eintritt.

Verleimung vorkonservierter Hölzer

319 J lässt sich auch auf Hölzern verarbeiten, die beispielsweise mit Aviatin E-Firnis vorkonserviert sind, ebenso auf solchen, die eine 319 J-Leimschicht aufweisen, Vor der Verwendung anderer Konservierungsmittel sind entsprechende Versuche durchzuführen.

Sonstige Hinweise.

Erhärtete Leimreste sind nicht mehr verwendbar, sie können auch durch Lösungsmittel oder sonstige Zusätze nicht wieder in verwertbare Form übergeführt werden. Am besten werden die noch frischen Leimreste nach Arbeitsschluss aus den Gefässen entfernt. Hart gewordene Leimreste können durch Auskochen mit alkalischen Reinigungsmitteln losgelöst werden.
Zur Händereinigung leistet ein Gemisch aus gleichen Teilen Wasser und Methyl-oder Äthylalkohol gute Dienste.

Field of Production of Chemical Works Albert

Wiesbaden-Biebrich

I) Work Albertstreet, Amoeneburg

A: Artifical resins; chiefly used as raw material for the fabrikation of varnishes, lacquers; artifical resins for varnishes.

1) Albertols: modified phenolic resins; combination of phenolic resins with natural rosin acid (abietic acid); Chief field of application: fabrication of oil-varnishes, combination lacquers, cellulose lacquers, spirit varnishes, printing inks, tinctures.

2) Alphenates: mixed ester of phenolic resins; for oil varnishes and cellulose lacquers.

3) Alresene: pure, oilsoluble phenolic resins; woodoil-reactive, pure alkylphenolic resins for varnishes with a special resistance against water, acids and alkalies.

4) Alnovols: pure, spirit soluble phenolic resins; for spirit varnishes, polishes, matting varnishes, adhesive-substances.

5) Phenodure: pure, spirit soluble, hardening phenolic resins; binding agent for different purposes e.g. for the production of grinding wheels, brake linings, carbon brushes.

6) Durophene: plastified, thermo-setting phenolic-resins; for stoving varnishes, enamels, electro-insulating varnishes, nitrocellulose lacquers.

7) Alresate: maleic acid resins; pale artifical resins; free of phenol, with excellent light and colourresistance; for oil varnishes and cellulose- and combination lacquers.

8) Alftalats: Alkyd resins i.e. mixed esters of dicarbonic acids (e.g. phthalic acid) and fatty acids; for air-drying and stoving varnishes of every kind, cellulose lacquers, combination lacquers, enamels, often in combination with Albertols and Alresates.

9) Resamine: Urea resins; for varnishes and enamels possessing a high heat resistance; combination with alkyd resins and nitrocellulose; binding agent for stoving enamels possessing a high gloss.

A branch factory for the types of artifical resins mentioned above exists at Schönebeck/Elbe.

B: Special Products based on artifical resins:

1) Rosin size for the paper industry based on natural rosin and artifical resin;
universal auxiliary agent for the fabrication of writing- packing- and similer papers to make them waterproof and to give them mechanical stability and smoothness; especially impregnating media for special papers with higher water resistance.

2) Glue for wood based on phenolic resins;
glues for wood for the hot and cold process obtaining a special water- heat- and tropical resistance; for the production of plywood, hardwood and for glueing purposes for aeroplane construction, wooden carriage bodies, boat building, furniture industries.

3) Emulsions for textiles, based on artifical resins; to impregnate yarns and fabrics, especially made of artifical silk, in order to improve the firmness when washing, to render it wind-proof and resisting against putrefaction, further to reduce shrinking of the material.

C: Moulding Powders:
Moulding powders are a combination of hardening artifical resins and fillers of different kind.

They are pressed in press moulds under the influence of heat and pressure to obtain the pressed articles.

1) Alberit-Quick pressing moulding powder type 1: Asbestos fiber used as filler; this moulding powder enables the preparation of high-heat resisting insulating material.

2) Alberit-Quick pressing moulding powder type S: wood powder used as filler; this moulding powder is used for the production of articles for daily use and for electrical insulating material.

3) Alberit-Quick pressing moulding powder type T: shavings and layers of textiles used as filler; apart from insulating properties high mechanical resistance.

4) Alberit-Quick pressing moulding powder type Z: cellulose used as filler; high mechanical resistance and insulating properties.

DEUTSCHE GASRUSSWERKE G.M.B.H., DORTMUND.

Two visits were made to this plant, - on September 21th and ~~November 9th 1945. The persons interviewed were Dir. Bache und~~ Dr. Herrmann. Damage to this plant by air raids was confined principally to the office building.

Anthracene residue, in the form of a coarse powder, is stored in large bins from which it is moved by an overhead crane to melting kettles equipped with mechanical stirrers. From the melters it is pumped to feed tanks from which the molten anthracene residue flows into gas heated vaporizerswhere the vapors of the residue are combined with preheated coke oven gas. The mixture of gas and vapors is circulated through pipes heated by gas flames to the machines which make the carbon black. In each machine is a rotating,water cooled roller beneath which is a row of burners for the gas-vapor mixture. Incomplete combustion of the mixture at the burner tips causes carbon black to be deposited on the roller. It is removed by a doctor blade and conveyed to a storage and bagging center. The process is shown diagrammatically in the accompanying flow diagram on page 150.

Peak production at this plant was reached in 1942 when its capacity was used to the extent of 90.1%. The quantities of carbon black made were 13,897 tons first quality and 166 tons second quality - total 14,063 tons. The quantities of raw materials used were 49,783,000 cubic meters coke oven gas, 21,798 tons (metric) of anthracene residue, and 33,614 tons of steam. The equivalent quantities per kilogram of carbon black are 3.54 cubic meters coke oven gas, 1.55 kilograms anthracene residue and 2.4 kilograms steam. Power consumption was 0.71 k.w.h. per kilogram of carbon black. The yield of carbon black is 72.5% when referred to anthracene residue actually vaporized or it is 64.4% of the original anthracene residue used. The balance is pitch residue (7.7%) and combustion loss (28%).

Prices paid in 1942 for raw materials were: anthracene residue 5.40 RM per 100 kilograms and coke oven gas 0,015 RM per cubic meter. The cost of the finished carbon black was 0,75 RM per kilogram.

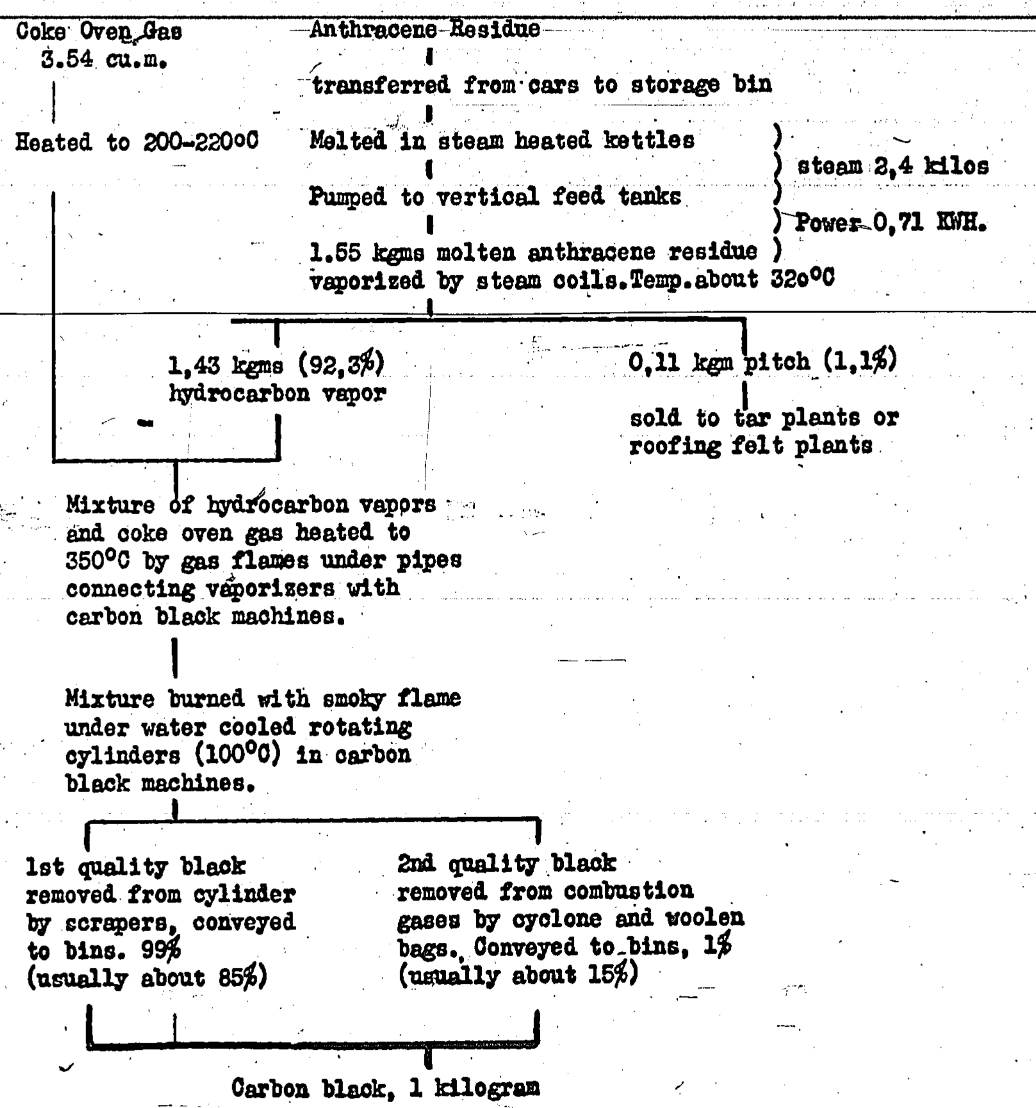
Anthracene oil can be used satisfactorily at the Dortmund plant but is much more expensive (12.00 RM per 100 kilograms).Naphthalene is a satisfactory raw material but in addition to being more expensive (9.00 RM per 100 kilograms for 76 degree naphthalene and 11,50 RM for hot pressed) some changes would have to be made in equipment and operating procedures at Dortmund to use naphthalene there. The Kalscheuren plant at Cologne, having 25% the capacity of the Dortmund plant, can use naphthalene and anthracene residue interchangeably.

Plants that produce lamp black by partial combusting of anthracene oil are located at Worms, Zons and elsewhere.

Carbon Black Production at Dortmund-Ruhr

Deutsche Gasrusswerke G.m.b.H.

(Average conditions in 1942 for production of 1 kilogram of product)



RUHRÖL G.m.b.H. BOTTROP-WELHEIM

The Bottrop-Welheim hydrogenation plant of Ruhröl G.m.b.H. was visited on September 21, 1945. The persons interviewed were Direktor Dr. Frese and Dr. Broche.

This plant was of particular interest because of

(1) its use of a mixture of 70% briquette pitch and 30% coal-tar oil as the feed stock for the hydrogenation plant,

(2) the digestion of coal in middle oil from the hydrogenation process followed by filtration through ceramic filters to obtain a coal extract, and

(3) the vacuum distillation of the coal extract to obtain a high melting point pitch suitable for charging directly into coke ovens to make low ash coke for carbon electrodes.

Also a residue from liquid phase hydrogenation (Entschlammung) went to the tar-distilling plant of the same organization for distillation to briquette pitch in admixture with coal tar.

The hydrogenation of the pitch-tar oil mixture, as it was carried out at this plant, has been discussed in detail by C. Cockram in CIOS Repot Item No. 30 File No. XXX-104.

The coal extraction and filtration process was also discussed by Cockram in the CIOS Report mentioned above, and by Lowry and Rose in CIOS Report, Item No. 30. Additional information about this type of filter is presented in a separate report by this investigator, covering a visit to Schumachersche Fabrik in Bietingheim where the ceramic filter rings are made. The following description of the filtering operation was furnished by Dr. Frese

"In a pressure vessel are mounted vertical tubes composed of porous stone rings, the so-called candles/or cartridges. The liquid pumped into this vessel flows around the/outsides of the candles. An absolute pressure of 4 to 5 atmospheres is gradually applied to the mixture, causing the liquid portion to penetrate into the interior of the tubes from which it is drawn off at the bottom, while the solid components of the mixture deposit on the exterior surfaces of the candles. When the filter cake has grown to a certain thickness, the raw product is poured back from the filter cake into the raw product feed tanks by means of CO_2. The filter case is then filled with washing filtrate, pure filtrate or solvent, depending on the state of the product and is forced through the filter cake into the candle, so that the residual carbon cake finally contains only solvent. After emptying the filter case the cake is dried by hot CO_2. Beneath the filter case there is a conical funnel sealed by a patented closure. The latter is opened and CO_2 is introduced into the interior of the candles under an absolute pressure of 8 atmospheres. By this means the cakes are pushed off the filter rings. They fall down into the so-called "protecting gas tank" from which the escaping CO_2 is conducted into the open air through pipes terminating on the roof. The lower part of the protecting gas tank is provided with a discharging device which carries the filter cake into a chain conveyor below. In the gas-tight compartment of this conveyor the residue coal is transferred to building 13a where the solvent which it contains is eliminated."

Each of the three filters used for the above described operation consisted of a steel case with cone bottom inside of which were 27 filter tubes or candles. Each candle was made up of 68 porous ceramic rings, clamped together to form a porous tube 2050 mm. long, 120 mm. O.D., and 80 mm. I.D. (each ring was 20 mm. high). After about 5000 filtrations of 15 minutes each, the candles were removed from the filter and new rings were substituted for those that had been in use.

The filtered extract was distilled in a special plant consisting of a gas-heated pipe still and two gas-heated batch stills with mechanical agitators. The filtered extract was pumped through the pipe still and two gas-heated batch stills with mechanical agitators. The filtered extract was pumped through the pipe still into a batch still where the vapors were released and the distillation was finished. The distillation residue was discharged to a continuous steel plate cooling conveyor which transferred the cooled pitch into cars.

The distillation of the residue from the Welheim hydrogenation plant in combination with coal tar at the tar plant of Mathias Stinnes is discussed by this investigator in a separate report.

RUHRCHEMIE A.G., STERKRADE-HOLTEN

The primary purpose of a visit to Ruhrchemie A.G., Sterkrade-Holten, on September 19, 1945, was to find out what coal tar products, if any, were used by that company, the investigator having heard that naphthalene had been employed in connection with the Fischer-Tropsch synthesis.

Director Martin, when interrogated about this matter, stated that some naphthalene from tar plants had been used with olefins from the Fischer-Tropsch synthesis in the making of lubricating oils by the aluminium chloride process. However, he stated that lubricating oil made from a mixture of naphthalene and Fischer-Tropsch olefins was inferior to that from Fischer-Tropsch olefins alone because of poorer thermal characteristics.

(Note: At a later date it was learned that the use of naphthalene in lubricating oil in the manner mentioned above was practiced at the Fischer-Tropsch plant of Rheinpreussen at Homberg-Moers instead of at Sterkrade-Holten. This has been reported by investigators Atwell and Schroeder in CIOS Report Item No. 30 File No. XXIV-9. According to that report, Fischer-Tropsch middle oil, boiling range 250-350°C, was chlorinate at 80-100°C to 20-25% chlorine by weight. Five volumes of the chlorinated oil were reacted with two volumes of naphthalene at 70-100°C in the presence of eight volumes of Fischer-Tropsch benzine and a small amount of $AlCl_3$ as catalyst. After withdrawal of sludge, neutralization with lime, bleaching, filtering and removal of benzine, the product was vacuum distilled to make spindle oil, turbine oil and cylinder stock. According to Atwell and Schroeder "all products were stated to have a high viscosity index and great resistance to oxidation, but tests on captured samples do not support these claims".)

In further conversation with Dr.Martin he mentioned the use of "vorlauf fettsäure")produced at soap factories from Fischer-Tropsch paraffins) with phthalic anhydride from naphthalene in the making of alkyd resins. He said that these were the only uses, as far as he knew, for coal tar products in connection with Fischer-Tropsch products. No use of tar products was contemplated in connection with the operation of the Oxo process.

Following the discussion outlined above, a brief visit was paid to the Fischer-Tropsch and Oxo plants. In the Fischer-Tropsch plant the buildings were badly damaged or destroyed; but according to Dr.Martin, at least 50% of the equipment can be used again. The Oxo plant was damaged only slightly. No detailed inspection of the Fischer-Tropsch and Oxo plants was made because they have been covered by previous investigators.

GASINSTITUTE DER TECHNISCHE HOCHSCHULE, KARLSRUHE

On December 1, 1945, a brief visit was paid to the Gasinstitute der Technische Hochschule, Karlsruhe, Germany, den Schlacthausstrasse 3 for the purpose of determining whether this research institution had carried on any investigational work in recent years on coal tar or products derived therefrom.

The persons interviewed were Professor Köerting and Professor Weyrich. Professor Bunte, former director of the Gasinstitute and well known authority on gas purification, gas production, etc., died last year. Professor Köerting explained that previously the work of the Gasinstitute was of two types. One phase of its activities was the teaching of gas utilization in the Technische Hochschule. This department was conducted by Professors Bunte and Köerting. Professor Köerting hopes that this work will be resumed soon by permission of the Military Government.

The other department or section of the Gasinstitute, according to Professor Koerting, resembled the American Gas Association. It dealt with research on and the practical testing of gas and water and was financed by the gas industry.

The Gasinstitute was housed in a separate building within the plant of the Statische Werke, Karlsruhe Gaswerk, Ost. This building was almost totally destroyed but a complete experimental gas plant consisting of two horizontal retorts and all the auxiliary equipment was undamaged. Also the analytical laboratories in the basement were not destroyed. Dr. Köerting hopes to be able to use these facilities for further research work if funds for such work can be secured.

At present, Professor Köerting, Professor Weyrich, and an assistant are occupying a small room in the office and laboratory building of the gas plant and are unable to carry on any of their usual activities.

When questioned about work on tar or tar products, neither Professor Köerting nor Professor Weyrich could remember having done anything along this line within the last ten years.

The Didier Werke, inclined chamber ovens at this gas plant were visited with Professor Köerting but no description of them will be given here because they are the subject of final FIAT Report No. 566.

KAISER WILHELM INSTITUTE FÜR KOHLENFORSCHUNG, MÜLHEIM, RUHR

This research laboratory was visited on November 21, 1945, with Mr. H. P. Stephenson of North German Coal Control and Major Henri Winkler. Neither the building nor any of the laboratory equipment had been damaged during the war and research is being resumed, with permission of the Military Government, on the production of higher paraffins by Fischer Tropsch synthesis methods. The apparatus used for this work was inspected briefly, as well as laboratory fractional distillation equipment. Of particular interest in the latter connection were the rotating band columns which were said to be highly effective fractionating devices, and because of low hold-up they were considered to be especially suitable for the distillation of small samples. A description of this type of column by H. Koch, F. Hilberath, and F. Weinrotter was published in Die Chemische Fabrik 14, 387 (1941) under the title "Eine Kolonne mit Rotierendem Metallband Zur Fraktionierten Destillation kleiner Substanzmengen". A copy of this article was obtained.

Dr. Koch, Dr. Peckler, and Dr. Siegler were asked if any research work on coal tar products had been done in their institution in recent years or in the laboratories of other research institutions in Germany. They were of the opinion that little or no work of that kind had been done outside the laboratories of the larger tar distilling companies and did not know how much had been done there.

APPENDIX

Quantities of Crude Tar Processed by German Tar Distillers in 1941, 1942 and 1943.

Origins of Crude Tar Supplies Received by the Principal Tar Distillers of Germany.

Quantities of Crude Tar Processed by German Tar Distillers in 1941, 1942 and 1943

Tar Distilling Company and Plant	Crude Tar Processed (Metrictons) 1941	1942	1943
Western Tar Plants			
Gesellschaft für Teerverwertung			
(2) Duisberg-Meiderich	772,130 (Duisberg-Meiderich, Alsdorf, Rauxel)	775,385 (Duisberg-Meiderich, Alsdorf, Rauxel)	292,887
Alsdorf			39,872
(2) Rauxel			473,978
(1) Bochum-Gerthe	27,773	25,888	22,666
Rütgerswerke A.G.			
(2) Rauxel	219,610	224,623	172,247
Duisberg			61,801
Gelsenkirchener Bergwerks A.G.			
(2) Bochum (Carolinenglück)			113,324
(2) Wanne Eickel (Pluto)	175,234	188,643	29,113
Duisburg-Hamborn (Thyssen)			4,352
Dortmund (Horder Hüttenverein)			8,023
(1) Bergbau A.G., Ewald König Ludwig, Herten	43,500	50,500	58,668
(2) Teerdestillation Math Stinnes Essen-Karnap	38,634	38,482	39,333
Harpener Bergbau A.G., Dortmund	4,260	146	97
(3) Gebr. Stumm G.m.b.H., Essen-Borbeck	18,695	19 435	4,192
(2) Gutehoffnungshütte Oberhausen-Sterkrade	72,674	72,656	73,556
Steinkohlenberg w. Rheinpreussen, Homberg	28,914	27,765	24,593
(1) Dr. F. Raschig G.m.b.H. Langendreer	38,505	38,388	34,833
Preuss Bergwerks u Hütten A.G., Barsinghausen	5,425	5,427	4,723

Tar Distilling Company and Plant	Crude Tar Processed (Metrictons) 1941	1942	1943
G. B. Ranke G.m.b.H., Dortmund	5,693	5,658	5,598
Totals for Western Plants	1,451,036	1,473,196	1,463,856

Northern Tar Plants

Tar Distilling Company and Plant	1941	1942	1943
(2) Hanseatische Teerproducten Fabrik, Hamburg	31,636	32,381	34,995
(1) Norddeutsche Kohlen u. Cokeswerke, Hamburg	7,280	6,734	4,007
Hochofenwerk Lübeck A.G., Lübeck	9,776	4,068	
(2) Akt. Ges. Johannes Jeserich, Hamburg-Eidelstedt	3,564	3,025	2,584
Stadtwerke Kiel, Kiel	2,833	3,693	4,939
Totals for Northern Plants	55,089	49,901	46,525

Southern Tar Plants

Tar Distilling Company and Plant	1941	1942	1943
Chem. Fabrik Weyl, Mannheim-Waldhof (2)	69,447	67,353	72,514
" " " , München-Pasing	17,302	17,521	19,474
Dr. F. Raschig G.m.b.H. Chem. Fab. Ludwigshafen (2)	46,595	49,849	47,493
Teerdestillation Stuttgart G.m.b.H., Stuttgart (1)	20,730	18,620	15,140
Oberrheinische Ölindustrie J. Glaser, Freiburg	6,111	6,557	8,635
Süddeutsche Teerindustrie Aug. Peters, Malsch	1,910	1,712	2,171
Chem. Fab. Badenia, Phil. Keilmann, Mannheim	8,020	9,446	9,000
Chem. Werke Worms Weinsheim G.m.b.H. Worms	7,441	7,355	7,411

Tar Distilling Company and Plant	Crude Tar Processed (Metrictons) 1941	1942	1943
Teerdestillation Nürnberg, Nürnberg	5,086	6,994	5,585
Totals for Southern Plants	182,642	185,406	187,423

Saar Tar Plants

Tar Distilling Company and Plant	1941	1942	1943
Neunkircher Eisenwerk A.G., Neunkircher	27,862	26,847	27,683
Röchlingsche Eisen u. Stahlwerke, Völklingen	38,527	33,797	46,937
Halbergerhütte G.m.b.H., Brebach	15,460	16,440	15,360
Saargruben A.G., Kok u. Chem. Werke, Saarbrücken	3,465	7,037	9,364
Ernst Hugo Sarg & Co., Saarbrücken	10,579	9,916	14,040
Eisen u. Stahlwerk "Carlshütte", Diedenhofen	3,128		
Hüttenver. Westmark G.m.b.H., Hagendingen	11,041	11,913	22,600
Teerdestillation Gaudach, Gaudach	8,508	16,579	10,831
Schock-Streng Nachf. Kg. Teerwerk, Esch	3,736	3,237	6,641
Totals for Saar Plants	122,306	125,766	143,455

Eastern Tar Plants

Tar Distilling Company and Plant	1941	1942	1943
Rütgerswerke A.G.			
(2) Erkner (near Berlin)	58,545	59,804	66,892
(3) Breslau	42,492	46,284	46,798
(4) Niederau	52,117	47,601	42,943
(5) Teerag A.G., Wien	30,325	31,694	28,153
(5) Borsig Kokswerke A.G., Hindenburg	66,591	65,133	59,884
(5) Schaffgotsch Bergwerks ges. m.b.H., Gleiwitz	25,512	25,909	26,709

Tar Distilling Company and Plant	Crude Tar Processed (Metrictons) 1941	1942	1943
(5) Oberhütten, ver. Oberschles Hüttenwerke, Gleiwitz	18,916	24,272	28,461
(5) Kokerei Vereinigung G.m.b.H. Kattowitz	82,182	76,634	65,375
(5) Bergwerksverwaltung Oberschles, Kattowitz	36,547	34,435	35,261
(1) Gewerkschaft Morgenstern, Zwickau	7,208	7,132	6,536
(1) Döbelner Chem. Fab. Oswald Greiner, Döbeln	8,398	8,282	8,424
(1) Chem. u. Teerproducten Fab. Schwieck & Co., Velten	9,820	10,550	9,670
(5) Imbusch u. Melzer, Waldenburg	4,584	4,649	4,592
(5) Küstriner Teerwerke, Küstrin-Neustadt	2,760	2,620	3,150
(5) Teerindustrie A.G., Danzig	9,915	8,615	9,550
(5) Teerwerk Preussen G.m.b.H., Königsberg	2,888*	5,753	5,518
Totals for Eastern Plants	455,912	459,367	447,496
Totals for all plants	2,267,017	2,293,636	2,288,728

(*) After June 1941
(1) Intact
(2) Damaged
(3) Destroyed
(4) Removed
(5) No longer German

ORIGINS OF CRUDE TAR SUPPLIES RECEIVED BY THE PRINCIPAL TAR DISTILLING PLANTS OF GERMANY (in 1942/43)

West

Gesellschaft für Teerverwertung works Meiderich	almost exclusively from coke ovens of the mines in the Ruhr district. Minor parts also from steel works coke ovens.
works Rauxel	100% from coke ovens of the mines in the Rhine-Ruhr area.
works Alsdorf	100% from coke ovens of mines in the Aachen area.
works Bochum-Gerthe	only from coke ovens of Bergbau AG Lothringen.
Rütgerswerke works Rauxel	from coke ovens of mines in the Rhine-Ruhr area.
works Hochfeld	from coke ovens of mines in the Rhine-Ruhr area, small parts from gas works in the Rhine province.
Gelsenkirchener Bergwerks AG works Carolinenglück, Bochum	only from coke ovens of mines belonging to the concern.
works Pluto Wilhelm, W-Eickel	" "
Ewald-König Ludwig, Herten	" "
Gutehoffnungshütte Oberhausen	" "
Gew. Mathias Stinnes, Essen-Karnap	" "
Rheinpreussen, Homberg	" "

Gebr. Stumm, Essen-Borbeck	only from coke ovens of mines belonging to the concern.
Dr. F. Raschig, Bo-Langendreer	chiefly from coke ovens of the mines in the Rhine-Ruhr area, smaller quantities also from steel works coke ovens and unimportant quantities from gas works.

East

Rütgerswerke works Erkner	main source municipal gas works Berlin, unimportant quantities from gas works of the province of Brandenburg.
works Mochbern	from coke ovens of the mines in Lower Silesia, besides this from municipal gas works of Breslau.
works Niederau	from the gas works of City of Dresden and cities in the vicinity.
"Teerag" Vienna	from the municipal gas works Vienna
Emmagrube, Kattowitz	only from coke ovens belonging to the concern.
Kokerei-Vereinigung, Kattowitz	from coke ovens of the mines in Western and Eastern Silesia.
Borsig-Kokswerke, Hindenburg	" "
Oberhütten, Gleiwitz	only from coke ovens belonging to the concern.
Schaffgotsch, Oderthal	" "

South

Weyl Mannheim	main source coke ovens of mines and steel works in the Saar area. Smaller parts also from municipal gas works Mannheim and cities in the vicinity.
Weyl Pasing	from gas works of city of Munich and cities in the vicinity.
Röchling'sche Eisen-u. Stahlwerke, Völklingen	only from coke ovens of own steel works.
Neunkircher Eisenwerk	" "
Halbergerhütte, Brebach	" "
Saargruben, Saarbrücken	from own coke ovens of the mines.
Dr. F. Raschig. Ludwigshafen	from coke ovens of mines and steel works in the Saar area; besides this smaller quantities from municipal gas works Ludwigshafen and cities in the vicinity.
Teerdestillation Stuttgart	from municipal gas works city of Stuttgart, besides this, smaller additional purchases from the Saar area.

North

Hanseatische Teerprodukten-Fabrik Haltermann. Hamburg	main source municipal gas works city of Hamburg, smaller quantities from gas works in the vicinity and from Bremen steel works.

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FIAT FINAL REPORT NO. 509

SF-36

UNCLASSIFIED

RECENT ENGINEERING DEVELOPMENTS IN SWITZERLAND ON GAS TURBINES AND STEAM GENERATORS

Rose, Harold J.

WARNING: Some products and processes described in this report may be the subject of U.S. patents. Accordingly, this publication cannot be held to give any protection against action for infringement.

UNCLASSIFIED

JOINT INTELLIGENCE OBJECTIVES AGENCY

WASHINGTON, D. C.

OFFICE OF MILITARY GOVERNMENT FOR GERMANY (U.S.)
Office of the Director of Intelligence

FIAT FINAL REPORT NO. 509 20 November 1945

RECENT ENGINEERING DEVELOPMENTS IN SWITZERLAND ON GAS TURBINES AND STEAM GENERATORS

BY

Harold J. Rose

Joint Intelligence Objectives Agency

FIELD INFORMATION AGENCY, TECHNICAL

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INTRODUCTION

Purpose and Scope of Investigation.

The purpose of this investigation was to determine what progress has been made in Switzerland during the war years, in developing equipment for the more effective utilization of coal. Particular attention was given to developments which may permit the use of coal for firing gas turbines.

Additional subjects of interest to the Solid Fuels Sub-Committee included:

Experimental burning of coal under pressure in Velox boilers.

Recent commercial developments in Monotube high-pressure boilers fired with coal.

Combined heating and power plants.

Commercial development of the "heat pump" for space heating and other uses.

Comfort heating by radiation from ceilings.

Three Swiss firms which have been prominent in designing and manufacturing equipment of this sort were visited, and the results of the interviews are presented in the following report under the individual firm names, arranged in alphabetical order, namely:

I. Brown, Boveri and Co.Ltd.
II. Escher Wyss Engineering Works Ltd.
III. Sulzer Brothers Ltd.

Dates of Investigation and Personnel.

Interviews with these companies covered the period September 18th to September 27th, 1945 and were made by Dr. Harold J. Rose (U.S.). It is understood that other U.S. missions have visited Switzerland with regard to gas turbines. However, so far as is known, the above investigator has been the only one who was primarily interested in the use of coal (which constitutes 98.9% of the United States' known reserves of mineral fuel, according to recent U.S. Bureau of Mines estimates of coal, petroleum, natural gas and oil shale reserves).

Most of the Swiss engineers interviewed spoke English well. Many of them had either worked in America, or had travelled on business in English-speaking countries. Company publications are commonly issued in English, German and French editions, though some of the publications were not available in English.

SUMMARY.

The economic situation in Switzerland during recent years has resulted in theoretical studies, engineering development work, and many commercial installations of ingenious methods for using energy more efficiently. The purpose of the present investigation was to learn of recent Swiss developments for the more effective use of coal, which comprises 98.9% of the remaining known mineral-fuel reserves of the United States.

Three firms were visited, namely: Brown, Boveri & Co.Ltd., Escher Wyss Engineering Works,Ltd., and Sulzer Bros.

Gas Turbines.

Each of these three firms is actively interested in gas turbines.

Brown,Boveri and Co.Ltd. is the only one which is actively experimenting with the use of coal for firing gas turbines. Considerable progress has been made experimentally in (a) burning coal under pressure to obtain combustion gases with a reduced content of solid particles, and (b) designing turbine blades which show minimum erosion from such particles. Engineering tests now in progress must probably be continued for some time before the company would be justified in designing a full-sized coal-fired combustion turbine plant for commercial trial and further development under practical conditions.

About 40 BBC-type gas turbines powered by combustion gas from the Houdry process, or by oil for power generation, have been built throughout the world, or are on order. This does not include gas turbines used in connection with Velox boiler plants. The oil-fired gas-turbine locomotive built for the Swiss Federal Railways was accepted after the required period of trial in actual service. BBC combustion turbines were built in Germany for blast-furnace-gas firing at metallurgical plants.

Escher Wyss Engineering Works, Ltd. This company has developed a closed-circuit "aerodynamic turbine" in which the working medium is air circulated under pressure. This feature permits the design of units of large output, and of nearly constant efficiency over a considerable load range. Combustion products ~~do not pass thru the turbine, and any fuel can be~~ used provided heating surfaces in the air heater can be kept satisfactorily clean and in good condition. Company publications contain references to the use of coal with a thermal efficiency as high as 35%, as a possibility after a period of future development.

The only aerodynamic turbine constructed to date is the 2000 kW. development unit at the company's plant. Performance tests in which the maximum average temperature of air entering the turbine was 1288°F. and maximum pressure was 343 lb./sq.in. showed a thermal efficiency of 31.5% at full load and 29.6% at half load, based on the net heating value of the oil fired.

Subject to establishing a satisfactory source of supply for alloy air-heater tubes, the company is prepared to accept orders for aerodynamic turbines of 6000, 12000 or 25000 kW. size. Still larger units are envisioned, such as a single-shaft output of 100,000 kW, with maximum air pressure not over 30 atm.

Sulzer Bros. This company has under construction a 7000 S.H.P. oil-fired marine combustion turbine with their own type of circuit, for trial purposes. Very little has been released on the design of this gas turbine. It is said to have certain advantages of a closed circuit without the necessity of a large air-heating chamber. Not much consideration has been given as yet to the use of coal as fuel for this type of turbine.

Compact Steam Generators.

Two novel types of steam generators (boilers) which were introduced before the war, have had further development and commercial acceptance.

Brown, Boveri Velox Steam Generator. In this type of boiler, fuel is burned under a pressure of 2 to 3.5 atm. absolute and the combustion products pass thru a gas turbine which drives a compressor supplying the required air. Steam is commonly produced at 25-35 atm., and 425 to 450°C. (797 to 842°F.).

To date, only oil and gas fuels have been used in Velox boilers, but Brown, Boveri have been studying the combustion of coal under pressure for several years. Development work on pulverized-coal firing of Velox boilers appears to be approaching a point where construction of a full-scale unit for industrial trial should be considered. This would promise a compact plant of high thermal efficiency, quick starting time, flexible automatic operation and stack gases free of smoke and flyash.

Sulzer Monotube Steam Generator. This design is intended for steam pressures above 80 atm. Most of the existing plants superheat to about 500°C. (932°F) and the present maximum working pressure is 140 atm. About 50 units have been built in eight European countries, of which 42 were fired with solid fuel - mostly pulverized coal or by means of travelling grates. Low-grade fuels with very high ash or moisture content, have been used successfully.

These plants offer very high-pressure steam generation in compact plants with automatic regulation of feed water, pressure, and temperature (of superheated steam) and prompt synchronization of heat input and removal under varying load conditions, when using a wide variety of solid fuels. As soon as known alloys are available the company is prepared to build Monotube boilers to produce steam at 600°C. (1112°F) and 160 atm. In a large plant, with reheat to the same temperature, thermal efficiencies up to 36% are expected.

Combined Heating and Power Plants. All three of the Swiss companies are interested in installations where the overall efficiency of heat utilization is considerably increased by using the energy partly for power and partly for heating purposes.

For example, steam may be used to generate power in a back-pressure steam turbine or steam engine, and the exhaust steam is then used for process work or comfort heating. Or the cooling water from certain gas-turbine plants may be drawn off at a high enough temperature for heating purposes or hot-water supply. The use of a heat pump in connection with power plants has also been considered.

Heat Pumps. Owing to a favorable combination of circumstances in Switzerland, "heat pumps" have received much commercial development recently for comfort heating and cooling, as well as for industrial uses. In this process electric power is used to drive a machine which extracts heat from cold lake or river water, and delivers the heat at a level high enough for comfort heating or other purposes.

In typical examples, 2/3 of the useful heat comes from the water supply, and only 1/3 must be supplied by the electric power used. With the lower temperatures used in "panel" or "radiation" heating it is said that as little as 1/5 of the energy needs to be supplied by the electric current. In industrial evaporation processes having much smaller temperature differentials, still higher ratios of useful heat to power input are well established.

In Switzerland, hydro-electric power is normally used for this purpose, and the heat-pump principle is used to multiply the heating effect of the electricity because of a coal shortage resulting from the war. It is obvious that electricity produced from coal could likewise be used to operate heat pumps. The relative efficiency and cost of producing electricity and heat from coal, do not encourage this idea in general. However, there will doubtless be cases where convenience, cleanliness or other reasons would lead to choice of a heat pump powered by electricity from coal instead of burning the coal directly for heat.

The recent heat-pump installations in public and private buildings in Switzerland are most interesting, but Swiss engineers are conservative as to commercial markets for such equipment. Over a large area such as the United States, with abundant coal reserves, only a limited percentage of cases might justify consideration of the heat-pump, and many of these cases would be economically feasible only when supplemented by more conventional methods of heating or cooling with energy derived from coal.

Radiation Heating from Ceilings. The Sulzer Co., has made a number of comfort heating (and cooling) installations in public and private buildings, by using pipe coils embedded in the ceilings.

Research Facilities. The companies visited maintain technical staffs and laboratories for research and development work in thermal engineering, and they also collaborate with faculty members of Swiss technical schools. Sulzer Bros. have just completed a large and well-equipped laboratory building which supplements their previous laboratories. Brown,Boveri & Co. expect to erect a separate thermal laboratory building as soon as the necessary materials can be obtained.

Documents and Publications. About 800 pages of technical publications, reports, etc., obtained from these companies (and which are referred to individually thruout this report) have been filed and microfilmed for convenient reference.

REPORT I - BROWN,BOVERI AND CO.LTD.

Location of Target.

The main office and factory of Brown,Boveri and Co. is in Baden, Switzerland, about 15 miles northwest of Zurich. The plant is just west of the railway station in Baden.

Date of Investigation.

The investigation occupied 2½ days and was made on September 18th, 19th and 20th,1945.

General Information and Personnel Met.

The following persons were met:

Dr.ing.Adolf Meyer, Director of the Heat Engineering Section, which deals with research, design and testing of thermal machines.

Mr.C.Seippel, Asst. to Dr.Meyer, and formerly in charge of the Gas Turbine Dept. (The present head of this department is Mr.K.Niehus).

Mr.Hans Dietler, who is in direct charge of the testing and development of coal-fired equipment.

Mr.Paul R.Sidler, American representative of B.B.C., who was in Baden at the time.

Mr.Walter Thut, who expects to go to the New York office soon to assist Mr.Sidler.

A number of other engineering executives and engineers of this company were also met. No attempt was made to discuss the electrical equipment manufactured by this company, but some of the research, testing and manufacturing facilities for electrical equipment were seen incidental to the investigation of the subjects covered by this report.

Information Obtained on Products.

Early Combustion Turbines Built by B.B.C. Dr.Meyer has described the early history and development of the gas turbine (See B.B.C. - Item 1. in list of "Documents and Publications" at the end of this section. These documents have been filed in Bag 1481, and have been microfilmed on Reel SF,6). In 1909 to 1913, Brown,Boveri and Co. built and tested to the order of Dr.Holzwarth, a gas turbine of the explosion type which had a net output of about 200 h.p. In 1928, they built a Holzwarth turbine with modified cycle, which was operated with blast-furnace gas in a German steel plant, and led to an order for a larger 5000 h.p. turbine from the German Brown,Boveri factory.

Since 1930, B.B.C. has used gas-turbine and compressor sets as an auxiliary to Velox boilers in which oil or gas fuel is burned under pressure produced by a compressor powered by the expansion of flue gases flowing continuously through a gas turbine. Since about 1936, gas turbines designed by this company, have been used on the hot exhaust gases of the Houdry oil-cracking process. In 1938 an oil-fired gas turbine of 4000 kW. size was ordered for the city of Neuchâtel, Switzerland, for emergency (bomb-proof) power supply. In 1941 an oil-fired gas-turbine-powered locomotive was completed for the Swiss Federal Railways. (See B.B.C. - Items 1, 2, 3, 8 and 18).

Coal-Fired Turbine Experiments. Realizing the potential economic advantages of using coal as a fuel for continuous-combustion gas turbines, Brown, Boveri and Co. have been studying this problem for about three years. The writer was privileged to discuss this subject with them at some length, and from the test apparatus and results seen, it is evident that they have made considerable progress along several lines. These experiments are still in progress, and have not reached the stage where a definite prediction as to their outcome is justified.

The engineering executives of the company feel that there is a good possibility that several months more of testing will bring the coal-fired combustion turbine development to the point where consideration will be justified of a full-sized experimental unit for industrial trial. Encouraging progress has been made in producing combustion gases which have a reduced content of fly ash when leaving the furnace chamber, and also in designing turbine blades which show far less erosion than formerly. However, tests now in progress or definitely planned, must be completed before these developments can be evaluated.

Brown,Boveri and Co. have not as yet published information on their experiments on coal firing of gas turbines. The following brief reference to the use of coal is from p.6 of B.B.C. - Item.7, written by Dipl.Ing.H.Pfenninger of B.B.C. in 1944.

> "(a) Power Plants for Producing Electrical Energy. The gas turbine can, for the present, only be employed with liquid or gaseous fuels. Experiments are now being made with pulverized coal, but these have not yet been completed. The range of load of the gas turbine lies here between 1000 and about 20,000 kW."
>
> " Recently, the problem of gasification of coal in gas generators has been again taken up. As the gas turbine is the cheapest form of power plant, the application of the gas generator is likely to be most remunerative here, so that the possibility of burning also solid fuels already exists today."

A paragraph in a German-language article (B.B.C. - Item 6, p.10) contains about the same comments on coal for combustion turbines.

Gas Turbine Publications Since the War Began. Copies of several publications dated later than 1939 were obtained.

Prof.Dr.A.Stodola of Zurich has summarized load tests of a B.B.C. combustion gas turbine of 4000 kW. size, constructed as a stand-by bomb-proof power station for the town of Neuchâtel, Switzerland. (See B.B.C. - Item 4). As this was intended only for emergency use, refinements such as a heat exchanger between exhaust gases and combustion air, were omitted. The electrical output of the generator had a heat equivalent of 17.38% of the fuel, in a test where the inlet temperature of gas to the turbine was 1,067°F. or 1026°F. (depending upon method of measurement) and the temperature at the gas turbine outlet was 532°F. Gas oil was used as fuel and no cooling water was required. Possible methods of increasing the efficiency are summarized in the paper.

Dr.W.G.Noack has discussed new methods of compressing and heating blast air for iron blast furnaces. (See B.B.C. - Item 5). This paper discusses various combinations in which the gas turbine is either the main machine, or else merely an auxiliary for fulfilling the "Velox" principle in which blast-furnace gas is burned under pressure, with very high rates of flue-gas flow, the necessary combustion pressure being obtained by a compressor coupled to a gas turbine driven by the flue gases. A Velox steam boiler or Velox blast heater may be used. Some cost comparisons are included. Dipl.Ing.H.Pfenninger of B.B.C. wrote an article in German in 1944, on the present position and economic outlook of the combustion turbine (B.B.C. - Item 6a). A translation of this article has been made. (B.B.C. - Item 6b).

An article in English by the same author in 1944, entitled "Present-Day Possibilities of the Combustion Turbine " (B.B.C. - Item 7) is somewhat shorter, and contains fewer diagrams than the preceding reference. It states that combustion turbines built up to the present time attain efficiencies of about 20%, which is sufficient only for special applications. By using air preheaters which are large enough, efficiencies of about 30% may be attained. Curves are shown for computing the most economical efficiency, allowing for amortization of heat exchanger costs and saving in fuel. The advantages of a single-stage combustion turbine are compared with (a) a steam plant, (b) a diesel engine plant and (c) a closed-cycle hot-air plant. The single-stage combustion-type turbine can be built today for outputs of 1000 - 6000 kW. No cooling water is required.

The two-stage combustion turbine has an efficiency of 21% without heat exchangers and 30% or more with heat exchangers. It can be started up from cold in about 10 minutes. This type consists of two compressors and two turbines in series. An order for a 10,000 kW. plant of this type has been received by B.B.C. They require 1/5 to 1/10 the cooling water of a steam turbine plant of the same output. Various fields of gas-turbine applications are discussed briefly in the paper.

Gas-turbine power plants are discussed in a German language article which is accompanied by a 2/3 page summary in English, as well as English titles for the illustrations (B.B.C. - Item 21). This discusses caloric power plants as emergency, supplementary and combined heating and power plants, with various cost comparisons. It includes the use of gas turbines, Velox boilers and conventional steam plants. Costs are estimated for Swiss conditions for a 50,000 kW. power plant. The lowest power cost is shown for powdered coal fuel and a steam turbine, next coal producer gas burned in a gas turbine, then oil fired in a Velox boiler with a steam turbine, and lastly oil fired in a gas turbine. Five-year fuel storage is cheaper for coal than for oil. The paper states, p.80:-

> "These tables show, that in modern caloric power plants the electric energy can even in Switzerland be produced at costs as low as in hydraulic accumulating plants."

Gas-Turbine Locomotive. The gas-turbine locomotive (oil fired) constructed by B.B.C. for the Swiss Federal Railways in 1941, was seen by the writer at Münchenstein near Basle, Switzerland. It had been run for a year in regular service, as the condition for acceptance. After acceptance, the locomotive was laid off on account of fuel-oil shortage.

Negotiations are now under way between the Swiss and French governments which may result in the loan of this locomotive to France for regular service on a much longer run than has been available in Switzerland.

"The First Gas-Turbine Locomotive" is the title of a paper by Dr.Ad.Meyer (B.B.C. - Item 8) which describes this locomotive in a general way, including the sequence of operations in a typical run. The economic prospects of the gas-turbine-electric locomotive are discussed by comparing it with steam and Diesel-electric locomotives. The paper is based on an oil-fired gas turbine. It is stated on p.12:-

> "If we succeed in solving the problems inherent to the pulverized-coal gas turbine, the future of the gas-turbine locomotive would be very bright indeed. We have a turbine developing 2000 H.P. of this type on our test bed and the results obtained justify our hopes of being able to put similar units on the market in the near future."

The reference B.B.C. - Item 9, dated July 1945, gives additional specifications and performance data on the first gas-turbine locomotive, constructed for the Swiss Federal Railways. The maximum output of the set measured at the coupling of the generator is 2200 H.P. at 5200/812 r.p.m. The power for starting up is produced by a Diesel generator set of 100 H.P. The electric transmission is the same as for Diesel-electric locomotives. In the shop test, thermal efficiency was 16% at full load and 17.7% at 3/4 load. In the period of regular service before acceptance, ending July 18, 1944, the locomotive ran 50,000 km. (31,000 mi.). During the 297 days, the combustion chamber was in operation for 1614 hours, and 1560 starts were made. A total of about 50 liters (13.2 gal.) of lubricating oil was consumed, or lost in overhauls, during these tests.

Summary of B.B.C. Gas Turbine Construction. It is understood that three gas turbines have been built for power uses, and 28 of the B.B.C. type for use in Houdry petroleum-processing plants. Eight more gas turbines are on order with B.B.C. at present. The above figures do not include the gas turbines used in Velox pressure-combustion installations, or blast-furnace-gas turbines built by the German B.B.C.

Velox Steam Boilers for Oil and Gas. Dr. Meyer stated in 1939 (p.5 of B.B.C. - Item 1):-

> "The work done by Messrs. Brown, Boveri in connexion with the Holzwarth gas turbine resulted in the development of the Velox boiler, whose principle is now well known to most engineers, but which nevertheless is referred to again here (Fig.8), because the experience gained with it led back to the combustion turbine. The Velox steam generator is a boiler fired under pressure, the pressure being produced by a compressor driven by a gas turbine, actuated by the exhaust gases of the boiler. Part of the pressure produced in the compressor is used to maintain high gas velocities in the heat-transmitting parts of the boiler, thus ensuring high rates of heat transfer. The remainder of the pressure head is used to drive the gas turbine."

Existing publications deal with oil - or gas-fired Velox boilers. A peak-load plant for Oslo, Norway is described in B.B.C. - Item 10. The burning of gas under pressure (including blast-furnace gas) is briefly discussed in B.B.C. - Item 11. A stand-by and peak-load power station built near Berne Switzerland in a horizontal tunnel, is described in B.B.C. - Item 12.

A general description of Velox steam generators for land purposes is given in B.B.C. - Item 13. This may be consulted for such items as characteristics and advantages, working principle and design, automatic governing, safety devices, starting time required, efficiency, constructional features and applications. Combustion takes place at a pressure of 2 to 3½ atmospheres, absolute, in a combustion space as small as 1/10 that of conventional boiler design.

Published accounts of Velox steam generators indicate that steam pressures of about 25 to 35 atm. are commonly used, and steam temperatures of 400 to 450 C. (752 to 842 F.). Plants have also been built for lower steam temperatures and pressures.

Additional references in English refer to a Velox installation at an Argentine oil refinery (B.B.C. - Item 14); a power station at Wellington, New Zealand (B.B.C. - Item 17); and the use of a Velox boiler or Velox blast heater in connection with compressing and heating blast air for iron blast furnaces (B.B.C. - Item 5).

Coal as Fuel for Velox Boilers. Brown, Boveri and Co. have been studying the combustion of coal under pressure for more than eight years. Development of a Velox boiler for burning pulverized coal under pressure appears to be approaching the point where construction of a full-scale unit for industrial trial should be considered. A gas turbine would be used on the combustion products to provide power for compressing the air for combustion. This development would promise very compact coal-fired power plants where space is at a premium; high thermal efficiency, quick starting time; flexible automatic regulation; and stack gases free of smoke and fly ash.

Both high-volatile high-oxygen coking coal from the Saar district, and medium-volatile-matter strongly-coking coals from the Ruhr have been used successfully in pilot-plant-scale Velox boilertests at Baden.

B.B.C. - Item 20, pp.24 and 25 illustrate a projected heating station which:-

> " ..... consists of a Velox steam generator fired with pulverized coal and producing approximately 48 tons of steam per hour."

All of the steam would be used for driving a heat pump by means of a steam turbine. For the arrangement described, the fuel consumption is said to be only 55 to 60% that of a low-pressure plant.

"The best possible utilization of the heat content of the fuel is obtained by a Velox steam generator burning pulverized coal with which more than 90% of the available heat in the fuel is utilized, and which can be readily adapted for automatic regulation of the boiler load."

Two German-language articles published in 1943 on Velox boilers, are listed as B.B.C. - Items 15 and 16.

Combined Heating and Power Plants. Several of the references cited discuss combination plants to produce both power, and heat for space heating or other uses. For example, B.B.C. - Item 21, refers to the use of exhaust steam or gases of a power plant for heating purposes. In the case of a steam turbine the temperature of the exit cooling water would be considerably higher than with a condenser, and the steam pressure at the exhaust of the turbine would also be higher. Such plants are, therefore, called "back-pressure" plants. Costs are estimated for Swiss conditions.

Applications of the heat pump for centralized heating plants and power plants, are discussed in B.B.C. - Item 20, pp.23-26.

Hot-water heating with Velox boilers includes a hot-water system under a pressure of about 12 atm. with an outgoing temperature of about 190°C. (374°F):-

"This type of boiler is frequently used for supplying heat to extensive systems, where the heat conveyed is transferred to low-pressure, low-temperature heating systems by means of calorifiers installed in proximity to the consuming centres". (B.B.C. - Item 13, p.20).

Heat Pumps. A heat pump is a mechanical device which uses power to cause heat to be absorbed at a lower temperature level, and to be given off at a higher temperature level. If used as a refrigerating machine, the useful output of the heat pump is the amount of heat absorbed at the lower temperature (the cooling effect), and the heat evolved at the higher temperature is thrown away. If used as a heating machine, the cooled material is thrown away, and the useful output of the heat pump is the amount of heat available at the higher temperature level (the heating effect). This possibility of using the heat pump as a heating machine has long been known to the engineering profession, but only a limited commercial use of the process has been made in the past. Actually, the same machine may be used for cooling in summer and heating in winter.

The principles of the heat pump used both as a refrigerating and heating machine, including the thermo-dynamic fundamentals, are discussed in B.B.C. - Item 20, pp.1 to 11.

Some applications of the heat pump as a heating machine are discussed in B.B.C. - Item 20, pp.12 to 33. These practical applications refer particularly to Switzerland, where there has been considerable recent development of the heat pump for comfort heating. Usually about two-thirds of the useful heat output is extracted from relatively cold river or lake water, and only one-third of the heat output is supplied by the electric power required to operate the machine. With some types of space heating, a higher ratio of heat to power can be obtained.

In Switzerland the object has been to save coal by using hydro-electric power, but it is obvious that the process may be operated with electricity made from coal when convenience, cleanliness, or other reasons lead to the choice of the heat pump process instead of burning the coal directly for heat.

The writer saw the heat pump used on river water to heat the Brown, Boveri plant at Baden. The reference just cited illustrates and describes heat pumps used for:

(a) Producing hot water at 58° to 70°C. (136 to 158°F.) from lake water for an artificial silk plant.

(b) Producing hot air for drying purposes, from moist warm air coming from the paper machine of a paper mill.

(c) Producing hot water at 68 to 74°C. (154 to 165°F.) from river water of 2 to 16°C. (36° to 61°F.) to supplement an existing central-heating power plant of the Swiss Federal Institute of Technology at Zurich, which heats a large group of public and private buildings. This heat-pump plant contains two Brown, Boveri thermoblocs.

The heating output of the heat pumps is about 15% of the total connected-up heating capacity. As a general rule, heat pumps for room heating are dimensioned for a given base load and peaks are covered by less expensive heat producers such as fuel-fired boilers.

(d) Air conditioning in one Zurich building is obtained by a heat pump supplying 50,000 kcal/hr. (ca. 200,000 Btu/hr.) when heating, and 27,000 kcal/hr. (ca. 108,000 Btu/hr.) when cooling. B.B.C. supplied part of the equipment.

(e) Evaporation in chemical plants, food preparation plants, etc., is usually much more favorable to the heat pump (thermo-compressor) process, than room-heating applications, owing to the smaller temperature differentials involved. This permits a higher ratio of useful heat to power consumed, and a lower equipment cost per unit of output.

A less technical discussion of applications of the heat-pump, with examples, is given by B.B.C. - Item 19.

Projected New Thermal Laboratory.

Brown, Boveri and Co. expect to erect a new building at Baden, for thermal research and testing, as soon as the necessary materials of construction are available and conditions permit.

Conclusions.

Brown, Boveri and Co. at Baden, Switzerland, are actively studying the combustion of coal under pressure, with respect to the direct use of coal for firing gas turbines and for firing Velox boilers. Engineering tests and design developments now in progress need to be completed before the technical prospects of this work can be evaluated. In a few months time the company may feel justified in considering the design of a full-size coal-fired turbine and a coal-fired Velox boiler for trial and further development under industrial conditions.

/ Of the Swiss firms covered by this report, B.B.C. is the only one which has experimented to date with coal firing of gas turbines. A number of turbines of their design, powered by gas or oil, have already been built.

The oil-fired gas-turbine locomotive built for the Swiss Federal Railways has passed the specified period of test in actual use, and has been accepted.

The company has proposed various interesting methods for improving heat economy of metallurgical plants, factories and central heat and power-stations. A separate thermal laboratory building is to be built at Baden when the necessary materials can be obtained.

Brown, Boveri have made recent installations of heat pumps in Switzerland for space heating, air conditioning, industrial drying and evaporation, etc. A combination of cheap electricity, abundant water supply, and wartime scarcity of imported coal has promoted this development.

DOCUMENTS AND PUBLICATIONS
Referred to in Report I, Brown, Boveri and Co.Ltd.

Filed in Bag 1481 and microfilmed on Reel SF.6, pp.000001 to 000259. (Some of the items are reprints from the "Brown, Boveri Review").

B.B.C. - Item 1 (000001 to 000029)
"The Combustion Gas Turbine: Its History, Development, and Prospects" by Adolf Meyer. Paper read at meeting of The Institution of Mechanical Engineers, London, Feb, 24, 1939, and issued in reprint form, with discussion. 26 pp. + 3.

B.B.C. - Item 2 (000030 to 000033)
"New Gas Turbines that Work" by S.A.Tucker. Reprint from Power, June 1939. 4 pp.

B.B.C. - Item 3 (000034 to 000037)
"Combustion Turbines Brown, Boveri". 4 pp. 1558E - II.9 (XII.39) (1244)

B.B.C. - Item 4 (000038 to 000042)
"Load Tests of a Combustion Gas-Turbine Built by Brown, Boveri & Co.Ltd., Baden, Switzerland" by Prof.Dr.A.Stodola. 5 pp. 1584E - II.9 (VI.40) (1244)

B.B.C. - Item 5 (000043 to 000049)
"New Ways and Means of Compressing and heating Blast Air in Ironworks" by Dr. W.G. Noack. 7 pp. 1787E - IX.12 (VI.44) L.2044.

B.B.C. - Item 6a (000050 to 000061)
"Der heutige Stand der Verbrennungsturbine und ihre wirtschaftlichen Aussichten (Today's Position of the Combustion Turbine and Its Economic Outlook) by Dipl.Ing.Hans Pfenninger. 1 pp. + 1. Reprint from Schweiz. Bauzeitung Bd.123, Nr.24. u.26, 10.u.24 Juni, 1944.

B.B.C. - Item 6b (Not microfilmed, nor in Bag 1481)
A translation of the preceding article Item 6a, made in the London office of J.I.O.A. - T.I.I.C. Document Center. Copy of this rough translation deposited with L.L. Newman, U.S. Bureau of Mines, Washington, D.C.

B.B.C. - Item 7 (000062 to 000069)
"Present-Day Possibilities of the Combustion Turbine" by H.Pfenninger. 8 pp. 1825E - II.9 (VIII.44) L.2044. This is a shorter article than Item 6, and contains fewer illustrations.

B.B.C. - Item 8 (000070 to 000081)
"The First Gas-Turbine Locomotive" by Dr.Ing.h.c.Ad.Meyer. 12 pp. 1668E - V.4 (IX.42) (L.2044). (This is a reprint from the Brown, Boveri Rev.1942, No.5, pp.115-126).

B.B.C. - Item 9 (000082 to 000095)
"The Gas-Turbine Locomotive. Specification of the Gas-Turbine Locomotive constructed by Brown, Boveri for the Swiss Federal Railways". July 1945. 14 pp.

B.B.C. - Item 10 (000096 to 000121)
"The Rosenkrantzgat steam power station of the Oslo Electricity Works. A Velox peak-load plant of considerable output" by A Fischer. Aug.1937. 26 pp. 1474E - II.1 (2044)

B.B.C. - Item 11 (000122 to 000123)
"The Burning of Gas Under Pressure" by J.G. Coutant. Reprint from American Gas Journal, Feb.1938, pp.23-24, 2 pp. N.Y.110.

B.B.C. - Item 12 (000124 to 000134)
"A Modern Stand-By and Peak Load Steam Power Station Built as a Bomb-Proof Plant" by U.Roth. 9 pp.+ 1. 1557E - II.1. (IX.39) (2044).

B.B.C. - Item 13 (000135 to 000155)
"The Brown,Boveri Velox Steam Generator for Land Purposes", no author stated. 21 pp. 1533E - II.8 (IX.40).

B.B.C. - Item 14 (000156 to 000162)
"The Velox Power-Plant at the San Lorenzo Refinery of the Argentine State Oil Fields", by H.S.Hvistendahl. 7 pp. 1625 E - II.8. (XI.40) (2044).

B.B.C. - Item 15 (000163 to 000178)
"Ein automatisch anfahrendes Velox-Kraftwerk von 10,000 kW. Leistung als Schnellreserve". (An Automatic Process Velox Power Plant for 10,000 kW. Output as Quick Reserve), no author stated. 15 +1 pp. 1706D - II.8. (VI.43) L.2944.

B.B.C. - Item 16 (000179 to 000187)
"Anwendungen des Aufladeverfahrens nach dem Velox-Prinzip". (Application of the Supercharging Process by the Velox Principle) by W.G.Noack. 9 pp. Reprint from Zeitschrift des Vereines deutscher Ingenieure im N.S.B.D.T. Bd.87 (1943) Nr.35/36. S.547 bis 555.

B.B.C. - Item 17 (000188 to 000194)
"The Velox Boilers at the Evans Bay Power Station, Wellington, New Zealand" by R.S.Maunder. (No publication reference) 7 pp.

B.B.C. - Item 18 (000195 to 000202)
"Actual Problems in the Production of Power in Thermal Power Stations" by Ad.Baumann/ W.Broggi. 8 pp. 1553E - II.1. (VIII.39).

B.B.C. - Item 19 (000203 to 000212)
"Brown,Boveri Heat Pumps for Heating and Cooling. Thermo-compressors for Concentration Plants. You're in a Quandary?". 10 pp. 1673E - IX.7 (III.43) L.2244, K.16, 17, 18, 21, 34.

B.B.C. - Item 20 (000213 to 000246)
"The Heat Pump as Refrigerating and Heating Machine" by A.Meldahl, 11 pp; "Some Applications of the Heat Pump as a Heating Machine" by Ad.Baumann and D.Marples, 22 pp. Total 33 + 1 pp. 1747E - XI.7. (XII.43) L.2044.

B.B.C. - Item 21 (000247 to 000259)
"Thermische Anlagen für Reserve -, Ergänzungs-und Heizkraftwerke mit besonderer Berücksichtigung der Turbine", pp.69 - 78. "Centrales thermiques de reserve, centrales auxiliaires et centrales chauffage-force matrice", pp.78-79. "Caloric power plants as emergency, supplementary and combined heating and power plants", pp.79-80, by P.Faber. Article in German with 2/3 page abstracts in French and English. From magazine Elektrizitäts Verwertung - L'Électrique - Electrical Service. 20th year, No.1-3, pp.69-80. 12 + 1 pp.

REPORT II - ESCHER WYSS ENGINEERING WORKS,LTD.

Location of Target.

The office and factory of Escher Wyss Engineering Works,Ltd., are located in Zurich, Switzerland, at Escher Wyss Platz.

Date of Investigation.

The investigation was made on September 21st and 22nd,1945.

General Information and Personnel Met.

The following persons were met:

Dr.Ing.C.Keller, Chief Engineer of the Research Dept. (Equivalent in U.S. to Director of Research and Development).

Mr.E.J.Meier, Mech.Eng. of the A.K.Dept. (Manager of the thermal section and formerly chief designer for turbines).

Information Obtained on Products.

Early Water and Steam Turbines. Escher Wyss have published "A Century of Turbines" which is the combined 1942/43 volumes of Escher Wyss News. See EW - Item 1a, in list of "Documents and Publications" at the end of the Escher Wyss part of this report. These documents have been filed in Bag 1481 and have been microfilmed on Reel SF.6.

The business of Escher Wyss is built around turbines of all types, and the history of this company in turbine construction since 1840 is summarized in EW - Item 1b. See also EW - Item 2c (Microfilm 000307-8) for a list of their publications on turbine research.

Aerodynamic Turbine. Escher Wyss have developed a gas turbine which they call an "Aerodynamic turbine with closed circuit", the principles of which are discussed in an article of that title by Prof.Dr.J.Ackeret and Dr.C.Keller (EW - Item 1c). It is compared with steam and gas turbines in an article by Dr.Keller (EW - Item 1d).

The working fluid is air, which circulates in a closed cycle under pressure, thus permitting smaller parts because of the denser working medium. The thermal efficiency is independent of the absolute pressures, and the choice of pressure level is determined by considerations of design. It is said that maximum pressures of 20 to 30 atm. represent satisfactory figures for even the largest unit outputs. The best ratio of inlet to outlet pressures is about four, as in open-cycle turbines. One of the advantages claimed for this system is the ability to control the output without much effect on thermal efficiency, by simply changing the pressure level of the air in the system. This changes the weight of the circulating medium without affecting temperatures or velocities.

The recirculated air is compressed by axial-flow compressors with water intercoolers (which require 1/10 to 1/5 as much cooling water as a steam turbine plant). The high-pressure air is then heated:-

(a) In a heat exchanger by hot air from the turbine exhaust.

(b) In a fuel-fired heater, in which the furnace chamber may be maintained at atmospheric pressure.

For other general references to this type of turbine, see EW - Items 2b and 8.

The writer saw the development model 2000 kW. aerodynamic turbine which has been built and tested at the Escher Wyss plant. Construction was begun in 1936, and the turbine was first operated in 1939.

Turbine Performance Tests. Official performance tests were made in December 1944 by Prof. H. Quiby of the Federal Polytechnic School at Zurich, and are described in a French-language article filed with this report (EW - Item 4). A two-page summary in English (EW - Item 5) has recently become available to American readers, so that an extensive summary is not required in this report.

In these tests, heated air at 1268°F. to 1288°F. and at 343 to 83.8 lb. pressure (depending on the desired load) was passed thru the high-pressure turbine which drove the compressor (consuming about 60% of the output), then to the low-pressure turbine which drove the generator consuming about 40% of the output. Exhaust air from the turbine had temperatures of 829 to 846°F. and the ratio of turbine inlet to outlet air pressure was about 3.56 : 1 in each case. The net thermal efficiency after allowing for all losses, was 31.5% at 2044 kW. output, 29.6% at half load, and 24.5% at one-fifth load, when figured on the net heating value of the fuel, in accordance with European practice. (Thermal efficiencies would be somewhat lower if figured on the gross heating value of the oil, as in American practice). Very cold cooling water was doubtless available in these December tests.

Solid Fuel and Derived Gaseous Fuels For Turbines. The closed-cycle turbine has not been tested with solid or gaseous fuels as yet, but Escher Wyss publications contain the following statements regarding the use of solid fuel, or gas derived from solid fuel;

> "Employment of gaseous and solid fuels. For the employment of gaseous fuels in open-circuit gas turbine plants with internal combustion, it is necessary to cool and sometimes purify same before the compression phase. In the case of closed-circuit installations with

external firing such cooling of the fuel is unnecessary; a separate gas compressor is eliminated, whilst the sensitivity of the plant to any solid matter in the fuel is, of course, also reduced. A much more difficult matter is the use of solid fuel, i.e., chiefly pulverized coal, in gas turbines designed for internal combustion, since soiling and wear both of the turbine and heat exchanger have to be dealt with. On the other hand the external combustion of pulverized coal, although presenting certain complications compared with oil firing, is much easier to realize, since similar experience in ordinary steam boiler plants is available. If it is possible to employ pulverized coal for the operation of aerodynamic turbines with the efficiencies later referred to an exceptionally economical power plant for large outputs will be available. The gasification of solid fuels in suitable gas producers followed by combustion of the gas in air heaters might also be considered." (EW - Item 1c, p.13).

Producer gas fuel is also referred to as follows:-

"When coal is employed as fuel it may, according to the nature and quality of the coal, prove convenient to adopt gas generators in place of furnaces, for producing the combustion gases for the air heater. In this connection no cleaning, cooling down or compression is necessary such as are called for when an open-cycle is adopted. The operation of up-to-date gas generators also makes adaptation and control of the heating an easy matter". (EW - Item 1d, p.40).

The same reference, in discussing the air heater, mentions pulverized coal firing:-

"Careful calculation of all these problems have led to a series of fundamental circulation layouts for the working medium to be heated and for the heating combustion gases. They permit, for a minimum loss of pressure and without the adoption of special measures, the attainment with all fuels, i.e., solid, liquid and gaseous, of maximum air temperatures lying far above those of the steam temperatures now employed, or which it is hoped to employ in the future.

In view of the fact that the waste gases from the air heater have relatively high temperatures, their waste heat can advantageously be employed for preheating the combustion air. The use of preheated combustion air leads to an increase in the furnace temperature, particularly in cases where solid fuels (coal) are used. For example, in plants burning pulverized coal such preheating of the secondary air is very desirable, since it permits a reduction in the size of the furnace as a consequence of the shorter combustion period. On the other hand the duty to which the tubes are subjected is more exacting where higher furnace temperatures are adopted. A simple means for compensating these conditions is a return circuit of the flue gases to the combustion chamber, whereby practically any desired temperature in this chamber can be adhered to". (EW - Item 1d, pp.30, 31). See EW - Item 5, p.2, for arrangement of an air heater for pulverized-coal firing, with recycled flue gas.

Curves are shown illustrating the advantage of the closed cycle with recirculated air under pressure, for keeping down the temperature of the air-heater tubes. Fig.38 (p.37) of EW - Item 1d, compares the cost of fuel at power stations when using various fuels and types of equipment. The legend states in part "Unlike open-circuit gas turbines and Diesel engines, aerodynamic plants can be operated with coal". The thermal efficiency for thermodynamic turbines fired with coal is assumed to be 35%, with a footnote indicating that this 35% is a:-

"Possible figure in large installations after a future period of development. In contradistinction to other thermal prime movers a considerable margin for further increases in efficiency exists in this case".

The use of waste gases from various industries is discussed in EW - Item 1d, pp.39-40. This calls attention to the large dimensions of other equipment to use such waste gases, and points out the advantage of using the gases as fuel for the air heater of an aerodynamic turbine. It discusses the use of such a turbine, fired with blast-furnace gas, to drive blast-furnace blowers which must operate under widely different speeds andloads. See also a German-language article (EW - Item 3) on the applications of aerodynamic turbines in metallurgical plants.

Availability of Aerodynamic Turbines. The only turbine of this type which has yet been constructed is the 2000 kW. development model at the Escher Wyss plant. Subject to establishing a satisfactory source of supply of alloy-metal tubes for the air heaters, the company is ready to accept commercial orders for oil-fired stationary or marine aerodynamic turbines in three sizes, namely 6000, 12000 and 25000 kW. Still larger units are envisioned for future plants, according to EW - Item 1d, p.36-37, which suggests single-shaft outputs of 100,000 kW. with maximum pressures not over 30 atm. Presumably the company would accept orders for producer-gas fired or blast-furnace-gas fired turbines also. Preliminary studies on application to locomotives are being made. In the case of coal-fired installations, the company would wish to collaborate with an engineering firm experienced in the combustion of coal.

Combined Heating and Power Plants. It is pointed out (EW - Item 1d, pp.40-41) that the cooling water from the pre-cooler and compressor intercoolers can be allowed to rise to 80°C (176°F) or more, without altering the temperatures in the circuit, or changing the work output of the aerodynamic turbine plant. This water can be distributed as hot-water supply or for distant heating, without the necessity of returning the water. Fig.4 of EW - Item 5, indicates that in a test about 46% of the total heat in the fuel burned was removed in the cooling water. (In a steam-turbine plant the necessity of maintaining a good vacuum usually results in the use of very large volumes of cooling water which may be raised only a few degrees, and thus is not directly suitable for hot-water supply or for heating purposes).

Heat Pumps. The use of electric-driven "heat pumps" for heating or refrigerating, has been briefly discussed in part I of this report. Both of these applications are covered by an Escher Wyss publication (EW - Items 6a, b and c). Item 6c is an article "Heating systems with heat pumps" by A.Astertag, which includes considerable technical information.

The yield of the heat pump is greatest when the temperature difference between the source of heat and the material to be heated is least. It is therefore well suited for radiation or "panel" heating of the type where the heating pipes are embedded in the ceiling or other room surfaces, and the circulated water is not as hot as that used in conventional radiators.

Owing to the high initial cost of heat pumps, and the highly variable daily heat requirements for comfort heating, it is considered preferable to design the heat pumps for the base heating load, and to use a supplementary heating system for occasional peak-load demands which form only a small percent of the total annual heat needed. An engineering study is required to determine whether this is a practical solution in specific cases. See examples in the text, based on Swiss weather and for various heating problems such as:-

(a) The Zurich town hall where the same heat pump, operating on rivet water, serves for heating in winter and cooling in summer. Heating-water storage tanks are provided for peak periods of heating. See Figs.10 to 13, p.36.

(b) The Zurich public baths where heat pumps heat the swimming bath water, showers and ventilating air, and provide radiation comfort heating.

The Lausanne artificial ice rink uses heat from the refrigerating plant to raise the temperature of the entire city water supply by 2.5°C (4.5°F) thus economizing on the heating of water for cooking, washing and bathing.

Another publication, (EW - 7a and b) in the German language, contains additional technical information and examples of heat-pump applications.

It has already been mentioned in part I of this report, that a special combination of circumstances has led to the adoption of heat-pumps for various cases of comfort heating in Switzerland. Such favorable conditions might represent only a small percentage of cases in much of the area of the United States.

In the past, Escher Wyss have made numerous installations of heat pumps for industrial evaporating purposes in Switzerland and elsewhere. It is said that the largest heat pump in the world is an Escher Wyss plant which evaporates 220,000 lb/hr. of water with a total power consumption of only 4000 kW.

Research Activities. Escher Wyss has issued a publication "Research on Turbo machinery" (EW - Item 2a). The 19 articles which it contains are listed on the index page (Microfilm p.000302). Only one of these refers specifically to aerodynamic turbines (EW - Item 2b). Publications of research work on Escher Wyss turbines of all types are listed at the end of the reference (EW - Item 2c, Microfilm pp.000307-8).

It is said that this company has for several years maintained about 25 graduate engineers on development research. One effect of the war on neutral Switzerland, was to give them time to perfect their ideas and designs for aerodynamic turbines for stationary and marine applications.

Conclusions.

The Escher Wyss Eng.Works Ltd. has developed an "aerodynamic" gas turbine in which the working fluid is air circulated under pressure. Because of the denser medium, the working parts are smaller, and units may be built in larger capacities than with open-circuit gas turbines. Outputs may be varied without much change in thermal efficiency by varying the pressure level in the system. A variety of fuels may be used, with combustion at atmospheric pressure, since the combustion gases do not pass thru the turbine, but serve to heat the recirculating air which is passed thru tubes in the air heater and heat exchanger. Cooling water requirements are about 1/5 to 1/10 that of a steam-turbine power plant.

The only turbine of this design which has been built to date is the 2000 kW. development unit at the Escher Wyss plant. With inlet air at about 1275°F. the net thermal efficiency after allowing for all losses was about 30% to 31.5% from half load to full load. (These efficiencies were based on the net calorific value of the oil used, and the cooling water was evidently very cold during the test).

Subject to obtaining a satisfactory source of supply for alloy air-heater tubes, Escher Wyss are prepared to accept orders for oil-fired (and presumably gas-fired) aerodynamic turbines for stationary or marine use in 6000, 12000 and 25000 kW. sizes. They have not yet tried pulverized coal or producer gas as fuel, but refer to both in their publications. Stationary power units of still larger size, fired with coal with a thermal efficiency of 35% or more, are mentioned as a possibility after a future period of development.

The cooling water may be drawn off at around 175°F. (without affecting turbine performance or output) and used as hot-water supply, or for heating purposes.

Escher Wyss have made many heat pump installations for industrial evaporation, and more recently have built heat pumps for comfort heating and air conditioning (winter heating and summer cooling).

DOCUMENTS AND PUBLICATIONS
Referred to in Report II - Escher Wyss Eng.Works,Ltd.

Filed in Bag 1481 and microfilmed on Reel SF.6,pp.000260 to 000382, and 000761 to 000771.
(Some of the items are reprints from Escher Wyss News).

EW - Item 1a (Microfilm pages 000260 to 000261).
Cover page and index of "A Century of Turbines", Escher Wyss News, Vol.XV/XVI, 1942/43. Pages 1 and 2. 2 pp.

EW - Item 1b (000262 to 000263)
"A Century of Turbines". A summary, with dates, of Escher Wyss work in turbine construction, beginning with 1840. Pages 3 and 4. 2 pp.

EW - Item 1c (000264 to 000278)
"Aerodynamic Turbine with Closed Circuit" by Prof.Dr.J.Ackeret and Dr.C.Keller. Pages 5 to 19. 15 pp.

EW - Item 1d (000279 to 000300)
"The Aerodynamic Turbine compared with Steam- and Gas-Turbines" by Dr.C.Keller. Pages 20 to 41. 22 pp.

EW - Item 2a (000301 to 000302)
Cover page and index of "Escher Wyss Research on Turbo-machinery". 23001(e). 2 pp.

EW - Item 2b (000303 to 000306)
"An aerodynamic heat-power plant" by Prof.Dr.J.Ackeret and chief-engineer Dr.C.Keller. Pages 82-85, 4 pp. Also published in "Schweizerische Bauzeitung", Vol.113, No.19, May 1939.

EW - Item 2c (000307 to 000308)
List of "Publications of research work for Escher Wyss designs" of turbines. Inside back cover. 2 pp.

EW - Item 3 (000309 to 000315)
"Die Aerodynamische Turbine im Hüttenwerk" (The Aerodynamic Turbine in Metallurgical Plants), by Dr.sc.techn.C.Keller and Dipl. Ing.R.Ruegg. Reprint from "Schweiz. Bauzeitung", Bd.122, Nr.1, 3 July,1943. 7 pp. 23012 (d).

EW - Item 4 (000316 to 000344)
"Compte-rendu des essais de la turbine aérodynamique Escher Wyss AK" (Account of trials of the aerodynamic turbine Escher Wyss AK"), by Prof.H.Quiby. Reprint from Vol.125, No.23 and 24, 9/16 June,1945. 28 + 1 pp. No.23021.

EW - Item 5 (000345 to 000346)
"Performance Tests of 2000-KW. Closed-Cycle Unit" Power, Aug.1945. 2 pp. A summary in English of EW - Item 4.

EW - Item 6a (000347 to 000348)
Cover page and index of "Escher Wyss Refrigerating Machines. Heat Pumps", 2 pp. 24006(e).

EW - Item 6b (000349)
"Heat Pumps" p.31. 1 p.

EW - Item 6c (000350 to 000364)
"Heating systems with heat pumps" by A.Ostertag. pp.32-46. 15 pp.

EW - Item 7a (000365 to 000366)
Cover and contents page of "Escher Wyss Kälte - Wärme" (Cold-Heat) 2 pp. 24010(d).

EW - Item 7b (000367 to 000382)
"Die Wärmepumpe in der Heiztechnik" (Heat Pumps in Heating Technique) by A.Ostertag and A.Kornfehl, pp.25-40. 16 pp.

EW - Item 8 (000761 to 000771)
"Die Aerodynamische Turbine 'Escher Wyss AK' - Anlasse; L'installation de turbine aérodynamique 'Escher Wyss'; The 'Escher Wyss AK' aerodynamic turbine power plant", by Dr.C.Keller. A German-language article with half-page summaries, and titles to figures, in French and English. "Elektrizitätsverwertung - L'Electrique - Electrical Service". 20th year, No.1-3, April-June 1945, pp.81-91, 12 pp.

REPORT III - SULZER BROS. LTD.

Location of Target.

The office and factory of Sulzer Bros.Ltd. is located at Winterthur, about 15 mi. north-east of Zurich, Switzerland. The plant is adjacent to and north-west of the railway station.

Date of Investigation.

The investigation was made on September 26th and 27th,1945.

General Information and Personnel Met.

The following persons were met;

Dr.Frederick Oederlin, Managing Director (of engineering).

Dr.Herbert Walfer, Director of Research.

Mr.Adolf Egli, in charge of gas turbine department

Mr.Max E.Trechsel, in charge of public relations, patents, etc.

Dir.J.Gastpar, Manager of boiler department.

Mr.Vogler, Chief engineer of boiler department.

Mr.Viktor Juzi, Chief engineer for high-pressure boiler plants.

Mr.F.Eicher, Engineer, boiler sales department.

Mr.Felber, Manager of comfort heating equipment,etc.

Mr.Wirth, Chief engineer of comfort heating equipment,etc.

Mr.Adolf Brunner, in charge of locomotive department.

Information Obtained on Products.

Sulzer Bros. make a considerable variety of products such as Diesel engines,pumps, fans and compressors, steam boilers, steam engines and turbines, heating and ventilating plants, refrigerating plants, sheet metal and foundry work,etc. An illustrated summary of products is given in Sulzer - Item 19. See list of "Documents and Publications" at the end of this report. These documents have been filed in Bag 1481 and have been microfilmed on Reel SF.6. Numerous examples of boiler and plate work are given in Sulzer - Item 18.

Heat-Engine Developments. Past activities of Sulzer Bros. in the power field have been mostly concerned with Diesel engines, boilers and steam engines. A special number of the Sulzer Technical Review, dated December 31st, 1941 (Sulzer - Item 1) is entitled "The supercharging of two-stroke Diesel engines". It refers to:-

(a) Supercharged two-stroke Diesel engines with exhaust-gas turbines.

(b) "Power-gas" Diesel engines in which the engine-compressor unit delivers no mechanical energy, but serves exclusively to supply exhaust or power gas to a gas turbine.

(c) Sulzer development work on a constant-pressure gas turbine of their own system as a thermal prime mover (no details given).

Sulzer Bros. kindly gave the writer a copy of a typed memo "New Sulzer designs in the domain of the heat engine", which it was said was to be released shortly to the technical press as a progress report (Sulzer - Item 2). It summarizes recent developments on the above and other items.

Constant-Pressure Combustion Turbine. Very little information has been released on the Sulzer gas turbine. The previous reference states, p.3-4:-

> "The special feature of the Sulzer gas turbine consists in the employment of a new type of circuit which retains the essential advantages of the known closed circuit without having its main disadvantage in the form of a large and heavy air heating chamber. The Sulzer air heater used instead is considerably smaller and lighter, which makes the plant particularly interesting for marine propulsion. The thermal efficiency exceeds that of the best marine steam turbines even at small powers, and with full utilization of all present advantages and inherent possibilities will be raised very near that of the Diesel engine.
>
> The trial plant of a marine turbine on this new system, to give an effective output of 7000 S.H.P., is at present under construction".

In the present stage of development, Sulzer Bros. have been considering oil-fired gas turbines, especially for marine use. However, these turbines could also be used for stationary power plants, and probably be adapted to locomotive use. Not much consideration has been given as yet to the possibility of firing direct with coal. However, this could be done whenever coal-combustion and turbine-blade technology reach the point where coal can be burned under considerable pressure to yield hot combustion products clean enough to pass directly through a turbine. Firing with pressure-producer gas is another possibility.

Monotube High-Pressure Steam Generators. Information was obtained on recent developments of the Sulzer Monotube high-pressure boiler. A list of these steam generators was obtained (Sulzer - Item 16) which shows a total of 51 units built or under construction in Switzerland, Great Britain, France, Holland, Germany, Italy, Hungary and Roumania. Forty two of these use solid fuel, of which 26 are fired with pulverized coal, 13 with travelling grates, and one each with underfeed stoker, mechanical overfeed stoker, and with coke. One is fired with blast-furnace gas, seven with oil, and in one case the firing method is not specified. Brief information on rating, steam temperature and pressure, and auxiliaries is given for each plant.

Since this type of steam generator was known a few years before the war, it is not necessary to describe it in detail here. For convenient reference, several of the earlier publications have been included in the documents filed with this report. (See Sulzer - Items 3, 5, 7). Also included is a copy of a typed report dated 1936 (Sulzer - Item 4) on a power plant of this type for a colliery in Wales. This describes a complete high-pressure back-pressure plant with two monotube steam generators which work in conjunction with an existing plant having medium pressures. Each monotube generator produces 50 or more tons of steam per hour (at 110 atm. gauge and 770°F) on the same floor space formerly used to generate 16 tons per hour (at 25 atm. gauge and 762°F). Pulverized coal firing is used. Sulzer - Item 6 is a mimeographed general description of the monotube steam generator, dated 1937.

Sulzer recommend this type of high-pressure steam generator for pressures over 80 atm. Each unit tube is capable of producing about 10 tons of steam per hour and consists of a continuous welded tube several thousand feet long in which the feed water is preheated and evaporated, and the resulting steam is superheated. This tube is bent to form banks of preheater and superheater tubes as well as furnace walls. For larger capacities several of the unit tubes are used and bent to lie in parallel. There is automatic regulation of feed water, pressure and temperature of superheated steam, and prompt synchronization of heat input and removal under varying load conditions. It is said that there has been considerable improvement and simplification of the controls.

At the end of the zone of evaporation there is a continuous blow-down device to maintain the concentration of salts within certain limits. A report on measurements of the salt content at various points in the circuit is given in Sulzer - Item 14. It was concluded that adequately pure steam (8 to 10 mg. per litre residue on evaporation, or 0.47 to 0.58 grains/gal.) could be obtained even though the blow-down device was discharging water of 500 mg./l. residue (29.2 grains/gal.) and the make-up condensate was impure, containing 150 mg./l. of residue (8.8 grains/gal.).

An interesting high-pressure steam plant in an English cardboard factory is described in Sulzer - Item 8. The steam generator andturbines of this coal-fired plant are all located in the same room. Coal is brought by Redler conveyors direct from the coal storage yard to the hoppers on travelling-grate stokers. The space below the grates is hermetically sealed off from the boiler and engine room to prevent the escape of dust. The combustion gases pass thru a van Tongeren dust collector of Sulzer design, before being discharged to the atmosphere. Three generators each with a capacity of 37 tons/hr. of steam at 100 atm. and 797°F. were installed. An eight-hour test on one of these, when raising about 30 tons/hr. of steam, showed 89% efficiency.

Drawings and photographs of a number of monotube steam generators are contained in Sulzer - Item 10. These show sectional views and plant arrangements, also detailed views of various elements and accessories for this sytem. Plant layouts and furnace wall details are shown for both powdered coal and chain-grate-stoker firing. A photograph is shown of a Sulzer van Tongeren dust separator on a boiler-house roof. For additional illustrations of boilers and accessories, see Sulzer - Items 18 and 19.

A well-illustrated 16 page publication in the German language, accompanied by a typed Sulzer translation of the text and titles of figures, makes up Sulzer Items 12a and 12b. Item 13 is a 4 page descriptive leaflet in English.

The most recent published article is one dated 1945 by Director Gaspar (Sulzer - Item 9f). The text is in the German language, but each figure has English and French titles, and there is also a half page summary of the article in each of these languages. The title is "Planning boiler and accumulator plants for industrial power stations with special reference to back-pressure energy production". A large drawing of a diagram of a high-pressure steam plant with reheater is filed as Sulzer - Item 11. It is similar to Fig.32 on p.52 of Sulzer - Item 9f.

The latest reference on this topic is a typed memorandum which was prepared for the writer on September 27th,1945, entitled "High pressure installations equipped with Sulzer Monotube Steam Boilers". (Sulzer - Item 15). It states, in part:-

"Most of the land-installations have been built as superimposed high pressure plants to existing steam generating equipment. The existing installations contained obsolete equipment running with a low overall plant efficiency, and the purpose of the superimposing of the high pressure plant was, besides of obtaining a larger output of electric power, to reduce the coal consumption. The Sulzer monotube steam boilers are best suited for pressures above 80 atm. and the highest pressure up to now in service is 140 atm., although from the point of view of design and construction of the monotube boiler there is practically no upper limit. The steam temperature at superheater outlet used in most of the now (sic) installations is in the neighbourhood of 500°C. (932°F.). Sulzer Bros. have on hand designs for monotube boilers working with steam temperatures up to 600°C. (1112°F.) and these can be put on the market as soon as the special material for the superheater portion of the boilers is available".

This statement is followed by a discussion of improvement in plant efficiency in some cases by reheating the exhaust steam from high-pressure turbines. This can be done by a separate oil or gas-fired unit, but the Sulzer monotube generator is said to permit cheaper and more efficient heating up to about 20 atm. and 750°F. by incorporating the reheater in the boiler itself.

The preheating of feedwater in several stages is another means of improving plant efficiency which can be incorporated in Sulzer high-pressure installations:-

"The number of stages for the preheating and its final temperature depends on the type of firing equipment as it is bound up withthe admissable air heating. With stoker-fired boilers where an air temperature of about 120°C. (248°F.) is generally adopted the feedwater is preheated up to about 150°C. (302°F.); for boilers with pulverized coal firing the air preheating can go as high as 300 or even 400°C. (572 to 752°F.) and the feed heating accordingly reaches about 200°C. (392°F.) for which usually four stages are provided".

The use of high-ash or high-moisture fuels is discussed as follows:-

"The monotube boiler is suitable for burning practically any type of fuel. Particularly in the past years, where the good coal has been used for other purposes, we have been compelled to build boilers for burning coals with ash and water contents up to 40 to 50%. No particular difficulties have been experienced in this respect with the monotube boilers built for coals of such low quality and Sulzer Bros. see no objection in using even coals with higher ash and water contents than those mentioned above. Furthermore the monotube boiler can easily be adapted to any firing equipment, be it stokers or pulverized firing installations. As regards the pulverized coal firing, however, Sulzer Bros. have adopted in most cases the tangential firing with the burners arranged in the corners, the system as developed by the Combustion Engineering Company. In order to avoid slag deposits on the tubes lining the combustion chamber or a choking up of the entire boiler, which may occur at high outputs when using coal, the ashes of which have a low melting point, the first tube bundle at the outlet of the combustion chamber is usually provided with a wide tube spacing. With the design of the combustion chamber as adopted by Sulzer Bros. there are no difficulties in choosing a low figure for the heat release in the combustion chamber (BTU/c.ft) which is particularly necessary when using coal of low quality."

The concluding statement is:-

"With a new large size plant working with 160 atm. 600°C. (1112°F.) and reheat to 600°C. (1112°F.) it will be possible to attain overall plant efficiencies of up to 36%, provided all components of the plant are properly designed."

It is stated in Item 2. p.5, that:-

"Steam power plants of this type offer efficiencies such as are expected from the known forms of gas turbine plant, and they are also quite able to compete with the latter in the matter of space requirements."

Combined Heating and Power Plants. Sulzer - Item 12b, p.2, discusses the advantages of using high-pressure steam first for power and then as low-pressure steam for industrial process work or heating:-

"It offers a particularly favorable solution for industrial concerns which need both power and heating steam in large quantities. The high-pressure steam then supplied by it can first generate power while being expanded to a suitable back pressure in a power unit, and afterwards can be used as heating steam, either directly or by the employment of a steam transformer. The energy thus obtained is a by-product and is accordingly cheaper than power generated by any other thermal means."

Sulzer Bros. consider the design and construction of such plants, including the superimposing of high-pressure plants on existing medium or low-pressure steam plants, to be one of their specialties. (Sulzer - Item 2, p.5).

Items 7 and 8 describe two coal-fired plants in which steam at 100 atm. is first used for power and then as relatively low-pressure process steam in a cardboard factory and a bobbin and spool works.

There is an extended discussion of various aspects of simultaneous heat and power production in Sulzer - Item 9f.

O.Walti, in a paper "The Past and Future of the Steam Engine" (Sulzer - Item 17e) states that the economic position of the steam engine is being restored by the use of high-pressure steam in engines for power followed by use of the steam for process work and heating.

H.Nyffenegger discusses "Combined heating and power plants with back-pressure piston type steam engine" in a German-language article accompanied by a half-page English summary and English titles to the figures.

Heat Pumps. Sulzer Bros. have for years manufactured evaporating plants for the chemical and food industries. The economy of such evaporating (concentrating) plants with and without heat pumps, is discussed in Item 17d by W.Wittwer. Among the factors which favor heat pumps are high fuel cost and a good load factor. The heat-pump plant has low cooling-water consumption. For an evaporator with contents boiling at about 212°F., as much as 44 to 55 lb. of water can be evaporated per kwh. of electric power supplied to the heat pump.

"Centrigual Pumps in Heat Pump Plants" by J.Sprecher is the title of Item 17c. Sulzer centrifugal pumps have been used to deliver water to a number of heat-pump plants including some described in part I of this report, in which Brown,Boveri "Thermoblocs" were installed.

The use of an oilfree reciprocating compressor as a heat pump in concentrating plants, where the vapors must not be contaminated by lubricating oil, is described in Sulzer - Item 17b.

Radiation Heating from Ceilings. Three publications were obtained, all in the German language, on comfort heating, air conditioning,etc., (Sulzer - Items 21, 22, 23). These contain very little detailed technical information. Item 21 describes the advantage of radiation heating and cooling from ceilings containing pipe coils. It contains many general views of public, industrial and private dwellings which have radiation heating by the "System Crittall". Page 16 shows an exterior view of the newly built (1942-43) city hospital of Basel, Switzerland which has 81 miles of pipe coil. This is said to be the largest radiation heating installation on the Continent, and it also provides cooling of the bed area in summer. Item 22 gives the names and addresses of nearly 100 of these installations, with length of concreted heating spiral. These include hospitals, office buildings, indoor swimming pools, schoolhouses, museums, dwellings,etc.

Item 23 contains many small photographs of buildings with Sulzer installations of central heating, air conditioning, distant heating, ventilating,etc.

In industrial plants, water may be heated up to 180°C. (357°F.) by waste steam and circulated under pressure to radiators for heating purposes.

Research Laboratories. Sulzer Bros. have recently completed a large new laboratory building for research, development and control work. A recent issue of the company's Technical Review is devoted to this building. (Sulzer - Item 20a). Floor plans and some of the major equipment are shown in Item 20d. The oscillographic equipment of the new physical laboratory is described in Item 20c. Of interest in connection with gas turbines is their investigation of "Brittleness and Toughness of Metals at High Temperatures" (Item 20b).

Adjoining existing buildings house the thermal laboratory and aerodynamic laboratory. From an inspection of the various laboratories it is evident that this company has excellent facilities for engineering research and control work in their field.

Conclusions.

Sulzer Bros. have investigated gas turbines powered by exhaust gases from Diesel engines, also constant-pressure combustion turbines as prime movers. They are constructing a 7000 S.H.P. oil-fired marine combustion turbine with their own type of circuit, for trial purposes.

Little information has been released on the Sulzer combustion turbine. It is claimed to have certain advantages of a closed circuit without the necessity of a large air-heating chamber. They have not yet given much consideration to the use of coal as gas turbine fuel.

About 50 monotube high-pressure steam generator units (mostly coal-fired) have been built in eight European countries. They recommend these for pressures over 80 atm. Most of the present plants superheat the steam to about 500°C. (932°F.) and the present maximum working pressure is 140 atm. However, as soon as known alloys are commercially available, they are prepared to build generators producing steam at 160 atm. and 600°C. (1112°F) with an expected overall plant efficiency up to 36%. Most of the present installations have been built as superimposed high-pressure plants to supplement existing medium-or low-pressure steam plants.

Much attention has been given to increasing fuel economy thru combined heating and power installations in which high-pressure steam is used in back-pressure steam turbines or steam engines, and the exhaust steam is used for heating or process purposes.

Sulzer Bros. manufacture plants for industrial evaporation and have supplied centrifugal pumps and other accessories to recent installations of "heat pumps" for comfort heating.

They have an extensive business in central heating, air conditioning, ventilating, etc., and have made many large installations of comfort heating and cooling by coils embedded in ceilings ("System Crittall").

They have just completed a large and admirably equipped laboratory for metallurgical, physical and chemical work. In combination with their existing thermal and aerodynamic laboratories, this gives them excellent facilities for engineering research, development and control work in their field.

Present Swiss Views on Energy Problems. A recent issue of the Swiss magazine "Electrical Service" is devoted to articles on the more effective use of energy sources. They believe that in Europe the demand for fuels and electricity will exceed the supply for some time to come.

Some of the articles in this issue have already been cited. Three general articles on energy problems, especially water power and coal, are mentioned in closing this report as they will help to explain the viewpoints and policies of Swiss development engineers.

For convenience in microfilming, these items have been listed with the Sulzer publications. For the cover page and contents page see Sulzer - Items 9a and 9b. The articles are in the German language, with half-page summaries in English and French.

The three articles of general interest are:-

"Water power and coal" - an editorial. (Item 9c).

"Energy as seen by modern science" by Prof.Dr.Franz Tank, Rector of the Federal Polytechnic School, Zurich. (Item 9d).

"Meeting the demand for energy by means of water power" by Prof.Dr.Bruno Bauer, Federal Polytechnic School, Zurich. (Item 9e).

A continuing shortage of imported coal has led to the exploitation of Swiss peat deposits, and the writer saw peat being tested as boiler fuel.

DOCUMENTS AND PUBLICATIONS.
Referred to in Report III - Sulzer Bros, Ltd.

Filed in Bag 1481 and microfilmed on Reel SF.6, pp.000383 to 000760, and 00772 to 00814.
(Some of the items are reprints from "Technical Review Sulzer").

Sulzer - Item 1 (000383 to 000406)
"The Supercharging of Two-Stroke Diesel Engines". Special number of the "Technical Review Sulzer". 31st December, 1941. 21 + 3 pp.

Sulzer - Item 2 (000407 - 000412)
"New Sulzer designs in the domain of the heat engine". Typed memo, undated, obtained September 27th, 1945. 6 pp.

Sulzer - Item 3 (000413 - 000431)
"The Sulzer Single-Tube Steam Generator" by Prof.A.Stodola. Reprinted from "The Engineer". Nov.17/24th, 1933. 19 pp. 5415e - 500 I. 34.

Sulzer - Item 4 (000432 - 000436)
"High Pressure Primary Plant with Sulzer Monotube Steam Generator for the Power Station of a British Colliery". Copy of typed memo with figures. 11th February, 1936. 3076 IX.1935.

Sulzer - Item 5 (000437 to 000443)
"The Monotube Boiler in S.S.'Kertosono'". Reprint from "The Engineer" 5th June, 1936. 7 pp. 5822e. VII. 1936. 500.

Sulzer - Item 6 (000444 to 000452)
"The Sulzer Monotube Steam Generator". Mimeographed text plus figure. 27th October, 1937. 9 pp.

Sulzer - Item 7 (000453 to 000461)
"100-Atm. Steam Power Plant with Sulzer Monotube Steam Generator and Reciprocating Steam Engines" by C.Veit. This article was originally published in "Zeitschrift des Vereins Deutscher Ingenieure" Berlin, Vol.81, No.81, and later in Technical Review Sulzer No.2, 1938. 9 pp. 5873e.

Sulzer - Item 8 (000462 to 000465)
"High Pressure Steam Plant with Sulzer Monotube Steam Generators in a Cardboard Factory in England". Reprinted from Sulzer Technical Review. No.2, 1940. 5921e.

~~Sulzer - Item 9a (00772)~~
Cover page of "Elektrizitätsverwertung - L'Électrique - Electrical Service", 20th year, No.1 - 3, April-June 1945. 1 p.

Sulzer - Item 9b. (00773)
Contents page of same. 1 p.

Sulzer - Item 9c (00774 to 00775)
"Wasserkraft und Kohle; Force hydraulique et charbon; Water power and coal". Editorial, pp.1-2. 2 pp.

Sulzer - Item 9d (00776 to 00781)
"Das Energieproblem im Lichte neuzeitlicher Forschung; Le problème de l'énergie, à la lumière des recherches scientifiques récentes; Energy as seen by modern science" by Prof.Dr.Franz Tank, ETH, Zurich. German-language article with half-page summaries in French and English. Pages 3 to 8. 6 pp.

Sulzer - Item 9e (00782 to 00785)
"Der Einsatz von Wasserkraft und Kohle in die Bedarfsdecking Betrachtungen zur schweizerischen Energiewirtschaftspolitik; L'emploi des forces hydrauliques et du charbon pour couvrir les besoins en énergie; Meeting the demand for energy by means of water power and coal" by Prof.Dr.Bruno Bauer, ETH, Zurich. German-language article with half-page summaries in French and English. Pages 9-12,59. 5 pp.

Sulzer - Item 9f (00786 to 00803)
"Planung von Kessel - und Speicheranlagen für industrielle Wärmezentralen Unter Berücksichtigung der Energieerzeugung im Gegendruckbetrieb; Les projets d'installations de chaudières et d'accumulateurs pour centrales industrielles de production de chaleur, compte tenu de production d'énergie en exploitation a contre-pression; Planning boiler and accumulator plants for industrial power stations with special reference to back-pressure energy production" by Dir.J.Gastpar, Winterthur. German-language article with half-page summaries, and titles to figures, in French and English. Pages 42-59. 18 pp.