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CENTRAL INTELLIGENCE AGENCY

© OUNTRY	East Germany		REPORT		
JBJECT	VEB Elektrochemisch	ies Kombinat Bitterf	eld DATE DISTR.	22 April	1954
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- 1. The organic department of the Bitterfeld Electrochemical Combine was organized into the benzol manufacturing section which produced benzol chloride and an insecticide called Gesarol, a phthalic acid ester called Palatinol, and phosphoric chloride, phosphorus trichloride, phosphorus oxychloride and phosphorus pentachloride; the softening agents section which produced tricresylphosphate and triphenyl phosphate; the section which produced carbon tetrachloride; and the special plants of Werk-Nord which produced oxalic acid and formic acid, and a plant in Werk-Sued which made benzoic acid and sodium benzoate.
- Hydrochloric acid was recovered from the combustion residues of chlorine gas in the inorganic department. Small quantities of hydrochloric acid were also recovered by chlorinating processes in the organic department. In late 1950, the Combine used about 10 percent of its production for its requirements, mainly for making calcium. The balance of the production of hydrochloric acid went almost entirely to Wismut A.G. which allegedly processed a 38 percent acid. The phosphorus section still had only one furnace and had no chance of getting a second one. The chloride manufacturing sections needed a monthly supply of 30 tons of phosphorus. Considerable quantities of phosphorus were sent to Piesteritz for the manufacture of phosphoric acid. Apatite from the Kola Peninsula, whose supply varied, was used in the manufacturing process and often caused difficulties, as only fine-grained material was available. A new method involving pulverized apatite had, therefore, been developed. The apatite was stirred with spent sulphide solution supplied from the Agfa plant in Wolfen and formed a thick paste which was subsequently granulated in a kiln. Ammonia from Leuna was the basic material for all nitrogen products made in Bitterfeld. The ammonia oxydation plant, which had a monthly capacity of about 2,200 tons of nitric acid, was equipped with seven V2A-steel double furnaces which dated from WW II and had remained undamaged. A new catalyzer was developed from cobalt aluminate based on research work started during the war. Between 1946 and 1949, the semimechanical experiments had made such progress that the appropriate

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conversion of the existing oxydation plants could be started and could be completed in 1950. The new method required that in addition to the previous platinum-net contact, a second one of aluminum oxide be fitted to each furnace, making it making it necessary for the flues to be arranged vertically to prevent the gases flowing past from blowing off the cobalt aluminate contact. As a result of this conversion, the previous losses of platinum were reduced, while the percentage of nitric acid remained the same. Aluminate of cobalt was obtained by treating aluminum sulphate with cobalt ammonia and heating it with hydrogen in a small rotary kiln. In this way the required granulation of the contact agent was obtained. Nitric acid was used mainly in producing ammonium nitrate and potassium ammonium nitrate certain quantities of amronium (Kalkammonsalpeter) for fertilizers. mitrate were also delivered to factories manufacturing explosives. Small quantities of nitrinitrate of natrium (sic) obtained were shipped to VEB Farbenfabrik Wolfen. Limited quantities of fluorine were produced in cooperation with the plant in Dohna and were used in Bitterfeld for the manufacture of an agent called Freon. Gaseous fluorine had not yet been obtained. Plans during recent years called for expansion of the fluorine industry with emphasis on fluor chloride Which, thermically

3. A new manufacturing method was developed at Bitterfeld during the period from 1946 to 1947, when, on Soviet orders, a large plant for producing metallic calcium was built. This new plant consisted of three manufacturing plants. The first, located in a building which belonged to the chlorate factory, burned lime with chlorated; (sic). The second melted the chlorinated lime in graphite-lined electric baths. Since the residual metallic calcium still contained a certain percentage of impusives, the third plant melted the calcium with metallic copper and distilled it under high vacuum pressure. With the copper binding all components in a calcium copper alloy, the calcium distillate had a high degree of purity. About 150 tons of distilled calcium metal were produced in 1949. In 1950, this plant was closed down, but was preserved for an emergency.

(sic) treated , produced high-grade plastics.

4. Since early 1950, a new plant, which had been built on a design completed in February 1949, was in operation for the manufacture of titanium dioxide. The designed production was about 80 tons of pure titanium dioxide per month and about 40 tons of titanium dioxide with impurities of iron. The equipment included 4 dissolution (sic) containers, 1 container for the manufacture of germinating (sic) solution, 2 filter presses, 4 hydrolytic containers, 1 rotary annealing furnace, and one rotary lying kiln (sic). The daily consumption of raw material and additional agents for the monthly output of 80 tons of pure TLO2 at the installation was about 10 tons of ilmenite, 0.8 tons of iron filings, 1.5 tons of sodium sulfate, 20 tons of hydrochloric acid gas, 80 percent pure, 0.7 tons of concentrated sulphuric acid, 0.4 tons of caustic soda and 20 tons of high-pressure steam for the hydrolytic process. About 30 kg of barium hydroxide, 50 kg of phosphoric acid, 30 kg of potassium bisulfate and 50 kg of potash were scheduled to be used as mineralor stabilizers of TLO2 pigments. About 750 cubic meters of water from the Mulde River and 150 cubic meters of purified drinking water were also expected. Since the average daily requirement of 20 tons of hydrochloric acid gas was not consumed at an equal rate during 24 hours of operation, the hydrochloric acid gas production has to be keyed to the process of dissolution of ilmenite, working usually with three tanks and leaving one in reserve. The hydrochloric acid gas feed pipes were lined with rubber to prevent corrosion and were large enough to guarantee an hourly flow of about 3 tons without loss of pressure. The four diasolution containers were also rubber-lined and had a layer of acid-proof stones on the rubber layer. The concentration of the dissolution agent in the containers was about 150 to 250 grams of free muriatic acid (sic). About 200 to 300 kgs of mud were set free by the dissolution process which was filtered off by two filter presses of rubberized component parts. Each of the four containers had a gross capacity of 12 cubic meters and a net capacity of 7 cubic meters. One served as a stand-by and, in case of emergency, could also be used as a storage tank. Each hydrolite charge could remain in the container for 8 hours, as 8 dissolution solutions had to be processed daily in three dissolution containers available for continuous operation. This period of 8 hours included 1 hour for charging, 1 hour for heating up, 3 hours for hydrogenation under high steam pressure, and from 2 1/2 to 3 hours for draining and simultaneous filtration of the contents of the containers through rotary filters. These were supplied by VEB Maschinenfabrik Sangerhausen . The daily output of the filtering plant was about 8 tons of wet m-titanic acid containing

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between 50 and 60 percent of water. The 803 content was between 2 and 4 percent with respect to the m-titanic acid. The wet titanic acid was mixed with pure water by means of a lead-lined helical stirring device, and the required stabilizers Metrog added from a small dosing container. The mass then flowed from the helical device into a rubber-lined container, fitted with a stirrer and, while being stirred, was conveyed to an annealing furnace over a rustproof trough. The compactness of the mass was between 1.2 and 1.4 (sic). The annealing furnace was designed for a three-hour operation and a maximum temperature of 1100° C. The rotary kiln had acid firebrick lining. The annealed material, which left the furnace at a temperature of between 400 and 600° C, was slightly agglomerated and was ground in balltype grinding device in a wet grinding process. These wet grinding machines were lined with stones containing titanic acid and were filled with grinding balls which also contained titanic acid and were supplied by VEB Hescho-Kabla, Hermsdorf, Thuringia. At the end of the manufacturing process, the broken mass was conveyed into a rotary drying oven, which was also lined with acid firebricks. Desulphurized generator gas was used for heating the annealing furnace and the drying oven to avoid decolorization of the final product. The hourly quantity of waste from the hydrolytic plant was between 120 and 150 cubic meters, with a daily average of between 10 and 20 tons of muriatic acid. Between 10 and 20 tons were required daily to neutralize this percentage of acid. Between 3 and 5 tons of mud were obtained daily from the hydrolytic operation and the waste. One or two tons from the hydrolytic operation were dumped daily on the waste heap. Most of the remaining mud residue of 2 or 3 tons daily from the Waste disposal system went into the drain system as floating material, while the deposited mud was also carried to the waste heap. Except for the production of titanic dioxide for paint, only the small quantities of titanium needed for producing ferrotitanium were processed at Werk Nord. The main consignees of titanic dioxide were the Bitterfeld plastic plants of the Combine, which consumed about 60 percent of the total for making pigment, and some small paint factories. The ferrotitanic dioxide was ordered mainly by the Kjellberg Elektroden & Maschinen GmbH, Finsterwalde, for welding electrodes which could stand a certain percentage of iron.

- 5. All research work carried out at the Electrochemical Combine had previously served manufacturing purposes and, prior to early 1950, each section of the plant had developed independently. In early 1950, a central research station was established at the instigation of Starostin(fnu), a Russian head engineer, who left the factory in mid-1951. This station, which was in the scientific laboratory, was designed to examine manufacturing problems and to sand results to the various factories for practical development. Although this plan was not carried out, inasmuch as research work was still done mainly in the laboratories of the factories, some kind of coordination was assured, as the central research station was kept informed on the most important work done at the factories. In research work, the hadds of the plants and their laboratory personnel, previously subordinated to their section chiefs, were placed under the head of the research station which drafted long-term research plans for major manufacturing projects.
- 6. Research projects, which were important for the production scheme of the Combine during the postwar years, included the development of a manufacturing method for methylene chloride. The Agfa film factory of SAG Fotoplenka in Wolfen was interested in this method for the manufacturing of noncombustible film foils. Methylene chloride was prepared from methanol chlorinated in iron distilling columns with the aid of various catalysts, e.g. zinc chloride. Methanol was introduced into the column from the top and chlorine gas from the bottom to react with the methanol. The column was fitted with plates to lengthen the reaction path. At the beginning of the reaction process, the apparatus was heated to between 50 and 60° C. by means of carbon resistors and, after a certain initial period, was switched off, as the process was exothermic. Methyl chloride which was recovered during the first stage of this process, in undergoing another chlorination process in a comparatively small similarly arranged set of apparatus, produced methylated chloride. A fractional separation of methyl chloride and methylene chloride was made at this point because of the fluctuations which occurred during the process. The separated methyl chloride was then again conveyed into the chlorination chamber. In the summer of 1952, in the chlorination process still occurred difficulties ince it was not always possible to control the process completely.

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Expensive chlorination resulted in the formation of chloroform. These failures were allegedly remedied later. An expension of the installation, which at that time, had a monthly, capacity of 10 tons, was scheduled.

- 7. Further development research concerned compounds of barium. Experiments for the manufacture of barium chloride which was needed for principitating sodium sulphate for the Combine's electrolytical purposes and, in small quantities for pest-killing agents, started in 1948. The barium salts required were obtained from West Germany. Heavy spar (ferruginous sulfate of barium) was reduced in an old rotary kiln. The barium sulfide recovered was chlorinated, and the sulphur monochloride formed was expelled and used for the plant's purposes. The final products was waterfree barium chloride. The chlorination plant worked at a temperature of between 800 and 900 C. It took a long time to overcome all difficulates, as the sulphur manachloride formed could not be separated without special manages. The chlorination apparatus was frequently choked and the required pressure of charine dropped to 5 or 6 kg above atmospheric pressure, cooling down the temperature of the apparatus and delaying the entire process. In 1950, the monthly production of barium chloride still fluctuated between 20 and 60 tons, while the minimum requirement of the Combine was 50 tons. After 1949, attempts to produce barium nitrate started. Heavy spar from Thuringia was put into a small autoclave with a capacity of 150 to 200 liters which worked under a pressure of between 120 and 150°C. (sic), and carbonic acid was added. Carbonate of barium was thus produced without difficulties. The barium carbonate was then rather easily dissolved with nitricacid. In mid-1950, when about two tons of barium could be produced monthly with this apparatus and the methods indicated, the construction of large scale equipment was started.
- 8. After 1946, the Bitterfeld plant worked on the development of new pesticides. A monthly production of 2 tons of benzol hexachloride was, with difficulty, made possible in mid-1950. In the spring of 1950, the construction of a large production plant started as a result of experimental work on the manufacture of hexachlorcyclohexane and other gamma products for pesticides in 1949 and 1950. Bayer products were duplicated in the field of phosphoric acid ester to be used as insecticides. Although some results were obtained in late 1950, the final results were rather unsatisfactory. The utilization of the proven methods based on phosphoric acid ester seemed rather doubtful, as the comparatively small quantities of phosphorus available had to be used for more important products. From 1946 to 1948, various experiments were made with insecticides containing arsenic. They were, however, given up later because of their toxic effects.
- 9. The procurement of aluminum ore was a problem for the aluminum department of the Combine when its pertinent installations had resumed operation. Deliveries of bauxite from Hungark and Yugoslavia were mostly insufficient as to quantity and, in some cases, quality. The deliveries from Yugoslavia ceased altogether after a while. Large stocks of bauxite had first been available at the Lauta-Werk and were concentrated in Bitterfeld. Although experiments to dissolve the bauxite with nitric acid clay obtained from East Germany were successful to some extent, they did not go beyond therexperimental stage in late 1950. Repeated demands for bauxite with low silicic acid cantent from Hungary seemed to indicate that domestic clay could not yet meet the requirements of the aluminum factory in Bitterfeld.
- 10. Experiments to obtain potassium persulfate and hydrogen peroxide which were made from 1945 to 1948 remained unsuccessful for financial reasons and because the consulting chemist had been eliminated as politically unreliable. When the experiments were discontinued, the hydrogen peroxide was still imperfect as it continued to dissociate into hydrogen and oxygen.
- ll. Experiments to improve the Hooker patent for the electrolysis of sodium peroxide which had started in early 1949 were successful. The improvement resulted from the tier arrangement of the electrolytic double cells and some interior alternation which eliminated the necessity of using mercury. The wall cells were later also used without rubber lining. These changes allegedly permitted the use of more current in the double bath and thus increased the concentration of energy. The tier arrangement also saved space. An experimental plant of 16 double cells, with a capacity of about 60 tons of sodium hydroxide, was put into operation near the engine house electrolysis plant No 1 at the northern plant.

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- 12. After several experiments, the chief of the oxygen section in mid-1949 started production of a mixture of nitric acid or ammonium of lime blended with phosphorus slag. No information was available on the results of fertilizing tests made with this mixture. Experiments on growth hormones for plants were successful to some extent. They involved dichlor phenylic vinegar (sic) which was sprayed on tomato plants during the bhossoming period. Horicultural experiments carried out with this material in the summer of 1949 and 1950 resulted in fruits twice as large as normal ones, with no stones or kernals and good flavor. It was believed that the enlargement of the fruits was due to a sort of cancerous growth. The difficulty of the method was the correct determination of the moment of spraying which otherwise had no effect.
- 13. Research work on softening agents also involved esters of the homologues of benzol. One ester compound of good qualities which was obtained was a success because of its anti-freezing properties. It was used for the casting of foils made from polyvinylchloride. This softener was thoroughly tested. Similar tests were initiated with other derivatives of benzol. A device for high-frequency welding of plastics, which was developed in 1948 and 1949, could allegedly be attached to any sewing machine in place of the needle to function as a moving welding electrode (sic).

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