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I.G. FARBENINDUSTRIE A.G. BUNAWERK SCHKOPAU, GERMANY

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COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

I.G. FARBENINDUSTRIE A.G. BUNAWERK
SCHKOPAU, GERMANY

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Reported by

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CIOS Black List Item - 22
Miscellaneous Chemicals

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE
G-2 DIVISION, SHAEF (REAR) APO 413

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I.G. FARBENINDUSTRIE BUNAWERK, SCHKOPAU, GERMANY.

1. INTRODUCTION.

This plant was visited on 14 May, 1945. It was somewhat damaged, chiefly by bombs, but was still capable of producing 4800 tons/mo of Buna S as compared with its original capacity of 6000 tons/mo. A comprehensive statement of original and current capacities is given in Appendix I.

Information on the organic chemicals, other than Buna, made at Schkopau, was obtained from Dr. Carl Wulff, Director of the plant.

2. RAW MATERIALS.

The raw materials purchased by this plant were as follows:-

Benzene
Coal, powdered
Lime, burnt
Coke
Anthracite
Salt, rock
Bauxite
Methanol
Naphthalene

Power was both generated and purchased, most of the latter being obtained from other IG plants in the district.

3. PRODUCTS AND PROCESSES.

a. Formaldehyde

This was made from methanol, obtained from Leuna, by a continuous process, using a crystal silver catalyst. The process was one developed by IG, and used by them for years. The product made was 30 - 40% formaldehyde solution, containing 2 - 3% methanol.

b. Acetylene

The dry process was used for the production of acetylene from calcium carbide, the method and equipment being identical with those used at Knapsack by A.G. fuer Stickstoffdunger, who built the acetylene plant at Schkopau.

The acetylene produced contained no oxygen, but did contain hydrogen sulphide and phospine derivatives, all of which were removed by treatment with chlorine water. Sometimes acetylene was purified by treatment in a tower filled with a special absorbent charcoal (Benzarbon).

c. Ethylene

Ethylene was made from acetylene by hydrogenation, using hydrogen from Leuna. The temperature used in the hydrogenation was 180 - 300°C, being raised as the activity of the nickel catalyst decreased, and the pressure was one atmosphere.

d. Styrene

The benzene purchased for this synthesis was carefully purified, employing both sulphuric acid washing and neutralization with caustic soda, and distillation. The purified benzene was reacted with ethylene (from acetylene) using anhydrous aluminum chloride as a catalyst, and the resulting ethyl benzene was then dehydrogenated to give styrene. The dehydrogenation catalyst was a complex one, comprising the oxides of zinc, calcium, aluminum, potassium, and chromium. The styrene was redistilled using hydroquinone as an inhibitor to prevent polymerization in the still. No trouble with accumulation of rosin in the still bottom was encountered when using this technique.

e. Polystyrene

Styrene was polymerized in emulsion, using potassium persulphate as a catalyst. As emulsifying agents there were used the synthetic fatty acid sodium salts, obtained from the IG plant at Oppau or from Henkel at Witten. Occasionally Mersolat (sodium alkyl sulphonatez) from Leuna were used, or Mersopon (a mixture of Mersolat and soap).

f. Acetone and Acetaldehyde.

Acetone was made from acetic acid by passing the vapor at 400°C over a catalyst consisting of cerium carbonate on pumice. Atmospheric pressure was used. Acetaldehyde was made from acetylene using a catalyst consisting of ferrous sulphate and mercuric sulphate in dilute sulphuric acid.

The catalyst, when spent, was regenerated with nitric acid.

g. Acetic Acid.

This was made by air oxidation of acetaldehyde, using a copper-manganese oleate catalyst. No ethylidene diacetate was obtained.

h. Aldol

By condensing acetaldehyde there was obtained a crude aldol containing crotonaldehyde, acetaldehyde, aldol, and "polymers" (tri and tetra acetaldehyde condensation products). The entire mixture was hydrogenated in order to simplify the separation by obtaining more stable products. The reactions are as follows:-

Crotonaldehyde → Butyl alcohol

$C_3H_5CHO + H_2 \longrightarrow C_3H_7CH_2OH$

Aldol → 1:3 Butylene glycol

$CH_3CHOHCH_2CHO + H_2 \longrightarrow CH_3CHOHCH_2CHOH$

Acetaldehyde → Ethanol

$CH_3CHO + H_2 \longrightarrow CH_3CH_2OH$

"Trimer" → Hexane triol

$CH_3CHOHCH_2CHOHCH_2CHO + H_2 \longrightarrow CH_3CHCHCH_2CHOHCH_2CHOH$

Octane tetrol was also obtained. The triol-tetrol etc were used in the production of alkyd resins, as substitutes for glycerine. The 1:3 butylene glycol was dehydrated to give butadiene for Buna. Butylene glycol was known in this plant as "Butol".

i. Nekal BX

This product was made by simultaneously condensing two butyl groups on naphthalene and sulphonating, so as to obtain dibutyl naphthalene sulphonic acid, and neutralizing with sodium hydroxide. The sodium salt is Nekal BX, (used as an emulsifying agent in making Buna).

j. Butadiene

Butylene glycol is dehydrated catalytically, the catalyst consisting of a mixture of sodium dihydrogen phosphate (NaH_2PO_4), orthophosphoric acid, and dibutyl ammonium phosphate ($(C_4H_9)_2NH_2 .H_2PO_4$), supported on graphite. The reaction is carried out at 280°C, and one

atmosphere pressure. Steel equipment is used throughout, as no corrosion problem is presented. The heating medium is steam at 60 atm pressure, obtained from the power plant which operates at 120 atm pressure. The reaction is exothermic, so that good heat transfer is necessary. The reaction tubes are also of steel.

k. Buna

Besides Buna S for miscellaneous used, this plant made 180 tons/mo of Buna 85 for hard rubber and 170 tons/mo of Buna 32 for a plasticizer.

l. Chlorine and Caustic

Salt was electrolyzed in mercury cells to produce chlorine and sodium hydroxide solution. The cells were similar to those at Bitterfeld. The chlorine was, for the most part, consumed at Schkopau to make other products.

m. Glycol

This product was made from ethylene by first treating with hypochlorous acid (chlorine and water) to obtain ethylenechlorohydrine, treating this with calcium hydroxide to produce ethylene oxide, and finally combining the latter with water to secure the glycol. Diglycol was made similarly, by obvious modifications.

n. Hydrochloric Acid

All hydrochloric acid needed for the production of vinyl chloride and ethyl chloride was made by the direct combination of chlorine and hydrogen. Ethyl chloride was made almost entirely for the tetraethyl lead plants at Gapel and Frose, a very small amount being sold for use as a local anaesthetic.

o. Vinyl Chloride and Polyvinyl chloride (PCU).

By the direct addition of hydrochloric acid to acetylene, the Schkopau plant produced vinyl chloride. The catalyst used was mercuric chloride (10%) on active carbon, and lasted about 3 - 5 mos. The reaction is carried out at a temperature of 120 - 200°C, but, as it is strongly exothermic, the cooling medium is held at 80 - 150°C, a temperature

differential of 40 - 50°C. Some mercuric chloride sublimes out of the catalyst chamber and is lost, but has never caused any trouble. The output was 2 kg of vinyl chloride per liter of catalyst per day, and the catalyst life about 200 kg per liter. The vinyl chloride is recovered by liquefying in a condenser cooled with brine at -30 to -40°C. The condensate is then fractionated, the products obtained being acetylene, vinyl chloride and 1,1 dichloroethane. The vinyl chloride is stored at -20°C, but is considered stable enough to ship in uninsulated tank cars at 7-8 atm pressure to Wolfen, a trip which was usually made in 24 hrs or less. No stabilizer of any sort is used in the vinyl chloride.

About 800 tons/mo of PCU (unchlorinated vinyl chloride polymer) were made at the Schkopau plant. Potassium persulphate or hydrogen peroxide were used as catalysts, the latter only when the PCU was to be used as an insulator. Mersolat, from Leuna, was used as the emulsifying agent in this polymerization. They did not make any plasticized material.

p. Lubricating Oil

SS 903 and SS 906, having Engler viscosities at 100°C of 3 and 6 respectively, were made by polymerizing ethylene with an $AlCl_3$ catalyst.

q. Tetrachloroethane

This was made by the direct addition of chlorine to acetylene, using tetrachloroethane as a solvent. Iron balls, in a tower mounted on the reactor, were used to supply ferric chloride, the active catalyst, and were renewed as consumed.

r. Trichloroethylene

By treating tetrachloroethane with calcium hydroxide in aqueous suspension this plant produced trichloroethylene, for solvent use.

s. Dichloroethylene, $CH_2 = CCl_2$

This was made from vinyl chloride by first chlorinating to 1-chloro 2:2 dichloroethane and then dehydrohalogenating with sodium hydroxide solution. Calcium hydroxide dehydrohalogenates to give a mixture of the symmetrical and the unsymmetrical isomers, CHCl = CHCl and $CH_2 = CCl_2$, while sodium hydroxide gives only the latter compound. The

APPENDIX I

STATEMENT OF CAPACITIES

Statement No. 1.

showing the capacity of production of the plant as follows:

a. Capacity before the first air raid attack.

b. Estimated capacity of the plant in its present condition.

c. Estimated capacity without supply of electric current from outside.

Capacity in tons per month.

Product	(a)	(b)	(c)
Buna S	6,000	4,800	4,000
Buna 32 and 85	270	270	-
Vinyl chloride	2,500	2,900	-
Polyvinyl chloride	600	200	-
Polystyrene	50	50	-
Ethyl alcohol	800	800	500
Butanol	500	500	350
Acetic acid	700	700	-
Acetone	200	200	-
Phthalic acid	540	270	-
Formaldehyde (100%)	800	800	-
Diglycol	700	700	-
SS oil (lubricating oil)	500	500	-
Tetrahydro furane	300	300	-
Trichloroethylene	400	400	-
Ethyl chloride	120	120	-
Waste lime (refuse used as manure)	14,000	14,000	8,000
Caustic soda lye (NaOH)	4,500	2,500	-
Aluminum chloride	700	700	-

Statement No. 2.

showing the products and the quantities which are required per month in case (c) and naming also the factories and places where they were manufactured so far.
Unless otherwise specified the quantities are given in tons required per month.

Product	Quantity	From where obtained
Brown coal	180,000	Geisel district (near Merseburg)
Coke)		Waldenburg (Silesia)
Anthracite)	10,000	Ruhr district
Burnt chalk	10,000	Harz district
Hydrogen	5,000 cubic meters per hour	Leuna
Benzene refined	1,000	Arbo at Bochum
Tar (or coal)	1,500	Leuna or I.G.-Bergwerke
Special electrodes for the carbide plant	15	Siemens-Plania at Ratibor
Tar	35	Raschig at Ludwigshafen
Pitch	30	Verkaufsgesellschaft für Teererzaugnisse at Essen
Blankets for electrodes	120 pieces per month	Mauser at Köln-Ehrenfeld
Chlorine	25	Ammöndorf (Placo) or I.G. at Bitterfeld
Caustic soda lye (NaOH)	130	Schkopau
Mercury	4	Italy
Nitric acid	40	Piesteritz (Place)
Sulphuric acid (SO_3)	140	I.G. at Wolfen or at Döberitz
Ferrous sulphate	40	I.G. at Leverkusen
Phosphoric acid (P_2O_5)	100	Piesteritz (Place)
Caustic potash lye (KOH)	50	I.G. at Bitterfeld
Caustic potash	10	I.G. at Bitterfeld
Butyl amine	10	I.G. at Ludwigshafen
Silica gel	12	I.G. at Oppau
A-coal	5	I.G. at Leverkusen
Calcium chloride fuzed	5	I.G. at Bitterfeld
Aluminum chloride	30	Schkopau
Naphthaline	85	Verkaufsgesellschaft für Teererzeugnisse at Essen

Statement No. 2 (Continued)

Product	Quantity	From where obtained
Oleum (SO_3)	60	I.G. at Wolfen
Potassium persulphate	30	Elektrochemische Werke at Höllriegelskreuth near München
Diproxide	6	I.G. at Griesheim
Calcium chloride	240	I.G. at Teutschenthal
Acetic acid	40	Schkopau
Phenyl β-naphthyl amine	120	I.G. at Ludwigshafen
Talcum powder	40	Naintsch at Wien or Goller at München
Synthetic fatty acids	40	Deutsche Fettsäure-Werke at Witten

Statement No. 3.

giving a list of the stocks of raw materials on hand with details on how long they will last in case (c).

Product	Tons	Will last for
Brown coal	-	-
Coke)		
Anthracite)	15,000	1½ months
Burnt chalk	-	-
Hydrogen	-	-
Benzene refined	150	4 days
Special electrodes	70	4 to 5 month
Tar	70	2 months
Pitch	90	3 months
Blankets for electrodes	100 pieces	25 days
Chlorine	-	-
Caustic soda lye (NaOH)	500	4 months
Mercury	60	1 year
Nitric acid	-	-
Sulphuric acid (SO_3)	50	10 days
Ferrous sulphate	70	1 3/4 months
Phosphoric acid (P_2O_5)	100	1 month
Caustic potash lye (KOH)	25	1/2 month
Caustic potash	10	1 month
Butyl amine	10	1 month
Silica gel	70	6 months
A-coal	15	3 months
Calcium chloride fuzed	5	1 month
Aluminum chloride	300	10 months
Naphthaline	250	3 months
Oleum (SO_3)	20	10 days
Potassium persulphate	10	10 days
Diproxide	25	4 months
Calcium chloride	50	6 days
Acetic acid	20	1/2 month
Phenyl β-naphthyl amine	700	6 months
Talcum powder	80	2 months
Synthetic fatty acids	50	1 month

Statement No. 4.

showing stocks of finished products on hand.

Product	Tons
Buna S	1,600
Buna 85	50
Igeten (rubber latex)	100
Buna 32	30
Polyvinyl chloride	370
Polystyrene	190
Ethyl alcohol	400
Butanol	130
Acetic acid	20
Acetone	30
Phthalic acid	200
Formaldehyde (100%)	30
Diglycol	250
SS oil (lubricating oil)	80
Tetrahydro furane	20
Trichloroethylene	60
Ethyl chloride	20
Caustic soda lye (NaOH)	500
Aluminum chloride	300