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PERSONNEL OF INSPECTION TEAM

Lt. Col. J. G. Boyne (Br)..........	M. of S.
Dr. P. J. Leaper	CWS, Hq ETOUSA
Dr. W. S. Calcott	CWS, Hq ETOUSA

I. G. FARBENINDUSTRIE
LEUNA, GERMANY

1. INTRODUCTION

This plant was visited on 10 May 1945. It has been very badly damaged by bombing, although it is estimated [illegible] remaining 15% of its synthetic ammonia capacity or [illegible]. Information on organic products of this plant was suppli[illegible] [illegible]ector in [illegible]

2. PROCESSES

a. The chief organic compounds made at this [illegible]

- Methanol, isobutanol, and higher alcohols
- Lubricating oils (from ethylene)
- Phenol, cresols, xylenols, pyrocatechol, and resorcinol
- Cyclohexanol and cyclohexanone
- Epsilon amino caproic acid (an intermediate for "Luran", the IG "Nylon")
- Adipic acid
- Alcohols containing up to 10 carbon atoms, with traces of C_{12} alcohols
- Tri-hydroxymethyl ethane
- Iso octane
- Alkylate (Referred to as ET 120)
- Mersol and Mersolat (soap substitutes, made from a Fischer-Tropsch oil fraction)
- Aldehydes (e.g. Iso-butyric Aldehyde)
- Esters from adipic acid and Leuna alcohols
- Acids from the alcohols
- Mono-, di-, and tri- methyl amines

b. Luran was not made at Leuna, the intermediates being shipped to other IG plants at Landsburg, Berlin, Premnitz, and Wolfen.

c. Triethanolamine was made by IG at Ludwigshafen, but not at Leuna.

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Report No. 187

OFFICE OF THE PUBLICATION BOARD

DEPARTMENT OF COMMERCE

WASHINGTON, D. C.

I. G. FARBENINDUSTRIE A. G.
LEUNA, GERMANY

Reported By

W. S. Calcott,
CWS. HQ. ETOUSA

[1945]

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Price $ .10

8 p

e. The autoclave reaction time, from charging to charging, was 8 hrs, or three charges per day.

f. Bismuth was removed by blowing the tetraethyl lead with air, settling, and then filtering. The bismuth residues were worked up for metallic bismuth, but the production was only 50 - 75 kg per month.

g. The ethyl chloride for this plant was supplied chiefly by the IG plants at Schkopau and at Ludwigshafen, 300 tons/mo being supplied by these two plants. The Frose plant made 60 tons/mo, using alcohol and the zinc chloride catalyst batch process.

h. The Frose plant also made 300 tons/mo of Oppanol (poly isobutylene).

i. A considerable stock of Canadian nickel ore was on hand at the beginning of the war, but most was shipped to other plants for treatment, only 50 tons of nickel having been produced at Frose. A small amount of ore was received from Finland, but was not used.

W. S. CALCOTT,
CWS, Hq
ETOUSA.

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PERSONNEL OF INSPECTION TEAM

Lt.Col.Max Woldenberg.....CWS, Hq ETOUSA
Dr. W. S. Calcott.........CWS, Hq ETOUSA

PLANT OF I.G. FARBENINDUSTRIE FROSE, GERMANY.

1. INTRODUCTION.

This plant was visited on 12 May, 1945. It is the property of the IG, and was put into operation in August, 1939, primarily for the production of tetraethyl lead and ethyl fluid. It operated continuously from August, 1939 to 12 April, 1945. It is entirely undamaged, and in condition to operate again as soon as raw materials are available.

Dr. Franz Ebster (an Austrian) the Assistant Manager of the plant, supplied the information concerning the operations carried on at Frose.

2. PROCESSES.

a. Three hundred tons (metric) per month of tetraethyl lead were made at the Frose plant, using the process of the Ethyl Gasoline Corp., as supplied by them. A copy of the operating instructions was obtained (Betriebs Vorschriften - Management Orders) and has been forwarded to MIRS for filing

b. The alloy was made by the batch process, 2700 kg of PbNa (Na content, 9.98 - 10.00%, Mg content 0.1%) being made in each lot. This was cast in conical molds (cast iron), cooled under heavy oil, crushed and put into the charging hoppers.

c. One half of each batch of alloy, or 1350 kg, was used as an autoclave charge, and was ethylated with 590 kg of ethyl chloride (of which 200 kg were recovered) to give 390-400 kg of tetraethyl lead, a yield (on sodium) of 81.5 - 84.0% of theory.

d. Soluble cutting oil, purchased, was used as a lead dispersing agent in the still. The emulsifying agent was originally Turkey Red Oil, but later, as castor oil became scarce, resin acid sccaps were substituted.

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Report No. 281

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I.B. FARBENINDUSTRIE PLANT
FROSE, GERMANY
FUELS AND LUBRICANTS

W. S. CALCOTT
CWS HQ ETOUSA

[1945]

This report has been declassified and released to the Office of the Publication Board by the War and Navy Departments.

4 p.

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DEPARTMENT OF COMMERCE

WASHINGTON, D. C.

I.G. FARBENINDUSTRIE PLANT

FROSE, GERMANY

FUELS AND LUBRICANTS

W. S. CALCOTT
CWS HQ ETOUSA

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and sent under its own pressure through a filter, (2), to remove traces of ice and or solid carbon dioxide. The filtered liquid then passes to a point in the upper fractionating column where the mixture composition corresponds to that mentioned above.

The upper column operates at about 0.2 atmos. above normal pressure and separates the feed liquid by nitrogen reflux at the top into "pure" gaseous nitrogen at the top end "pure" liquid oxygen at the bottom. (NB--Boiling points at atmospheric pressure are as follows: Nitrogen = -195.8° C; Oxygen = -183.0° C).

The heat for fractionation in the low pressure (upper) column comes from the condensation of gaseous nitrogen rising to the top of the pressure column directly below. The tubular heat exchanger at the base of the upper column, and integral with the top of the lower column, thus has a double purpose. It supplies the heat for fractionation in the upper column and furnishes the reflux nitrogen liquid for the lower column, the heat exchange being made possible because of the "inversion" of temperature caused by the pressures chosen for the two columns. Part of the condensed nitrogen liquid at the top of the lower column, where it serves as reflux and is in turn flash vaporized to form the above-mentioned pure gas product. This nitrogen gas leaves the system by way of the Frankl regenerator assigned to nitrogen service at a temperature only 3°- 4° C below surrounding temperature.

The boiling liquid at the base of the upper column is substantially pure oxygen, and the proper proportion of the gas rising therefrom is deflected as oxygen product through the corresponding one of the Frankl regenerators. It emerges from the Frankl as the product stream of oxygen and at only 3 or 4° C below atmospheric temperature.

Each of the product streams, i.e., the "pure" nitrogen and the "pure" oxygen, remove by evaporation from the corresponding Frankl regenerator, the ice and solid carbon dioxide left from cooling of the incoming air in the previous phase of their operation. Thus each of the Frankl regenerators is not only again reduced in temperature, but is also put in a clean condition for good heat transfer, as well as for the further removal of water vapor and carbon dioxide from air led through in the next succeeding cycle. The evaporation of solids is complete enough so that, ideally, there is no need ever to remove the precipitated components by allowing the Frankls to tically cracking coke oven gas to CO and H_2 and then catalytically reducing to CH_3CH. To this end the CO_2 is removed from the raw gas and the CO, CH_4 and C_2H_4 separated by liquification. This mixture is then cracked catalytically with water vapor giving CO and H_2. The catalyst used and the conditions of operation were not disclosed but have been patented. The resulting gas consists of 10% N_2, 30% CO, and 60% H_2. This is then catalytically reduced to methyl alcohol. Production is 20,000 liters per day. In order to be able to run the alcohol unit to capacity and be partially independent of fluctuations in the supply of coke oven gas a new water gas plant has been installed. This water gas plant has a production capacity of 80,000 cu m. per day. Up to now, however, CO and H_2 from coke oven gas has been used for their methyl alcohol production. Almost all the methyl alcohol produced is converted to formaldehyde. Little is [illegible] Company.

7. Ethyl alcohol is produced from the ethylene (present in coke oven gas to the extent of 2%) by absorption in sulphuric acid. The sulphate thus formed is hydrolized to alcohol. Almost all of this alcohol is used for the manufacture of acetone. Additional alcohol is purchased to supplement the [illegible] liters day produced from ethylene.

8. Acetone is made by a process original with this company. This process has been patented. The operating conditions and catalyst were not disclosed. The method consists of reacting alcohol and water over a catalyst in one step and producing acetone, CO_2 and H_2 according to the equation:-

$$CH_3\ CH_2OH + H_2O = CH_3CO\ CH_3 + CO_2 + H_2$$

Production amounts to 3000 kg/day. It is sold to Air Liquide's acetylene works and to a viscose plant at Tubrize near Brussels.

9. Formaldehyde is made by catalytic oxidation (Ag) of methanol. 12,000l/day of methanal are used giving 20 tons of 40% formaldehyde per day. This is mostly used for the preparation of plastics.

10. Other organic solvents produced in small amounts from the above materials are ethyl ether, methyl ether, methyl/acetate, ethyl acetate, isobutyl alcohol (as a by product only) and the dimethyl acetal of formaldehyde $HCH(OCH_3)_2$ which has use as a solvent.

11. Before the war the company had a pilot plant for

the hydrogenation of coal tar oils producing 85-90 octane gasoline (before leading). This plant was dismantled in 1940 but in 1939 a total of 600 tons of such gasoline was delivered to the Belgian Army. It was planned to hydrogenate coal in two stages, but first to a tar oil then to a light oil which would be rectified.

12. Coal tar oils (neutral or phenolic) were hydrogenated at 700 atmospheres catalytically and gave a lubricating oil of good viscosity index. This oil was tested by the Belgian Army before the war and proved satisfactory for airplane engine operation. The method was published prior to 1940 and was original with this company. The Germans did not learn of this from Company personnel during the occupation and did not inquire concerning this synthetic lubricant. The laboratory plant had a capacity of 1kg/day. It was stated that if hydrogenation is carried out at a sufficiently high pressure no catalyst is necessary.

13. In the laboratory vegetable oils were hydrogenated to give alcohols of from 12 to 20 carbon atoms. These alcohols found use in the textile, drug and soap industries.

14. This plant also was obliged by the Germans to use the emulsified oil and water as a lubricant. Their chemists tried to determine the nature of the agent but with no success. They believe it is not a sulphonated alcohol. Samples of emulsion containing the emulsifier have been secured from the Marly plant. Colonel Phelps can have this analyzed in London in a very short time.

15. Plastics of several types are made. The largest production is 'phenoplast' from phenol and formaldehyde. Phenol is purchased and was procured from Germany. Production of this resin amounted to 40 tons/month which gives about 80 tons of moulding powder. Production of "aminoplast", urea and formaldehyde product, amounted to 10 tons per month. By polymerizing urea and formaldehyde to a low degree of solid material is obtained that is used as a glue. This is sold either as a solid or a water solution. It is used as the adhesive in the manufacture of plywood. Another resin, manufactured in small amounts is made from formaldehyde and P - tert butyl phenol. This gives a resin soluble in China wood oil which solution is a high grade varnish used especially for electrical equipment. Melamine has been produced intermittently, some months as much as 25 tons having been made. This is polymerized with formaldehyde to give "resin melamine" which has a good color and chemical resistance. It was produced in place of "phenoplast" when phenol was not available. Further, by less extensive polymerization a glue was made. The calcium cynamide for making melamine was purchased from Societe Belge

SECRET

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Personnel of Team

Lt. Commander R.H. BULLARD, U.S.N.R.

22/45 Societe Belge de l'azote at Liege.

1. The Liege works of the Societe Belge de l'azote at produit chimique (Target 22/45) was visited. Several conferences were held and among the personnel interviewed were:

M. Leroux	General Manager
M. Victor Huban	Technical Manager
M. F. Braconier	Chief Chemist
M. G. Deschamps	Research Director
M. L. Hanegraef	Sales Manager

2. Whereas the Marly plant of this company (reported on 22/44 target previously) produces only inorganic materials, this plant at Ougree, near Liege, manufactures both the inorganics and also a line of organics. Their main raw material is coke oven gas which is obtained from the coke ovens of a steel plant, [illegible]

3. A bridge on the Paris-Berlin railroad was the target for American liberators on 12 May, 25 May and 25 August 1944. Since this plant is located near this bridge it received a total of 20 bombs (1000 lb.). Hence most of the plant is not capable of operating for about another month. Parts escaped severe damage and can operate as soon as coke oven gas is available. Labor difficulties locally are the cause of temporary delay.

4. The production of ammonia and nitric acid at this plant is identical with that at the Marly plant. In fact, the Marly plant was laid out and constructed from this one. Hydrogen from coke oven gas is purified in the same manner and all the hydrogen produced is used for making ammonia. All the nitrogen production is used for ammonia and the entire production of oxygen is used in the manufacture of nitric acid (50%). The gaseous oxygen production amounts to 1100 cu.m.per hr. Only gaseous oxygen is produced.

5. During the war this plant produced compressed, methane gas for use as an auto-fuel in place of gasolene. Coke oven gas, which contains CO_2, CO, CH_4, C_2H_4, and H_2 and N_2, was scrubbed to remove the CO_2. The gas is then cooled to liquify the CO, CH_4 and C_2H_4, thus separating from the H_2 and N_2. The CO is removed from the CH_4 and C_2H_4 by fractionation and the methane is compressed into cylinders for use. This gas has a calorific value of 8000 Cal/m3.

6. Methyl alcohol is made at this plant by cataly-

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Copy 1

Report No. 170

OFFICE OF THE PUBLICATION BOARD

DEPARTMENT OF COMMERCE

WASHINGTON, D. C.

SOCIETE BELGE L'AZOTE, LIEGE

[illegible] MISCELLANEOUS CHEMICALS

Lt. Commander R. H. BULLARD, U.S.N.R.

[1944?]

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6 p.

In November 1943 the Germans stated they wanted liquid oxygen for welding purposes only and would furnish Linde equipment free of charge. The Belgians were immediately suspicious. The equipment arrived at Hondeng, Tertre, and Willbroek in January 1944. The Belgians had not installed it by March ("slow-down"). The Germans demanded immediate installation and the three managers resigned. Linde men then came from Germany and installed the equipment but it never actually produced any liquid oxygen. It was thought by the Belgians that this urgent demand for liquid oxygen was for a new weapon - flying bomb - because in April 1944 an Italian workman working on V1 launching installations stopped over in Brussels, talked indescreetly to a local Italian boy, and stated that the new bombs were 3 meters long, the first third contains one ton explosive, second third fuel and liquid oxygen and last third combustion chamber. Also it was learned by the Belgians that the Germans were building 32 new liquid oxygen plants in Germany for new secret weapons. This information may not be too reliable technically.

UNION CHIMIQUE BELGE

A meeting was held at the offices of the Union Chimique Belge. Those present were Dr. Joseph Guillissen - Director of Research,

M. Lambert Wera	Tech. Mgr. Asst.
M. Massard	Sec. to Mgr.
Lt. Col. Chittick	
Dr. Sherwood	
Lt. Aldrich	
Lt. Comdr. R. H. Bullard	

This company has thirteen (13) plants in Belgium. The following general information was obtained:

No chemical warfare agents were made in Belgium during the German occupation. Such plants as were in operation prior to the war for the Belgium government were dismantled before German entry.

From their coal tar plants some benzol, xylol, naphthalene, phthalic, anhydride, phenols, carbazole and anthracene were sent to Germany. But because of the "slow-down" only small amounts were available for export.

No concentrated nitric acid was exported.

Germany took 2000 tons of sulphuric acid per month from Belgium which is one third the total Belgium production.

Germany took all the arsenic oxide from Hoboken at the beginning of the war leaving almost none for insecticide manufacture for Belgium.

A total of 150 tons of 30% hydrogen peroxide was sent to Germany to the Deutsche Gold and Silber Scheide Anshalt. The Germans especially desired hydrogen peroxide in early August 1944 of higher concentration but none was available.

No ethyl alcohol was sent to Germany because of the scarcity of beet sugar molasses, its source. Actually alcohol was imported from France.

Germany requested ethanolamines, chlorsulphonic acids and ammonium chloride. They further wanted pharmaceutical products such as sulphanilimide but none was available.

Ammonium nitrate was requested in August 1944 but none was shipped.

/s/ R. H. BULLARD,
Lt. Comdr., USNR.

I.G. Farben. Dr. Rumscheit was in charge of all nitrogen industry for N. France, Belgium and Holland. In January, 1944 he was promoted so he had charge of German nitrogen industry also. Whereas Dr. Rumscheit was very able, his assistants were mediocre technical men. Hence the Belgians were able to dissemble easily and were very effective in their "slow-down". Dr. Rumscheit's first assistant was a Dr. Hill who is now presumed to be in Paris since he left Brussels for Paris on 1 September. Dr. Hill was stated to be a very good Nazi. His second assistant was Herr Suhr. The Mechanical engineer was Herr Lurtz. Very little material from this plant was ever shipped to Germany as the products. fertilizers mostly, were used in Belgium. No oxygen for shipment was made. Before the Germans left Brussels critical parts of equipment, all Platinum catalyst were hidden. Hence nothing was removed from this plant. It can be in full operation in two weeks from the time coal is received from Liege.

It was learned that during the winter of 1943, lubricating oils became critical. Under orders from Dr. Struck, Deutsche Referat for Mineral oils (whose assistant was Dr. Reuschle) an emulsifier were added to lubricating oils and emulsions with water were used as lubricants. It was stated that up to 50% water could be used. These emulsions were used for almost all lubricating purposes in their plant including the high compression equipment. This was first used in February 1943 and has been used since that time. Such lubricants were reported to be highly successful and it is the intention of the engineers to use them in the future for reasons of economy. The emulsifier was placed in the oil by the Referat and not given to the Belgian operators. The material was made by IG Farben at Leverkusen. Arrangements were made for securing samples of the oil containing the agent. Lieutenant Aldrich will bring these samples on his return.

The subject of liquid oxygen was discussed. No liquid oxygen was made at this plant for Germany. All oxygen produced went into nitric acid or CO_2. It was stated however that the Germans were much interested in liquid oxygen and had converted three Belgian plants to this purpose. These three plants are as follows:

1. Willebroek (20 km from Brussels)
   This plant was ready to operate but did not get into production.
2. Hondeng - Goegnies near La Louviere.
   This plant was under German control but did not operate

3. Zandewoorde (near Ostend)
   The personnel of this plant were forced to stay on but this plant never operated.

At Angleur (near Liege) a liquid oxygen plant was built by the Germans by exclusively German labor. It was very recent and was guarded by machine guns. It started operating August 15. Linalux, the power company, kept records of its power consumption and found it to be small. Colonel Chittick, Dr. Sherwood, Cdr. den Hartog and Lieutenant Aldrich left by jeep to visit this plant 12 September.

COMPTOIR BELGE DE L'AZOTE

Thru the assistance of Lieutenant Biot, U.S.N.R., a conference was held on 11 September at the Comptoir Belge de l'Azote, 8 rue de la Suisse. Those present were:

M. Edmond Voituron,	Directeur General
Leon Tallier	Directeur
M. Leroux	Tech. Mgr. Soc. Belge de l'Azote-Liege.
M. Isaac	Mgr. Marly works.
M. Verhaggen	Mgr. Cobelaz
Col. Chittick, CWS.AUS	
Lt. Aldrich, U.S.N.R.	
Lt. Comdr. Bullard, USNR.	

This is a cooperative bureau for the nitrogen industry in Belgium. M. Voituron welcomes visits to any of their plants by military missions and such visits are expected. Much of the information obtained during this conference duplicated that given above. Detailed minutes of this meeting were taken by Dr. Sherwood and Colonel Chittick will furnish us with a copy. Additional information obtained was as follows:

Dr. Oster headed all German nitrogen industries;
Dr. Rumscheit being his assistant operating in Belgium.

Before 1940 Belgium made some concentrated nitric acid but the plants were entirely dismantled before German occupation and none was made during the war.

Some NH_3 was shipped both to France and Germany but the amount was small.

The current situation on sulphuric acid in Germany is bad. There are no imports of sulphur and the German pyrites is low grade.

The story on liquid oxygen was much the same as given above.

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Lieut. ALDRICH, U.S.N.R.
Lt. Col. CHITTICK, CWS. AUS.
Lieut DIXON, U.S.N.R.
Lieut. M. A. BIOT, U.S.N.R.
Colonel P. H. DANE, A.A.F.

SECRET

AMERICAN EMBASSY
OFFICE OF THE NAVAL ATTACHE
LONDON

11 September, 1944

INTRODUCTION:

The plant of Societe Belge de l'Azote et des produits chimiques do Marly located at Brussels was visited by Lt. Comdr. Bullard, Lieut. Aldrich and Lieut. Dixon 9 Sept 1944. M. Rene Lombaers, Technical [illegible] Gerad, Head Engineer were present.

CHEMICALS:

[illegible] the following chemicals a[illegible]

NH_3 aqueous and anhydrous

NH_3 anhydrous

NHyCl pure and com. grades

HNO_3 50% only (no conc)

$(NH_4)(_2SO_4$

NH_4 NO_3

$NaNO_3$

KNO_3

NaH CO_3

The other chemicals listed in their catalog are made at their Liege plant.

In the afternoon Colonel P. H. Dane, AAF and Lieutenant M.A. Biot, U.S.N.R. (BuAer) arrived at the Marly plant and joined the conference.

MARLY PLANT:

The Marly plant was operated throughout the occupation by the Belgian company under the direction of the German Referat for Nitrogen Industries, Dr. Rumscheit, a manager of

File II-8
Item 22

Copy 1

Report No. 168

OFFICE OF THE PUBLICATION BOARD
DEPARTMENT OF COMMERCE
WASHINGTON, D. C.

SOCIETE BELGE DE L'AZOTE

Reported by
Lt. Comdr., R. H. Bullard, U.S.N.R.

[1944]

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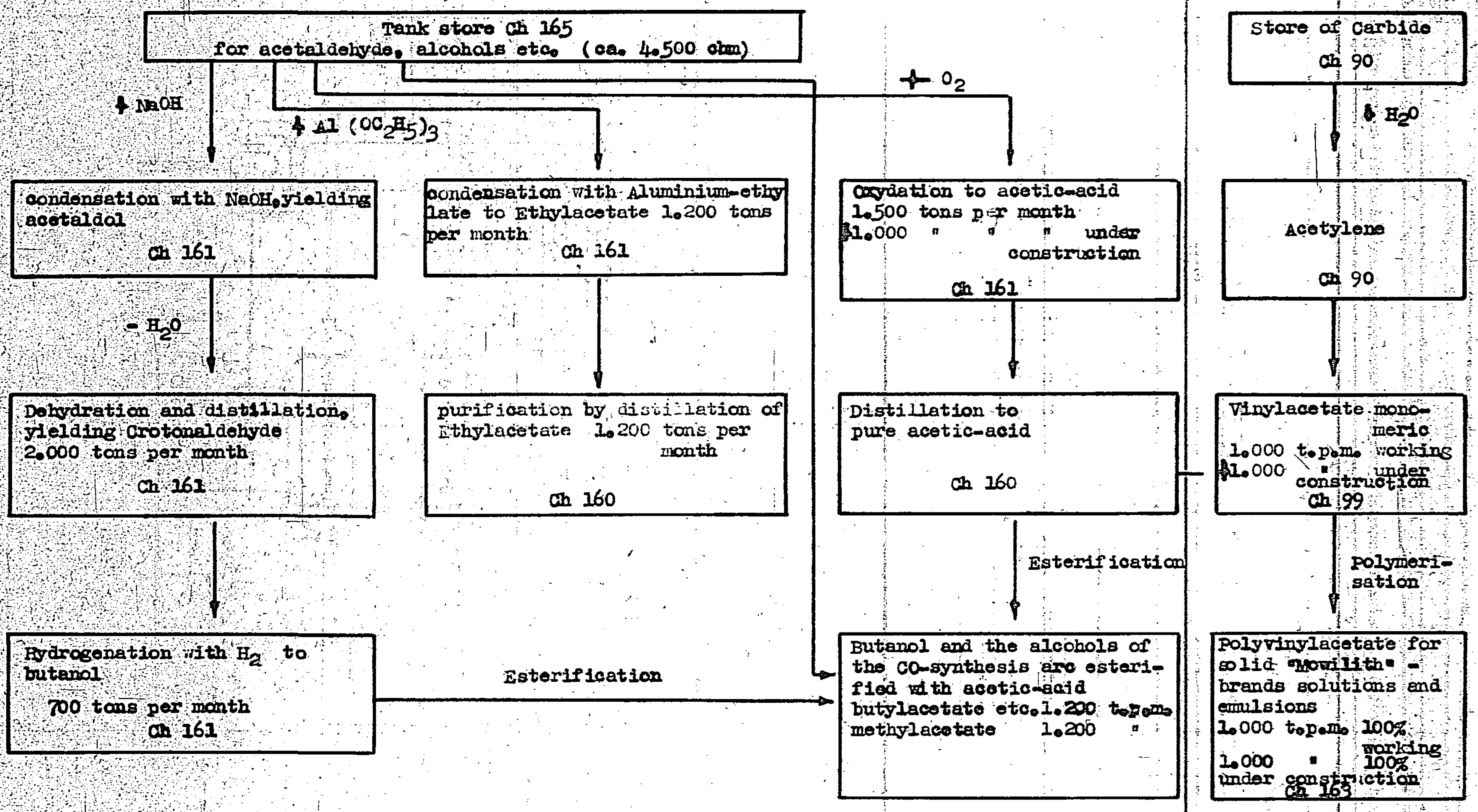
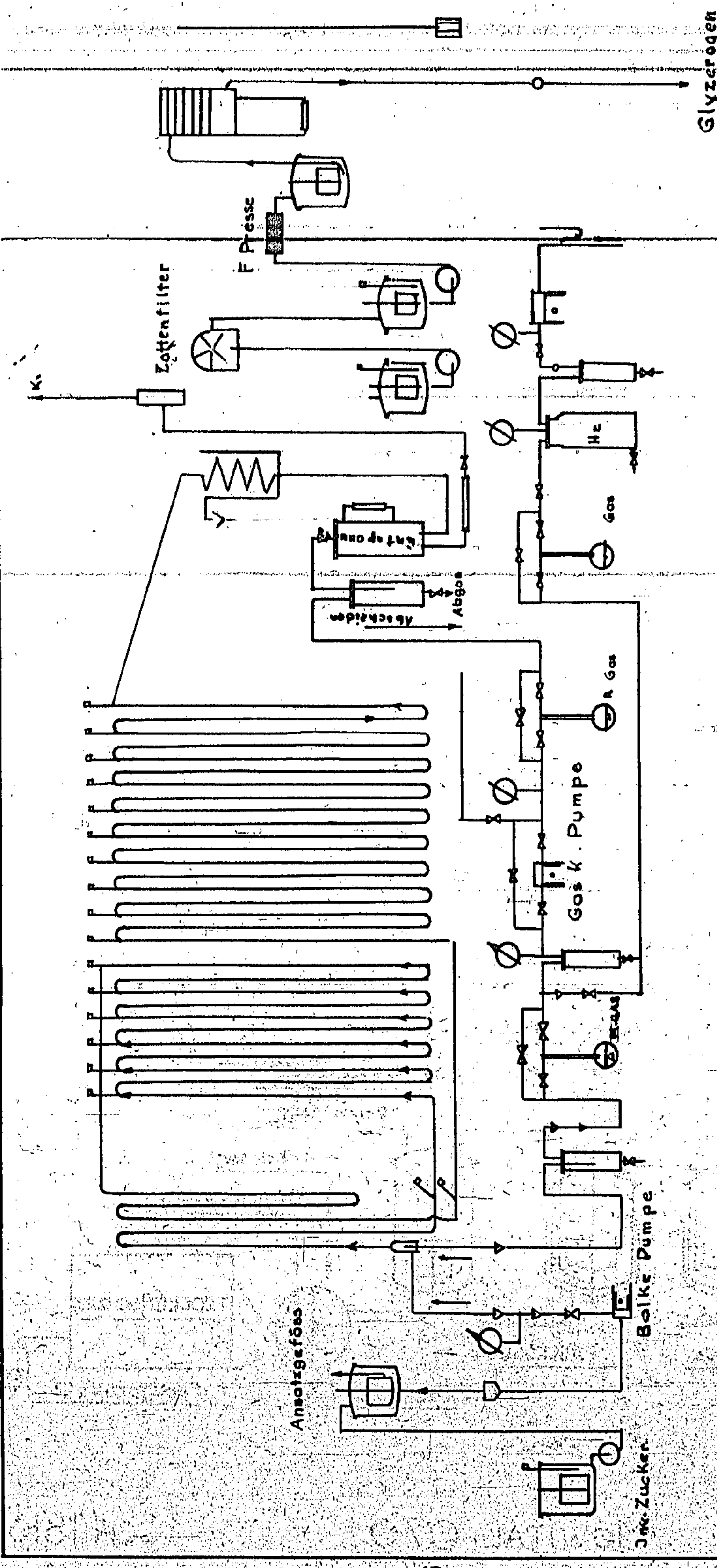
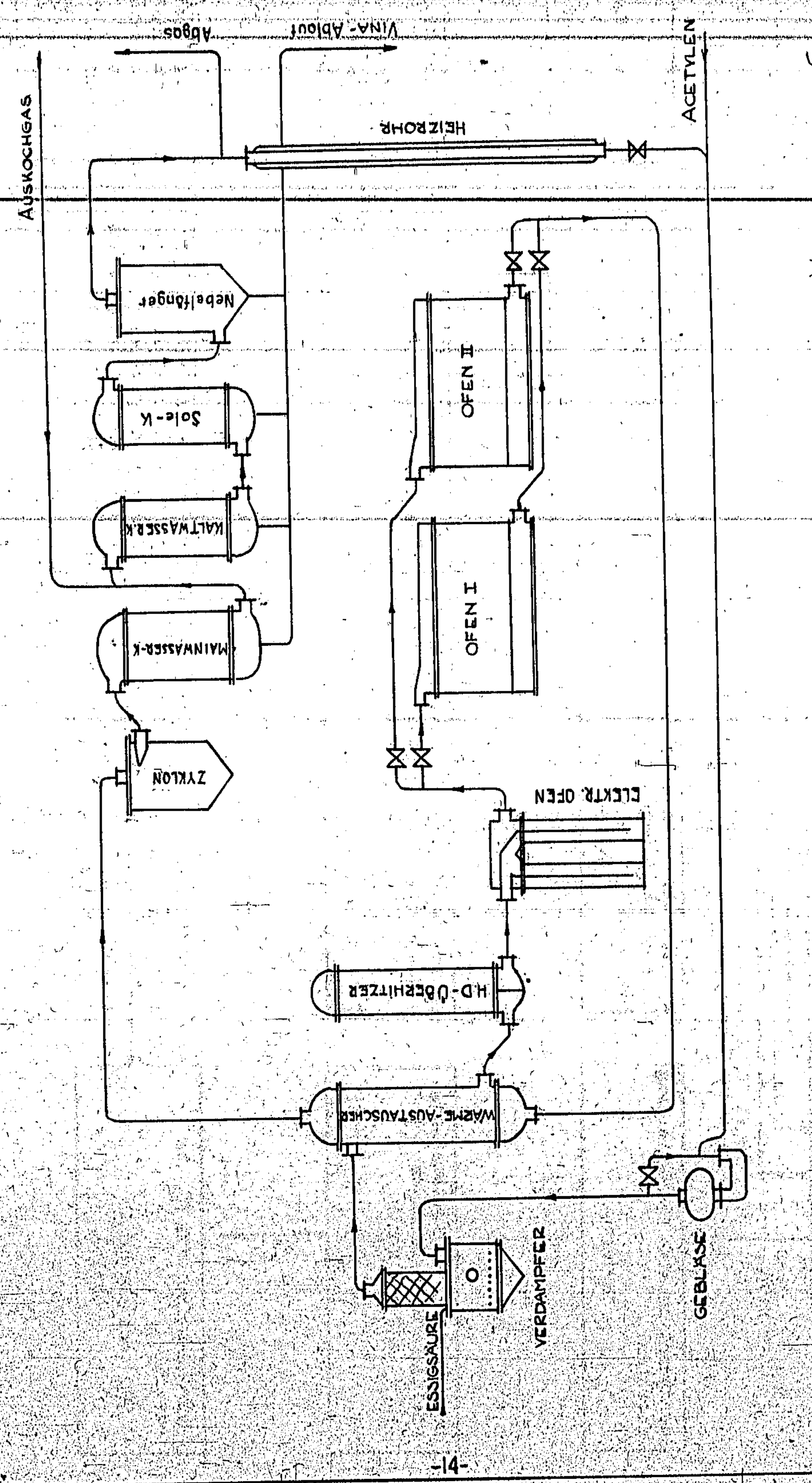
7 p

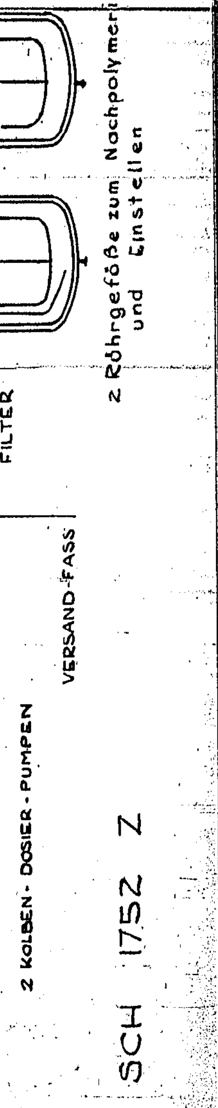

Tank store Ch 165
for acetaldehyde, alcohols etc. (ca. 4.500 cbm)
+ NaOH
+ Al $(OC_2H_5)_3$
+ O_2
Store of Carbide
Ch 90
+ H_2O
condensation with NaOH, yielding acetaldol
Ch 161
condensation with Aluminium-ethylate to Ethylacetate 1.200 tons per month
Ch 161
Oxydation to acetic-acid
1.500 tons per month
1.000 " " " under construction
Ch 161
Acetylene
Ch 90
- H_2O
Dehydration and distillation, yielding Crotonaldehyde
2.000 tons per month
Ch 161
purification by distillation of Ethylacetate 1.200 tons per month
Ch 160
Distillation to pure acetic-acid
Ch 160
Vinylacetate monomeric
1.000 t.p.m. working
1.000 " under construction
Ch 99
Esterification
Polymerisation
Hydrogenation with H_2 to butanol
700 tons per month
Ch 161
Esterification
Butanol and the alcohols of the CO-synthesis are esterified with acetic-acid
butylacetate etc. 1.200 t.p.m.
methylacetate 1.200 "
Polyvinylacetate for solid "Mowilith" - brands solutions and emulsions
1.000 t.p.m. 100% working
1.000 " 100% under construction
Ch 163



Schema der Glycerogenanlage

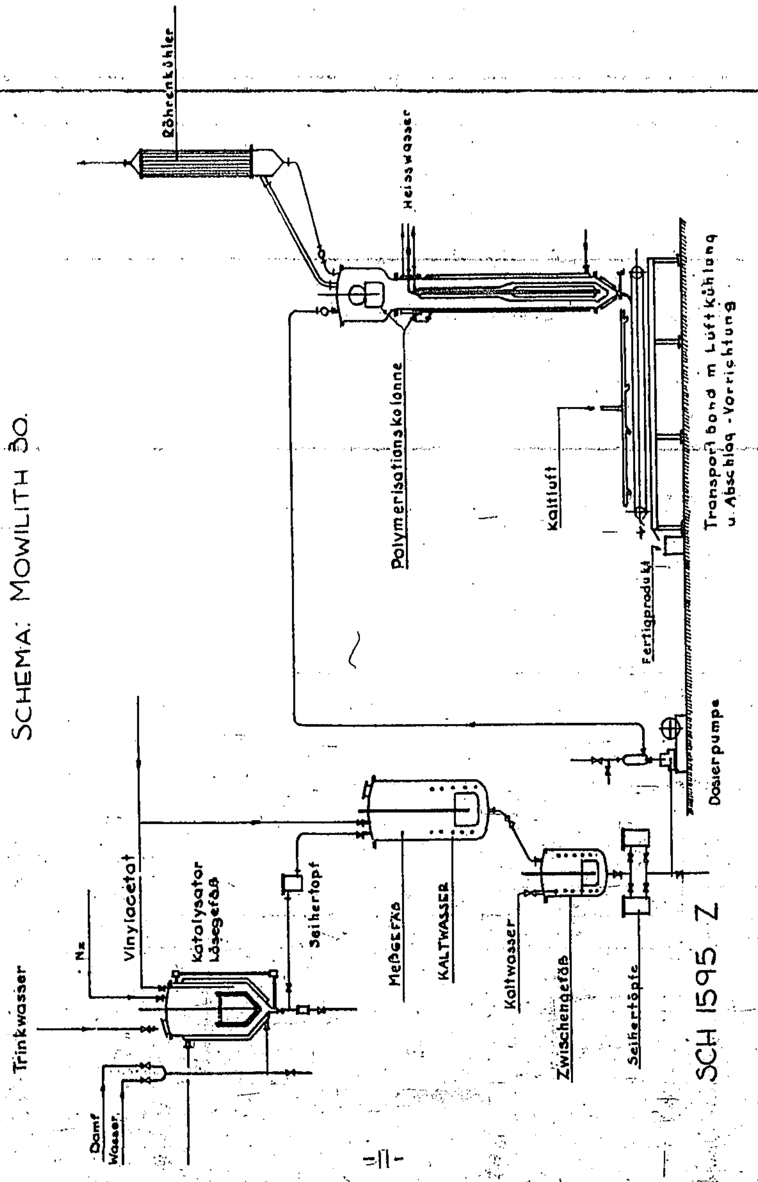


VINYLACETAT-SCHEMA



SCH 1752 Z

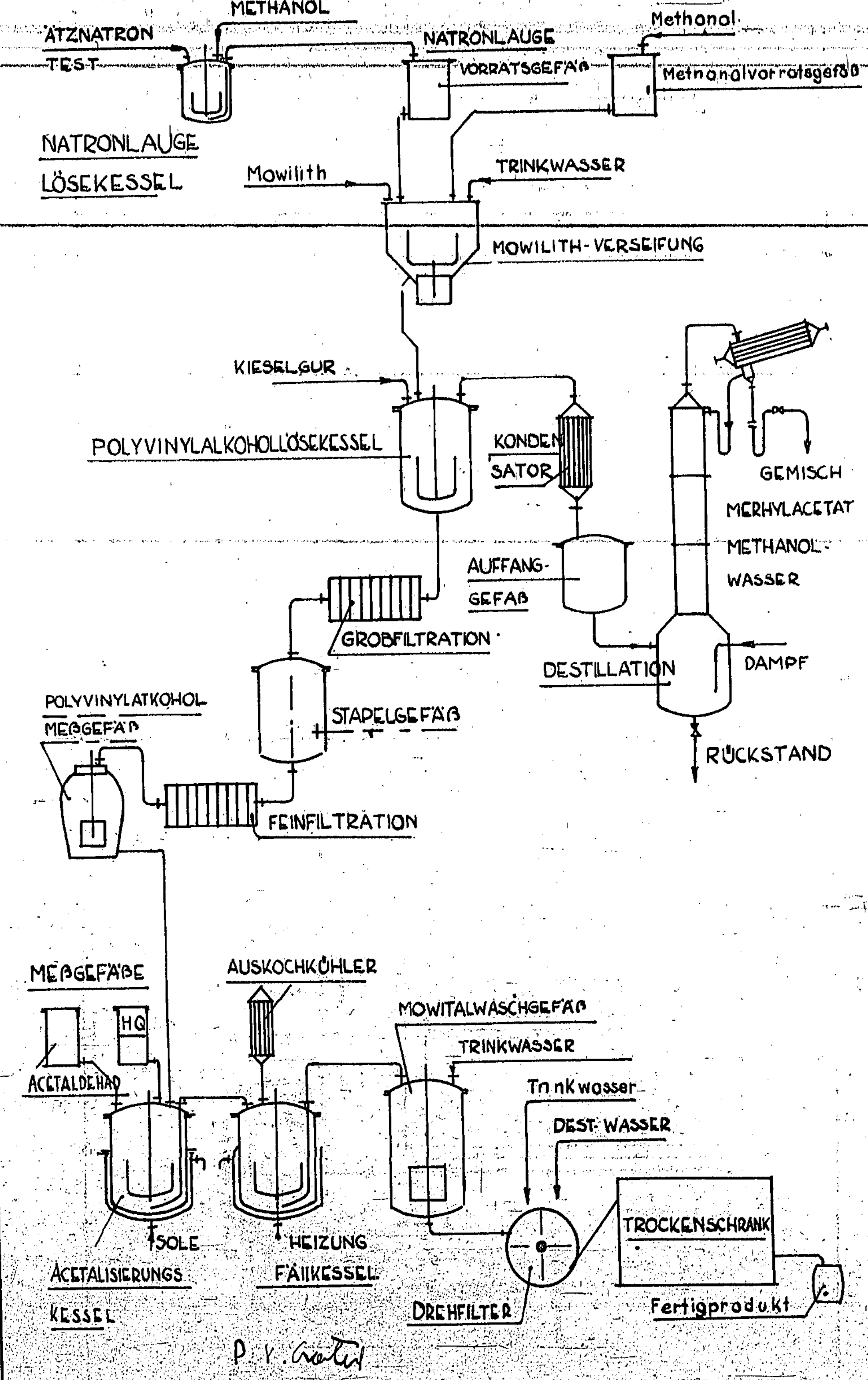
SCHEMA: MOWILITH 30.



SCH 1595 Z

Höchst

CONFIDENTIAL



P. v. Gotz

MOWITAL 070

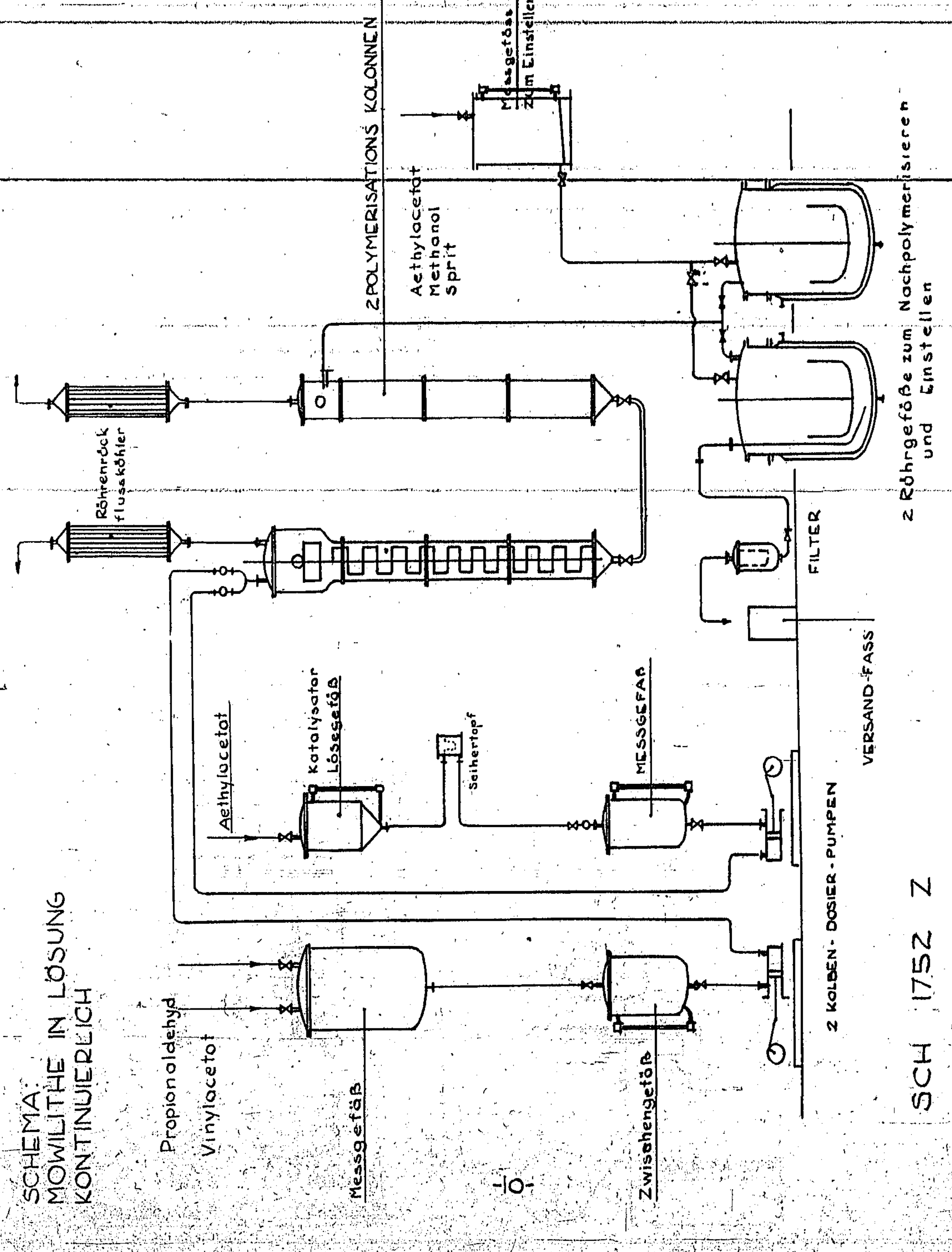
SCH 1810 Z.

mixture boiled under reflux for 24 hours. It is then cooled, centrifuged, washed with methanol, neutralized with sodium carbonate solution and dried in vacuo.

The alkaline hydrolysis is preferred to avoid the presence of H_2SO_4 in the product. The sulfate groups are particularly disturbing in making the acetals. The alkaline hydrolysis is shown on the drawing as well as the subsequent step of making the acetal as Mowilith 070. In carrying out the hydrolysis, 35% of polyvinyl acetate is dissolved in methanol and 0.02 mol. of alcoholic NaOH added per 100 mols. of PVA. The reaction is complete in 30 minutes.

The polyvinyl alcohol is dissolved in water, methanol and methyl acetate, filtered and can then be made into acetals.

R. L. Hasche



Polymerization in Solution

Polymers in the range of molecular weights of Mowilith 20 and 30 can also be made in solution. The accompanying print No. 1752Z shows the method employed.

Vinyl acetate and propionaldehyde are mixed in one vessel and the catalyst dissolved in ethyl acetate, the solvent employed, in another vessel. The two liquids then flow continuously into the two polymerization towers in series. The temperature is maintained at 75-85° C. by means of the reflux condensers and coils within the first tower. The rate of flow is so regulated that the polymer, in a 70% solution, leaving the towers contains about 5% of monomer.

The solution passes to agitated jacketed vessels where additional ethyl acetate is added to give a 60% solution and the monomer reduced to 2%. The entire process requires 12-15 hours. It is suitable for the production of Mowilith 15 and 2 but with type 30 difficulty is sometimes encountered due to separation of the polymer from solution.

C. Mowilith 50, 70 and 90

The Mowilith types 50, 70 and 90 are made by polymerization in suspension, which might properly be called head polymerization. A poor emulsifying agent is used, such as, for example, sodium polyacrylate or polystyrene of a low degree of polymerization. Benzoyl peroxide is the catalyst.

The polymerization is carried out in an enameled vessel provided with an agitator and a cooling jacket. Brine at 0-5° C. is circulated in the jacket. The material is formed in beads approximately 1 mm. in diameter.

An older method, which is still used on a reduced scale, was to massively polymerize the material in a vessel with a paper liner. It was provided with a shaft without propellor blades, a reflux condenser and cooling jacket. At the completion of the polymerization, the cover was removed and a core of solid polymer was withdrawn. The shaft in the middle of the core served as a spindle for a milling machine and the polymer was removed as shavings by a cutter. It was not possible to obtain a consistently uniform polymer by this method.

D. Mowilith ABC

Mowilith ABC has the following composition:

Polyvinyl acetate	81%
Polyvinyl benzoate	15%
Crotonic Acid	3-4%

The composition is mixed with phenol which gives a smooth coating for tin cans. It protects against acid and makes an excellent seal. The polyvinyl acetate imparts water resistance and the crotonic acid resistance and the crotonic acid resistance to acids but is soluable in alkalis.

E. Mowilith AB

Mowilith AB has the following composition:

Polyvinyl acetate	85%
Polyvinyl benzoate	15%

The polymer mixture was prepared in solution or emulsion. It was used as a water repellant for wrapping paper.

F. Appretan H

Appretan H consists of 95% polyvinyl acetate and 5% of crotonic aldehyde in solution of methyl, ethyl or isopropyl alcohols. When ammonia water is added, a thick milky emulsion is formed. It is used as a substitute for shellac and as a stiffener for hats.

G. Mowilith D-300

Mowilith D-300 is a copolymer consisting of 70% vinyl acetate and 30% vinyl chloride. It is used as an emulsion in 50% of water. It is compounded with pigments and plasticizer and makes an excellent washable wall paint.

H. Polyvinyl Alcohol and Acetals

Hydrolysis of polyvinyl acetate to polyvinyl alcohol is carried out both by the acid and alkali methods.

The process can be followed by reference to drawing No. 1810Z. In acid hydrolysis 15% of polyvinyl acetate is dissolved in a solvent consisting of 60% methanol and 40% methyl acetate. Sulfuric acid is added and the

After the reaction was complete, the solution was reduced to atmospheric pressure, allowed to settle and the catalyst filtered. The hydrogen was recirculated. The solution was bleached with charcoal and distilled. The composition of the product was approximately as follows:

Glycerol	40%
Proprylene Glycol	40%
C_6 Alcohols	20%

The yield was approximately 95%. The product was very stable and the redistilled glycerol fraction could be used as a substitute for the product from fats for all purposes.

5. SYNTHESIS OF N-BUTYL ALCOHOL

Normal butanol is synthesized at the Hoechst plant from acetaldehyde which is supplied by the I.G. plant at Knapsack/Kohn. The maximum capacity of the Hoechst plant is 700 tons per month, or approximately one-quarter of the German production of N-butanol. The plant is undamaged.

As the accompanying flow diagram indicates, acetaldehyde is condensed with caustic soda to form acetaldol. This reaction is carried out in a vertical tower with recirculation of the liquid through heat exchangers to absorb the heat. The catalyst is introduced into the suction of the circulating pumps.

Crotonaldehyde is produced in a continuous still from aldol employing acetic acid as the catalyst. The croton aldehyde is finally distilled under reduced pressure and less than 1% of acetoldehyde remains in the purified product.

The crotonaldehyde is reduced by hydrogen in the vapor phase over a catalyst consisting of copper deposited on pumice. The pumice 5-8 mm. in size is mixed with a concentrated copper carbonate solution in a wooden vat and the operation controlled so as to obtain 8% of copper carbonate on the support. A catalyst charge to the converter is 20 tons of pumice and 1.6 tons of copper carbonate. The volume of the converter is 50 cubic meters.

Since the reaction is exothermic, cooling tubes are provided in the catalyst bed and the temperature maintained within 5°. The operating temperature is 200° C. Water saturated with steam at 160° C. or 8 atmospheres pressure serves as cooling medium.

A two-fold excess of hydrogen is fed with the crotonaldehyde. Conversion is 98% to N-butanol, together with less than 1% of ethanol - the latter formed from traces of acetaldehyde in the crotonaldehyde feed.

6. MOWILITH POLYMERS OF VINYL ACETATE

The polymers of vinyl acetate, produced at Hoechst, were known to the trade as MOWILITH 20, 30, 50, 70 and 90 having a molecular weight with ascending numbers from 3,500 to 80,000.

Mowiliths 20 and 30 are made by massive polymerization; while types 50, 70 and 90 are emulsion polymers.

A. Mowilith 20

Mowilith 20 is made in a jacketed kettle with agitation. A reflex condenser serves to remove the heat of polymerization. That catalyst is 0.2-0.3% of benzoyl peroxide. A small amount of propionaldehyde is added to regulate the chain length.

Excess monomer is removed under reduced pressure and with agitation. The product is then poured while molten into molds.

B. Mowilith 30

Mowolith 30 is made by continuous polymerization in a tower. The operation may be followed by reference to drawing 1595Z.

Vinyl acetate is mixed with benzoyl peroxide and propionaldehyde in the same proportions as in the polymerization of Mowilith 20. It is then withdrawn through a measuring vessel into a pump and thence flows into the polymerization tower where it is maintained at 80° C. When the material at the bottom of the tower has attained the desired degree of polymerization it is discharged through slits and passes on to a conveyor belt in the form of bands. Cold air is blown in the material and at the discharge end of the belt it is cut by a rotary cutter.

The production of Mowilith 30 was 200-250 tons per month.

Butanol	Tons/Mo.	Tons/Mo.
Total German Capacity		3,250
Hoechst	700	
Ludwigshafen	1,000	
Schkopau	1,000	
Huels	300	
Wacker	250	
Methyl Acetate		
Total German Capacity		2,000
Hoechst	1,200	
Wacker (Burghausen and Muckenberg)	2,000	
Butyl Acetate (including Polysolvan)		
Total German Capacity		1,400
Hoechst	1,200	
Wacker	150	
Hiag	50	
Vinyl Acetate		
Total German Capacity		1,300
In Construction		(1,000)
Hoechst	1,000	
In Construction	(1,000)	
Wacker	300	
Mowilith-Emulsions		
Total German Capacity		1,600
In Construction		(400)
Hoechst	1,200	
In Construction	(400)	
Wacker	400	

3. MANUFACTURE OF VINYL ACETATE

The operation will be described by reference to the flow sheet prepared under the direction of Dr. Moeller. A vapor phase catalytic process is used. The catalyst is active charcoal upon which zinc acetate is deposited to give a zinc content of approximately 15%. The catalyst has a particle size of 3-5 mm.

Acetic acid of 98% purity is fed into an evaporator and thence passes through a heat exchanger, a superheater, an electric heater, and then enters two Fischer-Tropsch catalyst units operating in parallel. It will be noted that the acetic acid vapor is carried through the system with acetylene which is circulating and in excess. The reaction is exothermic and it is very important that a close temperature control be maintained. This is accomplished by return bend 1" diameter tubes running horizontally through the catalyst chamber. They are provided with vertical fins of common steel with a 25 mm. spacing. Temperature was controlled within 2-3°. The temperature with fresh catalyst was 180° and was raised to 205° before a fresh charge. The catalyst was discarded after the two units had produced 800 tons of vinyl acetate. Combined volume of catalyst was 28 cubic meters. Surface of fins per unit was approximately 1,000 square meters. The cooling fluid used in the catalyst unit was water under a pressure of 15 atmospheres or 200° C.

The contact time was approximately 30 seconds and the acetylene feed rate was 4,000 cubic meters per hour. Conversion per pass was 60% based on acetic acid.

Separation of vinyl acetate from the unconverted acetic acid was carried out in a stainless steel distillation column using thio diphemylamine as an inhibitor against polymerization. The product was stored without an inhibitor.

4. MANUFACTURE OF GLYCEROL BY REDUCTION OF HEXOSES

Glycerine was manufactured at Hoechst by a process which was developed due to the war-time emergency. The capacity of the plant was 180 tons per month.

Sugar was first inverted in a 70% aqueous solution with 0.1% oxalic acid at 70-80° C. After inversion the solution was neutralized with $CaCO_3$.

Reduction was carried out in a continuous manner in vertical heated return pipe assembly, as shown in the accompanying flow sheets prepared by Dr. Roth. The pipes were 20 mm. in diameter. Those through which the sugar solution and hydrogen gas ascended were jacketed while the down-coming pipes were bare.

Time of contact was approximately 8 hours, and the temperature 200° C. The pressure on the system was 300 atmospheres and the hydrogen velocity was ten times the rate of flow of the sugar solution.

The catalyst was 5% of nickel deposited as pumice and amounted to 1% of fresh and 4% regenerated, based on sugar solution.

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PERSONNEL OF INSPECTION TEAM

R. L. Hasche	TIIC, CIOS
R. H. Boundy	TIIC, CIOS

REPORT ON VISIT TO I.G. FARBEN INDUSTRIE PLANT AT HOECHST/MAIN

1. INTRODUCTION

This report contains information obtained on the manufacture of solvents and miscellaneous chemicals at the Hoechst-a-Main plant of the I.G. Farbenindustrie. Also included in the investigations was the polymerization of vinyl acetate to the various types of Mowilith.

Unlike the Ludwigshafen plant, Hoechst suffered negligible damage by bombing. Even the extensive dock system and storage facilities are intact. The plant could be operated at full capacity if coal could be obtained. However, since all of the main and Rhine bridges were destroyed, coal must come in by rail.

2. PRODUCTIVE CAPACITY

The Hoechst plant was the largest manufacturer of solvents and related chemicals in Germany. The figures which follow were obtained from Dr. Möller, Dr. Dickhaeuser, Dr. Roth and Dr. Schubert.

Following are figures of productive capacity at Hoechst as compared to the total for Germany:

Acetic Acid		Tons/Mo.	Tons/Mo.
Total German Capacity			10,000
Hoechst		1,500	
(In construction)		(1,000)	
Knapsack	2,500		
Schkopau	1,500		
Total	4,000	4,000	
Wacker		1,800	
Hcag (wood carbonization)		1,500	
Lonza		900	
Crotonaldehyde			
Total German Capacity			2,300
Hoechst		2,000	
Wacker		300	

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REPORT ON VISIT TO I.G. FARBEN INDUSTRIE PLANT AT HOECHST/MAIN

June 12, 1945

Reported by:

R. H. BOUNDY, U. S.
R. L. HASCHE, U. S.

On behalf of the

U.S. Technical Industrial Intelligence Committee

CIOS Target No. 22/1(g)
Miscellaneous Chemicals

June 27, 1945

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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I.G. FARBENINDUSTRIE PLANT
HOECHST/MAIN

Boundy & Hasche

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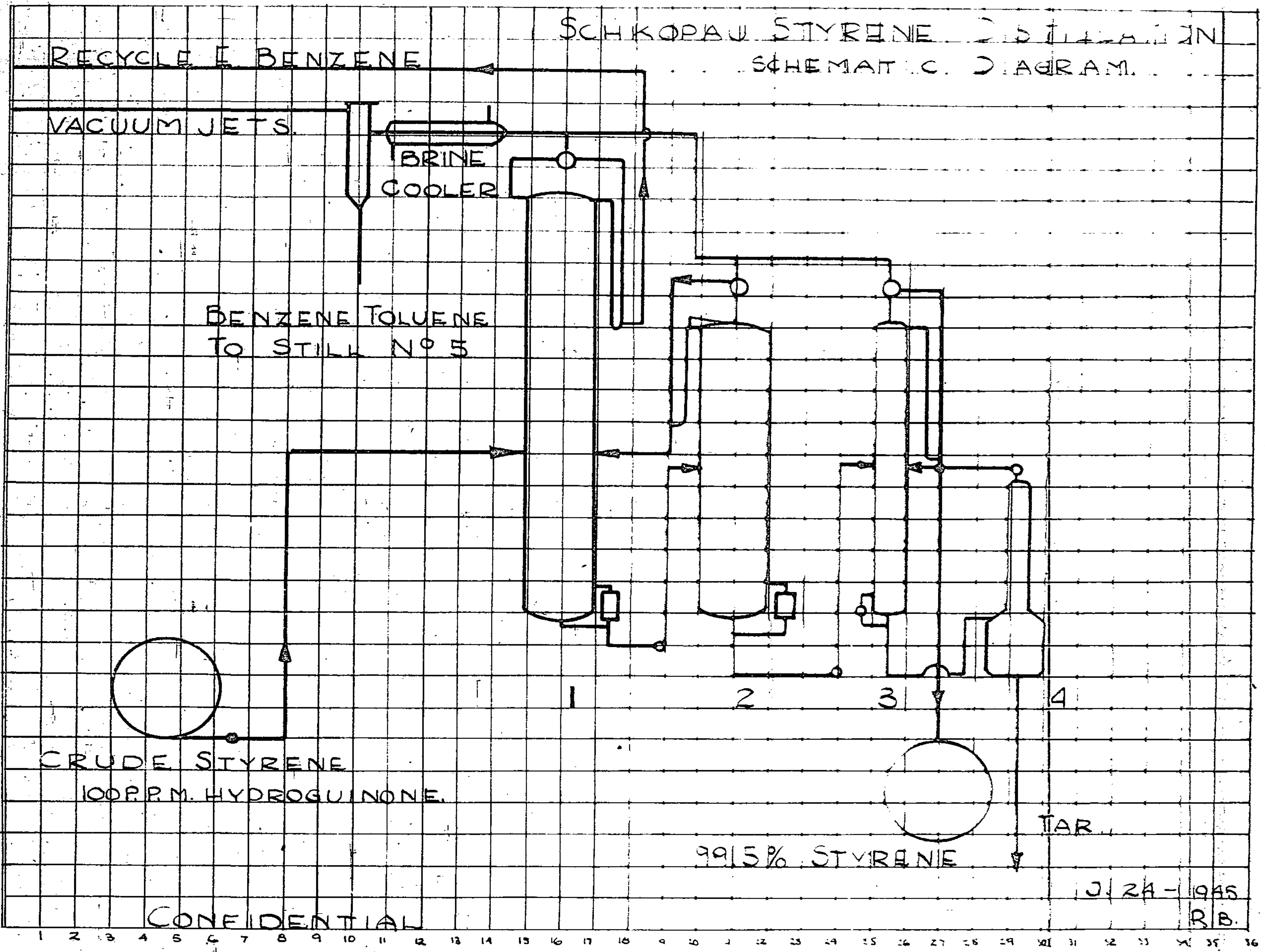
COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

SCHKOPAU STYRENE DISTILLATION
SCHEMATIC DIAGRAM.



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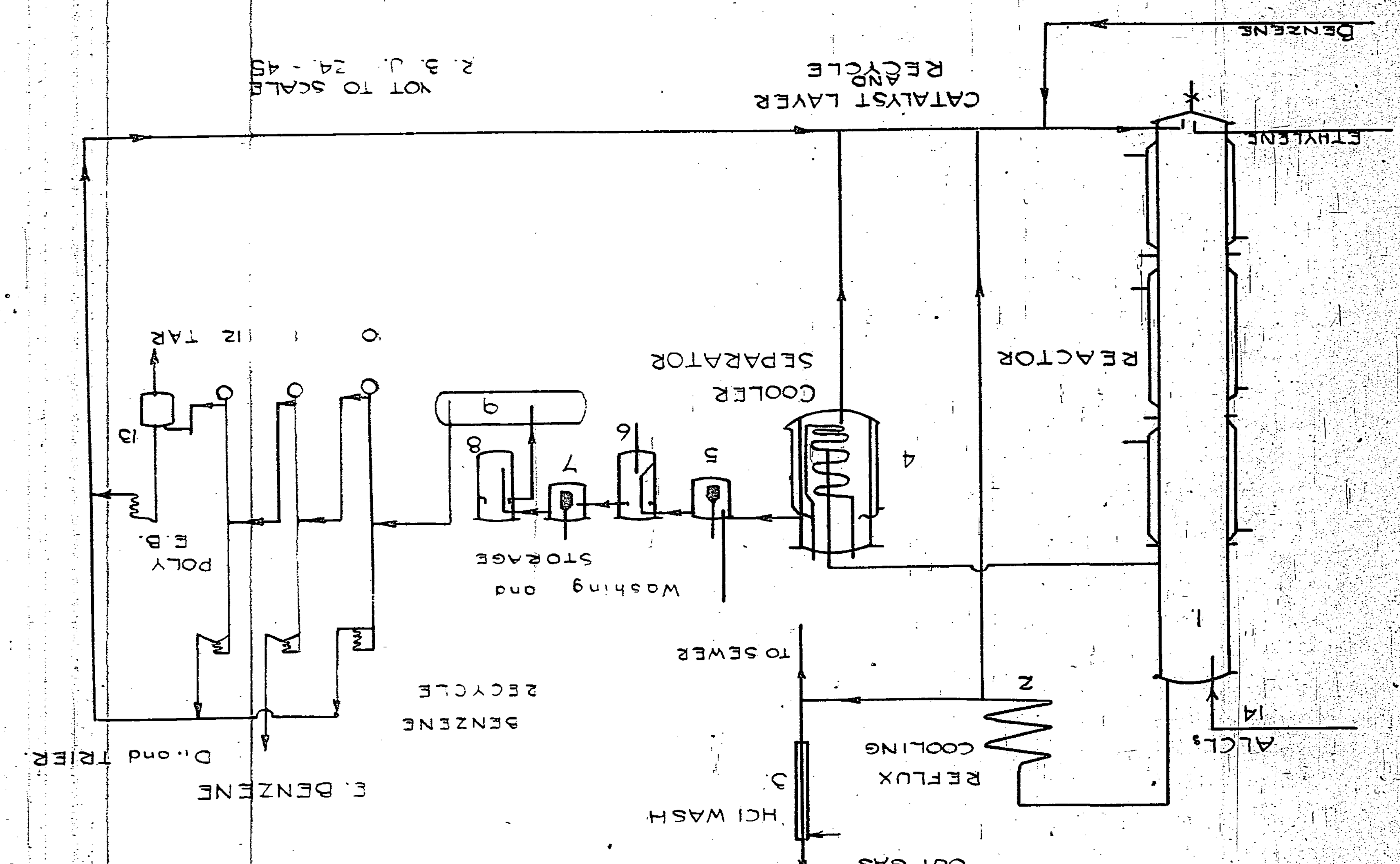
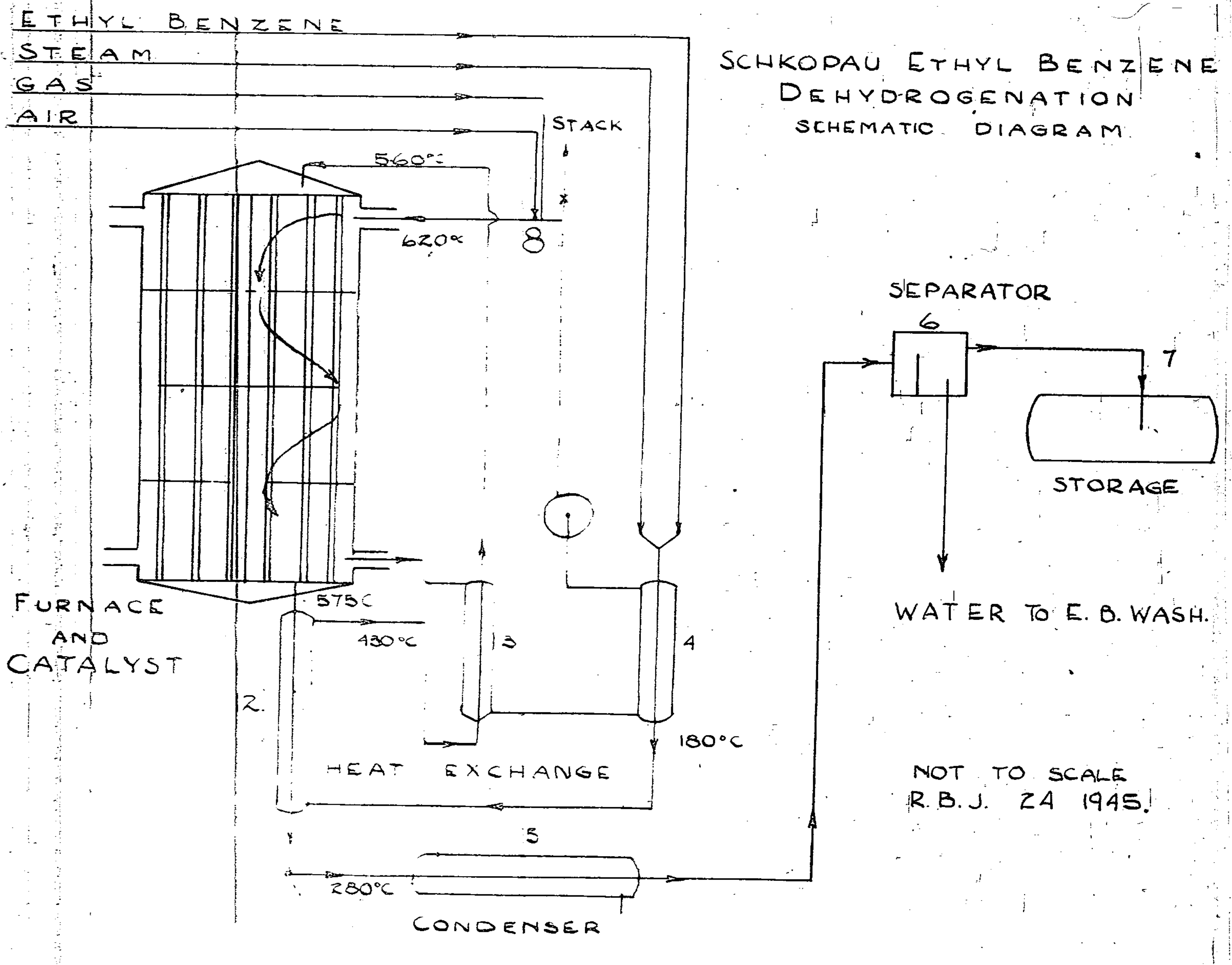


SCHKOPAU ETHYL BENZENE DIAGRAM

SCHKOPAU ETHYL BENZENE
DEHYDROGENATION
SCHEMATIC DIAGRAM



Water and emulsifier are fed into a 1500 gal. pfaudler type kettle made of Remonit (V24 steel) or nickel clad steel. Styrene and finally catalyst are added and the temperature increased to 75° C by means of a water jacket on the reactor. Complete polymerization requires 7 hours at 75° C. One to two percent monomer is then stripped off by introducing steam to the kettle while agitation continues. The emulsion is pumped to a second similar kettle where 1.5% formic acid (based on polystyrene content) is added with stirring to coagulate the polymer. The suspension is filtered on a continuous filter having a drum approximately 1.5' in diameter and a 1' face. The powder is continuously washed with pure water while being conveyed to a second filter similar to the first. From the second filter it is conveyed on a belt to a 30" diameter Aeroform press which compacts it to triangular pieces approximately 3/8" x 3/8" x 1". These particles are dense enough and strong enough to handle on a standard tray drier. The Aeroform press is a polished drum about 30" in diameter with a 2' face with polished cut triangular grooves about 3/8" at the top and 3/8" deep. The roll is heated to about 75° C. The damp power is fed onto the roll and compacted with two polished cold rolls about 10" in diameter. The resulting pellets are hard and dense. This preformer is also used for vinyl chloride polymer, but is not satisfactory as the polymer does not compress as well as polystyrene. Pellets formed by this method can be tray or belt dired in beds 3" or 4" deep with no difficulty. The air used for drying is heated to 75° C with steam coils. The pellets when dry are ground to a fine powder and packaged in 50 pound paper containers for shipment.

Moldings made from the above polystyrene have a heat distortion of about 90° C compared to 75° C for polystyrene (111) and because of the high molecular weight are quite tough and strong. The color, however, is slightly yellow and cloudy.

A 10 pound sample was obtained. Stock on hand is 190 tons ready for shipment.

No stabilizer, plasticizers, colors or other agents are added. Troisdorf is the principal user.

R. H. BOUNDY.

			Plate	Press		Temp		Reflex	Feed
Still	Plates	Diameter	Spacing	Top	Bottom	Top	Bottom	Ratio	Plate
No.1	45	72"	15"	25	175	45	100	4:1	18
No.2	25	60"	15"	15	75	35	85	8:1	15
No.3	20	48"	15"	10	50	35	85	5:1	10
	Also used one rasig rings in 35' x 36" tower.								
No.4	Batch, ring packed, about 20" diameter x 12' high.								
No.5	60	42"	18"	atmos.		100	140	1:12	28

Still No. 2

40% styrene off top feeds back to still No. 1; styrene with 2% tar off bottom feeds to still No. 3.

Still No. 3

99.5% or better styrene off top, tar plus small quantity styrene off bottom. Tar feeds to Still No. 4.

Still No. 4

90-98% styrene off top feeds to still No. 3. Tar off bottom is diluted with residue from the ethyl-benzene distillation and used as a rubber plastraizer.

Still No. 5

The feed is approximately 20% benzene, 30% toluene, 50% ethylbenzene. Benzene toluene overhead goes back to benzol refining. Ethylbenzene off bottom recycles to dehydrogenation.

Stills 1 and 2 are built up with individual plate sections approximately 15% deep. Each section is carefully machined and gasketed. Dr. Wulff stated they had no difficulty with air leakage through the gaskets. This was indicated too by the very small vacuum jets used for the entire distillation train. The lower sections (approx. 5 plates) in all stills except (5) must be dismantled and cleaned of polymer about once a year. They believe this is caused by divinyl benzene and occasionally by air in the system. They sometimes have polymerization in condensers but only infrequently. The tin lining on several of the plate sections we could see was almost entirely gone and rusty iron exposed in many places. Single reboilers were used on all stills. They were extremely small and of the vertical thermal circulation type. Reboilers on Stills land 2 were approximately 30" dia. x 5' high. The bubble caps were of pressed steel construction with the slots out very low to minimize pressure drop. Low pressure steam was used in the reboilers. Benzene and toluene were removed by operating the condensers in stills 1 and 2 fairly warm (about 40° C) and then refrigerating to 0° C the gases from these stills to the vacuum jets. The condensate contained 50% ethylbenzene, 15% benzene and 30% toluene and some styrene. This mixture was then frostinated in column No. 5, and the ethylbenzene recycled to the dehydrogenator. The benzene and toluene were further purified by recycle to the benzoe purification.

4. POLYSTYRENE PLANT

A. **Condition of Plant:** Ready to operate.

B. **Capacity:** 50 metric tons a month emulsion polystyrene (EF) of a very high molecular weight

Chemistry:

D. **Flow Diagram:**

99.5 Styrene

Ion Ex. Water

$K_2S_2O_8$

H_2O_2

Soap

HCOOH

E. **Materials Used:**

Styrene: 99.5 plus pure by refracture index. Less than .001 Divinyl benzene. Less than .001 methyl styrene. Mfg. at Schkopau.

Water : Purified by double ion exchange using phenol formaldehyde and aniline formaldehyde resins from Wolfen.

Potassium persulfate: Technical.

Hydrogen peroxide: 40% solution in carboys.

Soap: Sodium salt of a synthetic fatty acid made by the oxidation of straight chain paraffins at Oppau or Deutsche Fattsaure at Witten. Acid chain length C 11 to C 15. Paraffin is a by-product of the Fisher-Tropsch reaction.

Formic Acid: Technical.

F. **DESCRIPTION OF PROCESS:**

Two types of very high molecular weight polystyrene are made at Schkopau, the only difference being in the catalyst. Hydrogen peroxide is used where an electrical grade polymer is desired. It is easily washed out of the polymer. Potassium persulfate is more active and is used where electrical properties are not important. The polymerization cycle is about two hours less when potassium persulfate is used.

The polymerization formula is as follows:

50	parts pure water.
50	parts styrene.
.1%	$K_2S_2O_8$ (based on styrene)
1.0%	Soap (based on styrene)

The large quantity of spare fittings, pipe, equipment, etc. indicated the customary corrosion difficulties encountered in such a process. The elimination of all dealkylation (except in the reactor) simplified the operation and construction of the plant greatly. This is especially true in the elimination of many storage tanks of intermediates. Still No. 11 can be operated to produce pure diethyl benzene which was sold under the trade name "Kybol" and used for high octane gas. The plant is well engineered and modern in every respect though not nearly so highly instrumented as similar plants in the U.S.A.

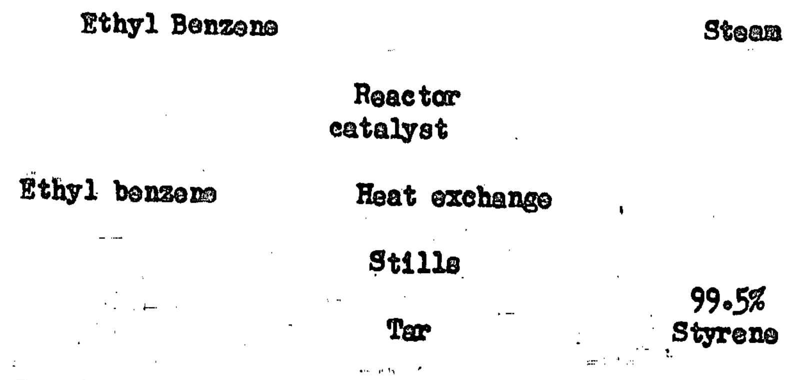
3. ETHYL BENZENE DEHYDROGENATION.

A. **Condition of Plant:** Ready for operation.

B. **Capacity:** 1600 M. ton/mo.

C. **Chemistry:**

D. **Flow Diagram:**



E. **Raw Materials:**

Ethyl benzene: Boiling range .5° C to dry point. Must be free of chlorides, diethyl benzene, toluene, and sulphur.

a. Steam. Low pressure steam.

b. Catalyst:

Zn 0	80%)	
$Al_2 03$	10%)	Wet mixed extruded and cut
Ca 0	5- 7%)	in approx. .25" x .25"
KOH	2- 3)	particles.
$Cr_2 O_3$	.5 to .7)	

F. **Process:**

Steam and ethyl benzene are mixed in ratio 1:1 and preheated by heat exchange to approximately 560° C and passed through the catalyst heated by combustion gases to 560°-610° C. Conversion per pass is about 40%. The composition of the reaction mixture is about:

40%	Styrene
1.0%	Toluene
.5%	Benzene
.3%	Tar
58%	E. Benzene

The reaction mixture after heat exchange is at 280° C, with copper alloy tubes. It is then condensed in a heat exchanger. The vest gases are then cooled to 2° C and passed through several small ring packed towers to remove all possible crude styrene. The out gases are finally piped to the power house where they are burned for fuel. 50 cu. m. are produced per 100 kg styrene produced. Approximate analysis is 85-90% H_2, 5-10% CO_2, 1-$2_1$0, C_2H_4, 1-2% CH_4 and a trace of CO. The mixture is separated from the water with gravity separators and settled in a 2,000 cu. ft. tank. After drying with caustics, the styrene mixture goes to storage in iron tanks and from there to the stills. The following cuts are made.

1. Benzene toluene.
2. E.B. with less than 2% styrene.
3. Styrene with less than .5% E.B.
4. Heavy tar for rubber plas.

After the condensation and separation (100 ppm based on styrene) of hydroquinone inhibitor is added. Maximum temperature in any still is 100° C. 99.5 styrene is produced with an overall yield of 90%.

G. **Equipment:**

a. **Furnaces:** 15 total - 22 catalyst tubes 8" diameter x 8' long in each. Vertical tube furnaces. Combustion gas heated. Iron heat exchange on incoming reactants with outgoing reactants and combustion gases. Tubes are 18-8 Cr-Ni-Fe alloy about 8' long and 8" inside diameter. Different shaped tubes have been used but round proved as good as any. The tubes and all parts of the furnace exposed to reactants are lined with Cu 3% Mn alloy to prevent carbon formation. The cover sheets, bottom, sides, etc. of the reactor are constructed of NCTS Chronium Nickel steel. The tubes are of FF6N steel. Heat exchange tubes and shell are of Sicronal 7. Admiralty metal is used for the condenser tubes. Total catalyst content perfurnace is about 60 cu. ft.

b. **Distillation:**
Storage in aluminum.
Distillation in tin lined stills.
Condensation in tin lined condensers.
Transfer in iron pipes.
Two distillation trains. See diagram for schematic sketch of one train.

Still No. 1
Ethylbenzene with less than 2% styrene off top. 80% styrene off bottom. Bottoms feed to still No. 2.

The ethylene and benzene react in an enameled-steel tower in the presence of Al Cl_3 catalyst. Reactor composition is:

45-50%	Benzene
35%	E. Benzene
15-17%	Higher alkyl benzenes

Reactor temperature is 90° C, controlled by reflux. The reaction mixture overflows from the reactor, is cooled to 50° C in a combined separator and cooler. The heavy catalyst layer recycles to the reactor and the top ethyl benzene layer is washed, first with water and finally with a 20% solution of sodium hydroxide. After drying with solid caustic the top layer is pumped to a 200 cu. m. iron storage tank which acts as an inventory between the reaction and distillation steps. The mixture is pumped to a series of continuous stills (see diagram).

No. 10 benzene recycle overhead and remainder bottoms.
No. 11 E.B. overhead and remainder bottoms.
No. 12 di and tri ethyl benzenes overhead and remainder bottoms; di and tri to recycle in reactor.
No. 13 Vacuum still - higher alkyl benzene overhead and tars to use for rubber plasticizer. Higher alkyl benzene to recycle in reactor.

Yield: On benzene: 95-96%
On ethylene: 92-94%
Ratio E.B. to Al Cl: Approx. 35 to 1.

The ethyl benzene must be pure. Di-ethyl benzene and toluene must be less than .002%. Boiling range .5° C to dry point.

G. Equipment: (See diagram)

a. The incoming benzene from the distillation of coal is refined before using. All equipment for this step is iron. The washing is done continuously in 30 cu. ft. stirred Pfaudler type kettles.

1. 96% H_2SO_4 wash.
2. Separator (approx. 100 cu. ft.).
3. Wash with 5% caustic.
4. Separator (approx. 100 cu. ft.).

Residual carbon disulphide and toluene are removed in two 30-plate x approx. 40" diameter stills. The highly refined benzene is then pumped to iron storage tanks.

b. Reactors. Total of four, three in use, one spare. The enameled steel reactor is 45" in diameter by 40' high. It is built in four sections, flanged and bolted. The first three sections from the bottom are jacketed for heating or cooling, while the top section is unjacketed. They experienced some difficulty with the gasketed sections leaking and also with the enamel chipping. The ethylene and benzene are bubbled into the reaction mixture at the bottom of the reactor with no effort to get good distribution. Both inlet pipes are turned down to keep the Al Cl_3 from settling to the bottom of the reactor. All parts in contact with the hot reaction mixture are either glass enameled or coated with a baked-on phenolic enamel. The reactor operates about 80% full. No separate dealkylators are necessary, as the polyalkyl benzenes are recycled to the reactor where an equilibrium is established. No H Cl is necessary since the ethylene gas is slightly wet. Dr. Wulff stated that at times they have had the ingredients so dry that the reaction ceased. They had no information on the Al Cl_3 dissolved in the E.B. layer but believed it to be extremely small. Iron equipment is used as soon as the reaction mixture is cooled to 40° C. Plug cocks are used throughout the plant. Pumps are centrifugal. Cooling is accomplished by reflux.

c. Separators and Coolers (4). Four in plant, three is use. They are approximately 6' in diameter x 8' high, jacketed for cooling and also cooled by internal heat exchange surface. The complete units are phenolic resin lined. Each unit contains approximately 150' of 1.5 inch cooling pipe.

Washers and Separators (5,6,7, 8 in diagram). The water wash and separation equipment is glass-lined while the caustic wash and subsequent separation is carried out in iron. Otherwise the units are duplicates. The washers are 3 0 cu. ft. Pfaudler type stirred kettles. Separation is done in 100 cu. ft. cylindrical vessels. The recovered condensate from the ethyl benzene dehydration plant is used for part of the wash water.

d. Stills (See diagram)

No. 10 Benzene off top. 1.6 meters dia. 50 plates.
No. 11 E.B. off top. " " " " "
No. 12 D.E. and Tri. E. benzene off top. Ring packed 25' high x 4' dia. (approx.). Operates at 100 mm. pressure.
No. 13 Poly E. benzene off top. Tar off bottom. Ring packed 25' high x 3' dia. Operates at 20 to 30 mm. pressure.

Reflux ratio in all cases 1:1 to 3:1 depending on conditions. The tar residue is used as a softener for Buna S. The process is continuous throughout. Standard steel construction is used except for the reactor and equipment in contact with the reaction mixture at temperature above 40° C. All steps except distillation are completely housed. Emergency outlets on the bottom of each reactor drain into storage tanks on the ground floor. Al Cl_3 is fed into the reactors either by a screw conveyor mechanism or merely by pouring about 10 pounds at a time into the reactor thru a double valved six inch pipe. The latter method was preferred as the conveyor had given much trouble.

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MANUFACTURE OF STYRENE AND POLYSTRENE AT I. G. FARBENINDUSTRIE, SCHKOPAU

1. INTRODUCTION

The styrene and polystyrene plants at I. G. Farbenindustrie, Schkopau, were visited May 26, May 30 and June 1, 1945 to obtain information on the above processes. Dr. Wulff, Dr. Moll, Dr. Klein and Dr. Weinbrenner were interviewed.

2. ETHYL BENZENE

A. Condition of Plant: Ready for operation.

B. Capacity: 2700 M. ton/mo. 80 ton/mo. rubber plas.

C. Chemistry:

H H
C - C
H H

al cls
cat

H H
C - CH
H H

D. Flow Diagram:

Benzene
Ethylene
Aluminum chloride
Reactor
Catalyst recycle
Diethyl and polyethyl benzene recycle
Separator
Washer
Ethyl benzene
Stills
Tar

E. Raw materials:

Benzene: Refined benzene from coal distillation.

Ethylene: 98.0% ethylene from hydrogenation of acetylene.

Al Cl_3: Powdered. Less than 0.1% Fe. Stored in tight iron drums. Manufactured from bauxite and phosgene in Schkopau.

F. Process: Refined benzene is further purified by washing with concentrated H_2SO_4 and distilling to remove CS_2 and toluene. Final benzene has boiling range .3-.5° C to dry. M.P. 5.1° C or higher. Benzene is dried in final distillation (azeotropic). Iron equipment is used for this stage. A caustic neutralization wash is used before distillation.

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REPORT ON
MANUFACTURE OF STYRENE AND POLYSTYRENE AT I. G. FARBENINDUSTRIE, SCHKOPAU

Reported by:

R. H. BOUNDY, U.S.
R. L. HASCHE, U.S.

on behalf of

U.S. Technical Industrial Intelligence Committee

CIOS Target No. 22/82
Miscellaneous Chemicals

1 June 1945

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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14 p. diagrs.

No. 198

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STYRENE

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Miscellaneous Chemicals

1 June 1945

[illegible]

ITEM No. 22
FILE No. XXVII—6

Classification Cancelled,
by authority of
The Joint Chiefs of Staff,
by Col. E. W. Gruhn.

~~COPY No.~~ 198

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MANUFACTURE OF STYRENE AND POLYSTYRENE
I.G.FARBENINDUSTRIE, SCHKOPAU

Boundy + Hasche

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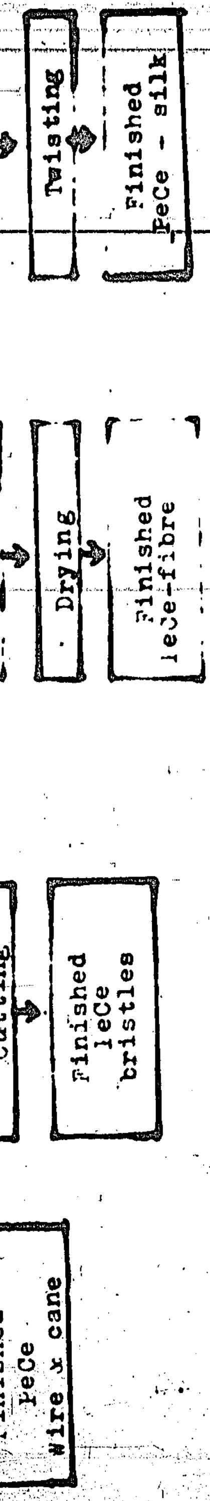
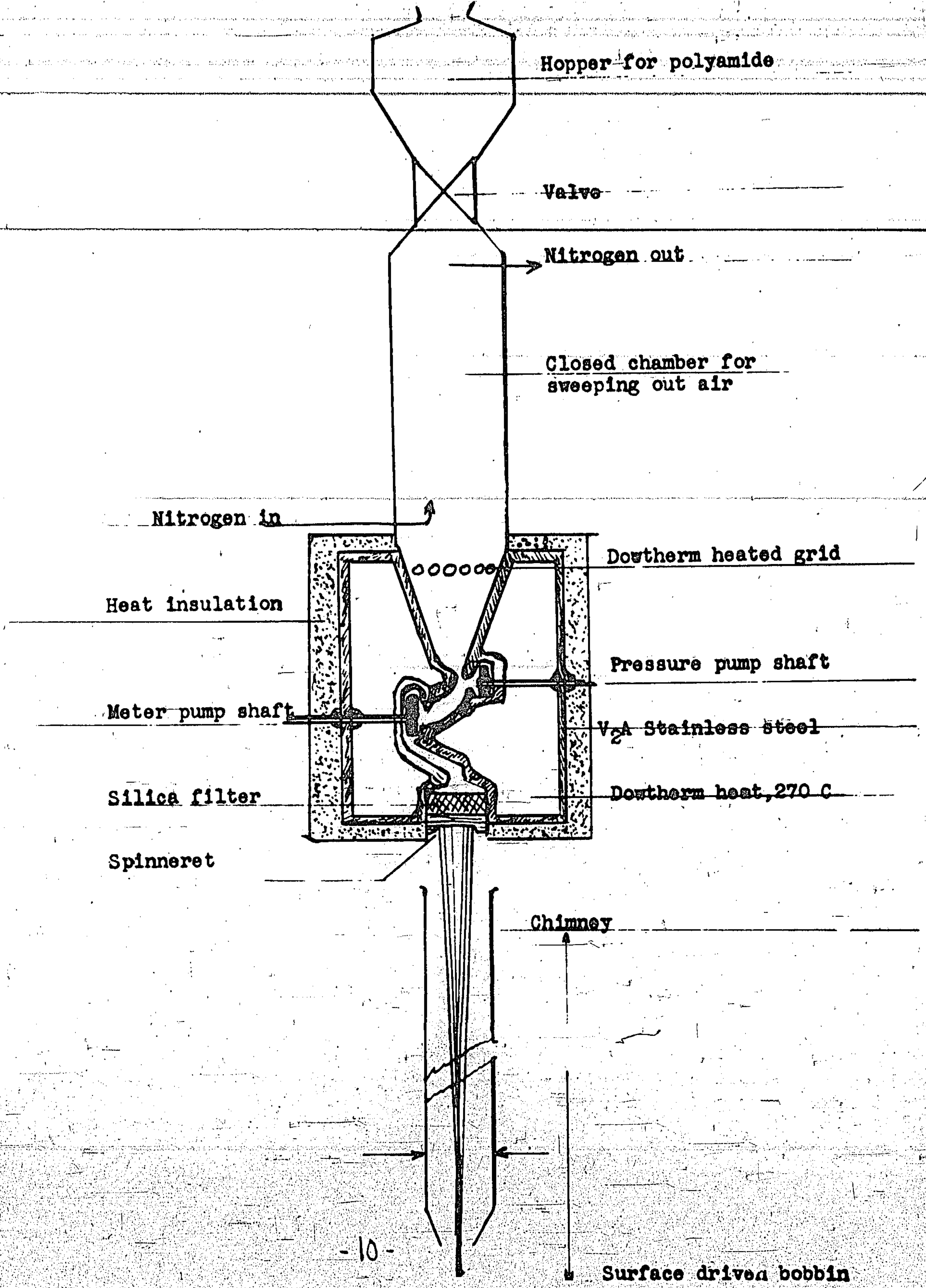
COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE



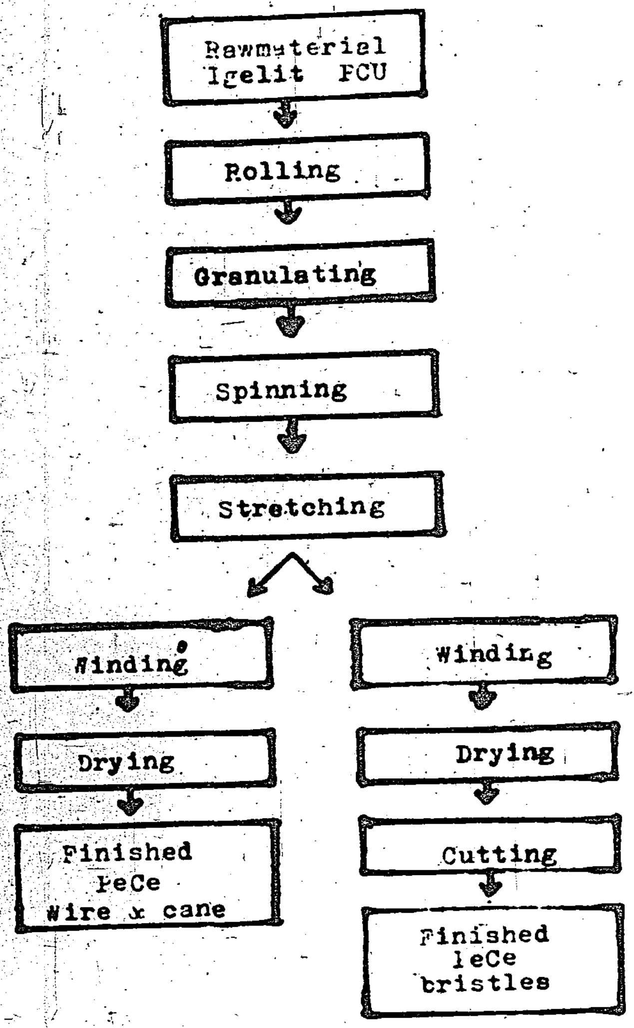
SKETCH OF GRID SPINNING UNIT

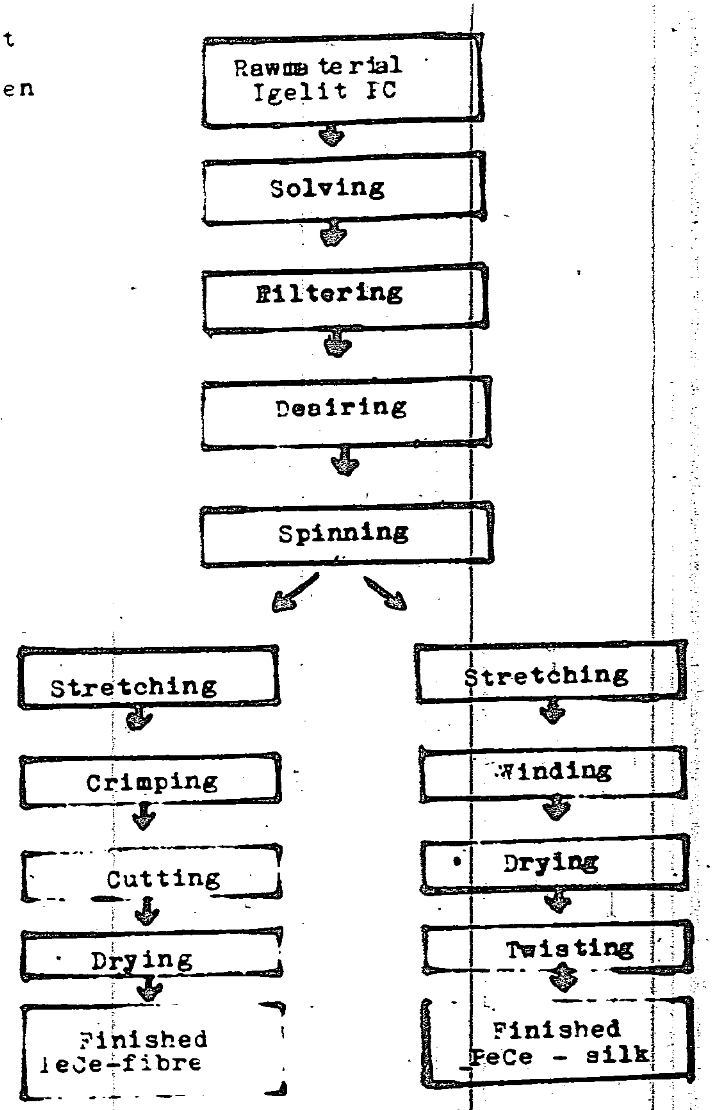


Flow-sheet

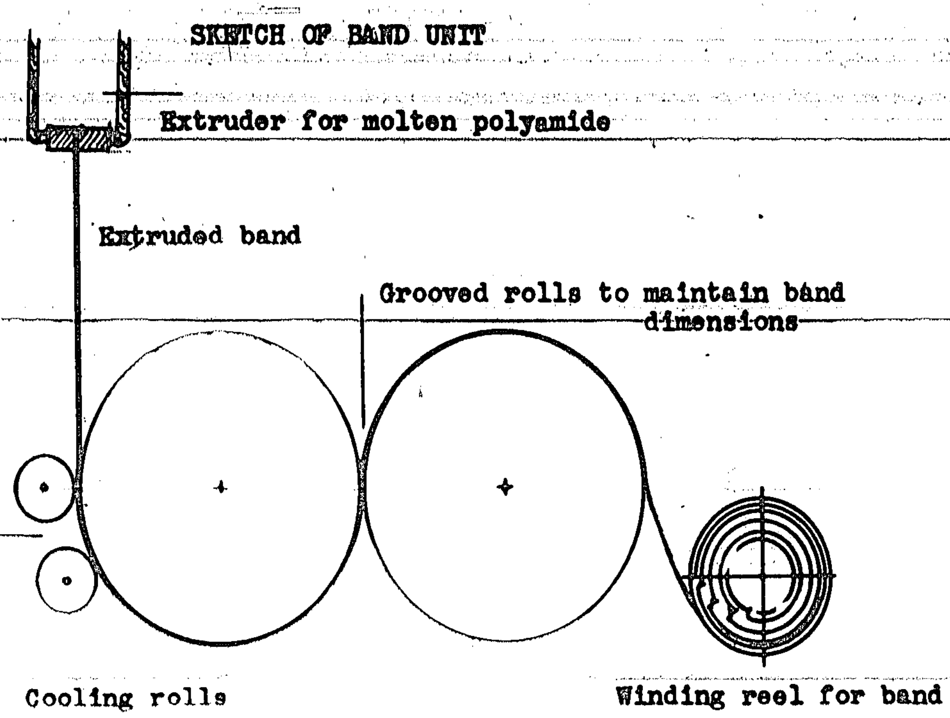
PeCe-Plant

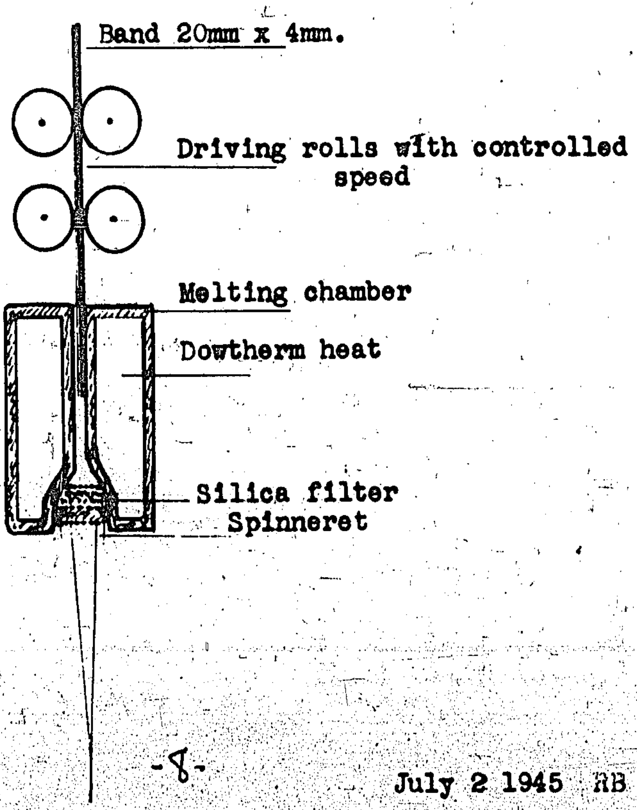
I.G. Wolfen





SKETCH OF BAND UNIT





polyamides, cellulose acetate-butyrate, and 56% acetyl cellulose. Each unit has a 100' copper or silvered copper mat finished band turning around 6' drums. Linear speed of the band is 480' to 650' an hour. Width of the film as cast is 48". The trimmed film is 45" wide. Total capacity is 325,000 sq.yds. a month but varies with the thickness of the sheet. Sheet up to .2 mm. can be cast but most of the stock appeared to be approx. .05 mm. Much of the cast polyamide sheet was reported to be used for gas proof capes.

Four grades of cast Lyafol were manufactured:

6A. 60% polyamid 6-6, 40% caprolactam
5A. 50% " "
6A. With a small quantity of isododecylphenol as a plasticizer
5A. With a little isododecylphenol as a plasticizer.

A representative casting formula is:

130	pounds	6-6
55	"	Caprolactam
300	liters	70-30 C_2H_5OH water solution.

The above composition is made up in a 1200 gal. tin lined copper jacketed agitated kettle and fed to two 600 gal. kettles of similar construction. From them it is metered onto the belt of the casting units.

5. CAPROLACTAM SPINNING AT WOLFEN

Caprolactam has a higher moisture absorption than Nylon 6-6 and for this reason is preferred for many textile applications. It was reported to have 20% elongation, 6 gms. per denier tensile strength, a water absorption of 14% and a melting point of 220° C.

Caprolactam is spun with 10 to 15% residual monomer to facilitate fabrication and stretching. The monomer is subsequently washed out with water at 80° C.

Two methods of spinning are used (see sketch). The conventional "grid" method and a newer "Band" method.

In "band" spinning a continuous tape .80" wide by .15" is used as feed for the spinneret. The band is formed by continuous hot extrusion and molded to accurate dimensions by grooves in two large wheels and smaller rollers which form the polyamide into the groove (see sketch). The band is then reeled and used as feed for the spinnerets. The band is fed by constant speed roller through a close fitting

opening and into the heating section which is rectangular. The fit of the band in the opening is so accurate that it acts as its own constant speed extrusion room for the melted material in the hot zone. At the end of the heated zone is a filter and spinneret (see sketch).

From the spinneret the extruded filaments are cooled and stretched 400% on differential speed rolls. Speed from the spinnerets is 1850' per minute. Both the grid and the band method extrude 40 g. per minute.

The grid spinning method was reported the same as used by Dupont before the war. With it coarse feed about 1/4"x1/2" is fed through a hopper into a vertical cylinder and thence to the melting chamber. Air is displaced from the vertical cylinder with nitrogen fed counter current to the igimid. The melting chamber consists of a jacketed hollow tubular grid which is heated to 270° C with Diphenyl. The molten polymer feeds to a stainless steel metering pump and then through a silian filter to the spinneret. The standard spinneret is V2A stainless steel 1.5" in diameter by 3/16" thick with 12 holes .30 mm. in diameter. Speed at the spinnerets is 2250' per minute. The 12 strand filament is approximately 160 denier at the spinneret and is reduced to 40 by 400% elongation (see sketch).

R. H. Boundy

E. Considerable development work had been carried out at Wolfen on the fabrication and use of Polyvinylidene chloride. The 85-15% and 85-20% vinylidene chloride vinyl chloride copolymers, made at Ludwigshaven, were used. They were given the tradename "Durit." Chief difficulties encountered were due to the lack of nickel or nickel alloys for the manufacture of fabricating equipment. They experienced much difficulty with decomposition of the plastic. No plasticizers, or stabilizers were added to the copolymers. ~~Fabricated bristles had no characteristic plasticizer odor.~~ Samples of the bristles were obtained.

3. IGELIT PC (CHLORINATED POLYVINYL CHLORIDE) - FINE FIBER PLANT.

A. Capacity : 5 metric tons a day cut staple fiber.
.6 metric tons a day continuous filament.

B. Raw Materials:

a. Igelit P C (Chlorinated Polyvinyl Chloride):
Manufactured at Bitterfeld and shipped to Wolfen in 50 pound paper bags.
Chlorine content is 62-64%.
Softening point is 100° C.
The igelit is a white powder. Particles vary from a few microns to one mm. in diameter.

b. Acetone: 99.8 pure and free of water.

c. Water: Purified by double ion exchange.

C. Steps in the Process:

a. Cut staple fiber : Dissolving, filtering, deairing, spinning, stretching, crimping, cutting, drying and packaging.

b. Continuous filaments: Dissolving, filtering, deairing, spinning, stretching, winding, drying, twisting, and packaging.

D. Description of Process:

No iron can be used; all equipment is either lead lined, bronze, or glass. The Igelit PC is dissolved (28%) in acetone in lead lined, agitated (250 rpm) 1500 gal. pfaudler type kettles. .5% (based on Igelit) of dioxydiethyl sulphide $(HO\text{-}C_2H_5)_2S$ is added to the solution as a stabilizer. The mixture is stirred for several hours and heated to 40° C with warm water in the kettle jacket.

After solution is complete the material is filtered once through cotton and once through linen. It is now deairated by standing warm (40° C) for 24 to 26 hours. After cooling to 20° C it is ready for spinning. At this stage the solution is a very viscous, slightly yellow material resembling honey in its physical appearance.

Two methods are used for spinning, one for continuous filaments and one for cut staple fiber.

Normal production of the endless Igelit yarn is .6 tons per day of 300 denier yarn, 120 filaments. From 90 to 1000 denier can be made. The solution is metered with small bronze gear pumps through the spinneret vertically into a parallel current of water at 16° C. The spinning rate is 60-65 meters per minute. Acetone is extracted from the spinning solution as the filaments are carried vertically with the water. Each spinneret and water bath is individual. A glass tube about 4" in diameter and 8' high makes up one unit. The filaments as they come from the water bath still contain about 7% acetone. The water as it overflows at the top of the glass tube contains 13% acetone. This mixture is distilled and the acetone recovered for recycle. The filaments are drawn from the water bath onto a set of differential speed pulleys directly at the top of the glass tube and stretched 300% in several stages. An anti-static and wetting agent treatment of (Tallosan) sulfonated fat was applied and the filament wound onto bobbins. Drying is accomplished with air at 50° C. Three spinning machines each having approximately 100 spinnerets were used for the total production.

The cut staple fiber was spun much simpler equipment and on a larger scale. Each spinneret extruded multi-filaments into a small horizontal trough with water at 16° C flowing parallel with the fibers. The trough was 12" wide x 8" deep x 10' long (approx.). Seven such troughs combine into a larger one and the washing of fibers from all seven spinnerets becomes counter-current. After washing the combined output of fibers is stretched 300% on large differential rolls in three stages. It was then crimped and cut wet and finally dried in a Procter Schwartz tunnel dryer at 45-75° C. The finished fibers have a strength of 1.8 to 2.2 gms. per denier and an elongation of 40% at break. Principal uses are for filter cloth, rope, tents, clothing, etc.

4. FILM PRODUCTION AT WOLFEN

Three large band casting units were used at Wolfen for the production of various types of film including

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REPORT ON FABRICATION OF PLASTICS AT I.G. FARBENINDUSTRIE, WOLFEN

1. INTRODUCTION

The I. G. Farbenindustrie at Wolfen was visited May 25 and 26, 1945 for the purpose of obtaining information on the methods used at Wolfen to fabricate plastics. The following men were interviewed:

Dr. Gajewsky
Dr. Rein
Dr. Esselman
Dr. Miller
Dr. Oppelt
Dr. Sedlach

The chief products were polyvinyl chloride monofilament and fine fiber, Nylon film and fibers, and cellulose acetate and mixed ester films. The main types of Nylon like products are Perlon L (Omega Caprolactam) Perlon T (adipic acid and hexamethylenediamine) and polyurethane. Caprolactam production is concentrated primarily at Landsberg where 7 machines with 48 spinnerets each produce 10,000 pounds a day. Lichtenberg has a capacity of 60 tons of bristles a month and 7,000 pounds a month of caprolactam silk.

The raw materials are supplied by:

Leuna	Diamines, diacids, caprolactam.
Ludwigshaven	Adipic acid, hexamethylenediamine.
Leverkusen	Isocyanates.

Polymers are shipped from Landsberg, Lichtenberg, and Wolfen.

Experimental spinning is done at Wolfen, Lichtenberg, Ludwigshaven, and Leverkusen.

Almost all the polyvinyl chloride (Igetit PC and PCU) bristles and fine fiber is manufactured at Wolfen.

Small quantities (up to 20 tons a month) of polyvinylidene chloride (durit) bristles and monofilaments were fabricated at Wolfen.

Many types of film were produced, both commercially and experimentally.

2. POLYVINYL CHLORIDE (PCU) MONOFIL PLANT

A. Capacity: 40 metric tons per month in condition for immediate operation.

B. Raw Materials: Unplasticized polyvinyl chloride, 56% chlorine, 1.38 density, finely divided (1. to 3. micron) white powder shipped in 50 pound paper bags from Bitterfeld or Schkopau. No stabilizers are added at Wolfen.

C. Steps in the Process: Rolling, Granulating, Extruding, stretching, winding, drying, cutting and packaging.

D. Description of the Process:

The powder is first processed and sheeted on a standard two roll mill (approx. 18"x48") at 120-150 ° C. After cooling it is ground to pea size in a "Condux" mill. The above steps are necessary to make a satisfactory feed for the small extruders. The extrusion is done with a battery of 24 small vertical machines, all duplicates. Each produces 100 to 150 pounds a day. One man operates six extruders. The screws are 1.2" in diameter x 18" long of stainless steel. The conversion ratio is 3.5 to 1. The cylinder is jacketed in two parts. The hopper end is heated to 120° C and the die end to 160° C. The melted plastic is extruded through a 16 to 40 hole die. The holes are .25 to 1.0 mm in diameter. The extruded monofils are cooled to 50° C in a water bath and immediately stretched 200 to 300%. The threads are cooled and stretched as a bundle, not individually. The stretching is accomplished continuously on differential speed drums set to stretch 200 to 300% as desired. The stretching is under water directly under the extruder. They are continuously wound on a rotating drum, dried for a short time at 60° C and cut across the drum to 24" lengths. They are packed in 2.2 pound bundles and packed in 200 to 250 pound boxes for shipment. They are used for brooms, brushes, etc. continuous cane, rattan, and monofil are also produced by merely omitting the cutting step and winding onto small reels. A total of 35 men operate this plant.

The strength was reported as 10-12 kg per sq. mm. Softening point is 120° C and recommended for service up to 60° C.

Two large (4 ") extruders were also housed in the same building. Their output is 600-650 pounds a day.

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REPORT ON
FABRICATION OF PLASTICS AT
I.G. FARBENINDUSTRIE, WOLFEN

Reported by:

R. H. Boundy, U.S.
R. L. Hasche, U.S.

on behalf of the

U.S. TECHNICAL INDUSTRIAL INTELLIGENCE COMMITTEE.

C.I.O.S. Target No. C22/1(j)
Miscellaneous Chemicals.

May 25, 1945.

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

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10 p. diagrs.

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ITEM No. 22
FILE No. XXVII-16

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The Joint Chiefs of Staff,
by Col. E. W. Gruhn.

COPY NO. 187

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FABRICATION OF PLASTICS
I.G. FARBENINDUSTRIE, WOLFEN

Boundy + Hasche

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

35% Vinyl chloride
3% Mersolate (based on vinyl chloride)
.5% Hydrogen peroxide or)based on vinyl chlo-
.1 to .2% Sodium persulfate)ride
Balance water.

Three streams are metered into the top of the reactor:

1. The mersolate and water which were previously mixed.
2. The vinyl chloride.
3. ~~The catalyst dissolved in a small quantity of water.~~

Emulsification takes place in the first or upper few feet of the reactor. The temperature is accurately controlled at 45° C from top to bottom of the reactor by means of a water jacket on the reactor. Reaction time is 3.5 hours with sodium persulfate and 5 hours with hydrogen peroxide. From the bottom of the reactor the emulsion now 85% polymerized is pumped to a helical spiral vacuum evaporator where the excess monomer is flashed off condensed and recycled to the polymerizers. The evaporator is about fifteen feet high and eight feet in diameter, jacketed and heat insulated. Exposed parts are of nickel. It is operated at 100 mm. pressure. As the emulsion flows slowly down the spiral path all excess monomer is evaporated without breaking the emulsion. The spiral trough is about 18-20" wide and 3-6" deep and makes one spiral in 12 to 18 inches. One evaporator has capacity for all production to the spray dryer.

The emulsion free of monomer is now either spray dried as at Bitterfeld (see drawings of unit) or coagulated and dried by conventional methods. When the coagulation method is used $Al_2(SO_4)_3$ is added in a glass lined kettle, the suspension settled, decanted and filtered on a continuous (oliver type) filter approximately 6' in diameter with a 3' face. The powder is washed on a belt as it passes to a second similar filter. Soda (.2 to .5%) is added at this stage as a stabilizer. The damp powder is then compacted on an aeroform continuous press. The resulting pellets are dried at 80° C on a link screen belt, ground to a fine powder and packaged in paper bags (50 pounds) for shipment. .5% alphaphenyl indole or .5% diphenyl urea are occasionally used for stabilizers.

R. H. Boundy

F. Description of Process

The incoming acetylene is thoroughly dried with KOH and the hydrochloric acid with concentrated H_2SO_4. It was emphasized that the reactants must be free of moisture and that catalyst life was dependent on this feature. The reactor is constructed entirely of iron. The catalyst reaction vessel is approximately ten feet high and six feet in diameter. It is mounted vertically and is filled with catalyst tubes each ten feet long by two and one-half inches inside diameter. Each furnace contains an estimated 100 to ~~150 tubes. Eighteen such units are required for the given~~ production of 3500 to 4000 tons per month. The temperature is accurately controlled by circulating cooling water around the catalysts tubes. The operating temperature with fresh catalyst is 120° C. This is gradually increased to as high as 200° C. over a period of six months to one year The catalyst is then discarded. It cannot be reactivated but the mercury is recovered. When a new furnace is put into operation all air is replaced with nitrogen, the furnace is brought up to temperature and the acetylene and hydrochloric acid introduced. The reaction is highly exothermic and good cooling is necessary. In past practice oil was circulated as the cooling medium. This was changed to water partially for simplicity and safety and partially for better heat transfer. It is common practice in Germany to use water for heating or cooling even at temperatures as high as 250° to 300° C. It is extremely simple to remove the heat from the water by flashing off steam.

HCl is always carried in 10% excess over the acetylene. The reaction is at atmospheric pressure. Pressure drop through the catalyst is less than one pound. The acetylene in the reaction mixture is approximately 10%. Excess HCl is removed with water and the vinyl chloride cooled and condensed by a spray of 25% $CaCl_2$ solution at -40° C. Distillation is carried out in two 20' high by 36" diameter ring pack columns. The first still takes vinyl chloride plus acetylene over the top and residual high boiling chlorinated compounds out the bottom. (Mostly unsymmetrical ethylene dichloride.) The second takes acetylene overhead and very pure vinylchloride off the bottom. Yield on acetylene 96-98%, on HCl 80-90%. The vinyl chloride is stored in iron tanks underground at -40 to -50° C. No inhibitor is added. When asked why it was necessary to store so cool when there was no danger of polymerization, they stated that they did not think it was necessary. The vinyl chloride is shipped in 60,000 pound iron tank cars without inhibitor. They are not insulated but do have a wooden housing over them to protect them from the sun. Nitrogen is always used as an inert gas over the vinyl chloride.

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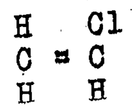
3. POLYVINYL CHLORIDE (PCU)

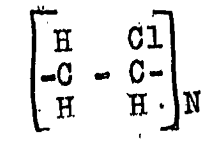
A. **Condition of Plant:** Can be operated at 350 metric tons per month in three months.

B. **Inventory:** 370 metric tons now on hand ready for shipment.

C. **Chemistry:**



$$\begin{matrix} H & & Cl \\ C & = & C \\ H & & H \end{matrix}$$



$$\left[\begin{matrix} & H & & Cl & \\ - & C & - & C & - \\ & H & & H & \end{matrix}\right]_N$$

N = 700 appx.

D. **Chemicals:**

a. Vinyl chloride: Pure vinyl chloride made at Schkopau containing no known impurities. Must be free of acetylene and higher chlorinated compounds.

b. Mersolate: The sodium salt of the product formed by reacting SO_2 and Cl_2 with a long chain (C 15 to C 20) hydrocarbon made at Leuna.

c. Sodium persulfate: Technical.

d. Hydrogen peroxide: 40% in water, shipped and stored in carboys.

E. **Description of Process**

Two types of polymer are made. One for electrical applications is polymerized with hydrogen peroxide catalyst. The second is polymerized with $Na_2S_2O_8$ catalyst and is used for all other purposes. Polymerization time is shorter when $Na_2S_2O_8$ is used and for this reason is preferred when electrical properties are not important. Two types of drying the polymer are used at Schkopau. First spray drying as described in the Bitterfeld report and second coagulating, filtering, washing, compressing and finally drying with hot air. 200 tons per month can be produced by the second method.

The polymer process is continuous in contrast to the batch units at Bitterfeld. Dr. Wulff claimed a more uniform product, a higher molecular weight and a simpler plant as advantages of the continuous process over batch. Eight polymerizing units are installed and produce 350 tons a month but by repairing some of the bomb damage these units can produce 600 tons per month. A total capacity of 1000 tons per month was planned with 12 units but bombing destroyed some of the equipment before production was started.

Each reactor is a jacketed, vertical glass lined cylinder 25 feet high by 5 feet in diameter with a paddle agitator in the top seven feet. Ratio of reactants are as follows:

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PERSONNEL OF INSPECTION TEAM

R. L. Hasche	TIIC, CIOS
R. H. Boundy	TIIC, CIOS

MANUFACTURE OF VINYL CHLORIDE AND POLYVINYL CHLORIDE AT I.G. FARBENINDUSTRIE, SCHKOPAU

1. INTRODUCTION

The vinyl chloride and polyvinyl chloride plants at the I.G. Farbenindustrie at Schkopau were visited May 23 1945 to obtain detailed information on the processes in. The following I.G. technical men were interviewed:

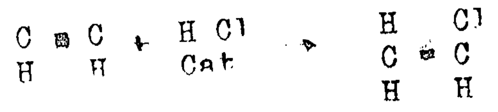
Dr. Wulff, Director
Dr. Moll, Director
Dr. Klein
Dr. Weinbrenner

VINYL CHLORIDE

A. **Condition of Plant:** Ready to operate.

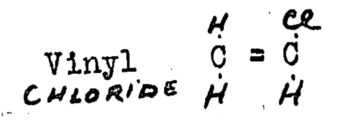
B. **Capacity:** 3500 metric tons per month.

C. **Chemistry:**

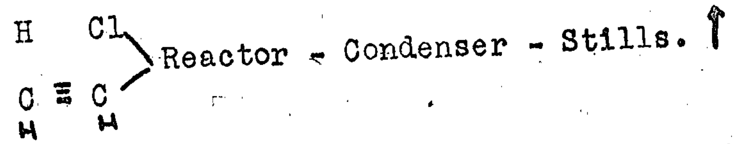


$$\underset{H}{C}\equiv\underset{H}{C} + \underset{Cat}{HCl} \rightarrow \overset{H}{\underset{H}{C}}=\overset{Cl}{\underset{H}{C}}$$

D. **Flow Diagram:**



$$\overset{H}{\underset{H}{C}}=\overset{Cl}{\underset{H}{C}}$$



E. **Materials Used:**

Acetylene:	98.0% Acetylene made from Ca C_2 at Schkopau. The acetylene must be dry.
HCl:	95-98% HCl made by burning hydrogen and chlorine at Schkopau. It must be dry.
Catalyst:	Mecuric chloride on activated carbon. 8 to 14 mesh carbon is soaked in a solution of mecuric chloride and dried. Analyses on dry basis 90% carbon and 10% mercuric chloride.
Calcium Chloride:	A 25-30% solution of calcium chloride for refrigeration purposes.

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MANUFACTURE OF VINYL CHLORIDE AND POLYVINYL CHLORIDE AT I.G. FARBENINDUSTRIE, SCHKOPAU

May 26, 1945

Reported by:

R. H. BOUNDY, U. S.
R. L. HASCHE, U. S.

On behalf of the

U. S. Technical Industrial Intelligence Committee

June 24, 1945

CIOS Target No. 22/82
Miscellaneous Chemicals

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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6 p.

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by authority of
The Joint Chiefs of Staff,
by Col. E. W. Gruhn.

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MANUFACTURE OF VINYL CLHORIDE AND POLYVINYL CHLORIDE I.G. FARBENINDUSTRIE, SCHKOPAU

Boundy + Hasche

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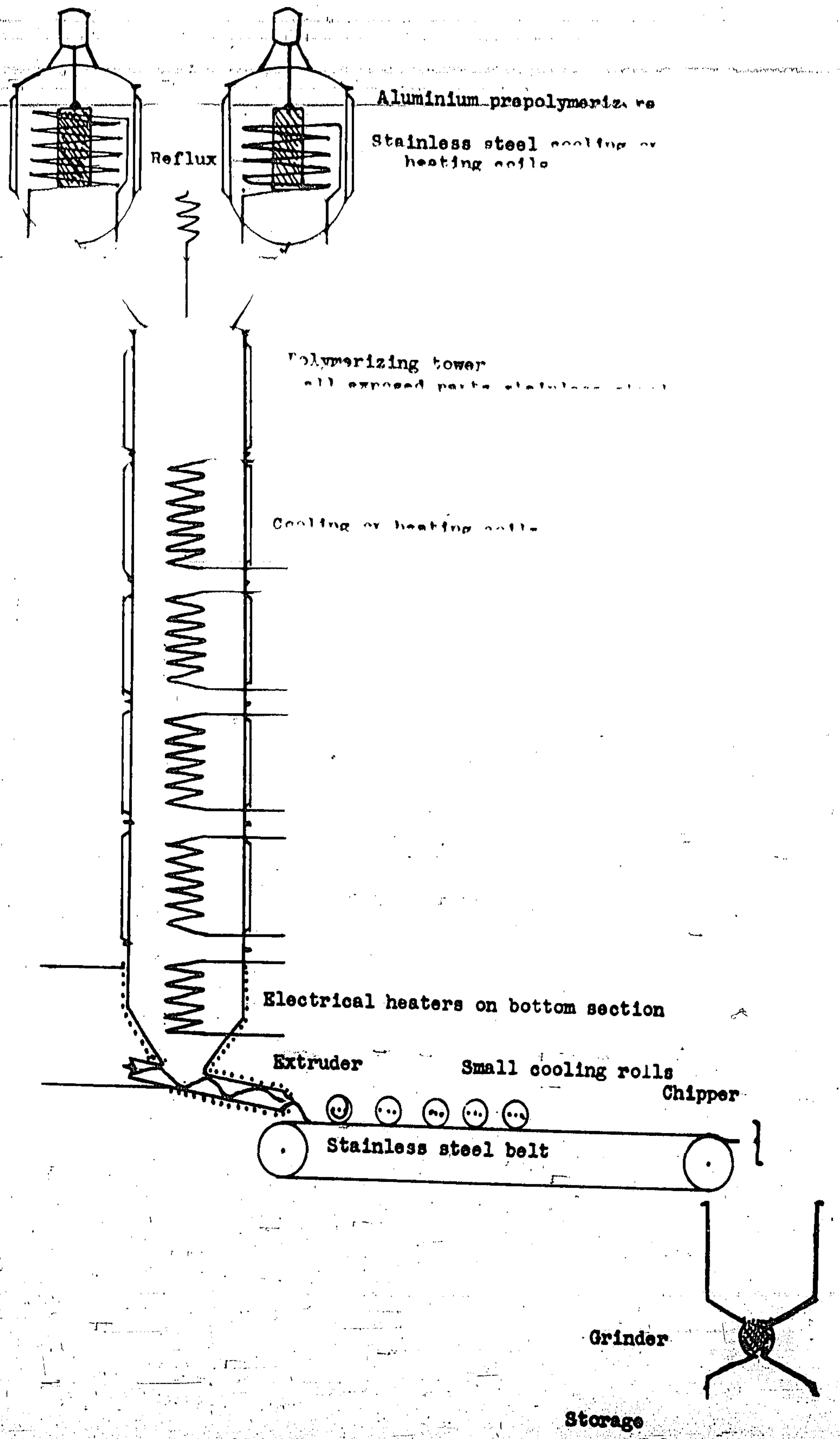
COMBINED INTELLIGENCE OBJECTIVES
SUB - COMMITTEE

SCHEMATIC DRAWING OF UNIT FOR POLYSTYRENE II AND III



July 1 1945 ES

polymer is milled in a special extrusion machine to remove the last trace of ethylene and to extrude the polymer in commercial sheets.

The plant houses four polymerization units, exact duplicates, each capable of producing 75 metric tons a month of polyisobutylene.

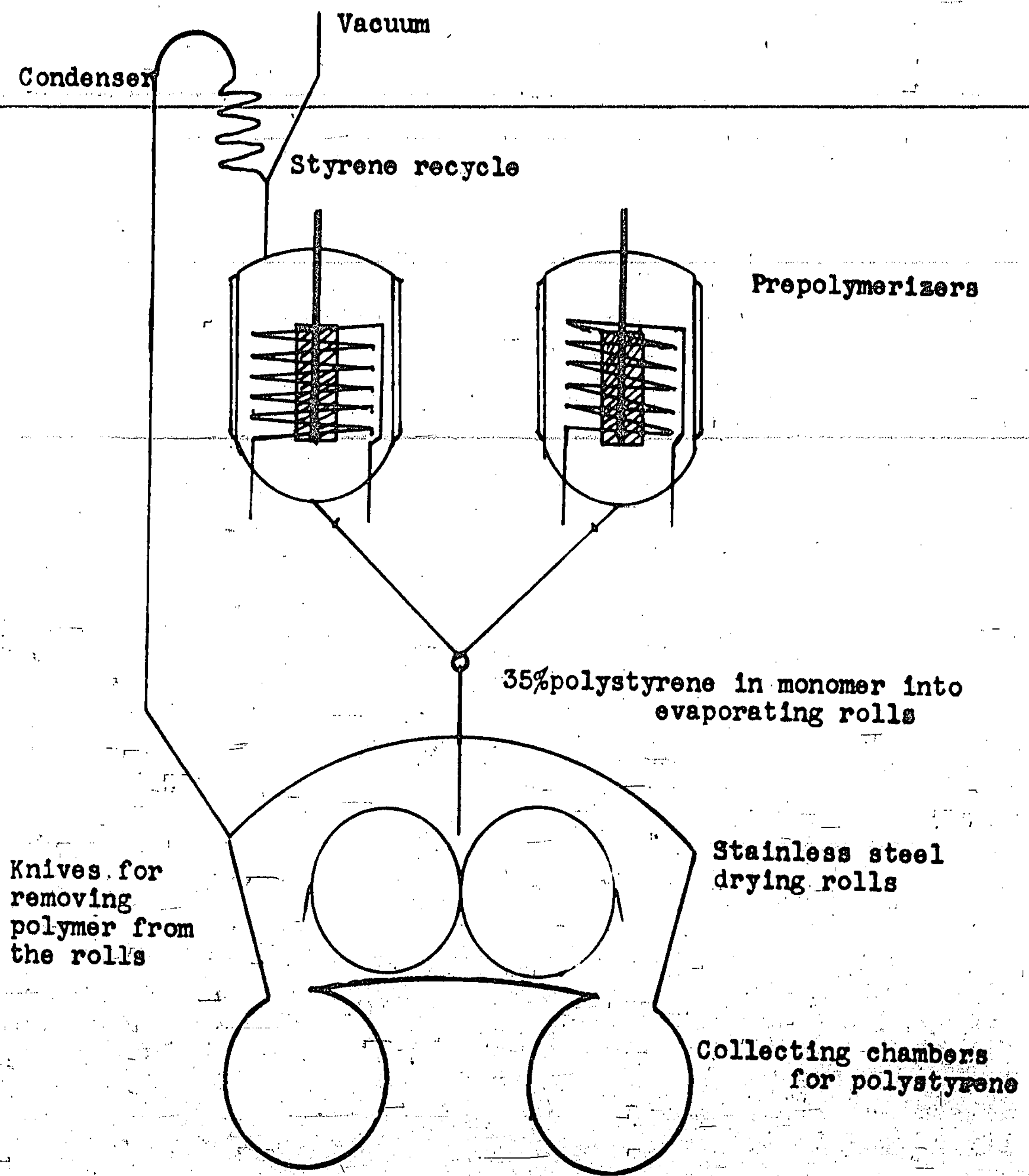
The reaction composition is as follows:

2 pounds liquid ethylene at -100° C with .1% Boron
trifluride at -100° C. (based on isobutylene).
1 pound isobutylene at 100° C.

Each unit is made up of a horizontal endless stainless steel belt approximately .025 inches thick by 12 inches wide by 20 feet long (running length). The top side of the belt is held cup shape by idlers so that the sides are three inches higher than the center. Thus the belt acts as a continuously moving container for the polymerizing mixture. The belt moves two feet per second. The belt is housed in a cylindrical steel shell 30 inches in diameter. The polymerization is at atmospheric pressure and so the housing need only be gas tight. The ethylene with dissolved catalyst is mixed with the isobutylene just as it pours on to moving belt in a layer one inch deep at the center of the belt. The polymerization is extremely rapid being practically complete in the first six feet of belt travel. The vaporized ethylene passes to a gas holder then compressed, condensed and recycled. The polymer is spongy because of ethylene vaporization and now completely fills the belt. The polyisobutylene is scraped from the belt at the extreme end of its travel and falls directly into the hopper of a large double screw extruder where it is milled and extruded into sheets about ten inches wide and one-half inch thick. The extruder is of iron and steel construction. Both screws or rolls are about 12 inches in diameter and 36 inches long. The depth of the flight is 3/4 inches. The width of the flight is 8-10 inches at the hopper and 2-3 inches at the output. The molecular weight of the polymer is controlled by the purity of the isobutylene and quantity of catalyst.

R. H. Boundy

SCHEMATIC DRAWING OF UNIT FOR POLYSTYRENE IV



Julyl 1945.
RB.

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MANUFACTURE OF POLYISOBUTYLENE AT I. G. FARBENINDUSTRIE, OPPAU

June 8, 1945

Reported by:

R. H. BOUNDY, U.S.
R. L. HASCHE, U.S.

On behalf of the
U.S. Technical Industrial Intelligence Committee

June 27, 1945

CIOS Target No. 22/517
Miscellaneous Chemicals

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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The Joint Chiefs of Staff,
by Col. E. W. Gruhn.

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MANUFACTURE OF POLYISOBUTYLENE
I.G. FARBENINDUSTRIE, OPPAU

Boundy + Hasche

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

atmospheres. Either gas or liquid ammonia could be used. The reaction is exothermic and requires cooling. The yield amounts to 90-95% of theoretical.

The reaction was carried out in an autoclave with a bonded stainless steel lining. Distillation as in the case of the first step, is carried out in aluminum equipment.

c. Dehydration of Formamide

The dehydration of formamide was carried out in the vapor phase at 200-300°. C over an activated alumina catalyst. The ammonia is used as diluent.

The HCN was washed from the diluent ammonia and then [illegible] distilled, [illegible]

R. L. Hasche

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PERSONNEL OF INSPECTION TEAM

R. . Boundy	TIIC, CIOS
R. L. Hasche	TIIC, CIOS

MANUFACTURE OF HYDROCYANIC ACID AT I. G. FARBENINDUSTRIE, OPPAU & LUDWIGSHAFEN

1. INTRODUCTION

The I. G. Farbenindustrie plant at Oppau has the only plant of its kind in Germany producing hydrocyanic acid from the interaction between methanol, ammonia and carbon monoxide. The manufacturing units for this process did not suffer bomb damage.

2. STEPS IN THE MANUFACTURE

The following steps are involved in the process:

a. Methanol reacts with carbon monoxide to form methyl formate.

$$CH3OH + CO = HCO2CH3$$

b. Methyl formate is reacted with ammonia to produce formamide,

$$HCO2CH3 + NH3 = HCONH2 + H2O$$

c. Formamide is dehydrated to hydrocyanic acid

$$HCONH2 = HCN + H2O$$

The first two steps in the syntheses are carried out at the Oppau plant where 850 tons per month of formamide was produced. The entire output was then dehydrated at Ludwigshafen to hydrocyanic acid.

3. OPERATION

a. Production of Methyl Formate.

The operation is carried out batchwise in a stainless lined autoclave. Methul alcohol is charged to the reaction vessel and 1% of NAOH added at catalyst. The mixture heated by a steam jacket to 100-110°C. Water gas is introduced slowly through the bottom until equilibrium is reached which is between 80 and 90% toward the ester. It requires about 4 hours to complete the operation. The reaction mixture is discharged and the ester separated from the unreacted methanol by distillation. The yield is about 90%.

b. Formation of the Amide

The reaction between Methyl formate and ammonia was carried out batchwise at a temperature of 60-70° C. and pressure of 10-15

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MANUFACTURE OF HYDROCYANIC ACID AT I. G. FARBENINDUSTRIE, OPPAU

June 8, 1945

Reported By:

R. H. BOUNDY, U.S.
R. L. HASCHE, U.S.

on behalf of the

U.S. TECHNICAL INDUSTRIAL INTELLIGENCE COMMITTEE

CIOS Target No. C22/517
Miscellaneous Chemicals

June 27, 1945

3 p

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF, (Rear), APO 413

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ITEM NO. 22

FILE NO. ~~XXXVII-64~~ XXVI-64

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MANUFACTURE OF HYDROCYANIC ACID I.G. FARBENINDUSTRIE, OPPAU

Boundy + Hasche

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

4. PCU FABRICATION AT BITTERFELD

The plant has facilities for milling, calendering, pressing flat sheets, press polishing sheets, extruding and welding. Large sheets and pipe are the two large production items.

Pipe (up to 8" diameter), solid cylinders (8" diameter) and special shapes were extruded by means of two large metallurgical type presses, one with a piston diameter of 10" and the other 16". Each one had two movable dies (transverse) so one could be cleaned while the other was in use. Rolled cylinders of hot preform from the milling rolls were fed directly into the extrusion cylinders. (Temperature 180° to 190° C.) No heat was added except to compensate for radiation and at the die. Pressures ranged from 300 to 600 atmos. Pipes approximately eight inch diameter x ten feet long x 1/2 inch wall were fabricated by this method.

Flat sheets were made either directly from a three roll calender or press polished on a flat plate press. Flexible sheet could be rolled on this calender to about .2 mm. thickness. They stated that a calender in operation in Ludwigshaven ~~is~~ used for sheets down to .03 mm. thickness.

PCU sheet, pipe, etc. is extensively fabricated by welding by means of a hot air torch using air at 200° C with technique similar to the acetylene welding of metals.

R. H. Boundy

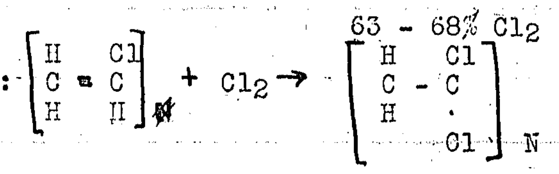
3. CHLORINATED POLYVINYL CHLORIDE (IGELIT PC)

A. Condition of Plant: Ready to operate.

B. Capacity: 120 metric tons per month.

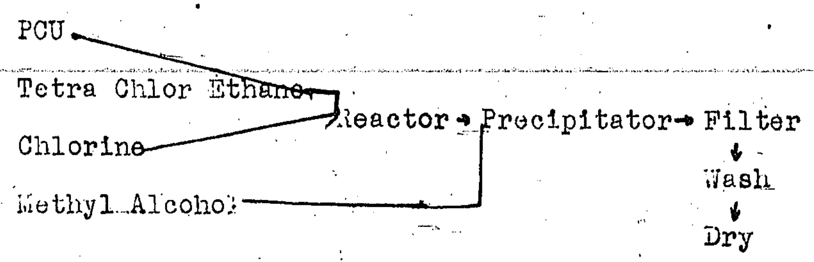
C. Inventory: 50 metric tons.

D. Chemistry:



$$\left[\begin{matrix} H & Cl \\ C & = C \\ H & H \end{matrix}\right]_N + Cl_2 \rightarrow \overset{63 - 68\%\ Cl_2}{\left[\begin{matrix} H & Cl \\ C & - C \\ H & \cdot \\ & Cl \end{matrix}\right]_N}$$

E. Flow Diagram:



F. Raw Materials:

PCU. Described in first part of this report.

Tetrachlorathane. Tech. grade Tetrachlorethane.

Chlorine. 96 - 99% Cl_2 from Mercury cells.

Methyl Alcohol. Tech. grade.

G. Description of Process:

1750 pounds of PCU and 17,000 pounds of tetrachlorethane are charged to the reactor and solution takes place with stirring. The temperature is raised to 90° C by means of lead steam coils and the addition of chlorine started. Chlorination continues for a period of 24 hours. Degree of chlorination is controlled by the Specific Gravity of the solution. Various grades are made with Cl_2 content from 63 to 68%. After chlorination the mixture is pumped to the precipitating kettle where approximately an equal volume of CH_3OH is added with constant stirring. The PC precipitates in spheres very small to .05" diameter. The mixture is filtered. The filterate is distilled for recycle and the precipitate washed twice with ion exchange water. It is vacuum dried 60° C and 100 mm. pressure for 12 hours and packaged in 50 pound paper bags. No addition agents or stabilizers are added. However, we were informed that fabricators frequently add about .5% $(C_2H_4OH)_2S$ as a light stabilizer. Glycerine may also be used as a stabilizer. The process is distinctly bath tub and could be greatly improved by good engineering.

H. Equipment:

a. Reactor (2) 280 cu. ft. acid brick lined. Lead lined stirrer. Lead heating and cooling coils. High grade brick lining.

b. Precipitator (1) 525 cu. ft. stirred kettle, no cooling coils, otherwise similar to reactor.

c. Filter (1) Earthenware, about 6' diameter x 6' high. Cotton filter cloth supported on a perforated plate. The PC was scraped off filter with a hoe into a wooden cart and transferred thereby to the dryer.

d. Dryer Horizontal rotating nickel cylinder about 4' diameter x 7' long, heated with horizontal nickel tubes 2" diameter spaced on 10" centers. Operated at 60° C and 100 mm. pressure. Circulated water used for heating.

I. Uses

Film, fibers, lacquers. Mostly at Wolfen.

F. Raw Materials:

a. Vinyl Chloride: The vinyl chloride is shipped from Schkopau in 60,000 pound steel tank cars under about 5 atmos. pressure. No inhibitor is added. Nitrogen pressure is used to load and to fill vapor space. Under these conditions vinyl chloride can be stored indefinitely without polymerization. Purity was expressed as 100.0%, with no known contaminates. Acetylene was less than .0009% by the copper test. Acidity .000%. Purity was also determined by freezing point. (-13.4°-13.5° C.)

b. Water: Water is purified by double ion exchange. The ion exchange resins are made at Wolfen. (Phenol formaldehyde and Aniline formaldehyde type.) The water contains less than 5.0 ppm. total salt.

c. Hydrogen Peroxide: 40% solution in water. Stored in carboys.

d. Merisol Emulsifier: The sodium salt of a 13 - 18 carbon straight chain paraffin treated with SO_2 and Cl_2.

G. Description of Process

Vinyl chloride is stored in iron tanks, room temperature, corresponding pressure with no inhibitor. The water and emulsifier are mixed before adding to the reactor in an agitated iron tank. The following is a charge for one reactor (15 total):

3500 pound vinyl chloride
3500 pound water
140 pound soap
14 pound hydrogen peroxide as H_2O_2.

They are charged to the reactor in the following order:

1. Water and soap premixed.
2. Vinyl chloride.
3. Catalyst.

Some of the reactors are nickel and some glass enamel lined. In either case they are 37' long x 40" in diameter. There are no baffles or coils inside. They are horizontal and rotated at 4.0 rpm. Both ends are flanged for easy cleaning. This is accomplished by hosing down with water about once a month. The temperature is controlled at 45.0° C by means of a water jacket the length of the reactor. The inhibition period is about 4 hours and polymerization is completed in 24 hours. The pressure drops at the end of the reaction and is used as a means of control. Conversion is 90%. The remaining vinyl chloride monomer is flashed to the atmosphere and wasted. The emulsion is then pumped (centrifugal pumps) directly to the drier. The two spray driers are standard iron driers (Nubilose made at Meissen near Dresden) approximately 30' diameter x 60' high. They are heated with air at 160° C. The air is heated by Fin type steam exchangers. The cold emulsion is sprayed tangentially through 30 sprays at the top of the drier. The dry finely divided PVC (1. to 3. micron) is collected by a cyclone (90%). The remaining 10% is removed by means of vertical cotton bags, approximately 10' long and 1' in diameter. The dry polymer is then conveyed with screw conveyors to large iron storage tanks (approximately 10' diameter x 30') and packaged from them in 50 pound paper bags. Some is fabricated at Bitterfeld, some at Wolfen. The remainder is shipped throughout Germany. .3% Na_2CO_3 is added to the emulsion after polymerization for stabilization of the polymer. No other addition agents are used and no washing is done. Urea (about 1.0%) is sometimes used as a stabilizer. The two spray driers are different sizes:

Small: Capacity 2500 kilos emulsion or 1250 kilos resin per hour. Volume approximately 500 cubic meter 90,000 cubic meter per hour of air is used for drying.

Large: Capacity 3600 kg. emulsion or 1800 kg. resin per hour. About 120,000 cubic meter of air per hour is used.

No control tests were run except stability in fabrication and viscosity in solution.

H. Uses

1. Calendering to sheets.
2. Extruding to pipe, shoestrings, tape, etc.
3. Battery boxes.
4. Chlorinating to Igelit PC.
5. General injection and compression molding.
6. Fabrication into valves, etc.

Sample: A 5 kg. sample of the above material was obtained for test purposes.

Note: 1. Two complete spray drying units were used.
2. High molecular weight was emphasized as a means of obtaining a tough plastic.
3. Only one type of polymer was manufactured.

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R. H. Boundy	TIIC, CIOS
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MANUFACTURE AND FABRICATION OF POLYVINYL CHLORIDE (PCU & PC) AT I.G. FARBENINDUSTRIE BITTERFELD

1. INTRODUCTION

The I.G. Farbenindustrie plant at Bitterfeld was visited on May 25, 1945 for the purpose of obtaining information on plastics and chemical intermediates for pastics manufactured at Bitterfeld. The following I.G. personnel were interviewed:

Dr. Burgen, I.G. Director in the Bitterfeld Wolfen area.
Dr. Schoenberg, Vinyl Chloride Plastics Production.
Dr. Ballman, Assistant to Schoenberg.
Dr. Weeber, Assistant to Burgen.

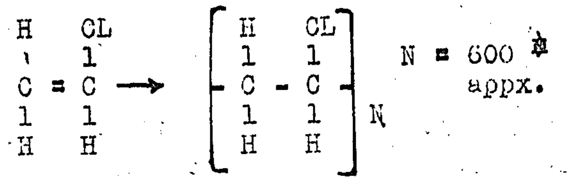
2. POLYVINYL CHLORIDE (IGELIT PCU) PLANT

A. **Condition of Plant:** Ready to operate.

B. **Capacity:** 1,500 metric tons per month.

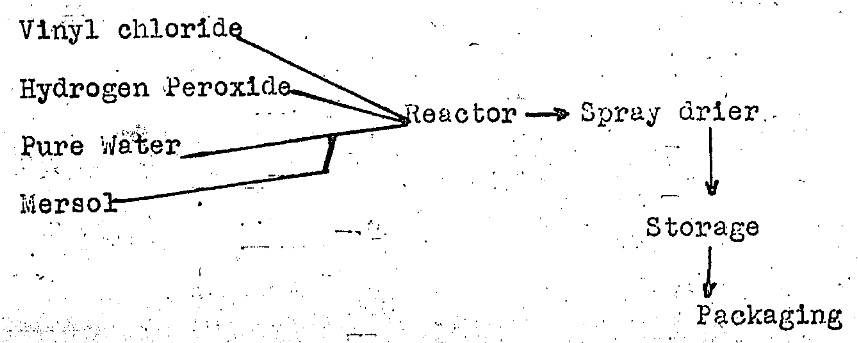
C. **Inventory:** 1,000 metric tons

D. **Chemistry:**



$$CH_2{=}CHCl \longrightarrow [-CH_2-CHCl-]_N \quad N = 600^{*} \text{ appx.}$$

E. **Flow Diagram:**



* This value questionable; it should be checked on sample obtained from plant.

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MANUFACTURE AND FABRICATION OF POLYVINYL CHLORIDE (PCU & PC) AT I.G. FARBENINDUSTRIE BITTERFELD

May 25, 1945

Reported by:

R. H. BOUNDY, U. S.
R. L. HASCHE, U. S.

On behalf of the

U.S. Technical Industrial Intelligence Committee

June 24, 1945

CIOS Target No. 22/1(b)
Miscellaneous Chemicals

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413.

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ITEM No. 22
FILE No. XXVI-52

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~~COPY No~~ 185

Classification Cancelled,
by authority of
The Joint Chiefs of Staff,
by Col. E. W. Gruhn.

~~CONFIDENTIAL~~

MANUFACTURE & FABRICATION OF POLYVINYL CHLORIDE

I.G. FARBENINDUSTRIE, BITTERFELD

Boundy + Hasche

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

CHART 2 (Continued)

ACETYLENE + ACETALDEHYDE

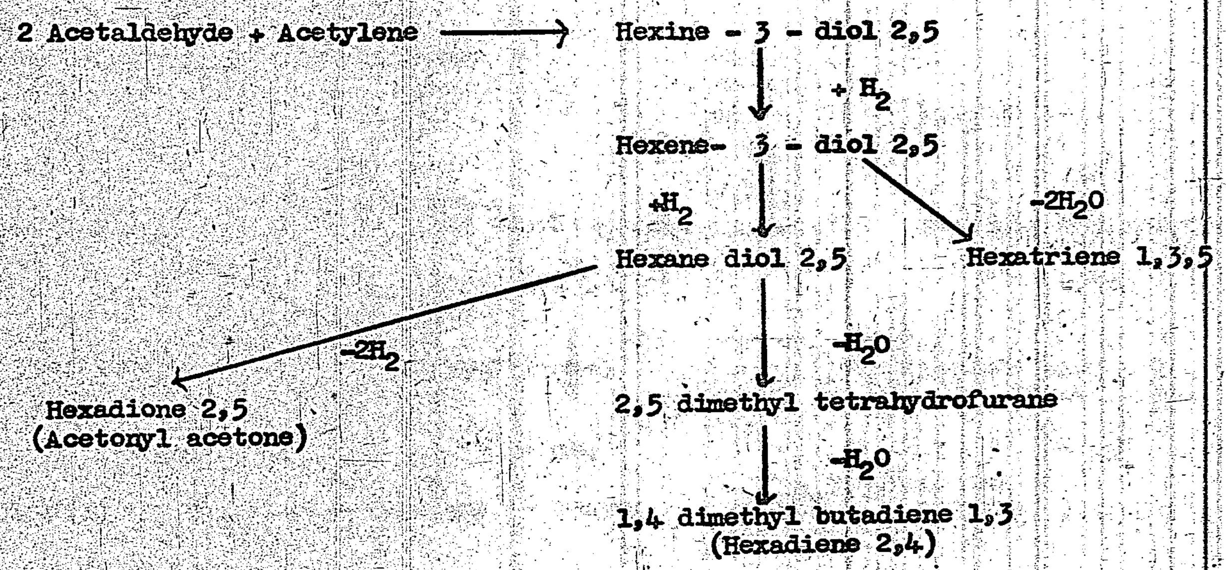


CHART 1 A (Continued)

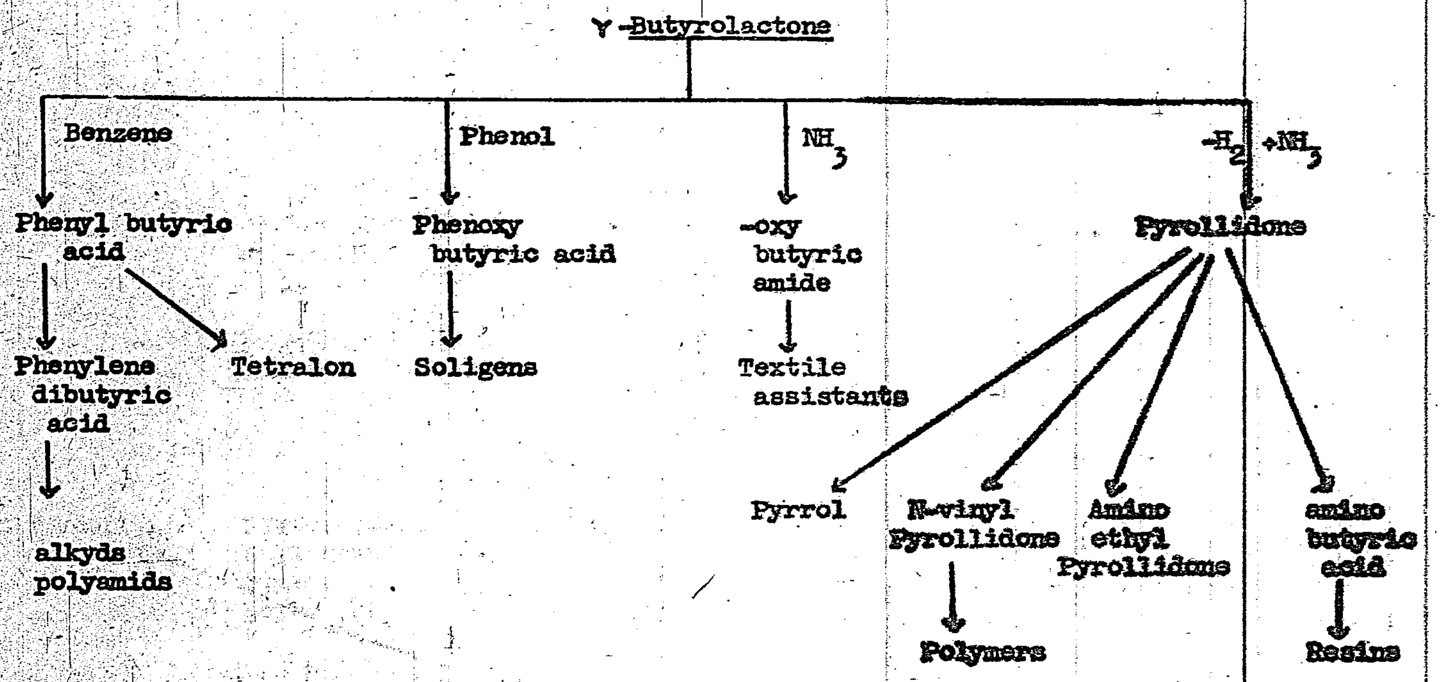


CHART 2

ACETYLENE + ACETALDEHYDE

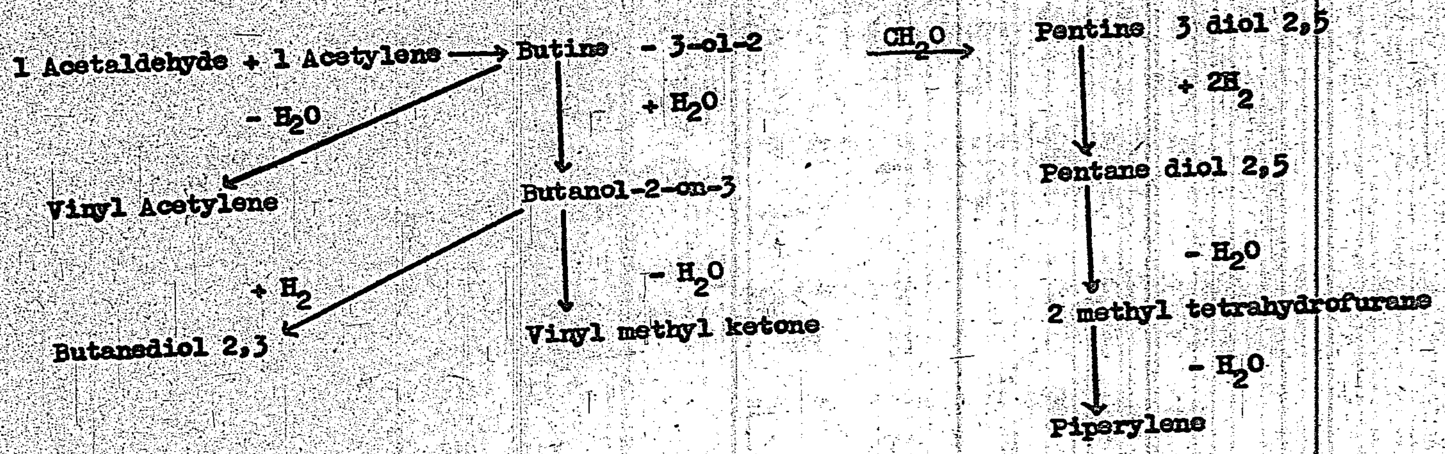


CHART 1 (Continued)

ACETYLENE + FORMALDEHYDE

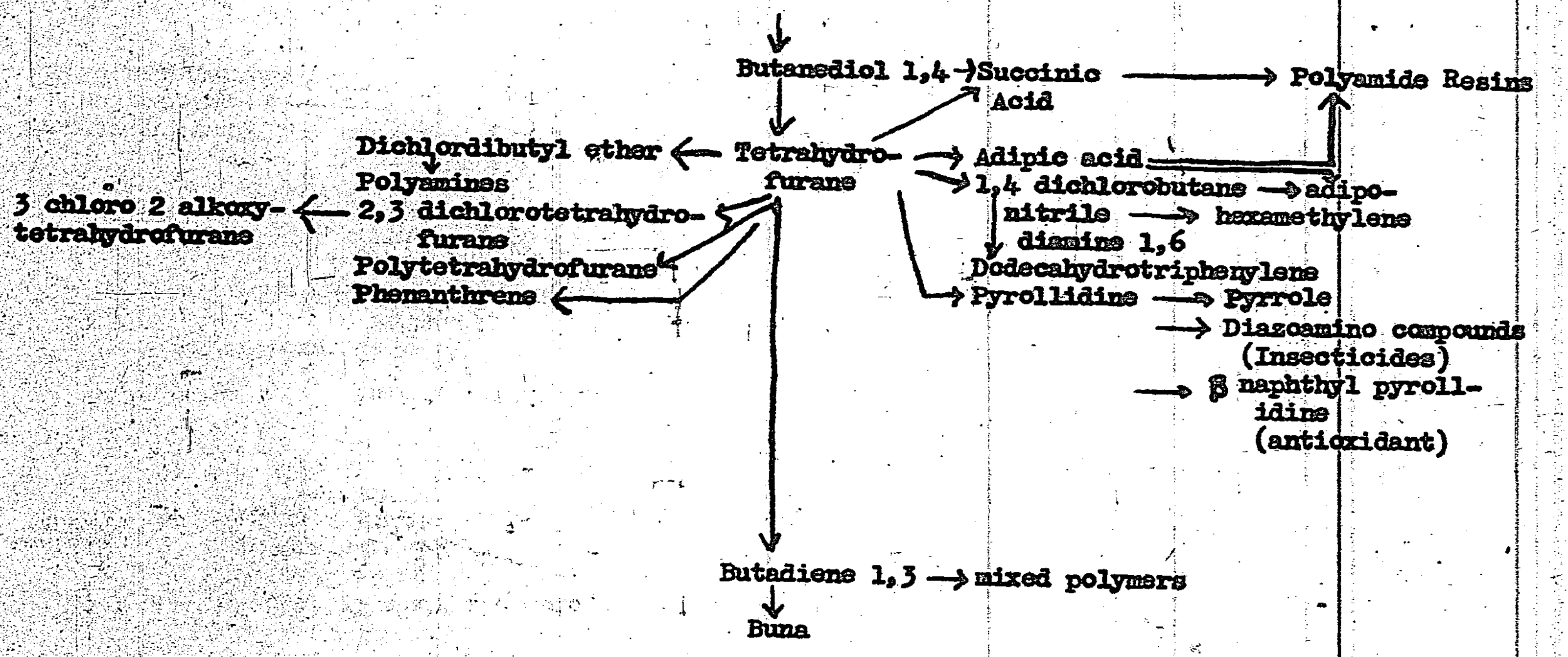
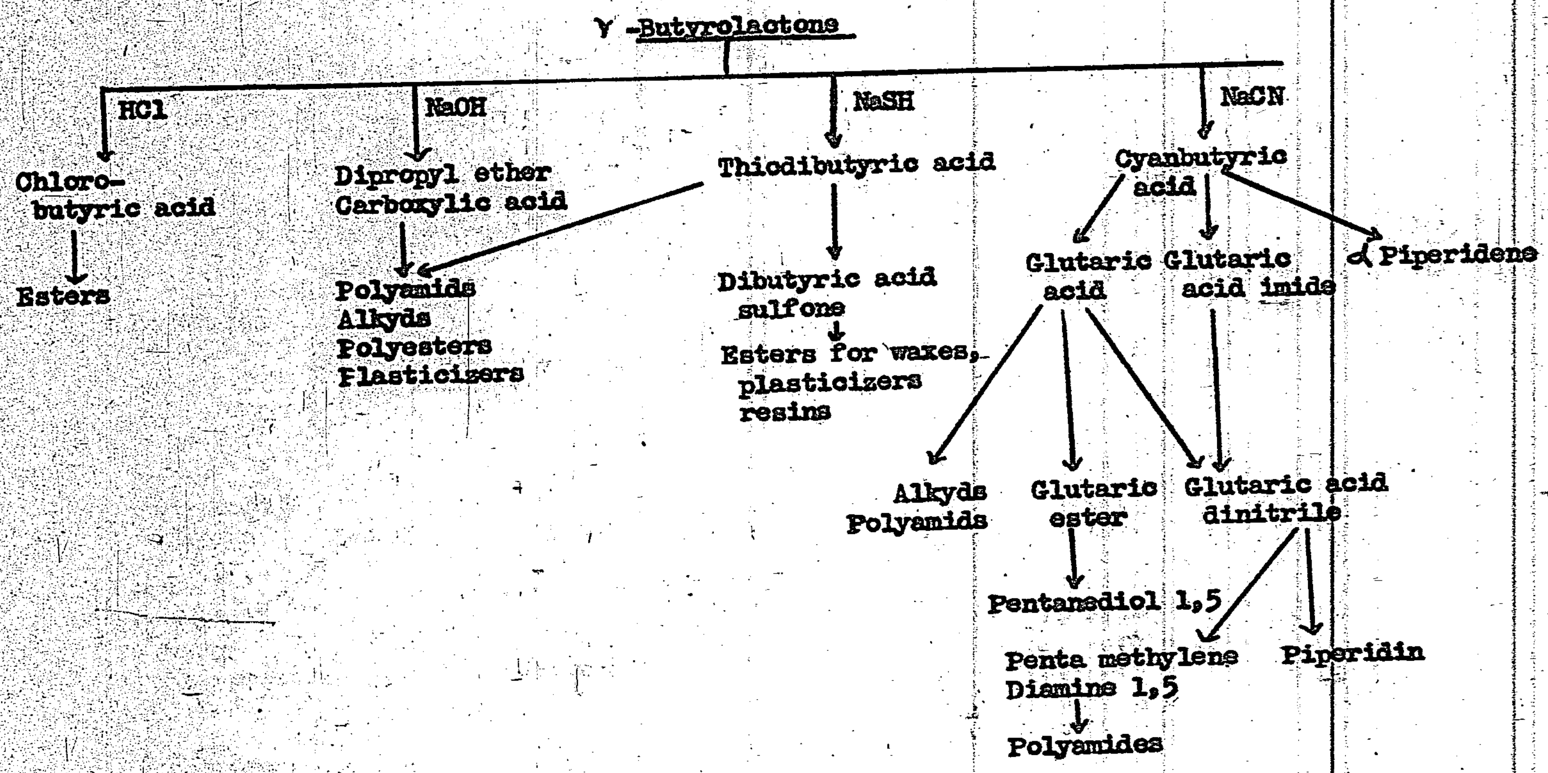


CHART 1 A



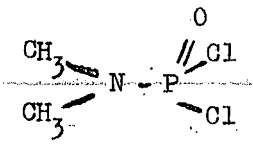
d. Ramasite

Ramasite is a water proofing agent consisting of an emulsion of paraffins (from brown coal) of different types hard and soft with less than 1% Nekal with a solution of basic aluminum acetate (or formate or chloride) and urea.

For use the emulsion is diluted 1 to 10 or 20 with water and the fabric drenched, then dried at over 100°C for the formates but at 70°C for the acetates. The water proofing effect is stated to be better than Velan (Zelan) but is removed by washing.

e. Blattan

Dr. Ambros mentioned a contact insecticide developed by Dr. Schroeder at Elberfeld -



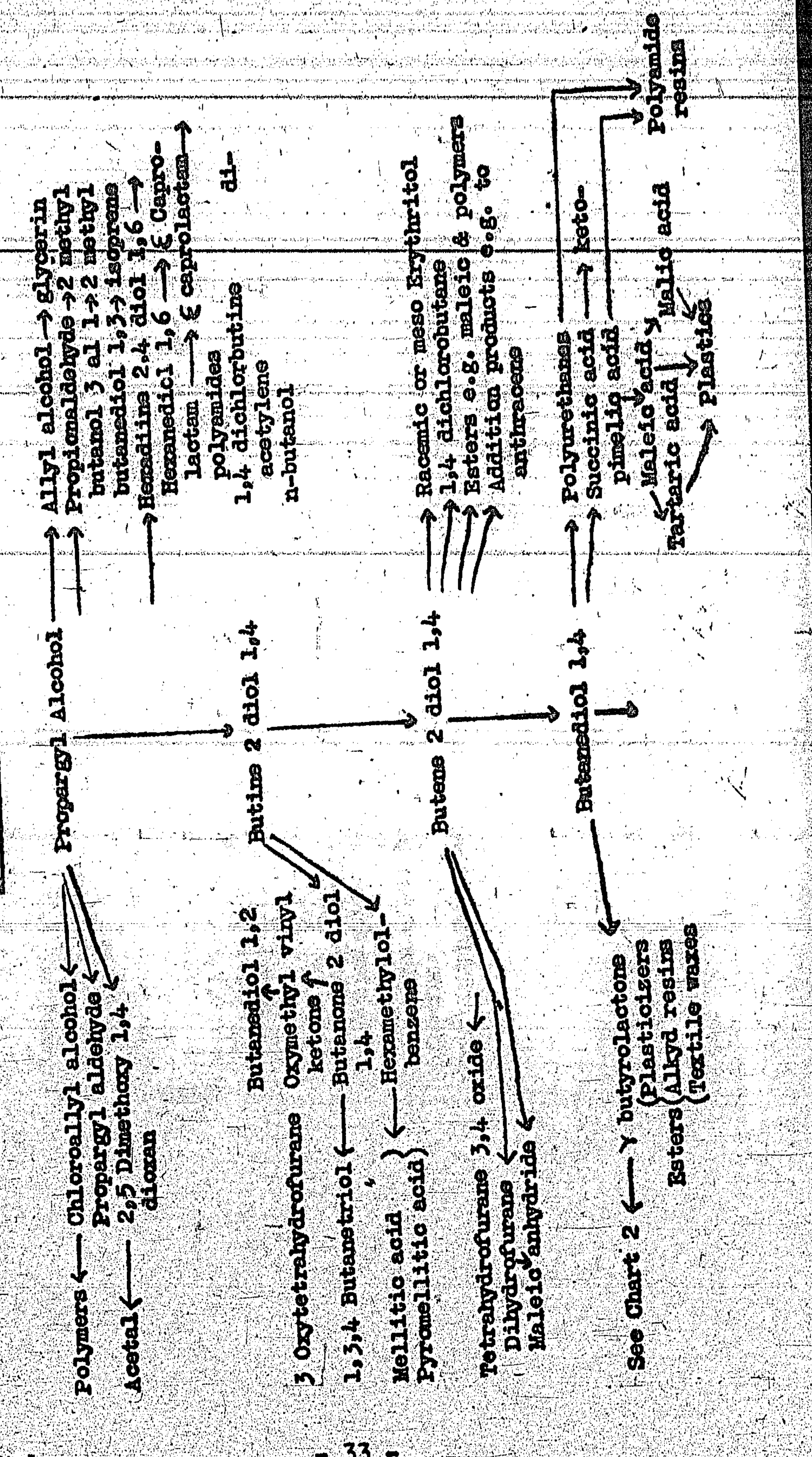
f. Polystyrene

A styrene polymerization tower had been shipped from Ludwigshafen to Gendorf and was lying in the freight yard. It was built of five steam jacketed sections and contained two steam coils in the lower sections. In practice the monomer styrene would pass to two prepolymerization kettles in parallel where it would be held with stirring at 50 - 80°C for two days without catalyst. From these kettles it passed to the top of the tower at 70°C and flowed slowly thru with a total time of sojourn in the tower of 24 hours. The temperature down the tower gradually rose to 200°C at the conical exit which was heated electrically. The tower was estimated to be 15 feet high x 2.5 feet diameter and to produce 1 ton polystyrene per day. From the conical outlet the molten polymer passed thru an electrically heated screw conveyor to a continuous metal belt carried on cooled rollers where it solidified and passed to the guiding system.

The chief grades of polystyrene produced were No. 3 with a molecular weight of 100,000 and No. 4 of 200,000. The No. 3 grade being the most important. It was stated that the styrene is completely polymerized.

CHART 1

ACETYLENE + FORMALDEHYDE



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f. Zephirol

Dimethyl benzoyl alkyl ammonium chloride.

A fatty alcohol such as octadecylalcohol is converted at 130°C in a corrosion-resisting autoclave with dry HCl gas under pressure to the corresponding alkyl chloride. The chlorohydrocarbon purified by distillation under high vacuum is brought into reaction at 100-150°C with 1.5 mols. dimethylamine in benzene solution. The hydrochloride obtained is crystallized from alcohol, slurried with ether and the free amine prepared by shaking with caustic potash solution. Finally the nitrogen of the dimethylamino octadecane is converted to quaternary by addition of 1 mol. of benzyl chloride.

g. Mitigal

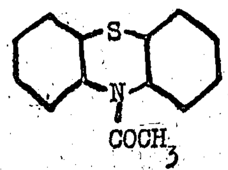
Dimethylthianthrene.

A solution of 600 gms SCl_2 in 1.2 liters toluene is allowed to flow into 2.8 liters of toluene and 400 gms aluminum chloride with heavy stirring. Stirring is continued at room temperature. The reaction product is poured on to ice and separated. The excess toluene is removed thru steam distillation and the reaction mixture given a fractional distillation. Chief product $BP_3 = 170 - 180°$.

h. Novocaine

P-nitro benzoyl chloride is condensed with diethylamino ethanol in boiling benzene. The p-nitrobenzoic diethylamino ethyl ester is reduced with a chrome nickel catalyst at 2 atmospheres pressure. The amino compound is converted into the hydrochloride, Novocaine.

i. N-Acetylphenothiazine



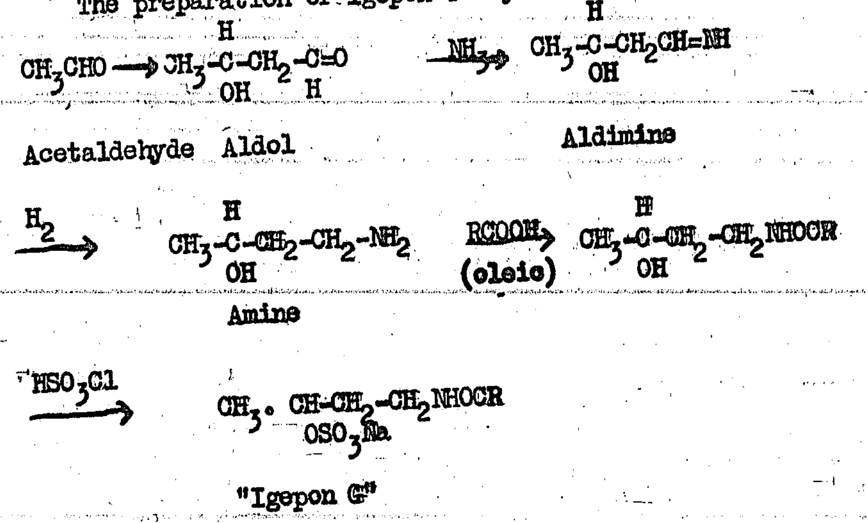
A mixture of diphenylamine and sulfur is heated whereby phenothiazine is obtained.

This is heated with acetic anhydride, whereby the nitrogen is acetylated. Actually a mixture of diphenylamine, sulfur and acetic anhydride can be heated together, and also obtain the N-acetylphenothiazine.

6. MISCELLANEOUS ITEMS

a. Igepon G.

The preparation of Igepon G may be outlined as follows:



$$CH_3CHO \longrightarrow CH_3\text{-}\overset{H}{\underset{OH}{C}}\text{-}CH_2\text{-}\underset{H}{C}{=}O \xrightarrow{NH_3} CH_3\text{-}\overset{H}{\underset{OH}{C}}\text{-}CH_2CH{=}NH$$

Acetaldehyde Aldol Aldimine

$$\xrightarrow{H_2} CH_3\text{-}\overset{H}{\underset{OH}{C}}\text{-}CH_2\text{-}CH_2\text{-}NH_2 \xrightarrow[\text{(oleic)}]{RCOOH} CH_3\text{-}\overset{H}{\underset{OH}{C}}\text{-}CH_2\text{-}CH_2NHOCR$$

Amine

$$\xrightarrow{HSO_3Cl} CH_3\cdot\underset{OSO_3Na}{CH}\text{-}CH_2\text{-}CH_2NHOCR$$

"Igepon G"

b. Water Softener - "Trilon"

The sodium salt of the compound prepared by the reaction

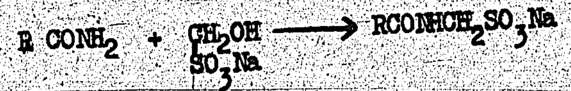
$$NH_3 + HCHO + HCN \longrightarrow N(CH_2COOH)_3$$

is a water softener sold as "Trilon."

Manufacture was started at Ludwigshafen just before the war.

c. Detergent

Dr. Reppe spoke of work done on a detergent developed by the Japanese, Dr. Otta, with whom, however, he claimed to have no direct contact. The product is made by reaction of a fatty acid amide and formaldehyde - sulfite -



$$R\ CONH_2 + \underset{SO_3Na}{CH_2OH} \longrightarrow RCONHCH_2SO_3Na$$

This is not as good as the Igepon but is cheaper and easier to manufacture.

Chlorinated polyvinylchloride resin under the trade name Vinoplex was made at the Bitterfeld plant of I.G. Farbenindustrie. It was produced by passing chlorine gas into a 20% solution of polyvinylchloride resin (52 to 56% Cl) in carbon tetrachloride at 30 to 40°C, resulting in the addition of 2 to 4% chlorine above the theoretical amount. The dried product (Vinoplex) is a white powder which is soluble in mixed solvents of ketones and aromatics and the cast films have excellent chemical resistance. This is the basis of IGELIT-PC.

Other products which are on the agenda of the laboratory for further study are the chlorinated Buna rubbers, chlorinated polystyrenes and polyethylene resins.

The interview was with Mr. Eichstadt, Chem. Engineer.

5. PHARMACEUTICALS

The work on pharmaceuticals at Gendorf is entirely in the laboratory stage. The following notes were given as indicating the directions of work:

a. Phanodorm and Evipan

Condensation of cyclohexanone with malonic ester in the presence of piperidine acetate gives cyclohexylidene malonic ester. Cyclohexenyl ethyl (or methyl) malonic ester is obtained by treatment with sodium ethylate followed by reaction with ethyl bromide or methyl iodide. Phanodorm (MP 171°C) is produced by condensation of the ethyl compound with urea and Evipan (MP 146°) by a similar reaction of the methyl compound with methyl urea.

b. Avertin (Tribromoethyl Alcohol)

Equimoleoular amounts of bromal and benzaldehyde are reacted with good cooling in the presence of about 30% on the weight of bromal of aluminum isopropylate in solution in absolute ether. After completion of the reaction, the batch is heated 1-2 hours on the water bath, the mixture is decomposed with 5% hydrobromic acid and then taken up in ether.

The ethereal solution is washed with pure water, then with dilute sodium carbonate solution and dried over sodium sulfate. After distilling off the ether the remainder is distilled under reduced pressure. Boiling Point = 92 - 94°C.

For further purification the avertin fraction is recrystallized from petroleum ether - MP = 80°C.

c. Adrenalin

The preparation of adrenalin takes place in three steps:

(1) Pyrocatechol is dissolved together with monochloracetic acid and phosphorus oxychloride in dry benzene and boiled
24 hours under a reflux condenser. Dioxyphenyl chloromethyl ketone
is formed and is obtained from the above mixture after volatilization of the benzene in vacuum and after heating up with carbon
tetrachloride by crystallization from water. M.P = 173°C.

(2) The powdered dioxyphenyl chlormethyl ketone in alcohol
suspension is shaken with a 40% aqueous solution of methyl amine
for 24 hours, then filtered and washed with cold alcohol. The
4-methyl amino acetopyrocatechol formed is purified by solution in
hydrochloric acid and reprecipitation with ammonia. The decomposition point is 230°C.

(3) In the last step the above compound is reduced, either
in the presence of aluminum amalgam or electrocatalytically by
means of a nickel or palladium electrode and addition of a
palladium chloride solution, to the optically inactive d,l adrenalin.

The inactive forms can be separated by means of the bitartarates into the optically active components.

d. Eunarcon

N-methyl-5 isopropyl bromoalkyl barbituric acid.

Molar amounts of isopropyl bromide and sodium malonic ester
are reacted. The isopropyl malonic diethyl ester is again converted to the sodium compound and decomposed with α bromalkyl
bromide to isopropyl bromalkyl malonic diethyl ester. The preparation of the α bromalkyl bromide takes place according to the
publication of Von Tollens by decomposition of 1, 2, 3 tribrompropane in ether solution with sodium. By condensation of the
isopropyl α bromalkyl malonic diethyl ester with methyl urea, ring
closure takes place to N-methyl-5-isopropyl bromoalkyl barbituric
acid.

e. Insulin

Extraction of the comminuted pancreas glands with dilute
60% alcohol containing HCl is followed by filtration and precipitation of impurities by ammonia. Sulfuric acid is added and
the alcohol distilled off in vacuum at a maximum temperature of 30°C.
The insulin is fractionally precipitated from the aqueous solution
with salt and finally by repeated precipitation at the isoelectric
point. The insulin is crystallized from phosphate buffer solution
after addition of zinc chloride.

very actively at 40-42°C with practically quantitative yields of acrylic derivatives.

In the laboratory process for the preparation of ester, alcohol is placed in a three-necked flask with the conc HCl, the air is swept out with acetylene and the necessary amount of nickel carbonyl allowed to drop out of a burette. Temperature is 40-42°C. It is important that sufficient acetylene be furnished, as otherwise the rate of absorption is so great as to form a vacuum. After distillation of the acrylic ester - alcohol mixture from the nickel chloride, the acrylic ester is obtained in pure form by washing and distillation.

Regeneration of the nickel carbonyl from nickel chloride is carried out by treating the chloride solution with a slight excess of ammonia above that necessary to form the complex hexamin nickel-2-chloride and then with CO at about 80°C and 50-100 atm. The nickel carbonyl is formed quantitatively, leaving in solution the excess ammonia, ammonium chloride and ammonium carbonate.

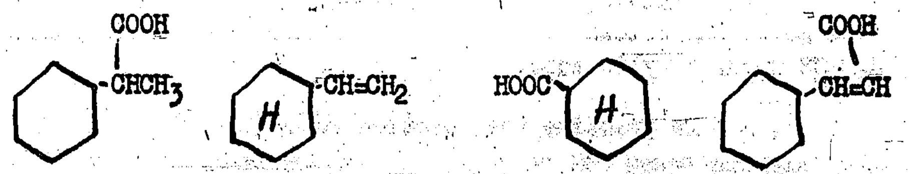
The acrylic acid process can be run continuously and without pressure except in the recovery of nickel.

(7) Products from Butadiene

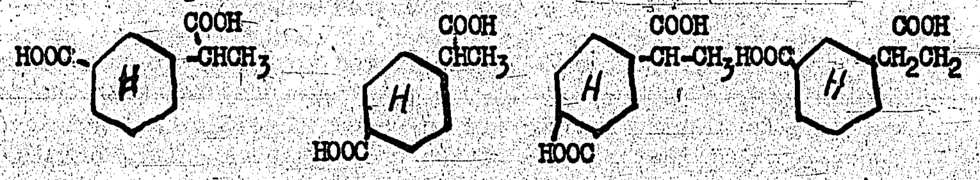
As butadiene in Germany is produced fundamentally from acetylene, Dr. Reppe's ideas on this subject are included herein.

Butadiene in benzene as a solvent is converted to vinyl cyclohexene at 270°C and 200 atmospheres with nickel carbonyl as a catalyst if no water is present. 70 - 90% yield is obtained.

In the presence of water four products are obtained:



With longer time a mixture of the following which cannot be separated is obtained:



The mixture serves as a base for plasticizers and when free of monocarboxylic acids, for polyamides.

4. APPLICATION DATA ON SURFACE COATINGS

Lacquers, varnishes, waxes and emulsions were discussed; none of these products were being made at Gendorf but were made by I.G. Farbenindustrie in Ludwigshafen. Essentially, they produced the basic materials for further processing by surface coating manufacturers.

Lacquer information possessed at the Gendorf plant is based on standard formulation using glycolesters as the active solvents for nitro cellulose, cellulose acetate, cellulose butyrate and cellulose tripropionate. The resin portion at this time is ester gum, although various synthetic resins will be employed when available. The diluent is benzene which is apparently permissible in Germany regardless of its toxicity.

Synthetic waxes were purchased from Ludwigshafen and the harder types were preferred in the 12C to 18C series, such as the palmitic esters.

Consideration is being given to the manufacture of resin emulsions and these will be based on modifications of methacrylate, polyvinylacetate and phthalic anhydride resins. Production of these products will depend upon the availability of the necessary raw materials at Gendorf.

Three grades of so-called varnishes, sold under the trade name Luphen, were made in Ludwigshafen. These are condensates of the phenol-formaldehyde type in butyl alcohol. The end product contains 75% resin and 25% butyl alcohol and is known as Luphen L. The process involves the reaction of approximately 1 part phenol, 1 part aldehyde in the presence of 4 to 6 parts butyl alcohol for 6 to 8 hours at 120 to 140°C. The addition of 1 to 5% HCl just prior to application is employed for final conversion of Luphen L resin. Ordinarily the resin solution was reduced to 50% solids with alcohol for spray purposes and was used where chemical resistance is required.

Lupen O H is Luphen L without hardener and is recommended for finishing wood or metal, either clear or pigmented.

The reaction for Luphen 145 is not carried as far as Luphen L and is soluble in ethyl alcohol. It was primarily used for can coatings which are baked at 165°C.

determined that the growth of certain pathogenic bacteria was completely suppressed in a dilution of 1:100,000 by the $C_{12}H_{12}$ fraction which because of small amounts of azulen (about 5%) was colored deep blue.

(3) Ethyl Alcohol

Dr. Reppe claimed that a practical process had been developed for direct hydration of ethylene which, in Germany, is likely to be produced from acetylene. The process is applicable also to propylene and butylene.

The olefine and water were run into the top of a tower packed with a catalyst consisting of a mixture of WO_2 and WO_3 made from ammonium wolframite and promoted with 5% zinc oxide. The catalyst is carried on silica gel and the mass contains 20% W. The pressure in the tower was 200-300 atmospheres and the temperature 300°C. In practice the whole system was kept under ethylene pressure as above, new ethylene being pumped in to replace that used up.

A 20% solution of ethyl alcohol flowed from the bottom of the tower to the rectifiers. The catalyst converted 1 liter ethanol per liter catalyst per hour.

(4) Acetylene and Alcohols

The type reaction for aliphatic alcohols is:

$$ROH + C_2H_2 = ROCH{:}\ CH_2$$

This reaction must be carried out with the alcohol in the liquid phase. The lower the molecular weight of the alcohol, the lower the optimum temperature of reaction, hence when the boiling point of the alcohol is lower than the reaction temperature, sufficient pressure must be maintained to keep the alcohol in the liquid phase.

(a) Acetaldehyde

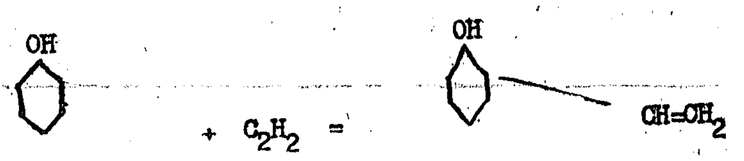
These principles are used in the Reppe process for acetaldehyde which is not new. Methanol is treated with acetylene at 20 atmospheres pressure and 90°C to form methyl vinyl ether. By treatment of the latter with water vapor at atmospheric pressure, hydrolysis takes place to methanol and vinyl alcohol, the latter immediately rearranging to acetaldehyde. The methanol is reused. Since the process uses no mercury, which became scarce in Germany, a plant for 200 tons/month was erected in Ludwigshafen, but was bombed out after being operated for a short while. Mercury poisoning troubles are eliminated, but Dr. Reppe admitted that the method was somewhat more expensive than the conventional mercury process.

(b) Polyvinyl oleyl Ether

The reaction of acetylene with oleyl alcohol takes place at 150-180°C with the use of 1% KOH as catalyst. No pressure is necessary. A 4 cu. m. tower capacity produced 10-20 tons/day at 99% yield. The polymerized vinyl oleyl ether is used as a pour point depressant for lubricating oils, but Dr. Reppe could furnish no details of its effectiveness.

(5) Vinyl Phenols

In the aromatic series phenols and naphthols react as follows:



Phenol was treated with acetylene in the presence of zinc naphthenate catalyst at 10 - 20 atmospheres and about at its boiling point (182°C). The vinyl phenol polymerizes directly. By reaction of these compounds with hexamethylenetetramine a series of resins were produced with properties varying from thermo plastic to thermosetting depending on whether 1, 2 or 3 molecules of acetylene are reacted with the phenol. Koresin for synthetic rubber was made similarly, starting with butyl phenol.

(6) Acrylic Acid

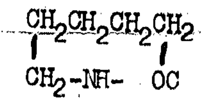
Acetylene in its isoform $H_2C = C<$ reacts with CO to give methylene ketene $H_2C{=}C{=}CO$ which in turn gives $H_2C{=}CH\ COOH$, acrylic acid.

In practice the reaction runs:

$$H_2O + C_2H_2 + 1/4\ Ni(CO) + 1/2\ HCl\ Aq =$$

$$H_2C = CH\ COOH + 1/4\ NiCl_2\ Aq + 1/4\ H_2.$$

Alcohols can be substituted for water to give the ester. Stoichiometric proportions of raw materials are used with the nickel carbonyl furnishing the CO; the presence of acid or halogen is necessary to bind the metal of the carbonyl as a salt. Technically 36% hydrochloric acid was used and the reaction ran

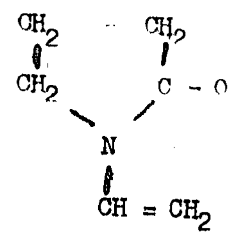


$CH_2CH_2CH_2CH_2$ / $CH_2-NH-\ OC$

for the polyamide known as Igamid B.

e. γ-Butyrolactone and N-vinyl Pyrollidone

1,4 butane diol may be dehydrogenated in a tower in the presence of copper strips at 200°C to form γ butyrolactone in 96% yield. Reaction with ammonia gives a 90% yield of pyrollidone which on treatment with acetylene furnished N-vinyl pyrollidone in 90% yield.



The N-vinyl pyrollidones are the bases of a new series of polymers, largely water soluble and of a character resembling albumen. They are used for glues and plastics but few uses have as yet been developed except the blood substitute "periston", described in report on the I.G. at Elberfeld. The degree of polymerization is controlled by the amount of hydrogen peroxide which is varied from 0.05 to 1.0%. The more peroxide used, the shorter is the polymer chain.

(1) Methods of Polymerization

(a) Block type

35 kg N-vinyl pyrollidone were mixed with 150 cc 30% peroxide solution and NH_3 equivalent to 1/2 the H_2O_2 and heated to 110°C. Without further heating the temperature went to 180-190°C. The hot polymer flowed out of the kettle and was cooled in blocks. The product has a yellowish color due to the high temperature, has a low K value (30-35) and contained about 10% monomer which was extracted with ether before solution of the final product in water and filtration.

"Periston" is a 2.5% solution of a product made in this way where the temperature is kept to 70-80°C.

These products can be used as glues and binders for films, for adhesives, thickeners for emulsions and solutions, and assistants in dyeing causing darkening of the color.

(b) Solution Type

To 30 parts N-vinyl pyrollidone in 70 parts water was added 0.5 parts 30% peroxide solution and 0.1% ammonia (100%) at 20°C. In presence of the oxygen of the air no reaction takes place, but in a stream of nitrogen it begins immediately and is complete in 2 hours. The product has a K value of 56.

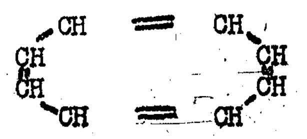
(2) Acetylene Polymers $(CH)_n$ Cyclopolyolefines

By condensation of an excess of acetylene under a total pressure of 10-20 atmospheres of which 5 atmospheres is nitrogen partial pressure and in the presence of a solvent such as tetrahydrofurane, cyclooctotetraene C_8H_8 was obtained at 60-70°C in liquid phase by means of a catalyst of nickel cyanide on a carrier with a yield of 90%. The reaction took place in a tower, both gas and liquid entering at the top. The liquid product was distilled and the tetrahydrofurane returned for reuse.

Small amounts of $C_{10}H_{10}$ and $C_{12}H_{12}$, soluble resins, and cuprene were also formed and particularly by raising the temperature the proportion of these can be increased. The best temperature for $C_{10}H_{10}$ is 80-90°C and for $C_{12}H_{12}$ about 130-140°C. Azulen forms as a by product when the temperature is 80-130°C.

Cyclooctatetraene	C_8H_8	BP_{760}	142-143°C	Golden Yellow
Cyclodecapentaene	$C_{10}H_{10}$	BP_2 BP_{760}	48-50°C 190-195°C	Deep Yellow
Cyclododecahexaene	$C_{12}H_{12}$	$BP_{0.5}$ BP_{760}	60-65°C 230-235°C	Bright Yellow
Azulen	$C_{10}H_8$	MP	99.5°C	Deep Blue

The constitutions of C_8H_8 and of Azulen are definitely determined, the former being:



The constitutions of $C_{10}H_{10}$ and $C_{12}H_{12}$ need further clarification.

The C_8H_8 and $C_{10}H_{10}$ have been investigated pharmacologically without finding any action. On the contrary, Prof. Kuhn

a. General Schemes for Development of Acetylene Compounds

Much of the framework of Dr. Reppe's ideas is contained in the three attached charts:

(1) Acetylene + Formaldehyde.

(a) γ- butyrolactone.

(2) Acetylene + Acetaldehyde.

b. Processes Reduced to Practice

(1) 1,4 butanediol (1,4 butylene glycol)

Several thousand tons/mo. of 1,4 butanediol were produced at Ludwigshafen, largely as an intermediate for butadiene. 1,4 butinediol was first made in a tower 18 m. high x 1.5 m. diameter containing 20 cu. m. of a contact mass of copper acetylide on silica gel where the copper compound is 10-12% of the contact mass. Five times the theoretical acetylene and formaldehyde diluted to 10% with liquor from the bottom of the tower were passed into the top of the tower under a pressure of 5 atmospheres of which 4 were due to acetylene and 1 to water vapor evaporated in the process. Temperature was 100°C. 1 cu. m. contact mass produced 1 ton of 100% butinediol per day.

The product was a 30% solution in water, which can be evaporated off and the diol crystallized from ethyl acetate. The diol can be distilled under ordinary pressure. Propargyl alcohol goes off in the first part of the distillation and can be recirculated with the formaldehyde.

The reaction is:

$$C_2H_2 + 2\ HCHO = HOCH_2\ C = CCH_2OH$$

A side reaction to give propargyl alcohol also takes place:

$$C_2H_2 + HCHO = HOCH_2C = CH$$

Yields are:

Acetylene	92% + 4% to propargyl alcohol
Formaldehyde	95%

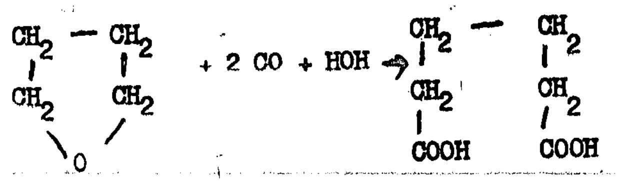
The 1,4 butinediol was hydrogenated by running it down a tower over copper nickel in the presence of hydrogen at 200-300 atmospheres and at 80-130°. A yield of 96% to 1,4 butanediol

was obtained with the balance going to butanol. Cost of 1,4 butanediol were 60 pfg/kg and it is expected that it may be reduced to 40.

c. Tetrahydrofurane and Adipic Acid

Tetrahydrofurane was produced from 1,4 butanediol in 92% yield by passing over a catalyst of phosphoric acid containing 1/2 - 1% phosphine at 260°C and 70 atmospheres pressure. The product is a good solvent and is known as Lösungsmittel T.

When tetrahydrofurane was reacted with two mols., carbon monoxide and one mol. water at 200 atm. and 270°C with 10% nickel carbonyl ($Ni(CO)_4$) a yield of 90% of adipic acid was obtained.



In practice the water containing 1% of its weight of iodine in the form of nickel iodide and the tetrahydrofurane in which the nickel carbonyl is dissolved were passed into the bottom of a tower with the CO gas. Adipic acid flowed out the top presumably with some tetrahydrofurane. The CO gas and nickel carbonyl passed to a separator to remove the latter and the CO returned to the tower. Throughput of the tower was 300-600 cu. m/hr. of the furane.

d. Propargyl Alcohol

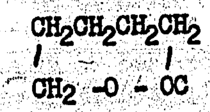
The reaction for 1,4 butinediol can be run to give 80% propargyl alcohol instead of 4% if desired. By hydrogenation with an iron catalyst under pressure, propargyl alcohol was converted to allyl alcohol and with a copper catalyst to propionaldehyde.

Oxidation of propargyl alcohol with air at 30°C over a cupric chloride catalyst gave hexadiindiol 1,6

$$HOH_2C - C \equiv C - C \equiv C - CH_2OH$$

which on further reduction goes to the hexanediol 1,6.

By conversion of the hexanediol to the double aldehyde followed by an internal Canizzaro reaction ε-caprolactone



is formed which is converted with ammonia to the ε-caprolactam

j. Thiodiglycol (Oxol)

The thiodiglycol plant had never been run and Dr. Hagen was unable to give any data as to production capacity. An aluminum tower, 6 m high x 0.66 m dia., packed with Raschig rings and equipped with cooling and heating coils is filled with thiodiglycol from previous manufacture. Hydrogen sulfide gas and ethylene oxide are passed in at the bottom maintaining a temperature of 90°C. The product overflows continuously to a cooler and to storage. Complete absorption of the gases is obtained.

If it is desired to purify the thiodiglycol, it is dried under vacuum and treated with just enough ethylene oxide to unite with the hydrogen sulfide present as determined by titration.

k. Acetaldehyde from Acetylene

Acetaldehyde was made from acetylene in two units and the crude product was purified in a continuous fractionating column.

Production from the two units amounted to 1800 T. aldehyde per month.

The essential reaction is:

$$HC \equiv CH + H_2O \longrightarrow CH_3CHO$$

This reaction was carried out in two vertical cylinders about 2 m. diameter by 8 m. high. These cylinders are made of V2A and contain no packing. The heads are designed to act as entrainment separators.

A layer of mercury stays in the bottom of the reactor at all times. The reactor was charged with a batch consisting of:

3000 kg $FeSO_4$
1600 kg H_2SO_4
8400 kg Water

Acetylene in the amount of 860 M^3 per hour and under a pressure of about 25 p.s.i.ga. was introduced below the level of the mercury which is thereby agitated until there was a mercury emulsion throughout the batch. Live steam was introduced near the bottom to help the agitation and to keep the batch temperature at about 95°C. Approximately 1 T. of steam was required per batch.

The vapors from the top of the reactor were cooled to 40°C by passage through a water cooled condenser. They then passed to a mercury separator from which mercury and condensate passed back to the reactor tower. Mercury lost by this process amounted to 1 kg per T of aldehyde produced.

Vapors from the mercury separator passed to a water scrubber packed with Raschig rings in which the aldehyde and a trace of acetic acid were washed from the gas. The scrubbing liquor from the bottom contained about 7.5% aldehyde plus a trace of acetic acid. It was sent to a continuous fractionating column. Gases from the top of the scrubber, chiefly acetylene, were recycled to the reactor.

The aldehyde solution corresponding to an acetylene feed rate of 860 M^3 per hour amounted to 21770 kg per hour. It passed through a heat exchanger, a steam heater, and was fed to the 21st plate of a 29 plate column. This column is 2 m in diameter by 10 m high, from the feed plate down, it is constructed of V2A. Above the feed plate it is cast iron. The top column temperature was 21°C, the boiling point of pure acetaldehyde. The reboiler temperature was 100°C. The residue from it was water plus a trace of acetic acid. The reflux ratio was constant at 2/1. The aldehyde taken off was degassed in a small column with a reboiler at the bottom. The acetylene recovered was put back in the recycle gas. The final product from the reboiler was 99.9% acetaldehyde.

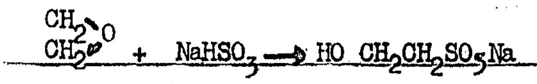
During the reaction the ferric iron content of the catalyst solution gradually decreased until a point is reached at which conversion fell off. The catalyst was then drawn off to a settling tank in which the mercury settled out. It then passed down through a brick lined tower packed with ceramic Raschig rings in which it was blown with live steam. Next it went to a second settling tank in which more mercury was recovered, and from there to the oxidizer. In the oxidizer the ferrous iron was oxidized continuously to ferric with nitric acid. On leaving the oxidizer, the solution was dark brown due to the presence of $FeSO_4.NO$ which was broken down by air blowing. The oxides of nitrogen from the blowing were recovered by water scrubbing in 3 towers in which the water was recycled counter current to the gas flow. The 15% nitric acid thus obtained was blended with fresh acid to a concentration of 25% and used again in the oxidizer. The catalyst solution after air blowing was ready for reuse.

3. CHEMICALS PROJECTED FROM ACETYLENE

Dr. Reppe removed his laboratory to Gendorf when it was bombed out at Ludwigshafen last fall. Dr. Reppe is a very creative research worker on the reactions of acetylene and has evolved a large number of schemes, few of which have been put into operation.

the ethylene oxide introduced until the batch shows the correct titre, sp.gr., mol. wt., etc.

The preparation of "Igepon O" may be outlined as follows:



$$\begin{matrix}CH_2\\CH_2\end{matrix}\!\!>O + NaHSO_3 \longrightarrow HO\ CH_2CH_2SO_3Na$$

$$RCOOH\ (\text{oleic acid}) + ClCOCl \rightarrow RCOCl + H_2CO_3$$

$$RCOCl + HO\ CH_2CH_2SO_3Na \longrightarrow RCOOCH_2CH_2SO_3Na$$

"Igepon A"

"Igepon A" decomposes at about 100°C; therefore it cannot be used if laundry must be boiled.

"Igepon G" was not made from ethylene oxide. Its preparation is outlined in section 6 of this report (miscellaneous chemicals).

g. Emulsifiers

Compounds made from 20 ethylene oxide molecules and one fatty alcohol molecule are emulsifiers sold as "Emulphor C" and were used in the preparation of emulsions used for spinning wool.

One oleic acid molecule combined with 6 ethylene oxide molecules gives Emulphor A, used for emulsifying machine oils like spindle oil.

Castor oil plus 40 ethylene oxide molecules gave "Emulphor E.L."

h. Textile Aids

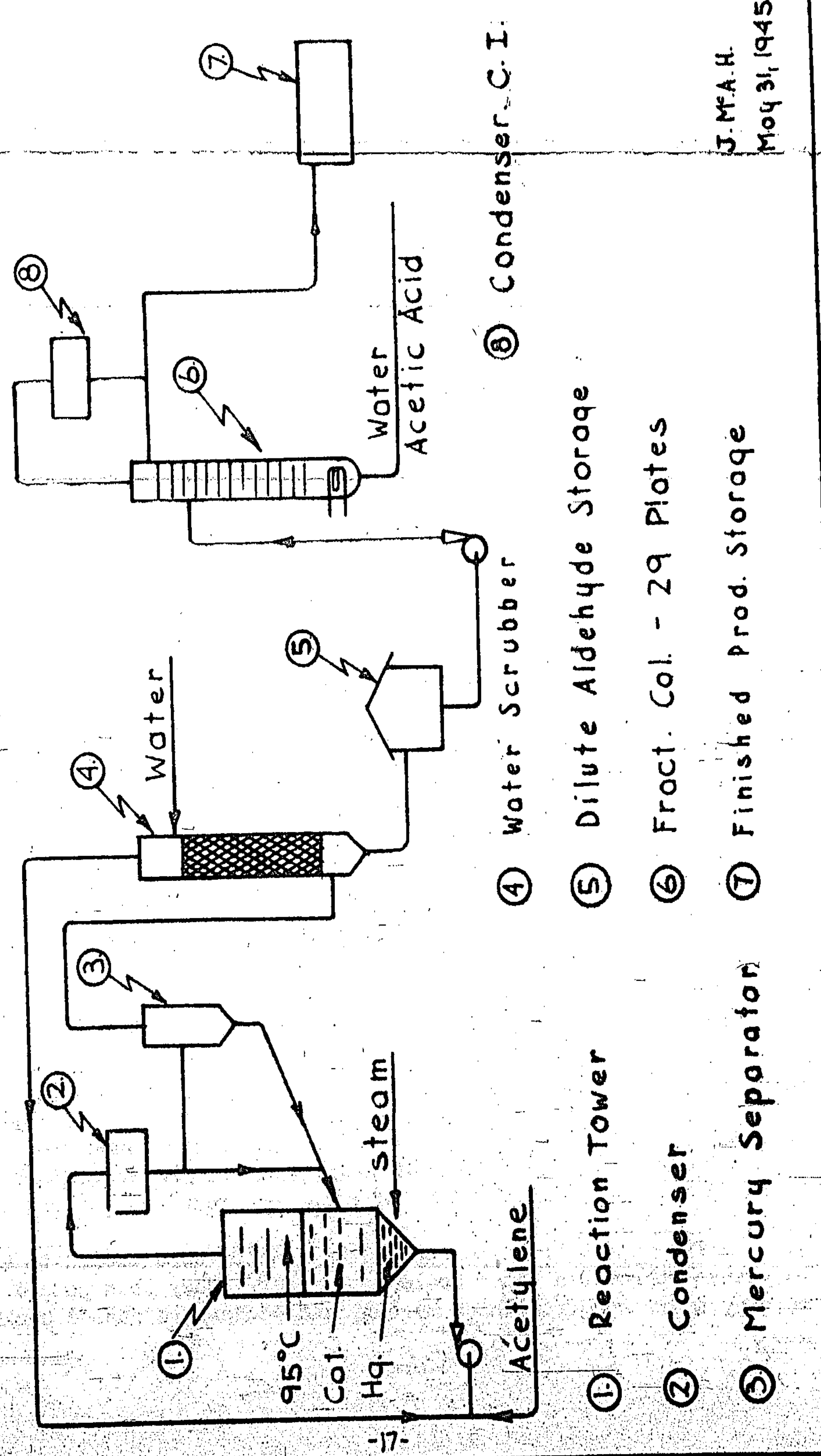
Compounds of 20 ethylene oxide molecules with one fatty alcohol molecule, were used as levelling agents for dyestuffs and were sold as "Polätinechtsolze." A 33% water solution of this material was sold as "Diazopon"; a 20% solution as "Peregal O."

Stearic acid combined with ethylene oxide gave "Soromin S.G.", which was sold as a softener for artificial silk.

i. Waxes

Waxes may be made by the polymerization of ethylene oxide - the so-called "polyethylene oxide waxes." None have been prepared at Gendorf lately.

Figure III
Acetaldehyde Equipment



Gendorf operating personnel believed that future chlorhydrin reactors should be made smaller and that all the chlorine should be introduced below the ethylene inlet to suppress the formation of dichlorethane. The latter modification is based on the theory that if all the chlorine has reacted with the water before ethylene is introduced, reactions I and II occur. If chlorine comes in direct contact with ethylene reaction III takes place.

d. Preparation of Ethylene Oxide

Ethylene oxide was prepared from the chlorhydrin by the following reaction:

$$\begin{array}{lllll} \text{I} & 2C_2H_4\,(OH)\,Cl & \longrightarrow 2C_2H_4O & + & 2HCl \\ \text{II} & 2HCl + Ca(OH)_2 & \longrightarrow CaCl_2 & + & 2H_2O \end{array}$$

The reaction was carried out in the equipment sketched in Figure II. There are four of these "saponifiers", each connected directly to one of the four chlorhydrin units described in Figure I. The chlorhydrin, HCl, water mixture and milk of lime were fed to the saponifier through concentric feed pipes in the proportion of 2 vol. chlorhydrin mixture to 1 vol. 12% $Ca(OH)_2$ slurry. The resulting reaction slurry was led through a long tortuous path in the saponifier on baffled, steam heated plates as shown. Ethylene oxide and steam distilled out the top of the reactor through the dephlegmator which removed some of the steam. A calcium chloride - calcium hydroxide solution was drawn off from the bottom.

The ethylene oxide-water mixture was fractionated in a three column, continuous distillation unit. The first two columns operated in parallel and received the ethylene oxide - water mixture from the "saponifier." They contain 50 bubble cap plates and received the feed on the 17th plate. The reboilers were steam heated to 55°C. The top column temperature was 12°C. These two columns were operated with a reflux ratio of 1/1 and produced 40-50 T. per day of 98% ethylene oxide.

Residues from the first two columns were fed to a third column having a reboiler at 80°C. The water was removed from this reboiler.

Vapors from the third column were fed to the first two columns on the 33rd plate.

The yield of ethylene oxide based on ethylene amounted to 70 - 75% of the theoretical.

All three columns were made of cast iron.

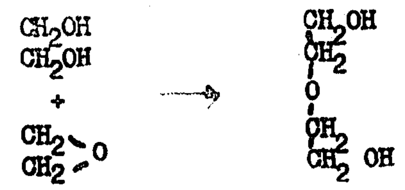
Dichlorethane was removed from the bottom of the third column, washed with sulfuric acid, hydrochloric acid, and caustic soda solution and finally distilled batchwise.

e. Preparation of Glycols

Ethylene glycol was prepared by the reaction.

$$C_2H_4O + H_2O \longrightarrow C_2H_4\,(OH)_2$$

Steam and ethylene oxide were passed up through an iron column packed with steel Raschig rings. The ethylene glycol produced was purified by fractionation. By altering the temperature, pressure, and feed ratios the same equipment can be used for the production of diglycols, for example:



f. Detergents

"Leonil" detergents were made from long chain alcohols and ethylene oxide. The type reaction is:

$$R.CH = CH\ .OH + C_2H_4O \longrightarrow RCH = CHO\ CH_2CH_2OH$$

The compound made from 8 ethylene oxide molecules and one alcohol molecule is water soluble and is a detergent. The compound made from stearyl alcohol was made up as a 30% water solution and sold as "Leonil O Lsg." or Genapol. It was actually being sold in considerable quantities because of the soap shortage. As it contains no sodium it cannot form salts and may, therefore, be used with hard or soft water. It is excellent for wool scouring in either acid or neutral media. When used for domestic laundry soda is added. It does not foam and therefore does not sell readily.

As of May 19, 1945, enough raw material was available at Gendorf to prepare 42 T of Genapol (weight of water included). For subsequent production it will be necessary to prepare the fatty alcohols by hydrogenation of fatty acids.

These compounds were prepared in an enamel lined kettle under 45 p.s.i.ga. and at 165°C. The fatty acid was charged and

The catalyst was silica gel containing traces of palladium: the exact amount of palladium not being known as the catalyst was prepared at Ludwigshafen. It was estimated at 4-8 parts per million. Catalyst life was about 12 months.

The exit gas after cooling contained 65% ethylene and was free from acetylene. It was dried over silica gel and liquefied by the Linde Process. A three column system was used. The partially liquefied mixture was fed to the middle of the first column from the bottom of which high boiling compounds (C_4-C_8) are removed. Hydrogen, methane and nitrogen were removed from the top of the second column from the bottom of which liquids passed to the side of the third column. Ethylene passed from the top of the third column and ethane from the bottom.

A second fractionation of the ethylene will give material free from ethane and having a purity of 98% but at an appreciable extra cost.

The output of the Gendorf plant was 25,000 to 30,000 T. ethylene per year.

c. Preparation of Ethylene Chlorhydrin

Ethylene chlorhydrin was prepared by the action of chlorine and water on ethylene. The equipment is sketched in Figure I. Ethylene was fed to the side of the reactor and chlorine at points above and below the ethylene. The following reactions occurred:

I $Cl_2 + H_2O \longrightarrow HClO + HCl$
II $HClO + C_2H_4 \longrightarrow C_2H_4(OH)Cl$ (ethylene chlorhydrin)

There was also a side reaction:

III $C_2H_4 + Cl_2 \longrightarrow C_2H_4Cl_2$ (dichlorethane)

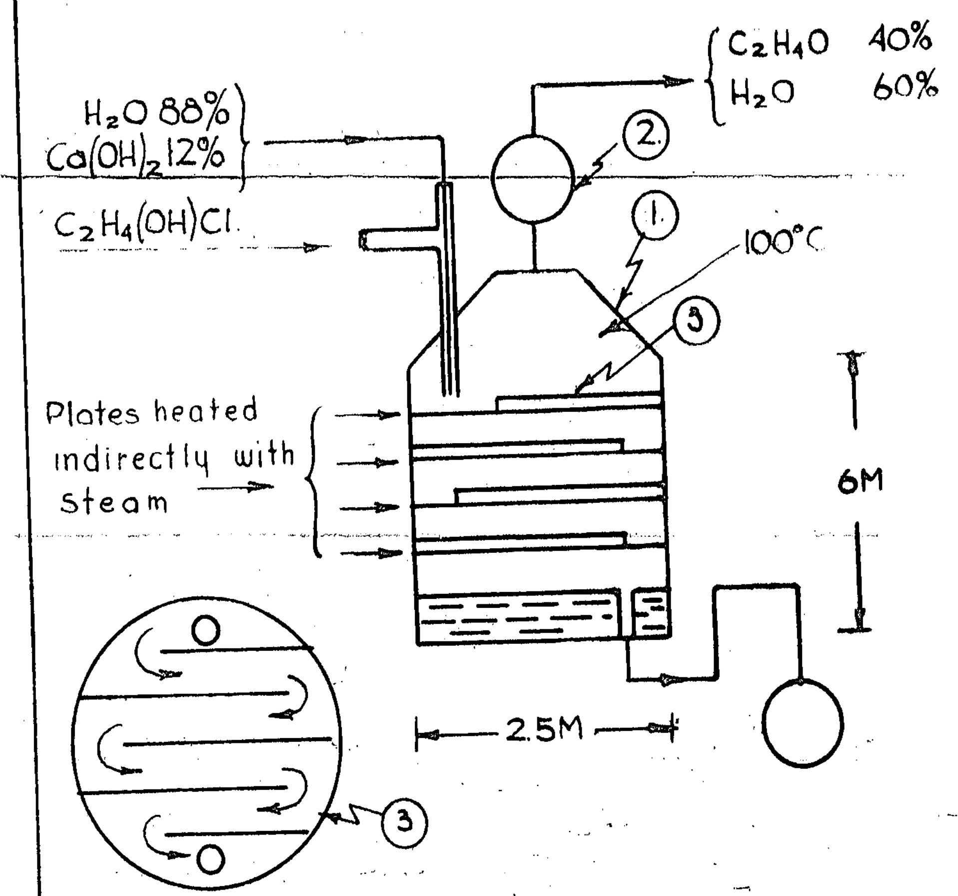
Four of the units described (Figure I) required per hour:

2000 M^3	Ethylene	(gas)
1000 M^3	Water	(liq.)
6000 kg	Chlorine	

The chlorhydrin produced was not isolated but was hydrolyzed immediately to ethylene oxide. The yields of chlorhydrin were not known but the above feed rate produced 2.7 T. of ethylene oxide per hour.

Dichlorethane in the amount of 0.7 T. per hour was produced simultaneously.

Figure II
Ethylene Oxide Equipment



① Saponifier C.I. 2.5MΦ x 6M
② Dephlegmatter to reduce loss of water with product
③ Plates, steam heated, with vertical baffles to give long tortuous path for reaction mixt.

J. McA. H.
May 30. 1945

the two cylinders. The $Ca(OH)_2$ discharged contained about 5% water and could be readily handled by a screw conveyor. The acetylene was dried by passage over the incoming carbide, passed through cyclone dust separators and finally emerged with a purity of 98%. The chief impurity was nitrogen with which the carbide was blanketed during shipment. According to Dr. Gruber, the operation of the "dry" generator had been unsatisfactory and several difficulties (e.g. the steady feeding of the carbide) had not been eliminated.

Acetylene from carbide was pure enough for the preparation of acetaldehyde, but before it could be hydrogenated to ethylene it must be purified as follows:

I - Scrubbed with dilute chlorine water to remove H_2S and PH_3.
II - Scrubbed with a solution of caustic soda to remove chlorine and for partial drying.

An attempt was made to purify the acetylene by passing it through active carbon but the process was unsuccessful and was abandoned.

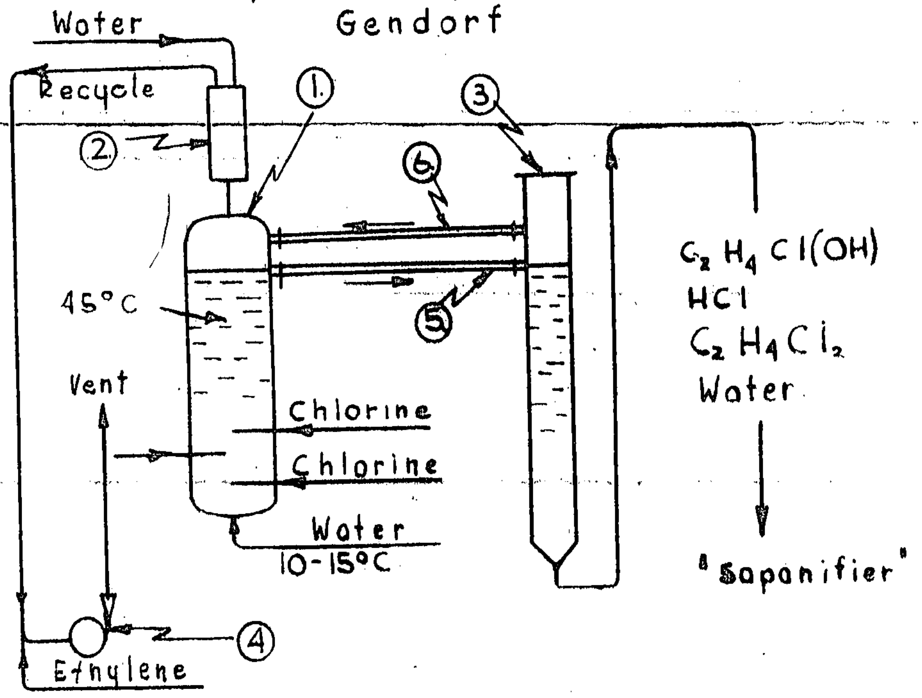
Carbide was used in the amount of 150,000 tons per year - equivalent to about 47,000 tons of acetylene.

b. Preparation of Ethylene from Acetylene

Ethylene was prepared by the hydrogenation of acetylene. The acetylene was prepared and purified as described above. The hydrogen was a mixture of electrolytic hydrogen and hydrogen prepared by the "steamiron" process. It required no purification as its chief impurity was carbon monoxide, which was hydrogenated to methane and did no harm.

The reaction was carried out in 22 converters arranged down two sides of a large building, 20 of these manifolded so that they may be operated in parallel. All the production of these 20 was passed through the other two in series for the removal of the last traces of acetylene. The converters were vertical cylinders about 10 ft. in diameter by 15 ft. high. Inside each cylinder were alternate layers of catalyst and bubble cap plates. The plates act as gas distributors and did not support any liquid. The gas mixture recycled through the converters contained about 5% acetylene, 8% hydrogen, 65% water vapor and 22% ethylene. The converters were operated at 7-10 p.s.i.ga. and at a temperature of 180-320°C depending on the age and activity of the catalyst.

Figure I
Ethylene Chlorhydrin Equipment
Gendorf



① Reactor, Bricklined C.I. 1.6Mϕ x 12M
② Condenser-Scrubber; Bricklined C.I.
③ Still Leg - for degasification of product
④ Blower - to recycle gases
⑤ Pipe conveying liquid-gas mixture ~~to still Leg~~.
⑥ Pipe returning gases to reactor.

J.McA.H.
May 28, 1945

(a) Main Laboratory

Pharmaceutical products:

Ether pro narcosi
Ethylchloride pro narcosi
Mitigal (Dimethylthianthrene)
Hartosol (Isopropylalcohol)
Kresival (Calcium salts of cresolsulfonic-acid)
Zephirol (Dimethyl-benzyl-alkyl-ammonium-chloride)
Phanodorm (5-ethyl-Δ-1-cyclohexenylbarbituric-acid)
Evipan (3.5-Dimethyl-5-Δ-1-cyclohexenyl-barbituric-acid)
Eunarcon (3-Methyl-5-β-bromallyl-5-isopropyl barbituric-acid)
Novocain
Adrenalin
Acetylphenothiazine
Avertin (1.1.1-Tribromoethanol-2)
Insulin
Postonal
Abasin (1-Acetyl-3-(bromodiethyl)-acetyl-carbamide)
Periston (Poly-N-vinylpyrrolidone)
Sulfonamide:

Prontosilum album
Pyrimal
Eleudron
Eubasin

Urotropine (Hexamethylentetramine)
Camphor oil
Barium sulphate

(b) Organic Intermediates:

Formaldehyde from methanol
Ethanol from Ethylene
Octadecylalcohol from vegetable oils by high pressure hydrogenation
Higher alcohols from long chained olefines
Ester of Oxymethylvinylketone from butin-2-diol-1.4

(c) Plasticizers, lacquers and plastics section:

Resins of phenol formaldehyde (Luphen-L) with or without plasticizers
Resins of phenol acetylene for plastics
Resins of melamine formaldehyde by reaction of dicyandiamide with melamine (nitrogen calcium)
Organic thioplastics (Thiokol a.s.o.)
Ethyl cellulose

(d) Textile auxiliaries:

Igepon A with intermediate products
Igepon T from Ethylamine including intermediates.
Igepon from monoethanolamine

(e) Inorganic auxiliaries:

S_2Cl_2 and SCl_2

Interviewed: Dr. Ambros.

2. CHEMICALS MANUFACTURED FROM ACETYLENE

a. Preparation of Acetylene

Acetylene was made at Gendorf from calcium carbide produced locally (Trostberg). In spite of the high cost of the carbide (180 RM per T) it is claimed that ethylene (See b below) made from acetylene was cheaper than ethylene made from ethanol.

Acetylene was generated by both the "wet" and the "dry" gasification processes. The "wet" process, in which carbide was fed to water in an agitated vessel and the resulting lime slurry thrown away, is old and needs no further description. The generator used for the "dry" process consisted of a rotating cylinder containing an inner wire mesh cylinder about 8 inches smaller in diameter. The two cylinders rotated about an axis inclined about 8° from the horizontal after the manner of a rotary kiln drier. Carbide was fed in lumps about the size of a walnut to the upper end of the generator and was tumbled about on the rotating screen. Water was added at five different points causing the evolution of acetylene and the lumps of carbide to crumble to a dust which passed through the wire mesh inner cylinder and rode to the lower discharge end of the generator through the annulus between

(8) Inorganic-Chemicals

Chlorine	2,500
Caustic soda	3,000

(9) Pharmaceuticals

Principal products in kg per month:

Ether	5,000
Ethylchloride	7,500
Hartosol	5,000
Postonal	1,000

Specialities in g per month:

Kresival	20,000
Zephirol	5,000
Phanodorm	1,000
Evipan	1,000
Eunarcon	500
Novocain	1,000
Adrenalin	1,000
Acetylphenothiazine	500
Avertin	1,000
Insulin	according to delivery of glands
Periston (Solution 3,5%)	according to delivery of butandiol-1,4.

f. Research

(1) Organic Section

Formation of Butadiene out of Ethanol
Glycol-ethers
Vinyl-chloride
Alcohols of Acetaldehyde
Dulcin
Textile auxiliaries
Ethylbenzene
Styrene
Polystyrene
Lacquers (Resins)
Ethylchloride
Chlorination of benzene
Chlorination of fatty acids

Polyethylene
Oxidation-experiments (ethylbenzene and others)
Rubber-compositions
Chloral
Insecticides (chloral and chlorobenzene)
Purification of crude ethylenechloride
Aldol
Ether-for-narcose
Crotonaldehyde
Crotonicacid

(2) Sales management

Preparation of varnishes and lacquers based upon

Nitrocellulose
Phenolic-resins
Polyvinylchlorides

Plasticizers
Solvents
Resins
Shoe-polish
Floor-polish
Glues
Esters of diglycolicacid as plasticizers
Polymerization of crude butylene
Textile auxiliaries as:

Detergents
Waterproofing-agents
Softeners

(3) Inorganic Section

Water electrolysis
Carbonmonoxide
Production of charcoal
Regeneration of potassium permanganate
Silver catalyst for formaldehyde synthesis
Basic Aluminium chloride
Phosgene (for organic synthesis)
Thionyl chloride (for organic synthesis)
Chlorosulfonic acid
Ammonium nitrate
Aluminium acetate

Soda	65.4	tons
Sodium bisulphite as SO_2 100%	19	"
Hydrochloric acid as HCl 100%	36	"
Graphite	290	"
Mercury	62.7	"
Sulphuric acid as SO_3	127	"
Chlorine	90	"
Caustic soda in solution as 100%	350	"
Ferrous sulphate	32.5	"
Nitric acid 62%	8.4	"
Methylated spirit	270.4	"
Phenylglycine	43.6	"
Coke	80	"
Diethylbenzene	122	"
Sulphur	950	"
Chloride of lime	27	"
Caustic potash	10	"
Calcium chloride	10	"
Methanol	0.3	"
Ethylene chloride crude	171	"
Ethylene chloride pure	208	"
Glycol	565	"
Diglycol	677	"

Aluminum chloride	61	tons
Copper sulphate	2.5	"
Adipic acid	30	"
Ethylglycol	11	"
Ceresin	2.5	"
Urea techn.	5	"
Resin	6.7	"
Naphthlene	2	"
Octadecylalcohol	2.5	"
Castor oil	9.3	"
Sesame oil	20	"
Rape-seed oil	4.5	"
Olive oil IIIrd quality	4.5	"
Paraffin	20	"
Stearic acid	11.6	"
Benzene	48	"
Active carbon	5	"
Orthodichlorobenzene	15	"
Carbon tetrachloride	2.5	"
Phosphorus-trichloride	2.3	"
Triglycol	114.8	"
Glysantin with tri. (Prestone)	65.7	"
Carbide	3.108	"
Acetaldehyde	380	"

d. <u>Supplies Needed</u>

	From:
Coal	Pensberg or Hausham
Coke	Munich or other municipal cokeries
Carbide	Hart
Salt	Heilbronn near Stuttgart
Oils	Stores in Passau, Töging and Regensburg
Toluene Benzene Phenol	Ruhr Territory
Ammonia Nitric acid	Middle Germany

Sulphuric acid	Kelheim near Regensburg
Nitrocellulose	Aschau
Peroxide of hydrogen	Kraiburg
Solvents	Burghausen
Plastics	Burghausen
Urea	Oppau
Formamide	Oppau
Methanol	Regensburg

<u>Production Capacities in tons/month</u>

(1) <u>Lacquers</u>

Luphene	35
Phtalopal	100

(2) <u>Plasticizers</u>

Palatinol	50
Soromine	10

(3) Plastics

Lupolene	5
Luvitherme	10

(4) <u>Solvents</u>

Glycol	1,000
Glycolether (Ethyl, butyl)	100

(5) <u>Detergents</u>

Leonil	50
Igepon	125

(6) <u>Waterproofing Agents</u>

Ramasite	20

(7) <u>Organic Chemicals</u>

Ethylchloride techn.	90
Ethylenechloride	400
Acetaldehyde	2,000

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PERSONNEL OF INSPECTION TEAM

Lt. Col. P.R. TARR	CWS,Hq ETOUSA
Col.J. H. ROONEY	M. of S.
Lt. Col. S.B. CORMACK	M. of S.
Lt. Col. J.W. CRAWFORD	M. of S.
Major T. LOVE	M. of S.
Major H.N. RYDON	M. of S.
Mr. V. C. BIDLACK	CWS,Hq ETOUSA
Mr. F. J. CURTIS	CWS,HQ ETOUSA
Mr. J. M. HARRIS	CWS,Hq ETOUSA
Mr. I. H. JONES	Petroleum Attache
Mr. G. M. KLINE	OD, Hq ETOUSA
Mr. E. B. PECK	Petroleum Attache

ANORGANA GmbH

WERK GENDORF

MISCELLANEOUS-CHEMICALS

1. GENERAL

Anorgana GmbH was started as a subsidiary of I.G. Farbenindustrie AG in June, 1940 and financed by Montan Industrie Werke AG, a subsidiary formed by the German Government to handle such matters. The cost of construction was 120,000,000 marks. During the war and while construction was going on 4000 workers were employed, partly foreign. It is estimated that normally 2000 workers would be needed for full operation and for the present situation about 1000 could be used.

a. Organization

Names and positions of officials of the Anorgana GmbH:

Managing director	Dir. Dr. M. Wittwer
Inorganic department	Dr. M. Gruber
Organic department	Dr. G. Hagen
Sales Service department	Dr. J. E. v. Klenck
Accounting	W. Jansen
Social department	Dr. E. Lederle
Ordnance department	Supt. K. Wurzler

Name and position of official of I.G. Farbenindustrie AG:

Research and pharmaceuticals --- Dir.Dr. W. Reppe

b. Present Daily Manufacture

Leonil (detergent)	300 kg
Luphen (lacquer)	1,000 kg
Ether (narcotic)	250 kg
Ethylchloride techn.	3,000 kg
Ethylchloride pro narcose	250 kg

c. Raw Materials and Finished Products on Hand

Common salt	2.477 tons	Formaldehyde 30%	30 tons
Barium carbonate	354 "	Ethylene oxide	52 "

RESTRICTED

ANORGANA GmbH WERK GENDORF

GENDORF, GERMANY

MISCELLANEOUS CHEMICALS

18-19 May 1945

Reported by:

V.C. BIDLACK - TIIC Chemicals
F. J. CURTIS - CWS, Hq ETOUSA
J. M. HARRIS - CWS, Hq ETOUSA

12 June 1945

CIOS Target Numbers 8/86, 22/1 (f), 22/534
Chemical Warfare

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

38 p. diagrs.

F.P. copy 1

ITEM No. 8.22

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Unclassified 10/15/45

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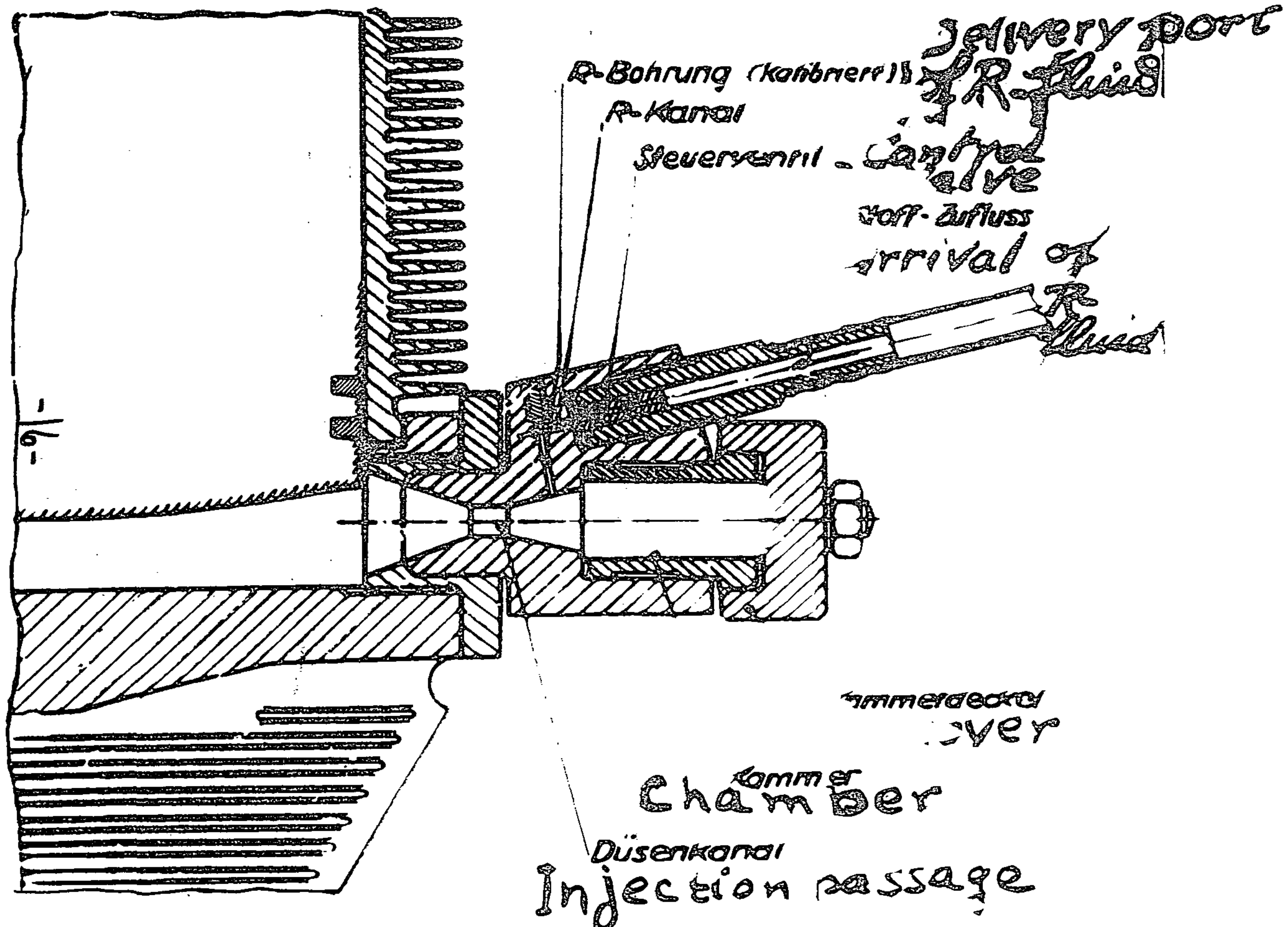
ANORGANA G.M.B.H. WERK GENDORF

Bidlack, V. C.

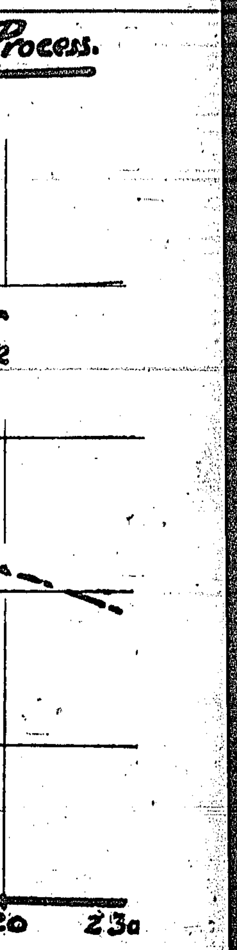
RESTRICTED

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE



Pumpenloses Einspritzgerät am Hirth …Zylinder

Figure 7. Pumpless Injection. Hirth [illegible] cylinder



3. Flight Tests with the Ring-Process. (b)(cont'd)

hour per cylinder, that is 15 to 23 mm3 per stroke. At no-load the use of R-fluid increased to 40 mm3 per stroke.

The Ring-process was used in the middle engine of a group of three on the plane. Tests showed that the high rate of cooling would cause difficulties, and a different set-up had to be used.

(c) The next series of tests was therefore run, also in a Junker JU-52 but with a BMW engine having a compression ratio of 1:9 instead of 1:8. Average fuel consumption was 185 grams/HP. R-fluid consumption was 18 to 23 mm3 per stroke per cylinder and again 40 mm3 at no-load. It can be said that the R-fluid consumption is from 5 to 10 grams per HP.

Unfortunately this plane could not go above altitudes of 5,000 meters, where difficulties were really expected due to excessive cooling, and further tests were planned. But in the winter of 1943-44 perhaps due to some improvement in the spark ignition technique in Germany, perhaps due to greater interest in other types of plane propulsion, it was decided to interrupt this work on account of what was termed "more pressing interests".

4. Problems Related to the Ring Process.

Two (2) problems were given consideration in connection with this process. The first is the problem of starting the engine, especially at low temperatures. It soon became clear that a set of auxiliary spark plugs, used only for starting, were an indispensable accessory of the Ring-process, together with battery, distributor, and all the electrical equipment necessary.

The second problem was one of reducing the cost of installation of the Ring-process, definitely higher than the cost of spark-ignition, by eliminating some of the injection equipment. The Hirth Motor Company adopted an injection system developed by Prosper l' Orange for small Diesel engines, and similar to the pre-gasification injection of the Cummins Engine Company in the U.S.A. It is illustrated on Figure 7.

4. Problems Related to the Ring-Process. (cont'd)

1. During the suction stroke, the operating valve opens and the delivery port is filled with R-fluid.
2. During the compression stroke, the gases passing through the Venturi into the chamber create a suction which causes the R-fluid to flow out of the delivery port into the chamber; there it is heated and ignites.
3. The flame created in the chamber comes out through the injection passage into the main combustion chamber and ignites the air-fuel mixture. The ignition of the R-fluid in the small chamber takes place at 10 to 20 degrees before Top Dead Center.

This solution worked out without mechanical difficulties, caused however a slightly higher consumption of R-fluid.

5. Conclusion.

It can be said that the main advantage of the Ring-process compared to the spark ignition method lies in the elimination of spark plug difficulties, serious in Europe at the time of the development of the process. A saving of fuel of approximately 5 percent can also be expected as a result of more knock-free performance and of more economical use of fuel in the "lean mixture operating periods. The fact that less cooling is necessary may be considered either an advantage or a disadvantage inasmuch as a careful control of the cooling process must be provided to avoid excessive cooling and resultant failures. The cost of installation of an R-fluid injection system must be compared to the cost of electrical ignition, especially when it is considered that electrical ignition is required for starting at the present stage of the development.

An exact evaluation of the process can only be made after giving due thought to all new processes related to power production for aircraft: Diesel engines, jet-propulsion, turbines. In that light, the Ring-process appears to be a minor development.

Prepared by:

R. J. Bender,
Lieut. USNR.

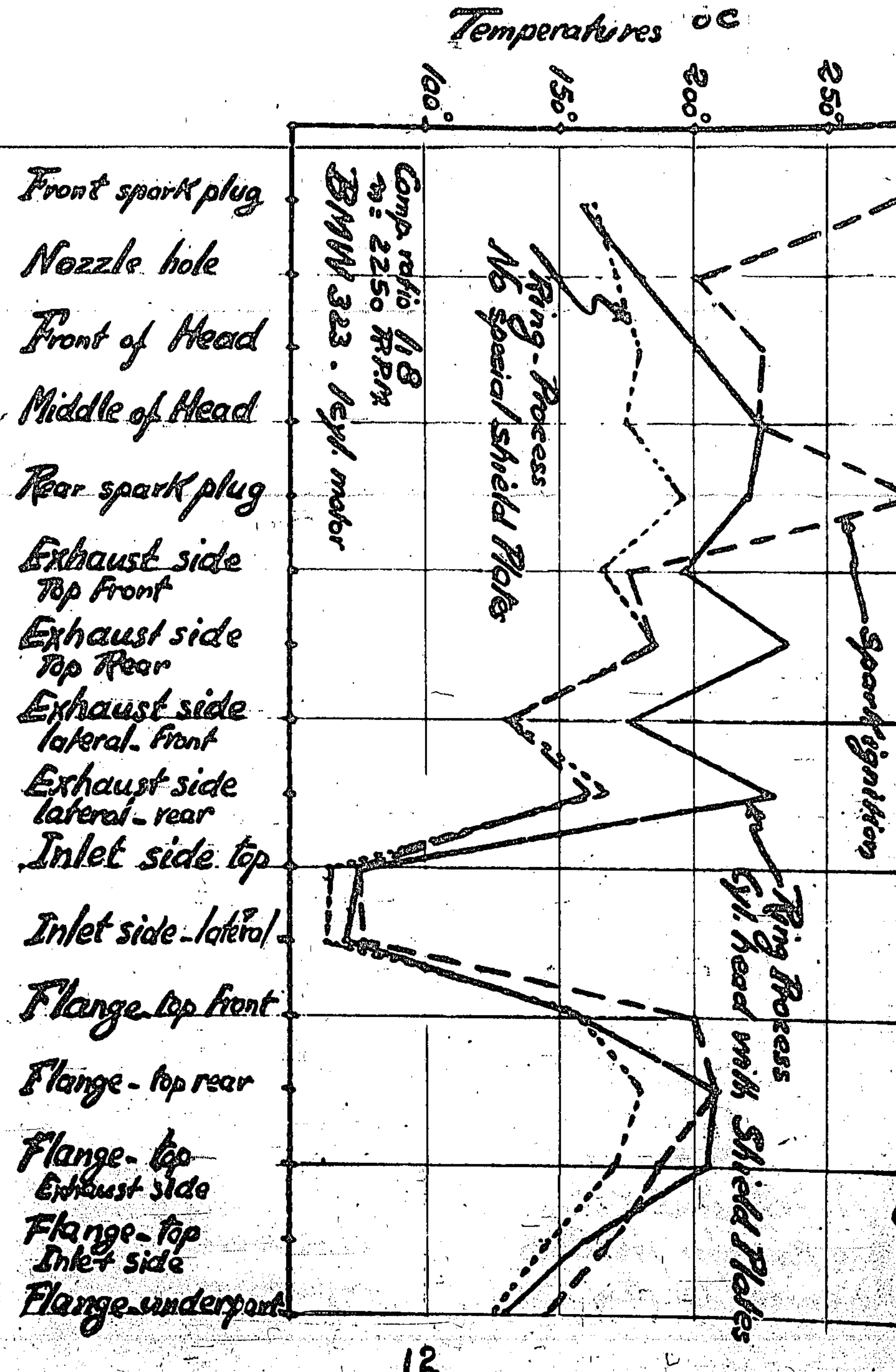


Figure 5.

Metal Temperatures.

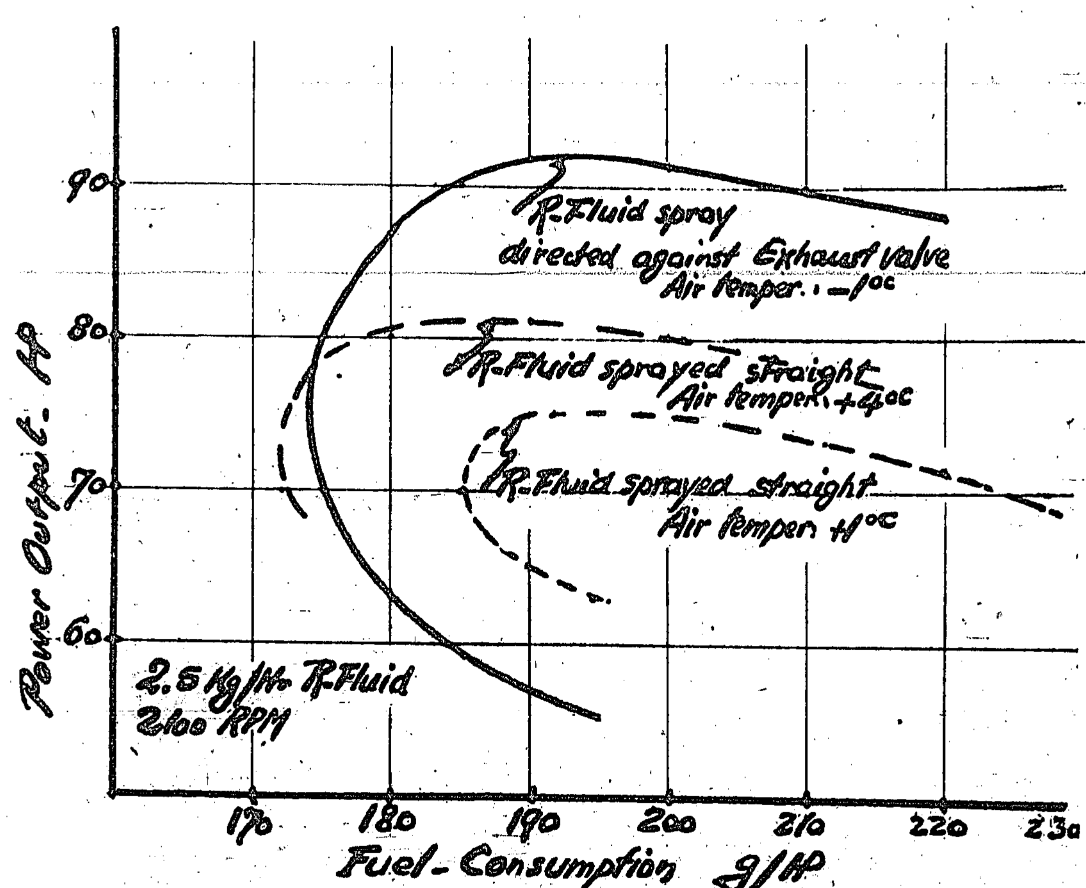


Figure 6.

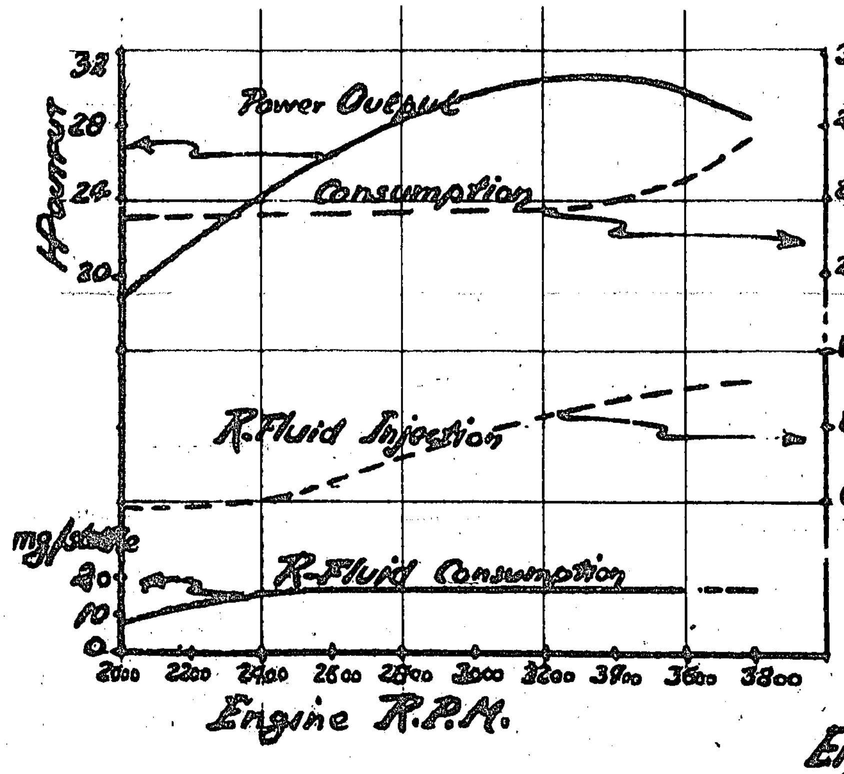
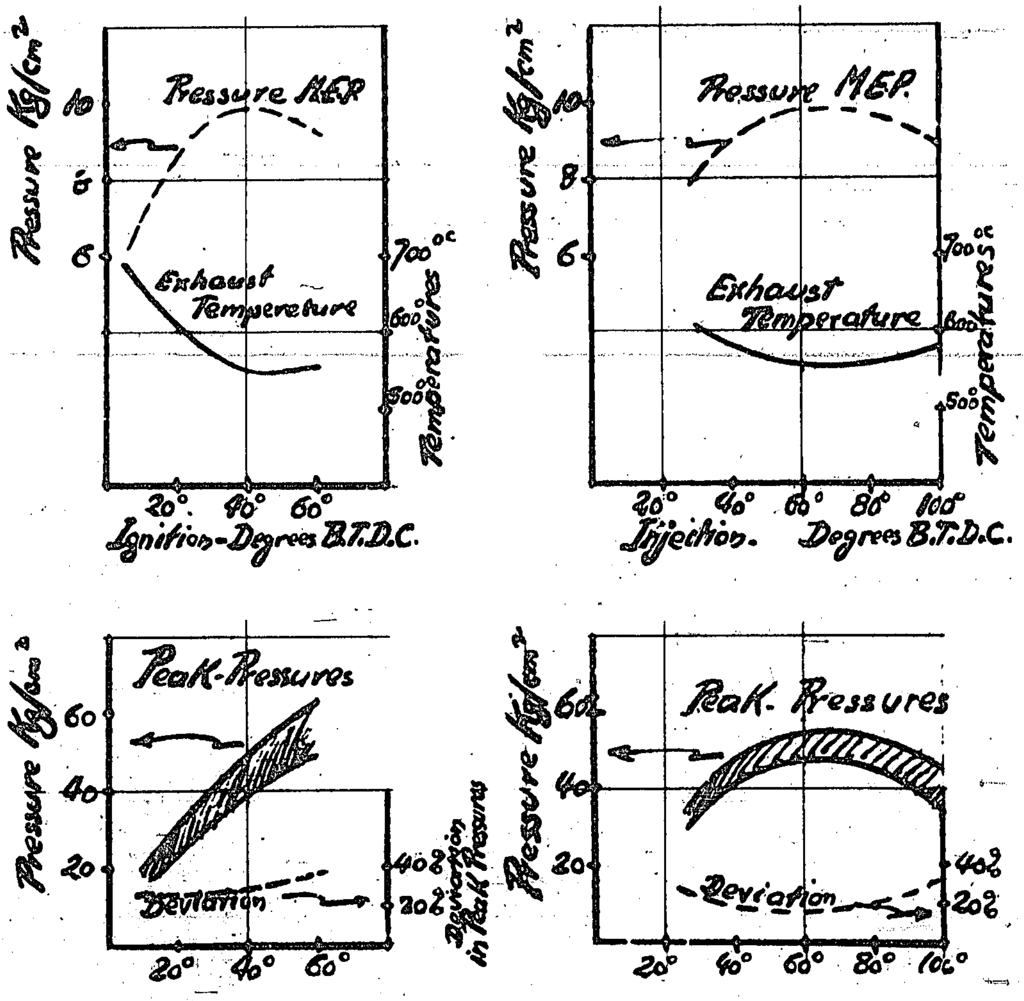


Figure 3.
Influence of
Engine Speed

Figure 3.



A- With Spark plugs.
Peak Pressures rise with
Advanced ignition.-

B- Ring-Process.
Peak Pressures are
proportional to M.E.P.
Figure 4.

2. Various Features of the Ring-Process (c)(cont'd)

pressures noted in the spark-ignition process and in the Ring-process, when varying ignition or R-fluid injection timing, is shown on Figure 4. Examination of the curves shows that, while the maximum M.E.P. indicative of maximum output of power is the same in both processes, peak pressures are different. In the Ring-process, M.E.P. and peak pressures are correlated, and independent from the timing.

(d) Ring-Process and Engine Temperatures.

Tests run with air-cooled engines as well as with liquid-cooled engines have demonstrated that the cylinder head temperatures were considerably lower with the Ring-process. In a liquid-cooled engine the heat carried away by the coolant was 15 to 25 percent lower with the Ring-process than with spark-plug ignition, at full load. Further tests were planned, but not completed, to determine how this difference in heat loss may be accounted for, particularly since the exhaust gases did not show an increased heat loss of equal amount.

(e) Ring-Process and Engine Speed.

Tests conducted at the HIRTH MOTOR CO. in Stuttgart Zuffenhausen proved that no difficulties in the injection of R-fluid need occur at any rate of speed. Velocities as high as 3800 rpm. were reached. The results of these tests are shown on Figure 3. Curves show that the timing of R-fluid injection must be slightly advanced at high speeds, as may well be expected, and that the fuel consumption expressed in grams per horsepower increases also with increasing speed, which is normal. A regular Diesel injection pump with a 6 m/m plunger will take care of the usual rate of R-fluid injection, which varies from 10 to 20 mm3 per stroke.

3. Flight Tests with the Ring-Process.

(a) Prior to the flight tests described below, preliminary tests were made on one cylinder of a 9-cylinder radial engine, the BMW 323, having a 3-liter cylinder capacity. An R-fluid injection nozzle was installed in place of the spark plug, and the

3. Flight Tests with the Ring-Process. (a)(Cont'd)

compression ratio was raised from 1:6.4 to 1:8 which is the minimum considered practical for self-ignition of R-300. No knocking was noticed with the various fuels used. Aviation gasoline C3 was selected for test purposes. The only difficulties experienced were an increase in fuel consumption and some ignition failures when the temperature of the air from the supercharger was below 50 degrees centigrade. This led to a study of the motor temperatures as it was expected that at high altitudes excessive cooling would cause further failures of the process. Figure 5 shows the temperatures at various places of this one cylinder engine.

1.-with spark ignition,
2.-with the Ring-process without any mechanical change,
3.-with the Ring-process after shield plates had been installed to protect the cylinder head from excessive cooling.

It also appeared advisable, in order to avoid ignition failures, to direct the spray of R-fluid against a real hot part of the combustion chamber. By causing the spray to impinge against the exhaust valve a considerable improvement was noticed throughout. This improvement, both in Fuel consumption and in Power output, is illustrated on Figure 6, where 3 conditions are shown: first, a straight spraying of R-300 into the combustion chamber, the air being cooled to 1°C; this condition is the worse of the three! Then the same direction of spray but with air slightly warmer; even this small difference shows an improvement. Finally, with air at -1 degree centigrade but with the spray of R-300 directed against the exhaust valve. A decided improvement is noticeable, power output higher, fuel consumption generally lower. Average consumption of R-300 was 2.5 kilograms per hour, which is rather high. The output, especially for rich mixture, was equally as good as for spark ignition, and the fuel consumption, especially at lean mixtures was decidedly better.

(b) Tests were then conducted with a BMW 323 9-cylinder engine in a Junker JU-52. Compression ratio was 1:8 Two-stage supercharger. Take-off power: 1000 to 1050 HP at 2500 rpm. Climbing power: 800 HP at 2250 rpm. The fuel consumption averaged 210 grams/HP. The R-fluid consumption was 1 to 1.2 per

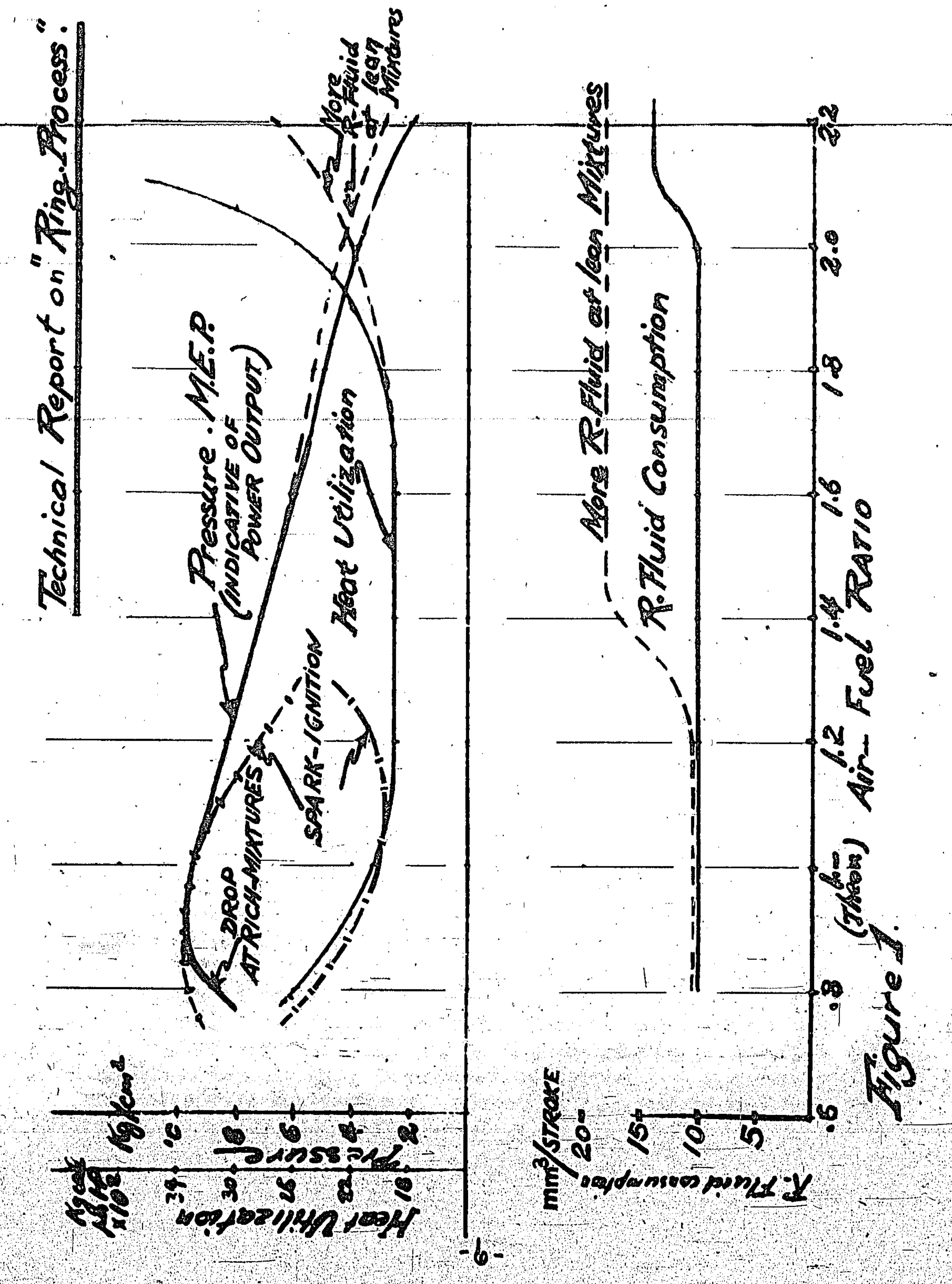


Figure 1

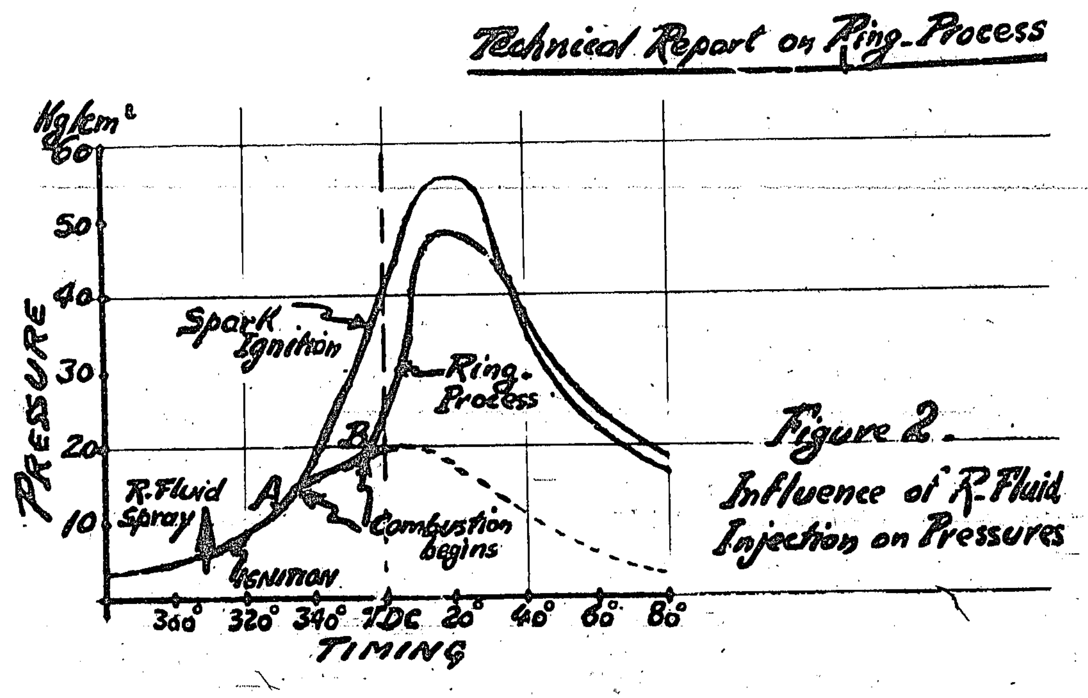


Figure 2.

1. Purpose of the Ring Process. (cont'd)

COMPARISON OF R-300 with AVIATION GASOLINE

	R-300	Aviation Gasoline
Spec. gravity	0.91	0.75
Viscosity CS at 20°C	1.5	0.6
Distillation E. P.	180°C	200°C
Calorific Value Kcal/kg.	6900	10400
Theoretical air required kg/kg.	9.3	14.8
Ignitability in Cetane rating	185	up to 35

It is to be noted that R-300 can also be used as starting fluid for Diesel engines, in which case it is designated as KS II.

The self-ignition quality of R-300 appears to be due to a rapid disintegration of the molecule under the action of heat, an exothermic reaction producing a rapid rise in temperature which causes the products of decomposition to ignite, at about 70 degrees centigrade.

On account of its high ignitability, R-300 is not particularly affected by changes in the compression-ratio, and operates down to a compression ratio of 1:7. In practice 1:8 is the minimum compression ratio selected.

2. Various Features of the Ring-Process.

(a) Ring-Process operates over entire range of air-fuel mixtures.

One of the interesting features of this process is that it operates with correct ignition from the leanest up to the richest air-fuel mixtures. This can be explained by the high energy of spray of R-fluid, estimated at 70 kcal. as against an energy release of 10^{-0}kcal. coming from a spark plug. The only condition for the ignition of very lean mixtures is a

slight increase in the quantity of R-Fluid. Figure 1 illustrates the economy of the process when the air-fuel ratio is varied. As slightly more R-fluid is used for the lean mixtures, the power output curve is also slightly raised. On the other hand for very rich mixtures the ignition delay seems to increase resulting in a power reduction. In other words, as the temperature of self-ignition of the R-Fluid is not reached quite as fast for a rich mixture, a slight power-loss occurs. To obviate this, the injection timing of the gasoline is slightly advanced so as to permit the R-fluid particles to completely permeate the air-fuel mixture.

(b) Ring-Process and Knock.

The Ring-process has proven to reduce to a considerable extent the knocking tendency of a fuel. This seems to be due to the fact that the particles of R-fluid penetrate thoroughly throughout the mass of air and fuel and, when they ignite, start the ignition uniformly. It is as if an infinitely large number of small spark plugs were all operating simultaneously.

(c) Ring-Process and Timing.

The timing of the injection of R-fluid into the cylinder does not seem to have the same effect upon the operation of the engine as the timing of the ignition has upon a spark plug engine. This is explained on Figure 2 on the theory that, as soon as the spark is produced ignition takes place and the rate of pressure rise is modified when the timing of the spark is changed. On the other hand the injection of the R-fluid does not instantaneously start ignition; ignition does not take place until near Top Dead Center. In other words a slight change in the timing of the injection of R-fluid does not affect the rate of pressure rise. A sudden rise in the pressure curve occurs at point A as soon as the spark is produced, in the spark-ignition engine while it does not occur until point B, for the Ring-process, regardless of the exact timing of the R-fluid injection; point B of the curve corresponds to the pressure and consequently temperature condition at which self-ignition of the R-fluid occurs. A comparison of the various

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SUMMARY

A method by which ignition, in an internal combustion engine, is obtained not by means of spark plugs but by self-ignition of a liquid finely atomized into the combustion chamber at the proper moment of the cycle.

This method is designated as "Ring-process".

Key Personnel Interviewed:

Dr. Fritz Penzig of the I.G. Farben Industrie. A.G. at Ungstein, near Bad-Durkheim.

Dr. K. Peter, of the Baierische Motoren Werke, At Munich.

July 1945

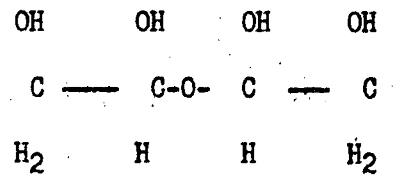
INTERNAL COMBUSTION ENGINES

1. Purpose of the Ring Process.

In the German aircraft gasoline engine it was recognized, up to very recently, that the weakest part especially for high-altitude operation was the spark-plug. Not only was it necessary to take unusual precautions to avoid ignition difficulties at high altitudes (for instance special high-altitude spark plugs, dual ignition, specially insulated cable, restricted air circulation) but it was also necessary to avoid fouling of the spark plugs by limiting the lead content of high-test gasoline. The "Ring-Process" is an invention for the elimination of the spark plugs. Ignition is produced by spraying into the combustion chamber, at the right moment of the gasoline-air compression stroke, a liquid that will spontaneously ignite at the temperature of the cylinder, and thus ignite the combustible charge.

The process, already mentioned in the writings of Dr. Rudolf Diesel in 1898 was developed by Dr. Fritz Penzig of I. G. Farben Industrie A.G. at Oppau in 1940-41 and perfected in cooperation with the Baierische Motoren Werke of Munich in 1942-43.

The ignition liquid used, called "R-Fluid" is an ETHER such as Budadiol diethyl ether, or Diethyl diglycol ether of formula:



$$\begin{array}{cccc} \mathrm{OH} & \mathrm{OH} & \mathrm{OH} & \mathrm{OH} \\ \mathrm{C} \text{ --- } & \mathrm{C{-}O{-}} & \mathrm{C} \text{ --- } & \mathrm{C} \\ \mathrm{H_2} & \mathrm{H} & \mathrm{H} & \mathrm{H_2} \end{array}$$

It is designated in Germany under the symbol R-300 and has the following properties:

~~RESTRICTED~~

INTERNAL COMBUSTION ENGINES
(Use of a chemical as ignition agent)

Reported By:

Lieut. R.J. BENDER, USNR
NavTecMisEu.

[1945]

CIOS Target Nos. 19/11a & 30/1.10.
Vehicles
Fuels and Lubricants

16 p, illus.

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

~~RESTRICTED~~

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Nav Tec Mis Eu 147-45

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INTERNAL COMBUSTION ENGINES

Bender, R. J.

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

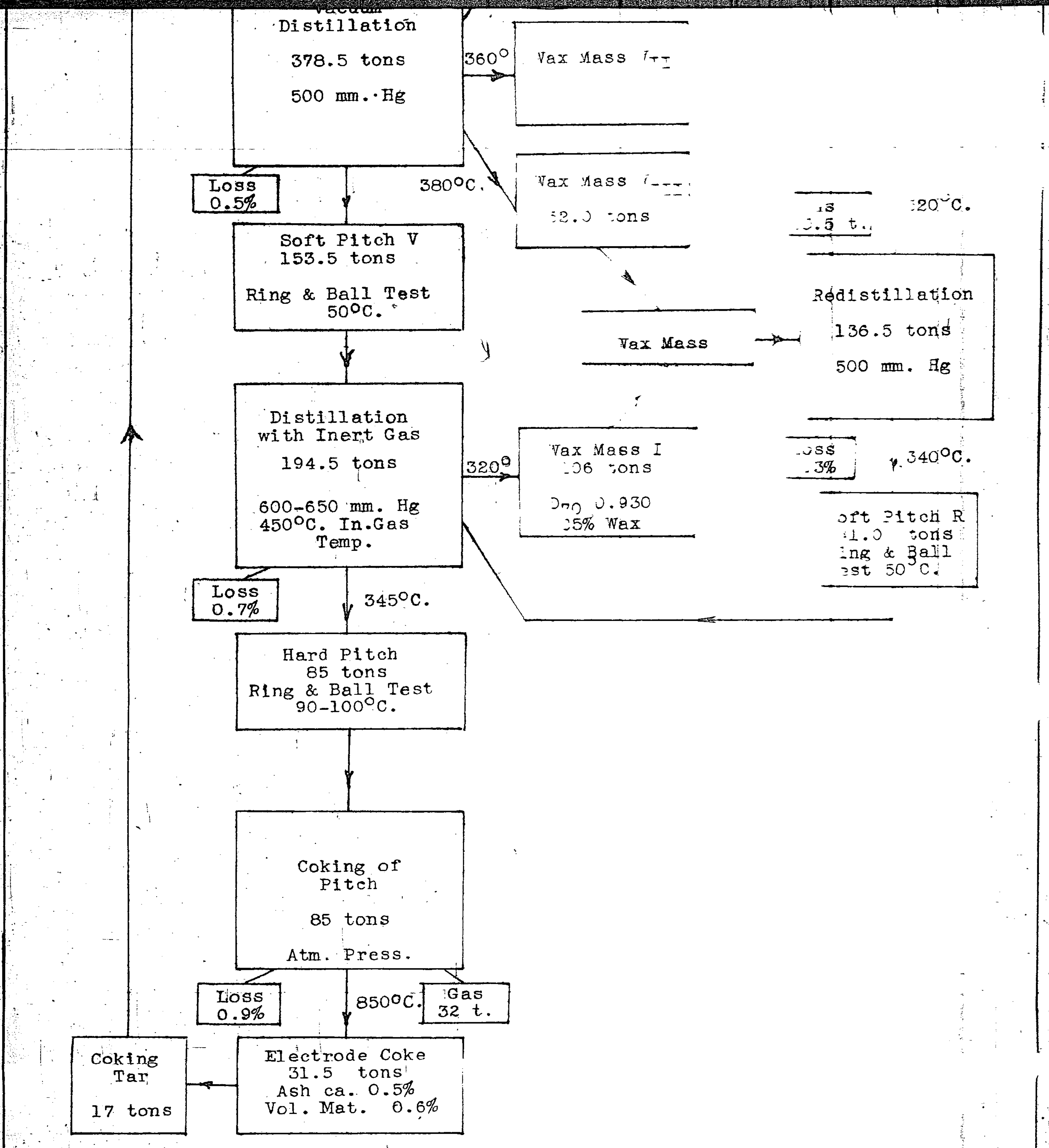


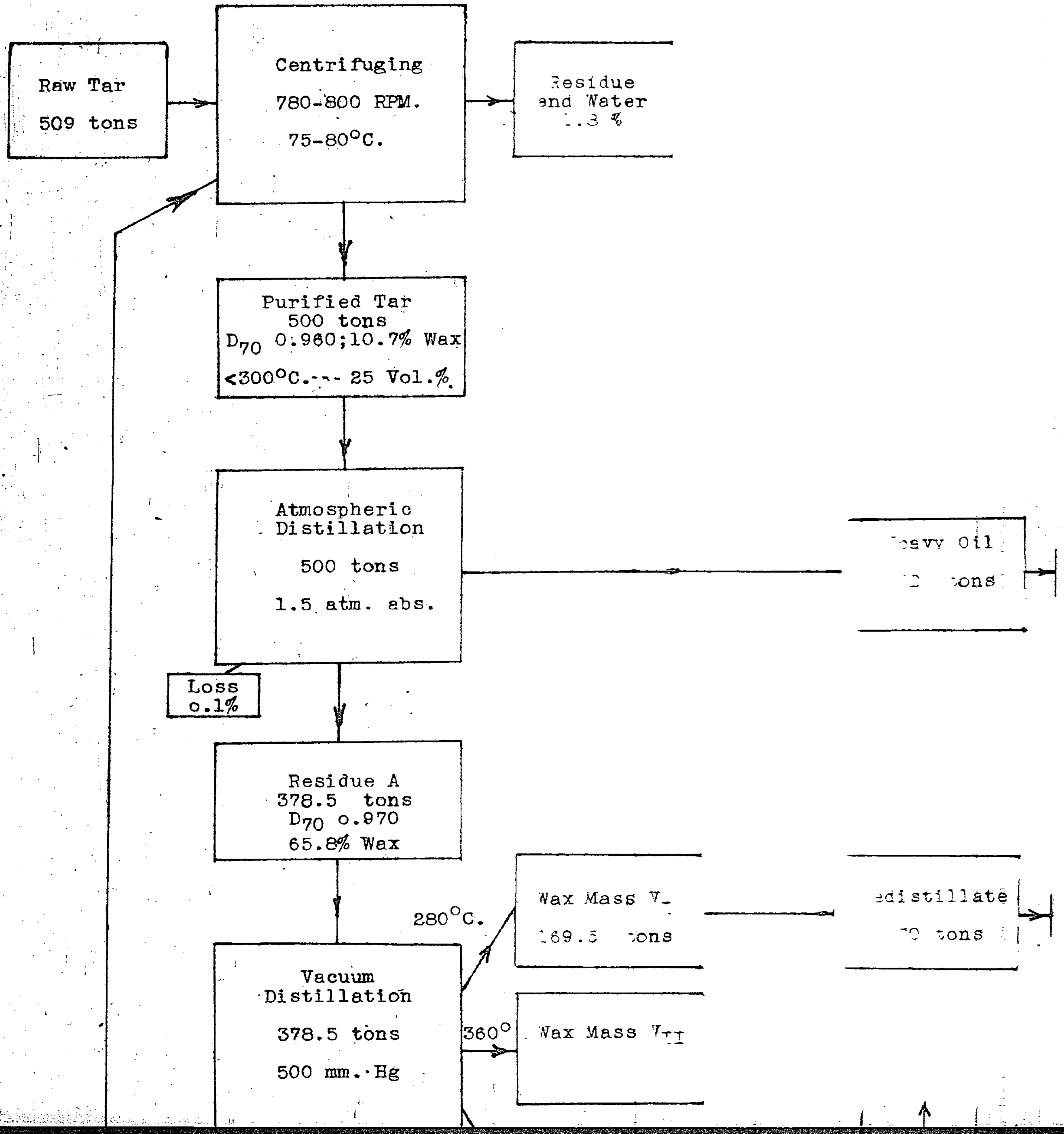
FIGURE IIA

P.1476

A.G.S. WERKE - ESPENHAIN

TAR DISTILLATION

on a daily basis



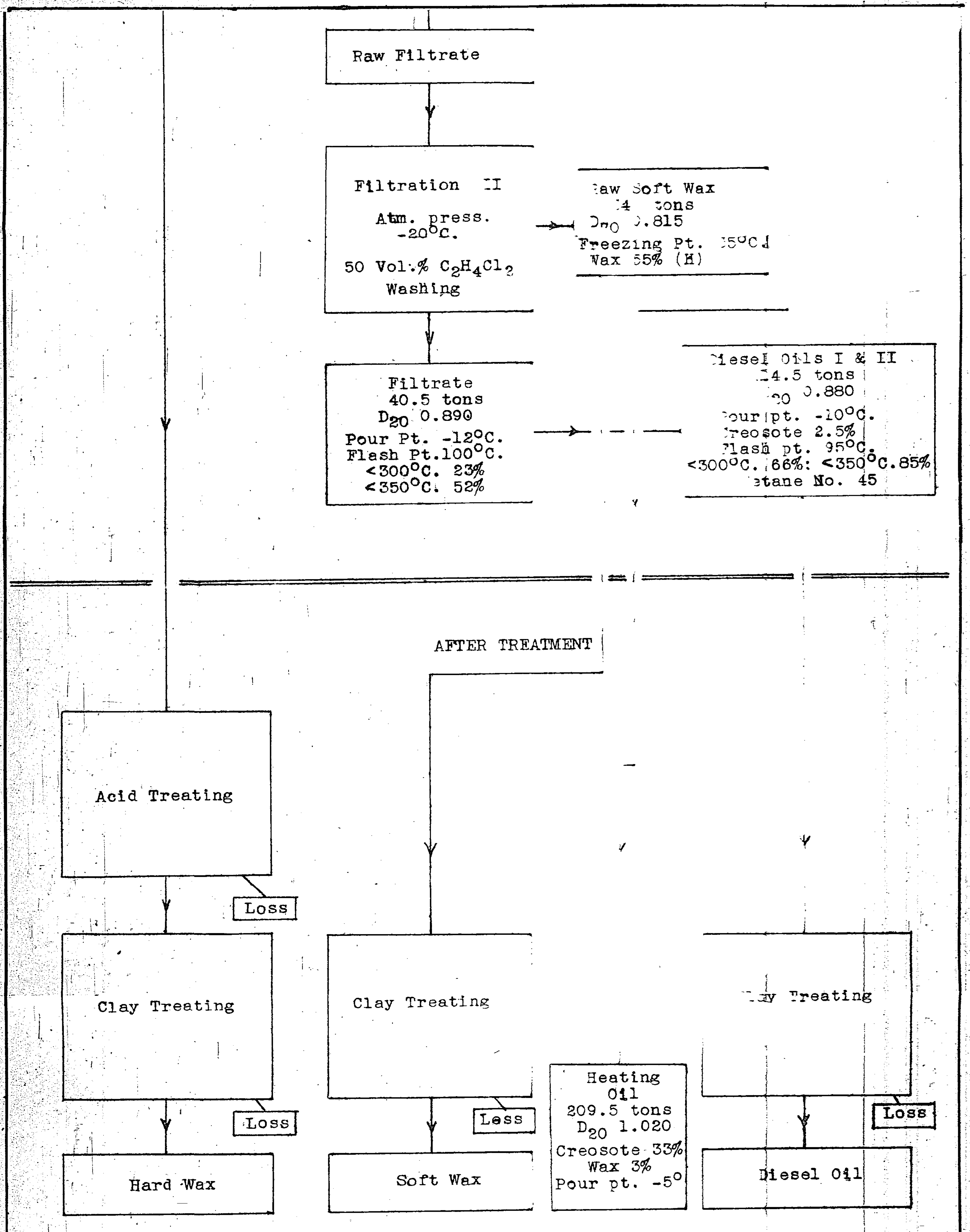


FIGURE IIB

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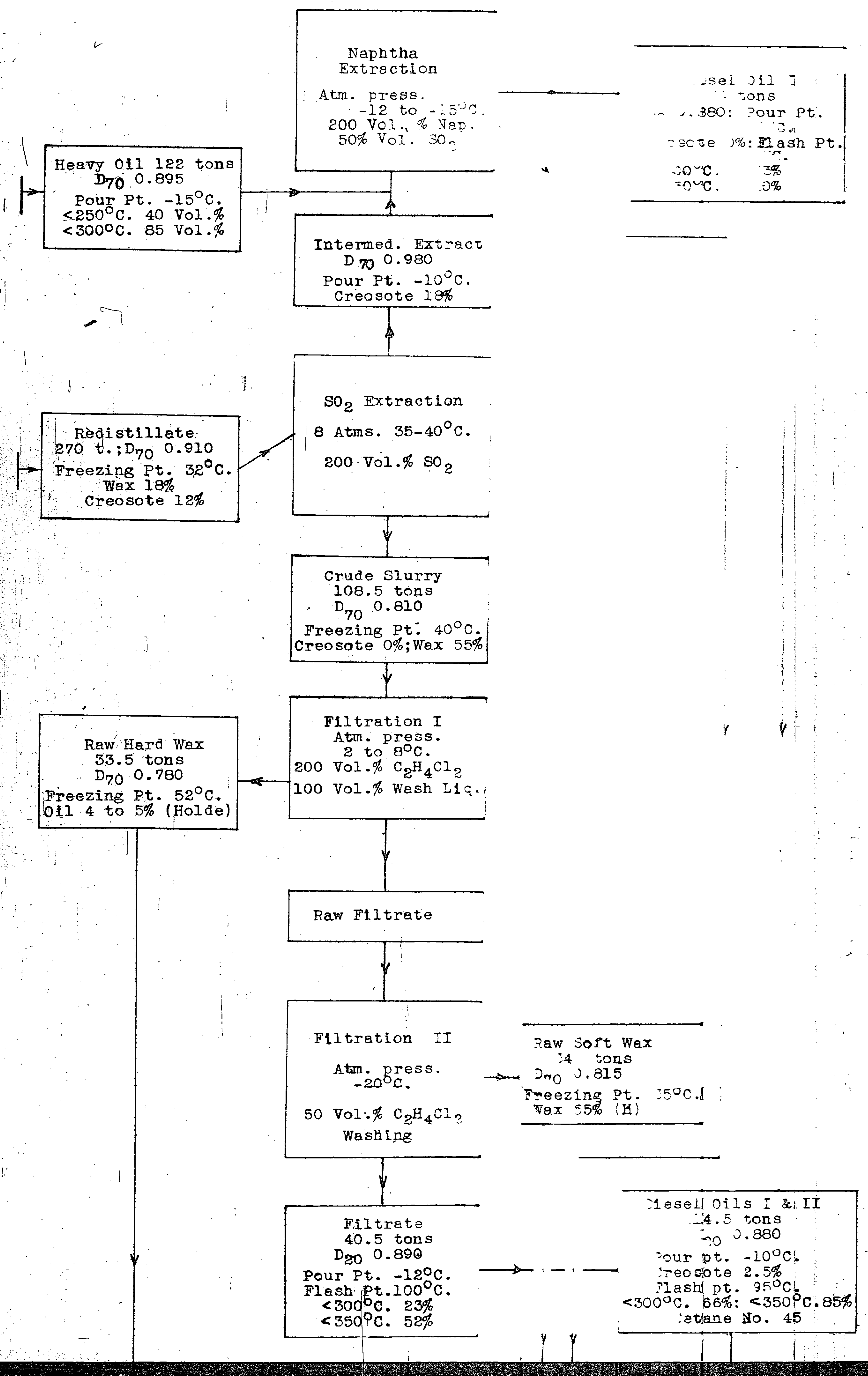
A. G. S. WERKE - ESPENHAIN

TAR SEPARATION

on a daily basis



A.G.S. WERKE - ESPENHAIN

PRODUCTION DIAGRAM

on a yearly basis

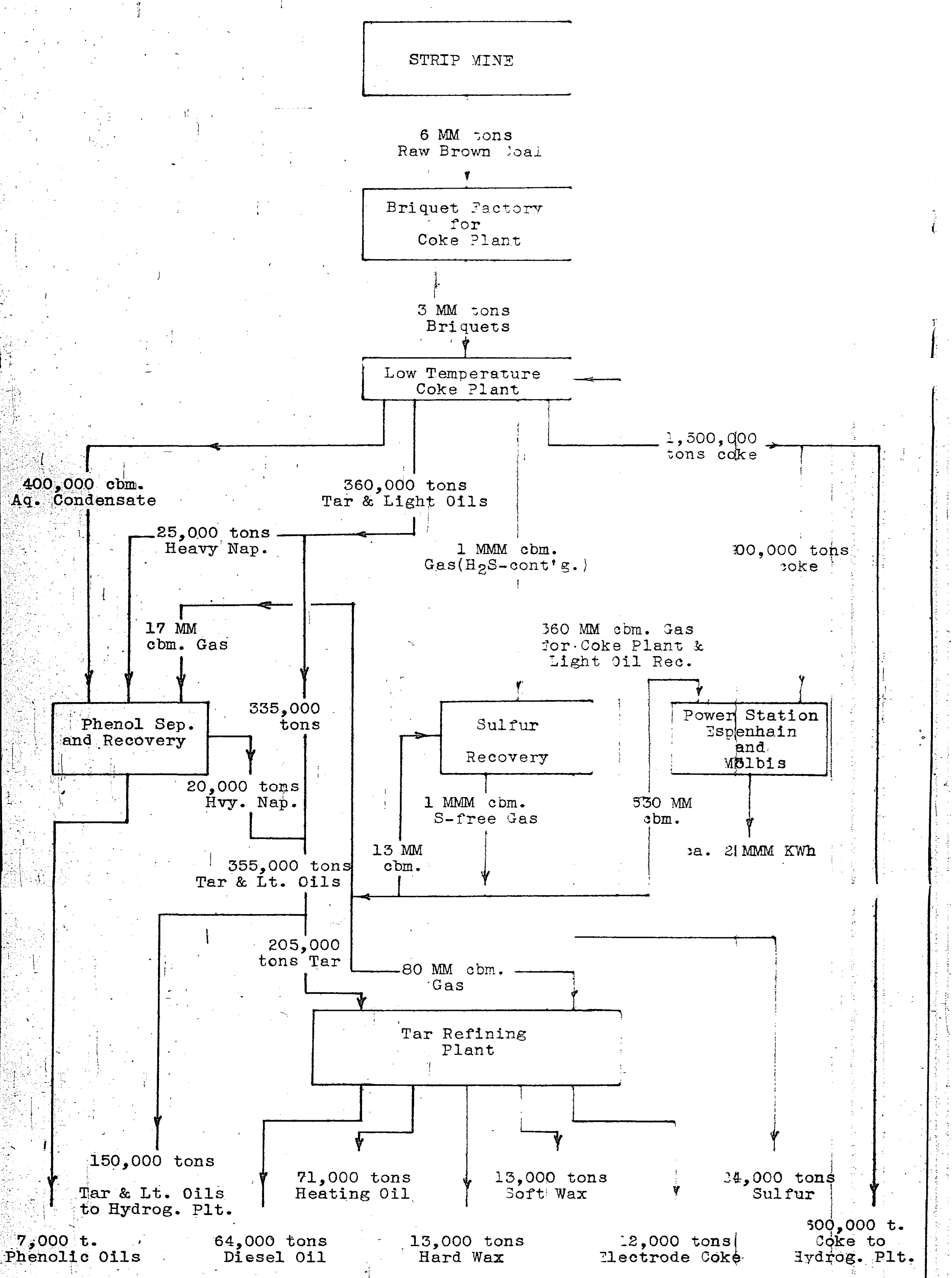


FIGURE I

P.1476

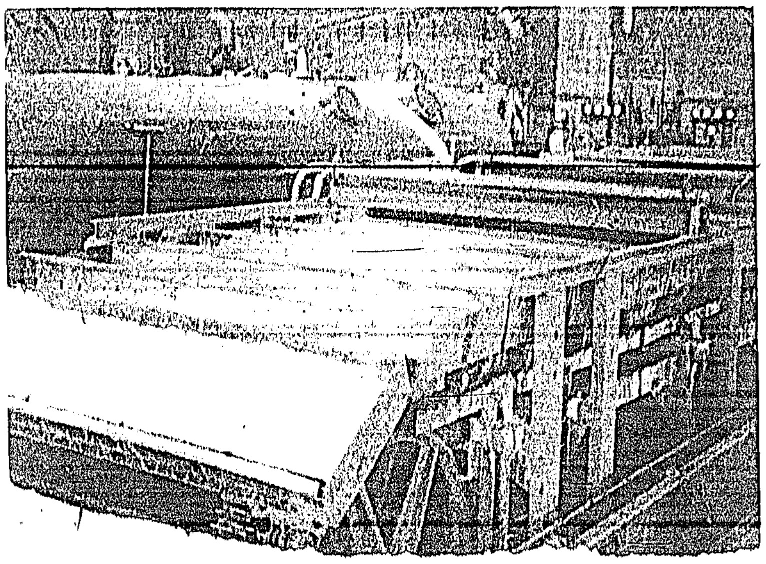


Fig. III

Continuous "Band Filter"
Foreground: A filter open for repairs
Left Background: Housed filter, as used