U. S. GOVERNMENT TECHNICAL OIL MISSION
INDEXES TO MICROFILMS

INDEX - MICROFILM REEL 240
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ITEM No. 22, 30, 31

SOME ASPECTS OF THE GERMAN PEAT INDUSTRY.

Robertson, R. H. S., Clements, A. G.

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BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

SOME ASPECTS OF THE GERMAN PEAT INDUSTRY

Reported by

R.H.S. Robertson,) for M.O.W. &
A.G. Clement,) D.S.I.R.

BIOS Trip No: 1957

BIOS Target Nos:

C22/2832
C22/2158
C30/390
C31/2098
C22/3272
C30/170
C31/2568
C22/2030
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C31/2565
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C22/3273
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C31/1788(a)

British Intelligence Objectives Sub-Committee

32 Bryanston Square, W.1.

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H. SUMMARY.

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A. PEAT INSULATION SLABS

Report 1. Torfoleum and Tektonwerke, Poggenhagen, Hannover.

022/2832

L53/X13

Personnel Interviewed (Dr. Eduard Dyckerhoff C22/2030
(Erich Kólpak C31/1369
16/3/46 (- Steinert, Works Manager C30/388

Dr. Dyckerhoff was the pioneer maker of peat insulation slabs from peat. The present factory was built about 1910 and the process has always been a commercial success.

Process of Manufacture

Light brown sphagnum peat, very similar to certain deposits in the Central Belt of Scotland, is brought 6 Kilometers by aerial ropeway from the bog. A large stack of peat is accumulated beside the works for winter processing; in summer part of the peat goes straight into the factory. The moisture content is then about 70 per cent The cut peat blocks are roughly torn in a mill (Reisswolf) and the resulting peat moss litter is transferred by elevator to a bath where it is pulped with a weak solution of phosphor acid by means of a paper beater. The beating makes natural adhesives in the fibres and the longer the beating the stronger the binding action. The sludge is passed through a trough to a hopper feeding four moulds, two on either side of a hydraulic press. The mould size is 1 x ½ m., and depth about 10 cm. compressing to 2 or 2½ cm. Dr. Dyckerhof has found it unsatisfactory to make plates larger than this in area. The expressed liquor returns to be used in th next batch. The pressed cake is transferred from the perforated false bottom of the mould by being turned over on to about 2 cm. mesh trays. 30 trays are accommodated on a truck, and 18 trucks are placed at one time in the tunnel drier, in 2 rows. Spare trucks are kept for filling. Air circulation is by fan and the air is driven in the direction of travel of the trucks to prevent warping of the slabs. Heating is by gilled steam pipes. The final temperature is such that if phosphoric acid had not been introduced the boards would char; this heat treatment was said to make the boards substantially waterproof. The final moisture content is well below the equilibrium moisture content of the boards. The phosphoric acid is also used to make the boards fireproof.

In the drier the moisture content is reduced from about 90 per cent. to about 10 per cent., and the coal consumption was said to be 9 Kilograms per sq. metre (2cm. thick) or 2250 Kilograms per tonne of product. In 1938 the price of coal was 17 R.m. / tonne.

Boards of greater thickness than 2½ cm. are made by glueing with sodium silicate solution. Some of the 2 cm. boards are finished by covering on both sides and on the edges with brown paper; the machines for doing this were designed by Dr. Dyckerhoff. The boards are then hot-pressed. The output is 100 to 120 sq. metres an hour.

The labour was estimated to be 11 man-hours per cubic metre, excluding the labour of winning the raw peat. At the press, 4 men fill and empty the moulds and one works the press; the production being 40 sq. metres an hour, or 320 sq. metres per 8-hour shift. They have sometimes worked day and night. The labour cost (1938) was from 5 to 8 RM per man per shift.

Less than 1 per cent. of the output was despatched in crates. Usually the boards were railed without packing in box wagons.

Uses of Peat Insulation Slabs

Dyckerhoff first introduced peat insulation into house building, later for insulating refrigerators and chilled rooms, and one of his staff built up a large business in installing Torfoleum slabs in ships.

Examples of Uses:-

Erich Koch-Siedlung, Dt. Eylau, Ostpreussen, Torfoleum hollow wall method for the external walls of cottages.

Pommersche Landsiedlung, Lauenburg, Pommern, ditto.

Kinderkrankenhaus, München-Schwabing, Oberbayern, Torfoleum for sound deadening covered by plaster.

Schlesische Leinenindustrie, Freiburg/Schlesien, Torfoleum used to prevent condensation in factory.

Westfalenhaus Dortmund, Westfalen-Süd, flat roof and floors insulated with Torfoleum.

Examples of steel-clad houses insulated with Torfoleum are shown as photographs in Professor Stiegel's book, Der Stahlhausbau, Alwin Fröhlich Verlag: Leipzig: Gohlis, (1938) pp. 74, 75.

Refrigerating chambers on over 140 ships including many of the Hamburg-Amerika Linie, private yachts built for the U.S.A. by Germaniawerft, Kiel. Elder-Dempster & Co.'s vessel "Burutu" was similarly treated.

Methods of use:-

Torfoleum slabs must be fixed to a rigid framework with pitch or by nailing. It can be plastered only if wire-netting is fixed to the surface of the slabs.

Commercial Information

The sale of Torfoleum has been affected by competition with other building materials. Cork was for a time very cheap, and wood-wool and gypsum or cement boards have replaced Torfoleum for certain applications.

The prices per sq. metre in 1939 were:-

	2cm.	2.5cm.	3cm.	4cm.	5cm.	
building contractor	1.40	1.70	1.85	2.15	2.64	RM
private contractor	1.80	2.10	2.30	2.70	3.20-3.40	RM

A selling price of 70 RM per cubic metre about 1938 was considered a rather poor price; but at that time 85 RM would have been a good price. The price is fixed now, so with the higher cost of coal and labour and the smaller output (25,000 sq. metres a month authorised) the profitability is doubtful.

Dr. Dyckerhoff stressed the importance of making more than one product from the raw material and from the plant.

From the peat he also makes peat moss litter, see page
The factory is also used for making TORFOTEKT, a wood-wool and gypsum building board, for which he has installed separate presses and 2 larger tunnel driers. 100,000 sq. metres of these boards a month have now been authorised.

The annual turnover of the firm includes all products:-

1933	267,361 RM
1936	3,657,763 RM
1943	2,860,797 RM
1944	2,566,088 RM

The maximum consumption of gypsum was said to be 1000 - 1500 tons a month, and of coal 450 tons, and a little cement. Labour required is 15 skilled, 205 unskilled workers and 84 office and other workers.

The technical development has been mostly the work of Dr. Dyckerhoff who held patents for the use of phosphoric acid and for the method of drying. He has employed a chemist before the war.

Report 2. Triangeler Torf-und Leichtbauplattenwerke, Gifhorn, Hannover
C22/2158 C30/389 G31/2564 M53/X93

Personnel Interviewed Hermann Koehler, owner

17/3/46

The process is essentially similar to that of Dyckerhoff. The factory was originally started in 1914 and finished in 1924, though the powerhouse was added in 1928. The section devoted to peat slab making was accidentally burnt down a few years ago, but it is hoped to rebuild it.

Process of Manufacture

Good fibrous light brown peat was cut 6 Kilometers from the works by about 6 men and brought in by rail after air drying to about 70 per cent. moisture. After milling, the peat is boiled in water in a wooden vat into which about 4

Kilograms of phosphoric acid per cubic metre of product and 5 Kilograms of low melting point bitumen and gatch are added. The hot slurry is pressed, and the product containing about 80 per cent. of water is transferred to tunnel driers. There are 6 of these, each holding 2 rows of 11 trucks. Each truck holds 26 trays. Air is circulated by fans across the direction of travel, and heating is by steam-heated gilled pipes. Steam comes from the exhaust of a high-pressure (28 atm.) steam turbine electricity station generating 500 KVA, in very good order. A steam-driven engine (150 h.p.) worked certain moving parts in the kilns and factory. The final temperature of drying was said to be 130 to 140° C. The boards are completely dried and subsequently conditioned to equilibrium moisture content. Phosphoric acid was stored in six 1000 litre earthenware vessels and pumped through earthenware pipes to the vat. The effluent expressed by the press was reused for the next batch. The press was made by the firm of Neustadt and Hort, and was installed by A. Beeck, Inh. R. Jung, Oldenburg, who supplied the hydraulic gear and fittings.

The normal size of insulation slabs was 1m. x ½m x 2½cm., but they could be made up to 6 cm. thick. Thicker slabs were made by planing and glueing with bitumen. Some slabs were covered with paper on a simple glueing machine or covered with pitch, outside the factory, for safety. The product is called TORFISOTHERM. Its specific gravity is 0.16 to 0.17.

The capacity of the plant was about 1,400 square metres per day of 2 shifts, and the main factory for its manufacture measures 80m. x 40m.

Herr Koehler stressed the importance of making wood wool and gypsum (or cement) boards (Leichtbauplatten) so as to take advantage of changes in the market. Cement was better than gypsum, but not always so easily obtained. They prepared their own wood wool from small logs some 45 cm. long. The product is called ISODIELE. The works could produce about 3100 square metres a day of these boards, but a partly built factory, which they hope to complete, will increase production by a further 8000 sq. metres.

The combined works used 24 tonnes of brown coal (5000 thermal units) a day or correspondingly less of bituminous coal (8000 thermal units) for making 4,500 square metres of the 2 products. This comparatively low consumption of fuel may be due to the efficient use of steam at different pressures for making electricity, driving engines and drying the product, but also because, in the peat section a hot slurry is pressed and the cake therefore contains less water than if a cold slurry had been pressed. The air circulation was also fairly efficient in the driers.

The factory was formerly owned by an Aktiengesellschaft, whose capital was about 900,000 RM, and the total cost of plant was about 1,300,000 RM. This company failed in 1930 or 1931 in the slump. The present owners are a private company. They also started the Latvian State Peat Industry's plant at Liepaja in 1935 and factories in Russia from 1930. Complete manufacturing details have been published in Russia, but not elsewhere.

The preferred method of using TORFISOTHERM is between two brick walls, but many other methods have been used, in breweries, slaughterhouses, cold stores, railway carriages, ships cold rooms (including HAPAG) The material is known to be as good as cork for thermal insulation. 12 cm. thickness . common in refrigerated chambers. Thousands of dwelling houses have been insulated with this material. For garages, peat slabs stuck on to masonite hard board was serviceable.

The company has no laboratory and are not interested in research. They make no peat moss litter and even buy this sometimes when the quality of their own peat is poor. They do not make peat for fuel although huge reserves lie on their land and the reserves of light brown peat are shrinking.

The price of the peat insulation boards was 2.25 RM per square metre (2½ cm. thick) in 1938 or 2.85 RM if papered.

Report No. 3. Otto Borggräfe, Oldenburg i. O. Alexanderstrasse 94.

C22/3198
C30/390
C31/2565

Works: Sedelsberg,

bei. Oldenburg

Personnel Interviewed Otto Borggräfe, Owner.

19/3/46

Borggräfe has built up a small works consisting of a peat moss litter section, a peat insulation slab section, a workshop and a 150 H.P. engine driven by gas generated from pressed peat (PRESSTORF from a Wielandt machine). Much of the plant has been designed, made and erected by Borggräfe.

A mill, similar to Dyckerhoff's is placed over a paper beater. The water is not heated nor is phosphoric acid added. They add as a water-proofing agent about 5 Kilograms of tar per cubic metre of product (mainly 3 cm. thick). The slurry is bucket elevated to a hopper which feeds portable moulds on one side of the press. The moulds are about 10cm. deep. The pressure in the press was said to be 100 atmospheres on a 20cm. diameter ram, and the boards are of the standard size, 1 metre x ½ metre. Three men work at the press; the moulds are shoved into the press through guides; there are stops on the far side of the press to position the moulds.

The pressed boards are inverted on a swinging gear on to wooden trays. They have 800 of these trays which are taken to a field where the boards dry in the wind for 7 to 14 days. The output is 200 to 300 square metres a day. The drying is finished in a day in one of two kilns, containing conventional trucks with wire mesh trays One kiln has 16, the other 28 trucks, each with 2 rows of 13 trays. Air is indirectly heated in a furnace burning peat and/or a little coke and is circulated by fan. The temperature in the drying chambers is only 50° C.

The applications of these boards do not require fire-proofing, and waterproofing is obtained by means of the tar. Consequently the boards are not heated to near charring-point as in Dyckerhoff's process. The boards appeared to be weaker than Dyckerhoff's.

The peat insulation has been used in a co-operative creamery 33 Kilometres from Oldenburg; a vegetable deep freezing plant near Osnabrück where 16 to 20 cm. thickness of material was used to maintain a temperature of -15 to -20° C. In ordinary chilled rooms, 12cm. is used. Quite large quantities have been used for insulating egg farm batteries in Oldenburg province.

If the war had not intervened, Borggräfe would have designed and supplied a peat insulation plant for a firm in Sweden.

Notes:-

Torfit-Werke. Hemelingen bei Bremen, Holzstrasse 49/57, appear to have made a peat building board called TORFIT, according to the Post Office Trade Directory for Bremen, 1941.

W.G. Wöltjen, Torfstreu-und Torfmullfabrik, also advertises peat insulation in the same Directory. The address is Falkenberg, über Lilienthal, (Bz. Bremen).

The binding properties of Hollander pulp were studied more recently by Dr. Barchfeld, BIOS 22/161, who independently discovered that fibre boards heated just below the charring-point became waterproof.

Karl Scheider, Kahl (Main), is given in "Wer Liefert Was" 1941 as a manufacturer of machines for making insulating slabs.

B. PEAT MOSS LITTER AND GRANULATED PEAT

Five firms making these products or the machinery for making them were visited, but only one, the Hannoversche Kolonisations A.G. was markedly in advance of current practice in this country.

Report 1. Eduard Dyckerhoff, Torfoleum u. Tektomwerke, Poggenhagen, Hannover.

This firm uses a type of tearing machine known as a "Reisswolf" for disintegrating the peat. The peat is then elevated by a drag conveyor and fed into the baling presses/

presses of which there are two. The dimensions of the bales were not measured, , but they appeared to be about 1 metre long by ½ metre square.

Another firm nearer to Neustadt a. Rübenberge is Moorverwertungs G.m.b.H. Here peat moss litter and granulated peat are made.

Report 3. Otto Borggräfe. Oldenburg; i.O., Alexanderstrasse 94

The works of this firm are at Sedelsberg, 35 Km. from Oldenberg and, unlike Dyckerhoff and Triangeler, peat moss litter is the main product and insulation boards the subsidiary one. The peat is hand cut and air-dried on the bog. It is brought in by small gauge railway, and the blocks discharged into a pit from which an elevator carries them to the mill. This, and the baling plant have been designed and built in Borggräfe's own workshop, and it is a simple device of hooked claws on a shaft. The claws mesh with a set of fixed bars giving the tearing action, and the rate at which it is run is believed to be about 200 r.p.m. The peat thus disintegrated is carried by elevator to the baling presses of which there are two mounted side by side. They are constructed largely in wood and are fairly primitive but quite efficient. The bales were not measured but appeared to be of the same size as those already seen.

Report 4. A. Beeck. Inhaber. R. Jung. Oldenburg i. O. Stau 28

C22/3214
C30/391
C31/2098

Robert Jung, owner of the firm was interviewed. This is an engineering firm which makes machinery for the manufacture of peat moss litter (Torfstreu) and Granulated Peat (Torfmull). Plants erected by Jung for these products include those at Torfwerk Mulmshorn Gunter Meiners, Mulmshorn/Rotenburg, Hann.; Neufabrik, Strockhausen, Mark; and a plant for the Glückner concern, (a steel company). This last appears to have been a model plant, but it was burnt down. It turned out per day 400 bales of 70-80 kg. weight and 0.96 metres by 0.56 metres square (the size was calculated to fit exactly into railway wagons).

It is very compact, requiring a building only 14 x 10 metres and the two baling presses are so arranged en echelon that they can be worked by the same team of men.

This firm installed the presses (made by Neustadt and Hort) at Triangeler's factory and supplied the hydraulic gear and fittings. Jung mentioned moss litter factories in the neighbourhood of Oldenburg at Ramsloh (belongs to Krupp and is the best in the neighbourhood), Scharrel, Heselberg, and Borggräfe's factory at Sedelsberg which he said was the poorest of the lot. All use peat from Westermoor.

Report 5. Hannoversche Kolonisation und Torfverwertungs A.G., Schwegermoor, Hunteburg, bei. Osnabrück, (Hannover).

C22/3271 C30/379
C31/2566

This firm is one of the largest and best organised in the German peat industry. It has a spacious and well laid out factory for peat moss litter and firelighters and at Schwegermoor. Their moss extends to 1800 hectares and consists of 1-1½ metres light brown peat on top of 2-2½ metres black peat. The brown peat is cut by hand and employs 100 men in the cutting season, the black peat is cut by machines, of which they have four. The annual production of brown peat is 10,000 tonnes in 240,000 bales of peat moss litter. They normally work three shifts and employ 300 men in winter and 600 in summer. Their bales are one third of a cubic metre in volume, that is, approximately the same size as Borggräfe's. They have specially designed railway wagons, about 10 metres long which hold 240 bales, and before the war they exported peat moss litter even to the U.S.A.

Their plant is completely mechanised. It begins with an elevator which takes the blocks tipped out of the wagons to the mill - "Reisswolf" type of tearing mill -; then another elevator carries the disintegrated peat to a storage hopper from which the baling presses (two mounted side by side) are fed. The plant has been largely designed and made by themselves. There is a very simple and ingenious gantry crane which handles six bales of peat at a time from the baling presses to the railway wagon.

We had heard that there was a peat fired electricity generating station on this moor, but Herr Janisch the manager who showed us round assured us that it was only experimental and was abandoned a number of years ago because it proved uneconomic. He told us that the only station generating electricity from peat in Germany was at Wiesmoor near Aurich.

Firelighters, the other products of this firm, and their mechanical methods of peat cutting are described elsewhere in this report.

Note:-

There are many other factories in Germany making peat moss litter and granulated peat. Among those existing up to 1941 are:-

Barnstorfer Torfwerk Aug. Boder, Barnstorf (Bz. Bremen).
Bourtanger Presstorfwerk GmbH., Alt Haren (üb Haren, Ems), et.
Georgsdorfer Torfwerk Gebr. Brill, Georgsdorf (Grafsch Bentheim).
Torfwerk G. Strenge, Ocholt (Oldbg.).
Bayerische Landestorfwerke GmbH., München 27, Trogerstr 54
Haskamp, B., & Co., Lohne, (Oldbg.).
Heseper Torfwerk GmbH., Meppen.
Staatliche Torfwerk, Schussenried, (Württmbg) and at Waldburg.
Most, Werner, Torfstreufabrik, Bremen, Vahrerstr 388.
Bremer Torfwerk A.G. Gnarrenburg (Bz. Bremen).
Torfwerk Pennigbüttel, Osterholz-Scharmbeck (n/r Bremen).
Torfstreu-und Mullfabrik der Fa. Eugen Stoph & Co. Sittensen (do).
Moostorfstreuwerk Zeven, Zeven (Bz. Bremen).
W.G. Wöltzen, Torfstreu und Torfmullfabrik. Falkenburg, über Lilienthal (Bz. Bremen).
Peper, H., Fischerhude (Bz. Bremen).
Torfstreufabrik Hahnen Knoop, Stotel.
Kohlener Torfwerk, Heinschenwalde über Bremerwörde, (Eurden)
"Klostermoor" Siedelungs-und Torfverwertungs - G.mb.H., Papenburg (Ems).
Torfwerk Teufelsmoor, Torfstreu und Torfmullwerk der "Turba" G.m.b.H., Bremen, Philosephenweg 8. Works at Teufelsmoor, über Osterholz-Scharmbeck.

Other manufacturers are mentioned in the reports.

C. PEAT COKING

Report 6. Dr. Wilhelm Wielandt. Oldenburg. Hochhauserstrasse 1.
Factory: Torfwerk Langenmoor. Elisabethfehn. C22/3272 C30/378
C31/2567

Dr. Wielandt's daughter explained how pressed peat was treated in a retort to yield gas, coke and tar at the works at Langenmoor. The coke found a number of industrial uses, particularly for gas generators used on motor cars, including the Doctor's car. The coke is very light, and fine-grained and rather soft. It is very reactive in a producer and the ash content is low. The sulphur content is also low.

The other product of peat coking is tar, which is partly used for impregnating peat fire lighters, and other local uses.

Note:-

K. Krapf and A. Schwinghammer, Chem. Fabr. (1939), 12, 195-9, described experiments on the low temperature carbonisation of peat, and the Deutsche Torf. G.m.b.H. is believed to have used the low temperature carbonising process in North Eastern Germany.

D. PEAT GAS GENERATION.

Report 7 Tiefstack Gasworks, Hamburg. C22/3273 C30/170
C31/2444

Personnel Interviewed, with the assistance of Mr. B. Marriage Officer in charge, Hamburg Gasworks.
-------Chief Enginner
18/3/46 Stief, works manager.

An experiment lasting four days on the gasification of peat briquettes was carried out at the Hamburg Tiefstack Gasworks in 1938 in the Pintsch-Hillebrand gas generator, which normally runs on brown coal. The coal experiment is described in full in Chemische Fabrik, (1939), 457-462, and the plant is described in Gas and Wasserfach, (1932), 581-586, (1935), 431 - 436, and (1940) 1-6.

Technically the experiment with peat was successful, though for running on peat the plant would require some modification such as widening the feeding chutes and gas outlets, because of the low bulk density of the raw material and the greater volume of steam in the gas.

The peat briquettes held their shape well during combustion, - an essential property. The ash was more easily handled because of the smaller quantity produced.

Peat was not used again because it was found impossible to obtain a sufficiently large supply, and the cost of transport from East Friesland was too great. Nine peat areas with 28 individual moors had been surveyed within 30 kilometers of Hamburg, totalling 3480 hectares and about 100 million cubic metres of peat, which quantity is not very different from that belonging to Torfwerks Vehnemoor in Oldenburg.

The Works Manager considered that the process would pay if the plant were situated near to a large peat moor.

Report 3. Otto Borggräfe, Oldenburg i.O., Alexanderstrasse 94

O. Borggräfe at his works at Sedelsberg i.O. uses pressed peat from a Wielandt black peat cutting machine after further drying in a stack beside the works. The blocks are elevated from a boot into a hopper feeding a simple vertical gas generating retort. The gas is washed thoroughly and used for driving a 150 H.P. engine. The engine has required to be overhauled only once in 3 years because the gas is so clean.

Note:-

Gustloff-Werke, Otto Eberhardt-Patronenfabrik Hirtenbert N-D Betrieb Liechtenwörth, Wiener Neustadt, Pottendorferstrasse 162, appear in the Reichs-Telefonbuch (Jan. 1942) as makers of peat gas generator installations.

E. PEAT WINNING

Reports 1, 4, 3 HAND CUTTING

Herren Dyckerhoff, Jung and Borggräfe confirmed that no successful machine had yet been invented for cutting light brown peat. It is still hand cut in blocks which are about 35 cm. x 15 cm. x 15 cm. on drying. The quantity cut per man can be estimated from the fact that 10,000 tonnes of peat moss litter are cut each year by 100 men at Schwegermoor. The season is about six months, hence we have a production of almost 4 tonnes per man per week. This 4 tonnes of peat contains about 70 per cent moisture and represents about 11 tonnnes at the moisture content at which it is usually cut (87-88 per cent.). The wages according to Dr. Dyckerhoff are from 5 to 8 Rm. a day, hence, the cost of cutting will be from 7½ to 12 Rm per tonne.

Report 1 MACHINE CUTTING

Dr. E. Dyckerhoff. Although no machine has yet been developed which cuts brown peat satisfactorily Dr. Dyckerhoff is attempting to develop such a machine. He informed us that he had made one machine some time ago which was tried out on the bog but was not satisfactory. A second machine part of which we saw had been made, and tried out without much more success, and he was now engaged in reconstructing it. It appeared to be a very heavy machine, but what its action was we did not discover. Herr Jung of Oldenburg mentioned this machine and said that it had an arm which came down, dug out a slab of peat, lifted it up, swung round and placed the peat to the side; it then moved on and cut another piece. He also confirmed that it was unsuccessful because it had not sufficient output.

The Wielandt machine. This machine was originally designed by Dr. W. Wielandt. Four of them are employed at Schwegermoor where they cut 40,000 tons in a six month season which begins in April. Each machine requires about 10 labourers and 3 foremen. From their figures one machine is equivalent to 100 men cutting by hand. We saw a further three of these maqhines on moors near Oldenburg. They are suitable only for black peat, and the product is in a form in which it can be used as fuel. Dredger buckets about 1.5 metre long and 10 cm. deep are used to excavate the peat and the conveyer on which they are mounted can be lowered to the depth (usually about 1 metre) to which it is desired to excavate.

The buckets deliver into a hopper in which a screw drives the peat to one end. A truncated cone is joined to this end and the peat driven into this is compressed to a solid rod which extrudes from the narrower end of the cone. This is chopped into 20 cm. lengths by a rotating knife and passes on to a conveyor. The conveyor, which may be of any length from 30 to 150 metres, consists of a series of horizontal plates. As soon as all of these are filled they all drop, depositing a row of peat blocks on the surface of the bog where they remain until air dried. The machine carries on until the conveyor is full again and deposits another row parallel to the first. These rows are, of course, at right angles to the direction in which the machine is travelling. A cable is fixed to a post some distance in front and the machine which is mounted on rollers pulls itself forward by winding up this cable on a windlass. The machines generally cut strips of from one to two kilometres long at a time and require a flat drying ground parallel to the cut face. We saw one of these machines driven by electricity and another by an internal combustion engine. The horsepower required is from 75 to 90. Again from Schwegermoor's figures, the quantity cut by a machine per day is from 65 to 70 tonnes expressed as the air dry weight or 260 to 280 tonnes as dug from the bog.

We also saw a smaller model without conveyor or cone which was used to strip the topsoil (heather, grass, etc.) and cast it to the side. The machines used at Schwegermoor were made by Heseper Torfwerk, Meppen, and those seen in Oldenburg province were made by Strenge, Gebh., Torfindustrie, Ocholt i.O. Other makers are listed in trade directories.

The product is known as PRESSTORF, pressed peat, and is used as a domestic fuel, for gas generation, and for coking.

Notes:-

A full description of the Wielandt machine may be found in Handbook on the Winning and Utilisation of Peat, by A. Hausding, published by the Fuel Research Station in 1921. The Peco process does not seem to have been used in Germany though Carl Schenk's plant may be similar.

The following notes are taken from observations made by previous BIOS teams:-

Torfwerk Rottau, Strafanstalt Bernau, Bavaria Director: Regierungsrat Dr. Weber. Products - peat since 1942. 10,000 tons a year planned, but 4,500 tons in 1944.

State owned and capitalised. Only hutments and 2 houses. 5 steam engines for cutting, each cuts 1,000 tons in 4 months season. 600 tons peat fuel a year required. Labour recently 10 men 15 women; required 300 men and 200 women. Stock 300 tons of peat. No laboratory.

Torfwerk Feilnbach, Feilnbach, bei. Aibling, Bavaria
Owner, Josef Fueglein. Make 5,500 tons of peat a year and hope to make 6,000 tons. Capital Rm 300,000. Hutments for drying the peat were noted, 6 steam engines each cutting 1,000 tons during the 4 month's season. Fuel requirements per annum are 700 tons of peat (1935-1944 average). Labour till recently only 16 men and 3 women; they need 60 men and 20 women. No laboratory. Stock 1,000 tons. Peat cutting is seasonal, and starts in April.

Peat machines are listed in "Wer Liefert Was"(1941) as under:-

Dziabaszewski, J., Posen, Margaretenstr 39.
Einberg, Andreas, of the same address
Osenberg, Ferdinand, Berlin-Lichtenberg, Herzbergstr 24/25.

F. MISCELLANEOUS USES

FIRELIGHTERS

Report 6. Dr. Wilhelm Wielandt, Oldenburg, i.o., Hochhauserstrasse, 1. works, Torfwerk Langenmoor. Dr. Wielandt's daughter was seen and she showed a sample of the firelighter which Wielandt makes. This is about 20 cm. by 10 cm. by 1 cm., is of light brown peat and is dipped in the tar obtained as a byproduct in Wielandt's distillation process. The tar penetrates a certain distance, but there is an untreated cone about 4 or 5 mm. thick. They are packed in packets of ten, but we did not discover the price.

Hannoversche Kolonisations und Torfverwertungs A.G. also make firelighters of the same size. They have a sawbench on which the incoming slabs are quickly trimmed to size and sliced to the appropriate thickness. The dust falls into a trough below where a screw conveyor carries it along to an elevator which takes it up to the peat moss litter baling plant. There is no wastage in this plant. This firm send away these slabs to be dipped in tar, but beginning April, 1946 they intend to carry out their own dipping and packing.

FIRELIGHTERS, EGG BOX FILLERS, ENTOMOLOGICAL SPECIMEN HOLDERS

C22/3197 C30/392
C31/2568

Report 8 Wilhelm Haar, Bremen, Donaustrasse 6 had a small business making firelighters from peat. This business is no longer in existence, but he now makes in his own house forms for packing eggs. These are merely dried slabs of light brown peat in which cups the size of an egg have been drilled, thus, in a slab about 25 cm. x 20 cm. x 5 cm. it is possible to drill cups for a dozen eggs. He also makes boards for mounting entomological specimens on. These are slabs of light brown peat of any desired size, - the one we saw was 30 cm. x 20 cm. x 2 cm. - covered with white paper. They are ideal for pinning insects to and he has supplied them to museums in Bremen and elsewhere.

COMPOUNDED FERTILISERS AND FLOWER POTS

Report 4 A Beeck inh. R. Jung. Herr Jung mentioned a type of peat fertiliser marketed in Germany under the name of HUMINAL. In this granulated peat (which was measured by volume) was mixed with unspecified chemical fertilisers (obtained from I.G.) in an ordinary scroll mixer, and baled in the usual way. This mixture had a very good market and quite a lot was exported. In a further development of this idea, flowerpots were moulded out of this fertiliser mixture. Plants could be grown very easily in them, only watering being required, and, when potting out, the pot is merely buried in the ground. The moulding appeared from his description to be hexagonal instead of circular, with only a small cavity for the plant in the centre.

HUMIC ACID

Report 9 Dr. Carl Daimler, I.G., Farbenindustrie A.G., Hoechst am Main.

C22/1g(a) C30/188(a)
C31/1788(a)

Dr. Daimler developed the use of humic acid as an emulsifying agent and emulsion stabiliser for road emulsions. In his original experiments he extracted it from peat with sodium hydroxide or sodium carbonate solutions, but found the extraction too costly and it was superseded by the discovery that in two places in Germany there are bands of almost pure humic acid in the brown coal deposits. Now they merely grind this up finely and use it direct. The mixture which it is used to emulsify contains tar, Mexico bitumen, and asphalt and is used as a road material.

Six per cent. humic acid is added, and it is said to act both as emulsifying agent and emulsion stabiliser. The humic acid is not water soluble. Dr. Daimler said sodium humate was not satisfactory for this purpose. Two other uses for this humic acid which he mentioned are in Kassel Brown a cheap pigment used for floor staining, and in pigmenting brown paper. The patent covering this use of humic acid was taken out in 1928 and has now expired.

G. PEAT, ADDITIONAL INFORMATION GATHERED BY BIOS TEAMS.

Agriculturchemisches Institut Weihenstephan der Technischen Hochschule Muenchen. Location: Weihenstephan, bei Freising, Bavaria. Measurements of soil fertility including that of low moor peat soils.

Süd-Chemie A.G., Moosburg, Bavaria, manufacture Nettolin from peat. Composting peat with nitrogenous materials and subsequent addition of PO_4, K_2O, and $CaCO_3$.

Institut für Pflanzenernährung und Bodenbiologie der Landwirtschaftlichen Hochschule, Hohenheim bei Stuttgart. Fertility of soils, research:-

(a) Products consisting of peat which has been subjected to treatment or enrichment with other substances: Huminal A (1.7 per cent. N), Huminal B (ca. 1-2 per cent. each of N, P_2O_5, and K_2O), Nettolin (ca. 3-4 per cent. each of ditto), Humunit, Gaeta (Gaertorf).

(b) Products consisting of peat mixed, with sewage or sewage sludge: Biohum, Almaghumus, Duengehum, Faedue (Faekalduenger), Faekaltorf, Torffakalduenger, Terrabona.

((c) Sewage sludge).
(d) Products consisting of animal wastes mixed with peat. Fihumin (mixture of fish waste and peat), Gaerhumin.
((e) Other organic substances)
((f) Unknown composition)

It has not yet been demonstrated that these products have a humus action on the soil as regards their physical, chemical, and biological effects. The composition of several of the products, together with the availability of the N, P_2O_5, and K_2O as determined by means of pot experiments, is as follows:-

Material	Composition %			Proportion readily utilised by plant %		
	N	P_2O_5	K_2O	N	P_2O_5	K_2O
(a) Huminal B	2.0	1.4	2.5	75	100	ca 100
(a) Nettolin	3.2	3.0	4.2	33	ca100	ca 100
(b) Almaghumus	0.9	1.6	0.1	10	75	12
(c) Elka-Duenger	1.8	1.3	0.9	10	50	20
(d) Fihumin	5.5	4.8	0.7	50	10	?

Dr. R. Runkel, Institute for Wood and Plant Chemistry of the Forest Research Institute and Soils Experimental Station Reinbeck, Hamburg

Because of the grave shortage of animal feeding-stuffs and the demands on German forests in Western Germany, Dr. Runkel has been working on modifying the Bergius process to use sphagnum peat as a raw material for saccharification. Fodder yeast has been grown experimentally on saccharified light peat about 1930 but the present economic conditions justify further investigations and trials at the yeast-from-saccharified-wood factory at Tornesch in Holstein (method Scholler), provided that the necessary nutrient chemicals, phosphates and nitrogen compounds, can be obtained.

A more promising method is thought to be the direct cultivation of yeasts on acid-washed peat impregnated with solutions containing phosphorus, nitrogen and potassium. The acid wash here removes bacterial substances, but does not actually saccharify the peat. It is recommended that priority be given to developing this process, as it is quicker and requires less complicated plant.

The following items connected with the peat industry require to be looked into and incorporated in a general summary of the German peat industry.

1. Peat used in magnesium production.
2. Peat used for making active carbon.

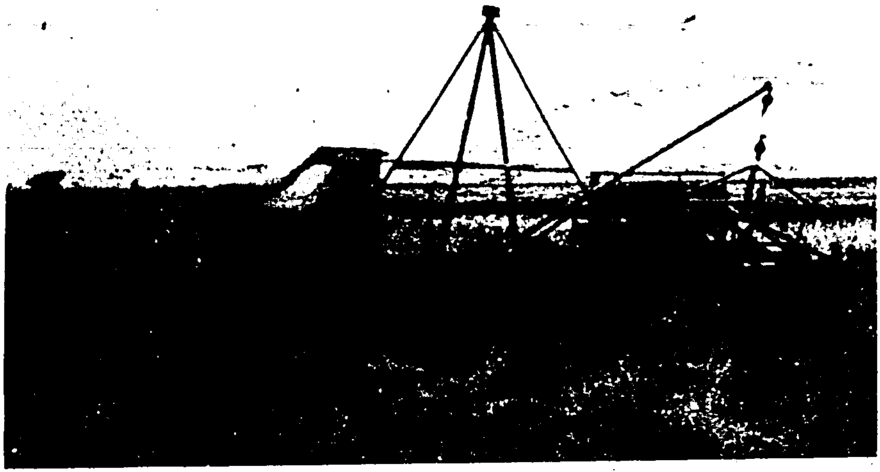
3. Peat used as a diluent in making asbestos rope.
4. Peat used for making hard building boards.
5. Peat used for making bricks and slabs by being mixed with clay and pressed, (DRP 708,659, F Helms).
6. Peat used for "Triumph" sanitary closets, Torfitwerke G.A., Haseke & Co., Bremen-Hemelingen, and Franz Genth, Kréfeld, Oppumer str. 83/85, "Universal"peat closets.
7. Process for drying peat, Madruck-Werke für maschinelle Druckentwasserung m.b.H., Staltach, Oberbay, Bavaria.
8. Torf-Forschung und Verwertung Ing. August Tanner, München 2, Rosental 19, (peat briquettes).
9. RKTL, Abteilung Torfteknik, Berlin.
10. Versuchsanstalt für technische Moorverwertung, Technische Hochschule, Hannover; Director, Professor G. Keppelér.
11. Agricultural peat investigation station, Bremen.
12. Carl Schenck, Eisengiesserei und Maschinfabrik, Darmstadt 6 m.b.H., Darmstadt, Lanwehr str. 55, manufacturers of machinery for:
    (a) the mechanical means of winning peat moss litter from the earth in a reasonably fine divided form (say ⅜ inch) so that it can subsequently be compressed into briquettes.
    (b) The mechanical means of cutting from the earth peat blocks of from 9 to 14 inches by 5 inches by 2 inches. Peat excavators, elevators and presses.
13. Maschinenfabrik und Eisengiesserei Vorm C. Haehne and Sohn G.m.b.H., Landsberg (Warthe) manufacturers of peat cutters and presses.
14. Torfwerk Teufelsmoor, Torf-, Torfstrew - und HUMINAL - Fabrikation, New St. Jüngen (über Osterholz-Scharmbeck), bei Bremen.

H. SUMMARY

Several facbries in Germany have made light-weight thermal insulation slabs from peat economically, and many firms make peat moss litter and granulated peat for agricultural purposes. Granulated peat fortified with artificial fertilisers are also fairly widely known.

In the peat areas pressed peat is produced by Wielandt or similar machines, but no satisfactory machines are known for cutting light brown peat. Press peat is widely used as fuel and less commonly for gas generation or coking.

Research and development work into other uses of peat is being conducted in many places, but many firms take little interest in research.



1) Strenge Turf Remover.
Ederwechterdamm, Oldenburg.

2) TRIANGEL, near Gifhorn.
Houses insulated with peat boards.

B.I.O.S. FINAL REPORT No. 597

ITEM No 30

INTERROGATION OF
DR. C. H. N. BENSMANN
OCTOBER 10th, 1945.

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Thomas, W. H.; Faragher, W. F.

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON-H.M. STATIONERY OFFICE

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LONDON-H.M. STATIONERY OFFICE

INTERROGATION OF DR. C.H.N. BENSMANN

OCTOBER 10th, 1945.

Reported by:

W.H. Thomas, British, Ministry of Fuel and Power.
W.F. Faragher, U.S. Petroleum Administration for War.

B.I.O.S.Target Number : C30/456
Fuels and Lubricants.

British Intelligence Objectives Sub-Committee,
32, Bryanston Square,
LONDON, W.1.

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PERSONNEL OF TEAM.

W.F. Faragher, U.S. Petroleum Administration for War.

W.H. Thomas, British, Ministry of Fuel and Power.

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1. SUMMARY.

Dr. Bensmann was interviewed with regard to his previous contacts with the Texas Company of America on Wednesday 10th October 1945 at Internment Camp 0.31, Nienburg.

Dr. Bensmann, a Doctor of Laws, is the proprietor of United Oils Patents Co. of Bremen. He has had practical refinery experience in Roumania and is a Patent Agent. Prior to the War, he was a member of the Reichstag and a Free Mason but during the war he served in the Intelligence Sections of the German Air Force and Navy but apparently had ample time to carry on his normal occupation of Patent Agent.

Dr. Bensmann provided an account of his contacts and business with the Texas Company and showed how the licensing of the Gray-process in Europe was effected. He also gave a review of the events which led to the Texas Company being appointed agent for the Fischer-Tropsch process in the United States.

2. INTRODUCTION.

Dr. Bensmann had intimated to the authorities in charge of Internment Camp 0.31 - Nienburg that he wished to consult a member of the United States Control Commission with regard to certain business which prior to the war had been handled by him on behalf of the Texas Company of America.

It was known that a party of British and American technical investigators would be working in the Hamburg area during the month of October 1945 and it was therefore arranged that two of the members thereof (one American and one British) should visit Dr. Bensmann for the purpose of ascertaining the nature of the communication which he wished to make.

The present report gives an account of the conversations which took place on that occasion.

3. ACCOUNT OF THE INTERVIEW.

Dr. Bensmann was interviewed by Dr. W.F. Faragher (U.S.A.) and Major W.H. Thomas (Br.) on Wednesday October 10, 1945 at Internment Camp 0.31, Nienburg. The following is an account of the interrogation which took place.

Dr. Bensmann's [illegible] and documents are [illegible] office at Bremen; [illegible] and prior to the War [illegible]. During the War he served in the Intelligence Section of the German Air Force and Navy, but apparently had ample time to carry on his normal occupation of Patent Agent.

He is the proprietor of a firm originally known as "Universal Oils Patents Co." Bremen, the title of which was changed in 1939, at the suggestion of the Texas Co. to "United Oils Patents Co.". The funds of the latter were held by the Deutsche Bank Filiale, Bremen.

Dr. Bensmann is a Doctor of Laws - Leipzig 1924; he has had practical refinery experience in Roumania Creditul and is now Patent Agent. His father was Hermann Bensmann who marketed American Fullers Earth (Floridan) in the Eastern Hemisphere.

In 1927 contact was made with the Texas Co., with reference to the Gray process and interviews were effected between Dr. Bensmann and Mr. T.T. Gray, R.J. Deerborn and W.F. Moore.

In 1931 the patent side of the Bensmann business was sufficient to warrant the formation of a separate Company (Universal Oils Patents Co.) to cover the main contacts the Texas Co., and Ruhr Chemie. The Gray process was taken over by the Texas Co., in 1933 and Dr. Bensmann then started to handle the Texas Co. patents in the German Patents Office but some of the work was done by a German Attorney (Kuhl of Berlin). The Texas Company covered the European patent work for a variety of firms (Standard, Phillips, Sun, Union, Shell, Pure, Humble and Bataafsche) on clay treatment, thermal cracking and vapour phase refining. Isomerisation was to be included but not polymerisation.

In 1935 Dr. Bensmann handled processes other than the Gray for the Texas Co. and solvent refining and dewaxing were included. Further contact with Ruhr Chemie was made in 1937 when a Gray license was sold to Fischer-Tropsch and the Gray plant was subsequently erected at Holten Oberhausen-Sterkrarde.

In 1938 the Texas Co. was appointed agent for the Fischer-Tropsch process in U.S.A. and all countries outside Germany. Subsequently negotiations took place for the erection of a Fischer-Tropsch plant in the U.S.A. and four engineers (two from the Texas Co., and two from the Standard of Indiana) spent two months at Holten in examining the process. R.J. Deerborn visited Germany in May 1938 and again in September 1938 and as a result, the Standard of Indiana, in order to avoid duplication, decided against purchase. The Texas Co. took a license and agency rights, the agreement for which was signed during September 1938.

In March 1939 Dr. Bensmann went with Prof. Martin and Dr. Tramm of Ruhr Chemie (head of Fischer process and now with Dr. Otto) to New York to sell the Ruhr Chemie patent rights in the U.S.A. to the Texas Co. and he exchanged with the latter certain rights in Germany; both contracts were independent. Payment to the Texas Co. was to be spread over 5 years; one and a quarter million dollars in cash and the remainder over 5 years.

Dr. Bensmann received a sale fee in addition to his charges as the Texas Co. representative and stated that during the War he had successfully protected the patents from seizure by the Nazi Party. He moreover maintained the patents and borrowed money from Ruhr Chemie to pay the patent fees.

He considers that infringement of these patents has probably occurred by order of the German Government and he has been unable to check refinery through-puts with the firms concerned. He observed that Gray treatment was essential in Germany because of shortage of sulphuric acid.

Dr. Bensmann sold six licenses to Still (Rechtlinghausen) and Wilke (Braunsweig) as a result of patent litigation and received payment on behalf of the Texas Co., of R.M. 60,000 then R.M. 15,000 and the remainder is still to be paid. During the War he was in touch with M. Legrand of La Gironde Cie., six or seven times in Paris and was last there in June 1944. Legrand paid no money to Dr. Bensmann but advised the latter to maintain the Texas patents. Dr. Bensmann stated that he had attempted to communicate with the U.S.A. via Sweden but with no success. He is hoping to make contact with the Texas Co. as soon as possible, but this will depend on his period and type of internment.

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B.I.O.S. FINAL REPORT No. 586

ITEM No 30

ERNST SCHLIEMANN'S OELWERKE UND EXPORT-CERECIN-FABRIK HAMBURG - GERMANY

LUBRICANTS AND MISCELLANEOUS PRODUCTS

Thomas, W. H., Withers, J. G.

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ERNST SCHLIEMANN'S OELWERKE UND EXPORT-CERESIN-FABRIK

HAMBURG - GERMANY

LUBRICANTS AND MISCELLANEOUS PRODUCTS

October 1st - 5th 1945.

Reported by:

W.H. Thomas, British, Ministry of Fuel and Power,

J.G. Withers, British, Ministry of Fuel and Power.

B.I.O.S. Target No.C30/405
Fuels and Lubricants

British Intelligence Objectives Sub-Committee
32 Bryanston Square,
London, W.1.

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PERSONNEL OF INTERROGATING TEAM.

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J.G. Allen - U.S. Petroleum Administration for War.

W.H. Thomas - British, Ministry of Fuel and Power.

J.G. Withers - British, Ministry of Fuel and Power.

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ACKNOWLEDGEMENT

German translations involved in the production of this report were made by Mr. K.G.C. Kirkpatrick.

1. SUMMARY.

This report gives information obtained from Ernst Schliemann's Oelwerke und Export-Cerecin-Fabrik regarding its products and methods of preparation thereof.

These products were very varied in type and included most of the known specialised lubricants, anti-corrosion oils, greases and plant spray oils. No aviation lubricants were made.

The information provided is in the form of answers to a series of questions designed to cover the manufacture and utilisation of lubricants and special products of similar character.

Fourteen samples of the Schliemann Company's products are listed at the end of this report; they are at present in the custody of the Fuel Research Station, Greenwich.

2. INTRODUCTION.

The information given in this report was obtained from Herr H. Grünewald - Chairman and Dr. H. Schünemann - Chief Technician of the Schliemann companies in the course of a visit (B.I.O.S. Trip No. 1248) to the Hamburg area during the period October 1st to 31st 1945.

The purpose of the interrogation was to ascertain what were the war-time activities of these firms and to obtain details of the various manufactured products. An additional object was to obtain samples of typical products for subsequent examination.

3. GENERAL.

Apart from the refineries at Grasbrook and Wilhelmsburg, the Schliemann firm owns Ernst Schliemann Export-Cerecin-Fabrik with works at Billbrook on the Elbe and a factory in Holland under the name of Witol.

The Grasbrook and Wilhelmsburg refineries were extensively damaged by air-attack. They possessed no distillation plant but were engaged in the refining and blending of lubricating oil and the preparation of greases.

During the war, they operated on existing stocks of residues from Russia and Roumania and on topped crude from Misburg (Wietze and Heide crudes), the products comprising an extensive range of transformer, turbine, medicinal and white oils, greases, mineral jelly, textile and cutting oils, horticultural and insecticide oils etc. Refining was by conventional acid and clay processes.

The naphthenic acids used for certain products were separated during the processing of the Russian and Roumanian residues and when stocks of the latter had been exhausted, reccurse was made to German petroleum residues which possess a much lower content of naphthenic acids and hence were much less satisfactory.

The refineries are now operating at 20% of original capacity on topped residue from Misburg using sulphuric acid and clay obtained from Munich as the chief refining agents.

The wax factory at Billbrook prepared wax products from hydrogenation and oil refining processes.

4. ANSWERS TO QUESTIONNAIRES.

QUESTIONNAIRE No. 1

FUELS

Products under this heading were not supplied by us and we can therefore give no pertinent information.

QUESTIONNAIRE No. 2

LUBRICANTS

A. Aviation

Those were not made by us.

B. Automotive.

Question 1. To what specifications are lubricants for petrol engines and high speed diesel engines produced, and what relative importance is assigned to the various properties?

Answer. Manufacture follows the "Guide to the Buying and Testing of Lubricants" issued by the Verein Deutscher Eisenhuttenleute (Union of German Ironmasters). These oils were not made to Army specifications, being delivered only to trade and industry.

Question 2. What are the chief sources of motor oils? What crudes are used, what refining methods and to what extent are synthetic oils incorporated?

Answer. In general, raffinates from different sources were mixed and blended with bright stock. Formerly, Russian and American raffinates were mixed with bright stocks of the same origin. Later, these foreign raffinates were replaced by German ones from the Hannover region and the Viennese basin. Synthetic oils were not processed.

Question 3. Are engine tests carried out before approval and if so, what are they?

Answer. Engine tests were not carried out.

Question 4. What laboratory oxidation tests, or sludge tests are used and how do they correlate with performance in an engine?

Answer. Laboratory oxidation tests were only carried out with Diesel engine oils. The method of Brown, Boveri & Co., was used (heating the oil at 110°C. for 70 hours in the presence of copper plate). If oils tested in this way showed no sludge formation, they gave no trouble during running. The Conradson coking value also, was determined with automotive oils.

Question 5. To what extent is Voltol used in motor oils? Is it more commonly used in motor oils than aviation oils and if so, why? What particular advantages in engine cleanliness, viscosity index, etc., have been found with Voltol?

Answer. We have no experience in this respect.

Question 6. Are detergents other than Voltol used in :-

(a) petrol engines?
(b) diesel engines?

Answer. No additives were made by us.

Question 7. Is trouble experienced with ring-sticking in diesel engines and if so what palliatives are used?

Answer. We have no experience of this.

Question 8. What type of filters are used in mechanically propelled vehicles? If chemical type filters are used, is any trouble experienced with them?

Answer. We have no experience of filters.

Question 9. What mileage is normally recommended between sump drainings?

Answer. Oil changing after 3-4000 Km. was always recommended.

Question 10. What inspections are carried out on used oils and how do the various types of oil compare on used oil conditions?

Answer. The insolubles, acid value and ash content of used oils were determined, as well as their properties after sulphuric acid regeneration.

The general experience was, that paraffin-base and pure naphthene-base oils behaved better than mixed and asphalt-base oils.

Question 11. What additives are used as pour point depressors, V.I improvers, anti-oxidants, antiwear additives, anti-corrosion additives and film strength improvers?

Answer. Paraflow was used to depress the pour point and a limited amount of Oppanol was occasionally employed as a V.I improver. No other additives were used.

Question 12. To what extent are fatty oils used in motor lubricants? What kind of fatty oils are used and what advantage have they?

Answer. In peace-time a motor oil with 3 per cent castor oil was made as well as unblended motor oil. Its special advantages were :- good pressure behaviour and better spreading qualities.

Question 13. Were any lubrication difficulties experienced with producer gas equipment (both wood and coal types) bottled gas, or acetylene driven vehicles?

Answer. No information is available.

C. <u>Marine.</u>

Question 1. What lubricating oils are specified for use with marine diesel equipment?

Answer. Good lubricating oil raffinates were usually recommended as diesel oils. For circulation lubricating, especially well refined oils were manufactured.

Question 2. What oils are used in steam turbine driven vessels? Are additives of any kind incorporated (anti-corrosion etc.)?

Answer. Exactly the same turbine oils were delivered as used on land. No additives were employed.

Question 3. To what extent are compounded cylinder oils used and what are the compounding agents employed?

Answer. Compounded cylinder oils were not supplied.

Question 4. To what specifications are stern-tube greases manufactured and what compounding agents are employed?

Answer. Stern-tube greases were not manufactured.

Question 5. What were the uses of aqueous emulsions of oil; was an addition of montan wax employed?

Answer. Aqueous emulsions were not supplied.

D. Transmission

Question 1. What lubricants are recommended and specified for tank gear boxes and car and truck gear boxes of normal and synchromesh types?

Answer. No gear oils or greases were supplied for use in tanks. High pressure gear oils and greases were supplied for normal automotive gearing.

Question 2. To what extent are Voltolised oils and compounded oils used for tank gear boxes? What compounding agents are employed?

Answer. We did not use voltolised oils in the manufacture of gear lubricants. For compounding gear oils having a viscosity of 20-30 Engler at 50°C., about 10 per cent of blown rape oil was added. In peace time a limited amount of high-pressure gear oil was made, with about 2-5 per cent of lead oleate.

Normal semi-solid greases were manufactured based on the above oils with the addition of about 10 per cent of sodium tallow soap.

Question 3. To what extent are additives of the nitrogen type, chlorine type etc., used in oils for gear boxes?

4. In what proportion are straight tooth, spiral bevel, hypoid and worm gears used in rear axles and what oils are specified in each type?

Answer. Chlorine and nitrogen containing additives were not used.

For normal auto-gearing, gear oils with a viscosity of 20-30 Engler at 50°C. were delivered, without any special additions. For helical and hypoid gears, the high pressure oils mentioned under (3) were used with the addition of lead oleate. The bulk of the gear oils supplied by us to the German market were without additives.

Question 5. What additives are used in extreme pressure oils?

Answer. Only lead soaps were added to extreme pressure oils.

Question 6. To what extent are compounded oils (fatty oil compounded) used for rear axle lubrication?

Answer. At times compounded oils with a limited addition of blown rape oil and train oil were ordered.

Question 7. What types of oil seals are used?

8. What temperature extremes are encountered in the different types of axle?

Answer. No information available.

E. Hydraulic, Recoil and Buffer Oils.

Question 1. To what extent are pure mineral oils used for this purpose? What speciifications apply with particular reference to V.I, pour point, etc.? From what crudes are they made and by what refining methods?

Answer. Pure mineral oils were recommended for industrial hydraulic purposes during the war. If the machinery was exposed to cold weather, low pour point products similar to transformer oil were advised.

The Luftwaffe had special specifications including pour point (-50°), viscosity (2°E. max. at 20°C.) and the swelling of Buna. These tests were carried out in the Luftwaffe's own laboratories. The only suitable crude oil available to us was a limited quantity of a light fraction from Russian 'Solarol'. Delivery was made for further processing, to the firm of Lotzin, Hamburg. We assume that for oils submitted to extremely trying conditions only synthetic products were available.

Question 2. To what extent are non-mineral oils used for this purpose? How much castor oil is used for this purpose?

Answer. Castor oil and amyl alcohol products were commercially available as hydraulic buffer oil. We ourselves did not manufacture such products.

Question 3. What solvents such as diacetone alcohol, methyl carbinol, etc. are used?

Answer. We know only of the use of amyl alcohol as an additive.

Question 4. To what extent are water and glycerol used in hydraulic fluids?

Answer. We did not make glycerine water mixtures. We have no information on the percentages used in practice.

Question 5. What work has been done on non-inflammable hydraulic oils?

Answer. We did not make non-inflammable hydraulic oils but emulsions of water soluble oils were often used for hydraulic presses. Such emulsions, containing 5 per cent of oil, are non-inflammable.

Question 6. What oils seals are used and has any trouble been experienced as a result of interaction between seals and fluids?

Answer. No information.

Question 7. To what extent are compounded mineral oils used and why?

Answer. We did not recommend fatted mineral oils for hydraulic purposes.

F. Railroad.

Question 1. What oils are used for steam cylinder lubrication (saturated and superheated) valves, axle boxes etc. and what compounding agents are used and why?

2. What types of crudes are preferred for this purpose? What asphaltene content is usual in such oils?

3. Are any special oils used in the lubrication of diesel locomotives and what additives, if any, are employed?

Answer. No information.

G. Industrial

Question 1. What types of oil are used in steam turbine lubrication? Are any troubles experienced with corrosion, emulsification and if so, how are they combatted? What additives are used, if any? What is the average life of a steam turbine oil?

Answer. Specially prepared raffinates were used for steam turbine lubrication, corresponding to the specifications in the "Richtlinien". Individual firms such as Brown, Boveri and A.E.G. had their own particular specifications. Oil decomposition, corrosion and emulsion formation were always found to originate in poor working conditions or improper treatment of the oils and could be remedied by removing the local sources of trouble. No special additives were used. The average life of turbine oils was about 40,000 hours. In plants where special care was exercised, the running time was up to 80,000 hours. The best results were given by raffinates of Russian origin.

Question 2. What types of oils are used for transformers? What electrical tests, if any, are carried out? Are any additives employed?

Answer. Raffinates from anti-cold spindle oil distillates were produced for transformer oils, corresponding to the specifications of the "Verband Deutscher Electrotechniker" and the "Wirtschaftsgruppe Elektrizitätsversorgung". Oils for export were prepared according to the B.B.C. Specifications (Switzerland), the Michie Test (England), the ASEA test (Sweden) etc.

The only electrical test made was for break down voltage. No special additives were used.

Question 3. To what extent are mineral, non-mineral, compounded oils and aqueous emulsions used in quenching? What types are preferred for small and large objects?

Answer. For tempering, the oils used were generally pure mineral oil raffinates, with viscosities between 2° and 6° Engler at 50°C. For annealing, cylinder oils with a high flash point were recommended, especially bright stocks.

For "polished surface" tempering (Blankhärten) selected raffinates were used, which formed no coking residue.

Mineral oil raffinates with viscosities of 2-4°E./50°C. were recommended for small objects. To meet certain requirements, the grade of purity chosen was that of transformer and turbine oils.

For dealing with large components, machine oil raffinates with a viscosity of 6.5°E./50°C. were employed.

Question 4. What types of cutting oils are used? To what extent are sulphurised and compounded oils used? Are corrosion inhibitors added and if so, what are they?

Answer. Spindle oil distillate with 1-2 per cent of added sulphur was preferred for use as cutting oil. For hard and tough steels, making a particularly big demand on the tool, such sulphurized oils were given an addition of up to 5 per cent fatty oil. No anti-corrosive media were added.

Question 5. What types of slushing oils are used? What additives are employed to give anti-corrosion properties?

Answer. Flotation oils were not manufactured.

Question 6. From what sources are textile oils refined? What research has been done on carcogenicity?

Answer. Textile oils were formerly prepared mainly from Russian oils and later, during the war, from German raw materials. No research was done on skin diseases.

Question 7. What oils are used for clock and instrument lubrication, especially for aviation use? What compounding agents are used?

Answer. As a base for clock oils, white oils of Russian origin were delivered to specialised manufacturers. No additives were made by us.

Question 8. What is "Radiol" and for what uses is it employed?

Answer. "Radiol" is unknown to us.

Question 9. What is the composition of compounded light machine oils?

Answer. For light, fatted, machine oils we believe neatsfoot oil and olein were employed as additives.

H. Greases

Question 1. To what extent are barium, lithium and aluminium soap greases used?

Answer. Barium and lithium soap greases were not produced. Special aluminium soap greases were made before the war for ball bearings.

Question 2. What fats are used for greases and what substitutes have been employed?

Answer. Technical fatty acids, such as those from train oil and tallow were used in the manufacture of machine greases. Synthetic final distillate fatty acids were used as substitutes during the war

Question 3. To what extent are waxes, especially montan wax used in grease manufacture?

Answer. Crude montan wax was used for making ordinary cup greases which were not put to any severe test. Refined montan wax was used for "hot-neck" greases.

Question 4. What types of greases are used for high temperature applications?

Answer. "Hot-neck" greases were made from the potassium and sodium soaps of tallow and refined montan wax.

Question 5. For what reason and to what extent are greases used in shock absorbers for aeroplane landing gear?

Answer. Shock absorber greases were not prepared.

Question 6. What material is employed for ship launching purposes?

Answer. Products made from crude paraffin wax and spindle oil were used for ship launching purposes.

I. General

Question 1. To what extent are aqueous emulsions used in industrial lubrication?

2. Has this type of lubricant ever been employed in engines of any type?

3. By what processes is Voltol made?

Answer. No information is available. Voltol was made by the Shell Company, Rhenania-Ossag.

QUESTIONNAIRE NO. 3

SPECIAL PRODUCTS

A. Bitumens

No information.

B. Extracts and Polymers

No information.

C. Waxes

Question 1. To what uses are waxes produced from petroleum brown coal oils and Fischer-Tropsch processes being applied and what particular properties are demanded by the user?

Answer. Synthetic wax from the Fischer-Tropsch process was used as an additive in vaseline manufacture. Otherwise such waxes were not used by us.

Question 2. What types of waxes (with specifications) are used in candle manufacture, paper impregnation, insulating, etc.?

Answer. No information.

Question 3. To what extent is slack wax from solvent dewaxing processed into a usable wax and how is this done? What proportion is used as a cracking stock?

Answer. The following types of waxes are bleached and refined:

(a) Slack waxes such as petrolatum and paraffin waxes from solvent dewaxing plants of mineral oil works.
(b) Wax residues (Röhrenwachs) from pipe lines and oil tank bottoms of German and foreign petroleum installations.
(c) Natural earth waxes (ozocerites) from Poland, Roumania and Russia.

The refining process for waxes is as follows :-

From 15 to 50 per cent of fullers earth is added to wax heated to 120-130° centigrade. The type of fullers earth used is the "Tonsil" brand of the Südchemie A.G. Munich, Bavaria. When the fullers earth is added the temperature is brought to 170° centigrade with continual stirring of the mass, which is finally filtered by pressing. The bleaching loss is equal to the quantity of fullers earth used and varies accordingly.

The wax absorbed by the fullers earth is extracted by solvents and the extracted wax must again be bleached in the same way as the crude wax.

The colour of the bleached waxes is between amber and light yellow.

Refined waxes of lighter colour can be obtained by bleaching with aluminium chloride which takes place in a container with an outlet at the bottom. From 10 to 15 per cent of aluminium chloride is added to the wax heated to 120° centigrade. The temperature is brought to 170-180° centigrade and stirring is continued for 4 hours. Tars separate and settle down within 1-2 hours and are drained off through the outlet. These tar residues are of no further use. The wax thus treated is bleached in another vessel with 10 to 20 per cent of fullers earth in the same way as already described. The final result is a refined wax of an ivory or light yellow colour.

If snow white waxes are to be made the wax must be refined with sulphuric acid as follows :-

The wax treated with aluminium chloride, but not with fullers earth, is brought to 120° centigrade in a directly heated vessel. From 20 to 40 per cent of monohydrate sulphuric acid are now slowly added. By further heating the temperature is brought to 220° centigrade, when the SO_2 gases will be driven off. The wax is cooled down to 170°centigrade and about 10 to 15 per cent of fullers earth is added with stirring and the mass is filtered at 130°centigrade by pressing. The wax remaining in the fullers earth is extracted by solvents and the extract is again bleached by fullers earth.

Natural crude ozocerite and "Röhrenwachs" (wax from oil pipes and from oil tank bottoms) are refined by sulphuric acid as described above. They are, however, not treated with aluminium chloride.

Yields.

(1) Bleaching with fullers earth gives net yields of about 92 per cent of bleached material.
(2) Refining with aluminium chloride gives yields between 70 and 78 per cent of refined wax.
(3) Refining with sulphuric acid gives yields of 48 to 92 per cent depending on the nature of the crude wax.

Question 4. What results have been obtained with the use of montan wax and synthetic substitutes in grease manufacture?

Answer. Montan wax was used with good results in the manufacture of cup greases and emulsifying greases. It was also used in combination with synthetic final distillate fatty acids for making cup greases.

Question 5. What work has been done on micro-crystalline (Staufferfette) waxes?

Answer. Micro-crystalline waxes (ceresines) were utilised for the production of vaseline and similar products.

D. Rust Preventatives

Question 1. What rust preventatives of the solvent type suitable for spray application are used and what is their composition, with particular reference to corrosion inhibitors?

Answer. For spray application, rust preventatives consisting of emulsions of boring oil and water were recommended. The boring oils, being based on naphthene sulphonic acid, had a good anti-rust action. No further additives were used.

Question 2. What corrosion preventatives of the petrolatum type for brush or swab application, of the transparent hard drying type and of the permanent aluminium paint type are used? Are any special additives used? What solvents are employed?

Answer. Petrolatums with drop points of about 50°C. were used as anti-rust greases without any additives. Solvents were not added.

Question 3. Are "fly-away" corrosion preventative oils used, and what is their composition?

Answer. No information.

Question 4. Have any special compounds been produced to combat cold corrosion in stored engines?

Answer. Protective oils, based on naphthene sulphonic soaps in mineral oil distillate, soluble in water, were used for this purpose.

Question 5. Are any corrosion preventative additives manufactured for addition to turbine oils, transformer oils, etc.?

Answer. No.

E. Cable Oils

Question 1. To what extent are oil-filled cables used? What oil specifications are laid down; what are the best sources for oils of this type?

Answer. We know that hollow, oil-filled cables were used as well as solid cables. Low viscosity, Russian based, transformer oils were preferred for this.

Question 2. Are any blending agents or additives employed (e.g. resins)?

Answer. No additives were used, as far as we know.

Question 3. To what extent are oil-impregnated insulated types used? What oils are used and what specifications have been laid down?

Answer. In the manufacture of solid cables, mixtures of resin and viscous mineral oil of the bright stock type were employed. The cable works issued the relevant specifications.

Question 4. What methods of test are used to determine the di-electric strength of such oils?

Answer. Electrical tests were used for ascertaining the break-down voltage and the voltage loss (VerlustWinkel).

F. Coolants and De-Icing Fluids

Question 1. What coolants are used in automotive engines, both for normal operation and low temperature and in aviation engines? Are corrosion inhibitors added? Are soluble oil emulsions ever employed?

Answer. Anti-corrosion oils were added to the usual coolants. The former were water soluble oils of boring oil type, based on naphthene sulphonic soap.

Question 2. What fluids or compounds have been developed for de-icing of aircraft and what additives are used? For what purposes are lithium and potassium soaps added to these compounds?

Answer. No information.

G. Miscellaneous

Question 1. Have detergents of the ester salts type been developed and how are they made?

Answer. No information.

Question 2. What developments have been made in products used for pest control?

Answer. A medium for winter spraying was prepared on a spindle oil distillate basis, with additives including naphthene sulphonic acid soap. Its composition was as follows:-

Spindle oil distillate, viscosity circa. 2°E. at 50°C.	....	70	parts
Naphthene sulphonic acid	....	19	"
Resin	....	3	"
Crude cresol	....	5	"
Alcohol	....	3	"
Caustic soda lye 40° Be	....	3.5	"

The components were mixed cold and gave a product which, with water, yielded a whitish emulsion. This product was used for combating the red spider, blood louse and cochineal insect on fruit trees and complied with the regulations of the Biologische Reichsanstalt.

It was used in the spring, before opening of the buds, in 5-8 per cent emulsion.

In peace time an emulsifying summer spray medium was prepared, based on oleic acid soap dissolved in white oil. This medium was used for combating the cochineal insect on citrus fruits and also for mildew control. It was used in 1-2 per cent emulsion.

QUESTIONNAIRE NO. 4

INDUSTRIAL LUBRICANTS

A. Turbine Oils

Question 1.
- (a) What type of oil has been used for this purpose and from what raw materials?
- (b) What changes have been made in this type of product during the war and for what reason?
- (c) What inhibitors and/or additives have been used in these oils?
- (d) What troubles have been experienced in their use?
- (e) What length of service has been common without oil change and what limits in deterioration are allowed before an oil change is considered necessary?
- (f) What specifications exist for these oils?

Answer.
- (a) Good quality light raffinates were used as turbine oils. The raw materials were preferably of Russian and American origin but later came from German sources.
- (b) Throughout the war the same oils as in peace time were used. After a certain time, raffinates of German origin were employed and towards the end of the war, "dry raffinates" were used.
- (c) No additives were used.
- (d) No information.
- (e) In general, the running time before changing oil was 40-50,000 working hours with a maximum of 80-90,000 hours. The oil was usually changed when the acid value reached 3.0 and the saponification value 6.0.
- (f) The specifications for turbine oils were laid down in the "Richtlinien". The Brown, Boveri firm had their own special specifications.

B. Railway Oils

Steam Cylinder Oils.

No information.

Axle Oils or Greases.

No information.

Railway Diesel Oils.

Question. (a) What oils and fuels have been used for this purpose?

(b) To what specifications were they manufactured?

(c) What changes in quality have occurred during the war?

(d) What troubles were experienced in their use?

(e) Have additives been used in these products; such as ignition promoters in the fuel and detergents in the oil?

(f) What operating times were common between oil drainage periods? Were these influenced by the quality of the products?

Answer. (a) Highly refined diesel engine circulating oils were used for high speed purposes.

(b) According to the delivery specifications of the "Deutsche Reichsbahn".

(c) Delivery stopped during the war.

(d)
(e) } No information.
(f)

C. Cutting Oils.

Neat Cutting Oils.

Question. (a) What type of products (with formulae) have been used for this work showing the different types used for different machining operations on different types of metal, including non-ferrous?

(b) Were these made to specification and if so, what?

(c) What changes have been made during the war either voluntarily or due to lack of the required raw materials?

(d) Were these materials satisfactory or could they have been improved?

(e) Were sulphur, phosphorous and chlorine additives used in these products as well as fatty oils?

(f) Have substitutes been employed for fatty oils?

(g) Were any troubles encountered in their use?

Answer. (a) The peace time products were pure spindle oil and spindle oil containing rape oil (25%) and blown rape oil (20%). During the war, cutting oil was only used in special cases. About 90 per cent of metal working was done using boring oil emulsion. For cutting oil, a product was made containing about 1 per cent of sulphur. For working with hard, toughened steels this sulphurized product was fatted with 3 per cent of rape oil.

For very delicate tools such as broaches (scraping cutters) fatting extended up to 25 per cent.

For non-ferrous metals, cutting oils were not used as far as we know. Aqueous boring oil emulsions were quite satisfactory in such cases.

(b) Cutting oils were divided into categories numbered 0-5. Category 0 included the water soluble oils which were made without any fatting.

Category (Bearbeitungsgruppe) B1 consisted of pure mineral oils without additives.

Categories B2 to B5 included the cutting oils, arranged in order of increasing fatty oil content.

(c) The shortage of fats led to the increasing use of sulphurized cutting oils without addition of fatty oils.

(d) The sulphurized oils supplied by us were found satisfactory in most cases.

(e) We used sulphur addition both to replace and to supplement fatty oils. Phosphorous and chlorine products were not utilised by us.

(f) Synthetic final distillate fatty acids were used as substitutes.

(g) No.

D. Soluble Oils.

Question.

(a) Show, with formulae, the changes made in these, products during the war.

(b) Were they used to a greater extent than pre-war as a measure of economy?

(c) Did the shortage of petroleum sulphonates greatly affect the properties of these products?

(d) What troubles were experienced in their use?

(e) Were these products made to Army, Navy or outside specifications? Give details of these.

(f) What additives were used to impart outstanding qualities, such as E.P., anti-rust, anti-foaming etc.?

(g) Indicate what operations they were used on and for what metals.

Answer.

(a) On the whole our soluble oils were made to the same formulae as pre-war, namely on a naphthene sulphonic acid soap basis which was available in our own works as a by-product of white oil refining. At times we used small quantities of the I.G. product "Hö", but only small deliveries in experimental quantities were made.

(b) Soluble oils were used during the war to a greater extent in the replacement of pure cutting oils than in peace time.

(c) The small amount of naphthene sulphonic acids available was made good by using the product "Hö". However, as we ourselves produced naphthene sulphonic acid soaps we did not suffer from any scarcity.

(d) No difficulties are known to us.

(e) We do not know whether soluble oils were used to supplement other specified applications.

(f) We made soluble oils of the naphthene sulphonic acid type as anti-rust additives. No further additive was necessary. Users were recommended however, to make an addition of soda up to pH 8 if the blends with oil were to be used for any considerable time. The addition of so-called "Ferropaboiv" was also advisable.

To increase the E.P. quality of soluble oils, experimental use was made of a product based on sulphurised (1%) spindle oil.

(g) Soluble oils were used for metal working operations such as boring, milling, turbing, screw-cutting and planing.

The soluble oil was composed roughly as follows :-

Naphthene sulphonic acid	15 per cent	by vol.
Spindle oil	76 "	"
Caustic soda lye 40° Be	2 "	"
Spirit	4 "	"
Water	3 "	"

It was used for the following purposes :-

1. As a soluble cutting oil.
2. As a boring oil.
3. As a general rust preventative.
4. As anti-rust oil.
5. As hydraulic oil.
6. As a cooling system anti-corrosion medium.
7. As a base for a winter spraying medium for fruit trees.
8. Textile oils for wool (shoddy).

For all the products named, the above composition was maintained with only slight deviations. Specially good lots which gave stable emulsions, were used as anti-corrosion oils.

The raw material was the naphthene sulphonic acid soap occurring in the refining of white oil. This was freed from water and salts either by dissolving in mineral oil and prolonged heating or by decomposing the soap by means of sulphonic acid and using the naphthene sulphonic acid produced in manufacture.

The best sulphonic acids were given by Russian distillates. Later Roumanian and German sulphonic acids or sulphonic soaps were also used but these were not so good; in such cases the soap content had to be increased from 15-25 per cent.

E. Drawing Oils

Metal Drawing.

No information.

Wire Drawing

Question. (a) What products were used for this purpose for the different types of wire?

(b) What were the specifications?

(c) What changes were made during the war?

(d) What troubles were encountered in their use?

(e) What additives were used in these products to impart special properties?

Answer. (a) In peace time large amounts of pure rape oil were used for wire drawing.

(b) The specifications are not available.

(c) During the war, products consisting of spindle oil with 25 per cent rape oil were used as substitute for rape oil itself. Such products were satisfactory even in the more difficult working processes. Later, the amount of rape oil was lowered to 3 per cent.

(d) When using such weakly fatted oils, consumers complained of high wear on the drawing holes and diamonds.

(e) To some extent drawing oils were sulphurized in order to give E.P. properties.

F. Rolling Oils.

No information.

G. Quenching Oils.

Question. (a) What were the types of product used and their mode of application?

(b) To what specification were they made?

(c) What changes in their composition have been made during the war and for what reason?

(d) Have these products always been satisfactory and if not, why not?

(e) Have any additives been used in these products and for what purpose?

(f) Were different products used according to the size of the part being quenched or was a different technique used?

Answer. (a) Pure mineral oil raffinates with viscosity about 2-6°E. were used in simple or cooled containers for quenching the glowing metal components.

(b) We had no special specifications. However, high quality raffinates were generally used, where it was a matter of "polished" quenching oils (Blankhärteole). For small components the viscosity used was about 2°E./50°C. For heavy parts, a machine oil raffinate, viscosity 6.5°E./50°C. was used.

(c) There was little change during the war.

(d) No such difficulties are known to us.

(e) No special additives were used in our quenching oils.

(f) With small parts, low viscosity oils were used and with large parts, higher viscosity oils.

H. Core Oils

Question. (a) What products were used for this purpose?

(b) To what specifications were they made?

(c) What changes have been made in their composition and the reason?

(d) Was any trouble encountered in the use of substitute materials?

(e) Were any additives used to impart special properties?

Answer. No information.

I. Rust Preventatives.

Question. (a) What products have been used for this purpose, indicating the application for each type of material?

(b) Which of these were peculiar to your Company?

(c) To what specification were they made?

(d) What changes have been made during the war? give the reason.

(e) Did the use of substitute materials improve or degrade the properties?

(f) What troubles have been experienced in their use?

(g) What special materials were added to give increased effectiveness?

Answer.

(a) Two main types of rust preventatives were used in the metal working industry: soluble anti-corrosion oil based on naphthene sulphonic acid soap and anti-corrosion grease based on petrolatum without special additives.

The use of such oils was not widespread in civil industry. The Luftwaffe, however, prescribed the addition of ½ per cent of anti-corrosion oil to the coolant. The common trade products were soluble oils based on naphthene sulphonic acid soaps with spindle oil raffinate.

(b) We did not prepare special products. The products already mentioned were in common commercial use.

(c) The soluble oils of the naphthene sulphonic acid soap type were made like boring oils. The greases consisted of petrolatum with a melting point of about 50°C.

The Luftwaffe specifications laid down that the protective oil must give a cloudy solution in glycol water mixtures. Further, the product had to have a cold test of -10°C. On melting, the product had still to have good solubility after one hour. Adequate anti-rust properties were also demanded. Testing was carried out in the Luftwaffe laboratories.

(d) As the war progressed, composition was changed in that spindle oil distillates replaced spindle oil raffinates and petrolatums with a lower degree of refining were used in the greases.

(e) The use of such less refined raw materials did not prove detrimental in use and quality as related to the intended use of such products.

(f) There were no difficulties in use.

(g) Special additives were not used.

J. Slushing Oils.

These were not made by us.

K. Ink Oils.

Such oils were not made by us.

L. Textile Oils.

Wool and Cloth Oils

Question.

(a) What products were used for this purpose, giving their composition?

(b) What specifications existed for this type of product?

(c) What changes have been made during the war and for what reason?

(d) How was the shortage of fatty oils overcome?

(e) What troubles have been experienced in their use?

(f) What additives have been employed to improve scouring, etc. or to impart special properties?

Answer.

Ripping and mill oils (Reiss-u-Schmelzöle)

For these purposes we supplied soluble oils in the first year of the war. It was then decided by the "Reichstelle für Industrielle Fettbewirtschaftung" that only the oil "Rotton" and "Grunton" developed by I.G. could be used. Their composition is unknown to us.

Textile Mill Lubricants

Question.

(a) What products have been used for these purposes?

(b) To what specifications were they manufactured?

(c) What changes have been made in their composition during the war and for what reason?

(d) What troubles were encountered?

(e) What additions were made to impart special properties?

Answer.

(a) In peace time we supplied a whole range of different weaving, traveller and cotton weaving oils.

(b) The chief property desired was a high degree of ease of removal by washing. This was attained by adding 20-25 per cent of neats'-foot oil.

(c) It was forbidden to use fatted oils for this purpose during the war. Pure mineral oil raffinates were supplied.

(d) In consequence more difficulty was experienced in washing out. However, only minor complaints were received.

M. Leather Oils

Question.

(a) What products have been used for this purpose?

(b) To what specification were they manufactured?

(c) What changes have been made in their composition during war and for what reason?

(d) Did the use of substitute materials result in a decrease in quality in the finished leather?

(e) To what extent were synthetic wetting agents used (indicate their type)?

(f) What troubles were experienced in their use?

(g) What additions were made?

Answer.

Before the war we supplied Turkey red oil, derived from sulphurized castor oil, for the leather industry. When castor oil was no longer imported this product could not be manufactured, so that we have no further experience in this line.

N. Cable Oils

Question.

(a) What type of oils were used for both the light and heavy types of cable oil?

(b) To what specifications were they manufactured?

(c) What changes in composition had to be made during the war?

(d) What troubles were experienced?

(e) Were any additions made to increase dielectric properties or to stop hydrogen formation?

(f) Were any additions made to impart special properties?

Answer.

(a) Thin oils of the transformer oil type, specially resistant to oxidation were used for filling hollow cables.

(b) The oils were derived from Russian spindle oil distillate refined with 10-20 per cent sulphuric acid. They were produced to the specification of the "Verein deutscher Elektrotechniker".

(c) On the whole, production was not altered during the war. When no more Russian oils were available, German spindle oil distillates also acid-refined, were substituted.

(d) Any difficulties are unknown to us.

(e) No additives were used.

(f) No additives were used.

O. Marine Oils

Question.

(a) What products were used for this purpose?

(b) To what specifications were they produced?

(c) Were Voltol products used in these oils?

(d) What changes have been made in their composition during the war and for what reason?

(e) What difficulties were experienced in their use?

(f) Were any additions made to impart special properties?

Answer.

(a) In the manufacture of so-called "ships' machine oils" (Schiffsmaschinenöle), machine oil raffinates of the desired viscosity were usually blended with about 3 per cent of blown rape oil.

(b) Such oils were supposed to give only a moderate emulsion when rubbed with water.

(c) Voltol was not used by us.

(d) During the war we delivered a small amount of "ships' machine oil" with 1 per cent of Emulphor FM (an I.G. product).

(e) Any difficulties are unknown to us.

(f) Emulsifying additive FM (I.G. product).

P. Refrigerating Oils

Question.

(a) What products (and from what sources) were used for this application, either for SO_2, CO_2, NH_3, Freon, etc.?

(b) To what specifications were they produced?

(c) What changes in composition have been necessary during the war and for what reason?

(d) What troubles have been experienced in their use?

(e) What additions have been used to overcome sludging and/or corrosion?

Answer. (a) Machine oil for CO_2 and NH_3 plant was supplied to specification (Richtlinien). Light white oils, earlier on from Russian sources and later from Rumanian, was produced for SO_2 and Freon.

(b) Products for NH_3 and CO_2 machines were to the requirements of the "Richtlinien". Those for SO_2 machines and chlorinated hydrocarbons were tried out by the consumer firms and on their approval, the oils were delivered to that particular formula.

(c) When Russian sources were no longer open, Rumanian oils were used.

(d) No difficulties are known to us.

(e) No special additives were used. The greatest stress was laid on the absolute dryness of such products however.

Q. Insulating Oils.

Question. (a) What products (and from what sources) were used for this application?

(b) To what specifications were they produced?

(c) What changes in composition have been necessary during the war and for what reason?

(d) What troubles have been experienced in their use?

(e) What additives, e.g. anti-oxidants or metal passifiers have been used?

(f) Have any non-petroleum products been used for this purpose?

Answer. (a) At the beginning of the war, insulating oils were prepared by sulphuric acid (5-10%) refining of Russian light distillates. Later, Rumanian and German oils had to be used.

(b) VdE and WEV specifications.

(c) Russian oil was no longer available, so that Rumanian and German sources were drawn on.

(d) None are known to us.

(e) No special additives were used.

(f) This is not known by us. However, we have heard that the AEG and Siemens, Berlin, carried out such experiments.

R. Specialities.

Question. (a) Have any oils been developed for high temperature lubrication? If so, describe them fully as outlined in the above question.

(b) Describe the uses of wire rope lubricants, their composition etc. Have refinery extracts found application in this field?

(c) What oils have been used as mould lubricants? Describe their use, composition, etc., as outlined above.

(d) What products have been used as air tool lubricants? Describe their use and changes in composition as outlined in the above question.

(e) What type of products have been used as general machinery oils and black oils? Describe their use, composition, etc. To what extent have oils been replaced by emulsions in the former case and to what extent have solvent extracts been used in the latter?

Answer. (a) No information.

(b) We did not make wire rope greases. We know that pure petrolatums were in use for such purposes.

(c) No information.

(d) As far as we know, ordinary machine oils were used during the war. In any case, we did not make special oils.

(e) For general machinery oils and dark oils, unrefined distillates were supplied during the war.

S. Greases.

Question. (a) What type of products have been used (with their compositions) for the following purposes?

(i) Ball and roller bearing greases, for high and low speed, high and low temperature and for heavily loaded applications.
(ii) General lubrication greases.
(iii) Block greases for steel mills, paper machinery crushers etc.
(iv) Greases or oils for open gears.
(v) Greases for Marine applications.
(vi) Any other types of greases.
(vii) Extreme pressure greases.

(b) With each of the above give the relevant specification.

(c) What changes have been made in composition during the war and for what reason?

(d) What troubles have been experienced in their use?

(e) What additions have been made for special purposes?

Answer. (a) (i) For ball bearing grease we delivered only a product based on spindle oil distillate with 13 per cent sodium soap from a synthetic fatty acid. The drop point was about 110°. No grease for heavily loaded bearings was delivered by us during the war.

(ii) As a general chassis grease, suitable for most lubricating purposes, a product was prepared consisting of spindle oil distillate with 5 per cent crude montan wax and 7 per cent synthetic final distillate fatty acid.

(iii) For paper machines, we supplied block greases based on spindle oil, with 5 per cent resin and 10 per cent fatty acid (saponified with NaOH). Manufacture had to be discontinued however, because of lack of raw material.

(iv) Gear greases were based on machine oil, with the addition of 10 per cent of tallow sodium soap.

(v) We did not make special marine greases.

(vi) At times individual boilings were made of special greases containing 10 per cent refined montan wax and 5 per cent fatty acid sodium soap as base. Such greases were used for ball bearings.

(vii) E.P. gear greases were prepared to a small extent in peace time, based on lead oleate in machine oil. Because of production difficulties they were dropped, however. We also prepared a machine tallow substitute for high pressure use (see Questionnaire

No. 3, Special Products - Supplementary Information).

5. RESEARCH AND DEVELOPMENT.

Question. For Items 1-18 inclusive indicate any research or development work which has been carried out, particularly of a more fundamental nature to elucidate properties, etc.

Answer. We did not carry out fundamental scientific research. Our laboratory work restricted itself to testing the suitability of the available raw materials and controlling the uniformity and quality of deliveries.

6. CONTROL TESTS.

Question. For Items 1-18 inclusive give the control tests carried out to ensure uniformity of manufacture. Where these are of a special nature, give full details.

Answer. The control tests used by us are all described in the books detailed below*. Further, specifications from the German Navy and Air Force were used. All our data in this connection were destroyed with our laboratories in the last phase of the war.

* (1) Richtlinien für Einkauf v. Prüfung von Schmierstoffen" (Guide to the Buying and Testing of Lubricants) 8th Edn. 1939 - Stahleisen m.b.H. Press, Dusseldorf. Beuth-Vertrieb G.m.b.H., Berlin, S.W.68.

(2) D. Holde "Kohlenwasserstofföle u. Fette" (Hydrocarbon Oils and Greases) 7th Edn. Springer, Berlin, 1933.

(3) "Vorschriften für Schalter u. Transformatorenöle" (Specifications for Switch gear and Transformer oils). V.D.E. 0370/1936.

(4) "Die Oelbewirtschaftung" (The Economical Use of Oil) - Directions for Testing, Supervision and Ease of Insulating and Steam Turbine Oils 1930, Vereinigung der Elektrizitatswerke EV., Berlin W.62.

7. SAMPLES.

The following samples of Schliemann products were obtained and have been placed at the Ministry of Fuel and Power, London.

Lubricants.

Sample No.	Description	Quantity
138/84a	Turbine Oil	4 gallons
139/84b	Transformer Oil	"
140/84d	White Oil for Ointments	"
141/84e	Cutting Oil + Sulphur	"
142/84f	Cutting Oil + Sulphur + Fatty Acid	"
143/84g	Cutting Oil - Water Soluble	"
144/84h	Refrigerator Oil - Light	"
145/84j	Refrigerator Oil - Medium	"
146/84k	Tallow Substitute	1 keg
147/81 1	Vaseline	"
148/61	"Schutzfett" 40	"
149/66	"Schutzöl" 39	4 gallons
150/111	Winter Spray Oil	"
151/109	Naphthenic Acid - Sodium Salt	"

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B.I.O.S.—FINAL REPORT No. 635

ITEM No. 30

MINERALÖLWERKE ALBRECHT

LUBRICANTS

Thomas, W. H., + Withers, J. D.

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BRITISH INTELLIGENCE OBJECTIVES

SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

MINERALÖLWERKE ALBRECHT

LUBRICANTS

October 2nd 1945

Reported by:

W.H. Thomas, British, Ministry of Fuel and Power.

J.G. Withers, British, Ministry of Fuel and Power.

BIOS Trip No. 1248

B.I.O.S. Target No. C30/59

Fuels and Lubricants

British Intelligence Objectives Sub-Committee,
32 Bryanston Square,
London, W.1.

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Mr. J.G. Allen, U.S. Petroleum Administration for War.
Major W.H. Thomas, British, Ministry of Fuel and Power.
Mr. J.G. Withers, British, Ministry of Fuel and Power.

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ACKNOWLEDGEMENT.

German translations involved in the preparation of this report were made by Mr. K. G. C. Kirkpatrick.

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1. SUMMARY.

This report presents the information obtained as result of the interrogation of the Manager of the firm of Mineralölwerke Albrecht.

The information is in general provided in the form of answers to a comprehensive questionnaire and covers the manufacture of normal types of lubricants and of certain specialised products such as cutting oils.

Perusal of the answers leads to the conclusion that the refining and blending processes were normal. No outstanding technical advances are evident in the operations as described.

2. INTRODUCTION.

The information given in this report was obtained from Johann Albrecht - Manager of Mineralölwerke Albrecht and Rudolph Braun his assistant, in the course of a visit (B.I.O.S. Trip No. 1248) to the Hamburg area during the period October 1st - 31st 1945.

Mineralölwerke Albrecht is stated to be a "sister company" to Rhenania-Ossag and to be affiliated to the Royal Dutch group of petroleum concerns.

The purposes of the interrogation, which took place at the Albrecht Company's office, Wallhof, Hamburg, were to ascertain details of the refining and blending procedures employed and to obtain data respecting the quality of the finished products.

3. GENERAL.

The firm of Mineralölwerke Albrecht possessed a refinery in the Hamburg area which was severely damaged during the war by air attack. It had no distillation equipment but was designed for the refining, blending and storage of lubricating oil and for the manufacture of greases (1800 tons/year), cutting oils (1000 tons/year) and certain heavy lubricants.

Conventional acid and clay treatments were employed and greases were of lime and soda soap types. Speciality products were turbine, transformer and white oils and a small amount of compounding of marine engine oils was carried out.

Prior to the war, feed stocks were of American or Russian origin but during the war they were obtained from Greater Germany - 80 per cent from Rhenania-Ossag and the remainder from other German refineries.

With regard to research and development work, the laboratory services were limited to the control of product quality and no fundamental investigations were carried out. Control tests were those given in Richtlinien für Einkauf und Prüfung von Schmierstoffen" 7th Edition 1939.

Transformer oils were tested according to VDE 0370/1946 regulations. The special tests for turbine oils corresponded to those in the "Oelbewirtschaftung" and for thermally stable oils (D.L. Oils), colour stability was tested using the MAN procedure (50 hours at 155°C.) followed by an estimation of hard asphalt formation. It was permissible for the Union-Colour of the oils, after ageing for 50 hours at 135°C. to change from 2 to approximately 4½.

4. ANSWERS TO QUESTIONNAIRE.

LUBRICANTS

A. Aviation

No information.

B. Wehrmacht and Civilian Grades.

Question 1. How were these oils manufactured and from what stocks?

Answer. Mainly by refining of distillates and raffinate blending. The main stocks for processing were semi-finished products from the Rhenania-Ossag, Harburg, as well as spindle oil from Curacao and to a lesser extent, American and Russian oils. During the war mainly German, Roumanian and Russian distillates were processed.

Question 2. What were the refining and finishing methods?

Answer. Normal acid and clay treatments.

Question 3. To whom were the oils despatched?

Answer. Primarily to factors at home and abroad, as well as to the Rhenania-Ossag Mineralölwerke, A.G. Hamburg.

Question 4 What quantities were produced?

Answer. Up to 50,000 tons per year.

Question 5. Who were the Government Departments concerned?

Answer. Reichswirtschaftsministerium - Mineralöl - Abteilung, Berlin. Reichstelle fur Mineralöl, Berlin.

Question 6. What additives were used?

Answer. For engine lubricants Oppanol and Paraflow were used but for metal-working oils, sulphur, animal and vegetable fats and vulcanised oils (Faktis) were employed.

Question 7. From whom were the additives purchased?

Answer. Oppanol from I.G. Farben-Industrie, Ludwigshafen.
Paraflow from the Deutsch-Amerikanischen Petroleum-Gesellschaft, Hamburg.
Fats from various factors.
Crude rape oil with a small content of free fatty acid from the Märkische Oelwerke A.G. formerly Herz-Oelfabriken, Wittenberge.
Sulphur from various factors.
Vulcanised oils (Faktis) from Deutschen Oil-Fabrik Dr. Grandel & Co., Hamburg.

C. Automotive.

Question 1. To what specifications are lubricants for petrol engines and high speed diesel engines produced.

Answer. The specifications were as follows:-

(a) Diesel engine oil components 3.5°E. at 50°C.

Sp. gr. at 20°C.	circa	0.912
Flash point	"	205°C.
Pour point	"	-20°C.
Hard asphalt		0.0
Carbon residue	"	0.06%
Ash		trace
Acid value	below	0.05
Colour		-2
Viscosity at 20°C.	circa	16.3°E.
" at 50°C.	"	3.5°E.
" at 100°C.	"	1.43°E.
" Poleheight		2.5

Diesel engine oil component 9°E. at 50°C.

Sp. gr. at 20°C.	circa	0.917
Flash point	"	220°C.
Pour point	"	-10°C.
Hard asphalt		0.0
Carbon residue	"	0.3
Ash		trace

continued ...

Acid value	not above	0.05
Saponification value	" "	0.15
Tar value (Verteerungszahl)	circa	0.06%
Colour		2 - 2½
Viscosity at 50°C.	"	9°E.

Diesel special 12°E. at 50°C.

Sp. gr.	circa	0.916
Flash point	"	250°C.
Pour point	"	-18°C.
Hard asphalt		absent
Carbon residue	"	0.65%
Ash		trace
Acid value	not above	0.05
Saponification value	" "	0.15
Colour		3
Viscosity at 50°C.	circa	12.5
" at 100°C. ?		

Blends of these oils were also used, together with additives, i.e. Paraflow and/or Oppanol.

(b) Engine oils. Sp.gr. 0.890

	Viscosity, 5°E.	Viscosity, 20°E.
Sp. gr. at 20°C.	circa 0.889	circa 0.894
Flash point	" 220°C.	" 262°C.
Pour point	" -25°C.	" -18°C.
Conradson test	" 0.27%	" 0.45%
Viscosity at 20°C.	" 25.9°E.	" 154°E.
" at 50°C.	" 5.0°E.	" 20.0°E.
" at 100°C.	" 1.59°E.	" 2.88°E.
" Poleheight	" 2.3	" 2.15

Blends of these oils were also used.

(c) Engine oils. Sp.gr. 0.900

	Viscosity, 5°E.	Viscosity, 20°E.
Sp. gr. at 20°C.	circa 0.900	circa 0.903
Flash point	" 216°C.	" 263°C.
Pour Point	" -25°C.	" -14°C.
Conradson test	" 0.26%	" 1.32%
Viscosity at 20°C.	" 27.2°E.	" 165°E.
" at 50°C.	" 5.0°E.	" 20.0°E.
" at 100°C.	" 1.58°E.	" 2.79°E.
" Poleheight	" 2.4	" 2.25

Blends of these oils were also used.

(d) **Engine Oils**. Sp.gr. 0.910.

	Viscosity 5°E.	Viscosity 20°E.
Sp. gr. at 20°C.	circa 0.910	circa 0.910
Flash point	" 217°C.	" 257°C.
Pour point	" -25°C.	" -14°C.
Carbon Residue	" 0.20%	" 1.22%
Viscosity at 20°C.	" 29.0°E.	" 176°E.
" at 50°C.	" 5.0°E.	" 20.0°E.
" at 100°C.	" 1.56°E.	" 2.70°E.
" Poleheight	" 2.74	" 2.45

Blends of these oils were also used.

Question 2. What are the chief sources of motor oils? What crudes and refining methods are used and to what extent are synthetic oils incorporated?

Answer. No information.

Question 3. Are engine tests carried out before approval and if so, what are they?

Answer. No information.

Question 4. What laboratory oxidation tests or sludge tests are used and how do they correlate with performance in an engine?

Answer. The M.A.N. test was used; this consisted of ageing for 50 hours at 155°C., followed by estimation of the amount of hard asphalt formed and of the change in colour using the Union Colorimeter. The Sleigh Oxidation Test (A.S.T.M.) was also employed. It was generally found that oils satisfying the above requirements gave no trouble.

Question 5. To what extent is Voltol used in motor oils? Is it more commonly used in motor oils than aviation oils and if so why? What particular advantages in engine cleanliness, viscosity index, etc., have been found with Voltol?

Answer. No information.

Question 6. Are detergents other than Voltol used in -
(a) petrol engines?
(b) diesel engines?

Answer. Oppanol has also been used in both petrol and diesel engine lubricants.

Acid value	not above	0.05
Saponification value	" "	0.15
Tar value (Verteerungszahl)	circa	0.05%
Colour		2 - 2½
Viscosity at 50°C.	"	9°E.

<u>Diesel special 12°E. at 50°C.</u>

Sp. gr.	circa	0.916
Flash point	"	250°C.
Pour point	"	-18°C.
Hard asphalt		absent
Carbon residue	"	0.65%
Ash		trace
Acid value	not above	0.05
Saponification value	" "	0.15
Colour		3
Viscosity at 50°C.	circa	12.5
" at 100°C. ?		

Blends of these oils were also used, together with additives, i.e. Paraflow and/or Oppanol.

(b) <u>Engine oils</u>. <u>Sp.gr. 0.890</u>

	Viscosity, 5°E.	Viscosity, 20°E.
Sp. gr. at 20°C.	circa 0.889	circa 0.894
Flash point	" 220°C.	" 262°C.
Pour point	" -25°C.	" -18°C.
Conradson test	" 0.27%	" 0.45%
Viscosity at 20°C.	" 25.9°E.	" 154°E.
" at 50°C.	" 5.0°E.	" 20.0°E.
" at 100°C.	" 1.59°E.	" 2.88°E.
" Poleheight	" 2.3	" 2.15

Blends of these oils were also used.

(c) <u>Engine oils</u>. <u>Sp.gr. 0.900</u>

	Viscosity, 5°E.	Viscosity, 20°E.
Sp. gr. at 20°C.	circa 0.900	circa 0.903
Flash point	" 218°C.	" 263°C.
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Viscosity at 20°C.	" 27.2°E.	" 165°E.
" at 50°C.	" 5.0°E.	" 20.0°E.
" at 100°C.	" 1.58°E.	" 2.79°E.
" Poleheight	" 2.4	" 2.25

Blends of these oils were also used.

(d) <u>Engine Oils</u>. Sp.gr. 0.910.

	Viscosity 5°E.	Viscosity 20°E.
Sp. gr. at 20°C.	circa 0.910	circa 0.910
Flash point	" 217°C.	" 257°C.
Pour point	" -25°C.	" -14°C.
Carbon Residue	" 0.20%	" 1.22%
Viscosity at 20°C.	" 29.0°E.	" 176°E.
" at 50°C.	" 5.0°E.	" 20.0°E.
" at 100°C.	" 1.56°E.	" 2.70°E.
" Poleheight	" 2.7	" 2.45

Blends of these oils were also used.

Question 2. What are the chief sources of motor oils? What crudes and refining methods are used and to what extent are synthetic oils incorporated?

Answer. No information.

Question 3. Are engine tests carried out before approval and if so, what are they?

Answer. No information.

Question 4. What laboratory oxidation tests or sludge tests are used and how do they correlate with performance in an engine?

Answer. The M.A.N. test was used; this consisted of ageing for 50 hours at 155°C., followed by estimation of the amount of hard asphalt formed and of the change in colour using the Union Colorimeter. The Sleigh Oxidation Test (A.S.T.M.) was also employed. It was generally found that oils satisfying the above requirements gave no trouble.

Question 5. To what extent is Voltol used in motor oils? Is it more commonly used in motor oils than aviation oils and if so why? What particular advantages in engine cleanliness, viscosity index, etc., have been found with Voltol?

Answer. No information.

Question 6. Are detergents other than Voltol used in -
(a) petrol engines?
(b) diesel engines?

Answer. Oppanol has also been used in both petrol and diesel engine lubricants.

Question 7. Is trouble experienced with ring sticking in diesel engines and if so what palliatives are used?

Answer. No information.

Question 8. What type of filters are used in mechanically propelled vehicles? If chemical type filters are used, is any trouble experienced with them?

Answer. No information.

Question 9. What mileage is normally recommended between sump drainings?

Answer. 2,000 - 3,000 Km.

Question 10. What inspections are carried out on used oils?

Answer. Estimation of the asphalt formation and increase in acidity are determined.

Question 11. What additives are used as pour point depressors, V.I. improvers, anti-oxidants, anti-wear additives, anti-corrosion additives and film strength improvers?

Answer. Pour point improvers; Paraflow and Paraflow Extra.
To improve viscosity index; Oppanol.
As oxidation inhibitor; - }
As anti-wear additive; - } No information.
As anti-corrosion medium; - }

Question 12. To what extent are fatty oils used in motor lubricants? What kind of fatty oils are used and what advantage have they?

Answer. Castor oil was used to a limited extent to improve "oiliness".

Question 13. Were any lubrication difficulties experienced with producer gas equipment (both wood and coal types), bottled gas or acetylene driven vehicles?

Answer. Any such difficulties are unknown to us.

D. <u>Marine</u>

Question 1. What lubricating oils are specified for use with marine diesel equipment?

Answer. The same as for automotive use.

Question 2. **What oils are used in steam turbine driven vessels? Are additives of any kind incorporated (anti-corrosion etc.)?**

Answer. **Steam turbine oils without additives, viscosity 6°E. at 50°C.**

Question 3. **To what extent are compounded cylinder oils used and what are the compounding agents employed?**

Answer. **No information.**

Question 4. **To what specifications are stern-tube greases manufactured and what compounding agents are employed?**

Answer. **No information.**

Question 5. For what uses are aqueous emulsions of oil, with the addition of montan wax, employed?

Answer. No information.

E. <u>Transmission</u>

Question 1. What lubricants are recommended and specified for tank gear boxes and car and truck gear boxes of normal and synchromesh types?

Answer. Gear oil of the following composition were used -
75% Saturated steam cylinder oil 280°E. at 85°C.
24% Machine oil distillate, 4.1°E. at 50°C.
1% Hexachlorethane.

Question 2. To what extent are Voltolised oils and compounded oils used for tank gear boxes? What compounding agents are employed?

Answer. No information.

Question 3. To what extent are additives of the nitrogen or chlorine type etc., used in oils for gear boxes?

Answer. Hexachlorethane up to 1% was employed.

Question 4. In what proportion are straight tooth, spiral bevel, hypoid and worm gears used in rear axles and what oils are specified in each type?

Answer. No information.

Question 5. **What additives are used in extreme pressure oils?**

Answer. Hexachloroethane was used.

Question 6. **To what extent are compounded oils (fatty oil compounded) used for rear axle lubrication?**

Answer. No information.

Question 7. What type of oil seals are used?

Answer. No information.

Question 8. What temperature extremes are encountered in the different types of axle?

Answer. No information.

F. Hydraulic, Recoil and Buffer Oils.

No information.

G. Railroad

Question 1. What oils are used for steam cylinder lubrication (satd. and superheat) valves, axle boxes etc?,

Answer. Saturated steam and super-heated steam cylinder oils were employed.

Question 2. What type of crudes are preferred for this purpose? What asphaltene content is usual in such oils?

Answer. After stocks of imported oils had been exhausted, oils of German origin with an asphalt content of 0.3 per cent by wt. maximum were used.

Question 3. Are any special oils used in the lubrication of diesel locomotives and what additives, if any, are employed?

Answer. No information.

H. Industrial

Question 1. What types of oil are used in steam turbine lubrication? Are any troubles experienced with corrosion and emulsification and if so, how are they combatted? What additives are used, if any? What is the average life of a steam-turbine oil?

Answer. **Steam-turbine oils were found satisfactory in use if they complied with the specifications.**

Additives were not used.

The average life was about 50,000 working hours.

Question 2. **What types of oils are used for transformers? What electrical tests, if any, are carried out? Are any additives employed?**

Answer. **Transformer oils having a breakdown voltage not below 300 Kv. were used Paraflow was added to lower the pour point.**

Question 3. To what extent are mineral, non-mineral, compounded oils and aqueous emulsions used in quenching? What types are preferred for small and large objects?

Answer. No information.

Question 4. What types of cutting oils are used? To what extent are sulphurised and compounded oils used? Are corrosion inhibitors added and if so, what are they?

Answer. See "Industrial Lubricants" section which follows.

Question 5. What types of slushing oils are used? What additives are employed to give anti-corrosion properties?

Answer. Mineral oil raffinates without additives were used.

Question 6. From what sources are textile oils refined? What research has been done on carcogenicity?

Answer. No information.

Question 7. What oils are used for clock and instrument lubrication, especially for aviation use? What compounding agents are used?

Answer. No information.

Question 8. What is "Radiol" and for what uses is it employed?

Answer. No information.

Question 9. What is the composition of compounded light machine oils?

Answer. Light machine oil raffinate, viscosity 2 - 4.5°E. at 50°C., with the addition of crude rape oil (3, 15 or 30%) thickened rape oil (3%) and Olein (10 or 40%).

I. Greases

Question 1. To what extent are barium, lithium and aluminium soap greases used?

Answer. No information.

Question 2. What fats are used for greases and what substitutes have been employed?

Answer. Animal and vegetable fats and fatty acids, synthetic fatty acids, low molecular weight fatty acids (acetic acid), saponifiers, lime and caustic soda were employed.

Question 3. To what extent are waxes, especially montan wax used in grease manufacture?

Answer. Crude montan wax was added in quantities up to 8 per cent; other waxes were not used.

Question 4. What types of greases are used for high temperature applications?

Answer. Hot neck greases were used.

Question 5. For what reason and to what extent are greases used in shock absorbers for aeroplane landing gear?

Answer. No information.

Question 6. What material is employed for ship launching purposes?

Answer. No information.

J. General

Question 1. To what extent are aqueous emulsions used in industrial lubrication?

Answer. The production of these was of minor importance.

Question 2. Has this type of lubricant ever been employed in engines of any type?

Answer. For bearing lubrication.

Question 3. By what processes is Voltol made?

Answer. No information.

SPECIAL PRODUCTS.

A. Bitumens

No information.

B. Extracts and Polymers.

No information.

C. Waxes.

No information.

D. Rust Preventatives.

No information.

E. Cable Oils.

Question 1. To what extent are oil filled cables used; what oil specifications are laid down; what are the best sources for oils of this type?

Answer. Transformer oils were used and were prepared to conform to normal specifications.

Question 2. Are any blending agents or additives employed (e.g. resins)?

Answer. No information.

Question 3. To what extent are oil impregnated insulated types of cable used? What oils are used and what specifications have been laid down?

Answer. No information.

Question 4. What methods of test are used to determine dielectric strength and electrical performance of such oils?

Answer. No information.

F. Coolants and De-Icing Fluids.

No information.

INDUSTRIAL LUBRICANTS.

A. Turbine Oils.

Question 1. What type of oil has been used for this purpose and from what raw material was it obtained?

Answer. Special raffinate from Russian oils, Astraöl 4000, re-distillate 4.5 - 12.5°E. at 50°C. (Sp. gr. at 20°C. circa 0.931, flash point 185°C., pour point -20°C., viscosity 4.1°E. at 50°C., sp. gr. at 20°C., circa 0.942, flash point 220°C., pour point -20°C., acid value 1, viscosity 12.5 - 13°E. at 50°C.) and blends of these oils.

Question 2. What inhibitors and/or additives have been used in these oils?

Answer. No inhibitors or additives were used.

Question 3. What troubles have been experienced in their use?

Answer. No troubles were experienced in their use.

Question 4. What length of service has been common without oil change and what limits in deterioration are allowed before an oil change is considered necessary?

Answer. Oil change was not undertaken before at least 50,000 running hours.

Further to control the suitability of the oil, tests were made with respect to changes in acid value and saponification value. The limiting values were :-

Acid value - not above 3.0
Sap. value - " " 6.0

Question 5. What specifications exist for these oils?

Answer. The specifications were as follows :-

		(1)		(2)		(3)
Sp. gr. at 20°C.		0.920		0.894		0.909
Flash point		200°C.		200°C.		215°C.
Pour point		-12°C.		-28°C.		-14°C.
Carbon residue		0.06%		0.04%		0.21%
Acid value	not above	0.05	not above	0.05%	not above	0.05
Sap. value	" "	0.15	" "	0.15	" "	0.15
Tar value (Verteerungszahl)	" "	-	" "	0.10	" "	0.10
Colour		2		2		2-2.5
Visc. at 20°C.		21.5°E.		-		-
" at 50°C.		4°E.		3°E.		6°E.
" at 100°C.		1.46°E.		-		-
Hard asphalt		-		0		0
Ash		-		trace		trace

B. Railway Oils.

Steam Cylinder Oils.

Question 1. What oils have been used for this purpose?

Answer. Saturated and super-heated steam cylinder oils of American origin.

Question 2. What changes have been made in quality during the war and for what reason?

Answer. After American cylinder oil stocks had been used up a change was made to oils of German origin. These had high specific gravities, lower flash points, high pour points and higher asphalt contents than the oils formerly used. As delivery specifications remained unchanged no official deliveries were made.

Question 3. What troubles were experienced in the use of these oils?

Answer. Complaints were made because of deposit formation. However, it could be shown in almost all cases that running troubles which occurred were not due to the oil.

Question 4. What are the specifications which cover these types of oil?

Question 4. What methods of test are used to determine dielectric strength and electrical performance of such oils?

Answer. No information.

F. Coolants and De-Icing Fluids.

No information.

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Question 2. What inhibitors and/or additives have been used in these oils?

Answer. No inhibitors or additives were used.

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Answer. No troubles were experienced in their use.

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Answer. The specifications were as follows :-

		(1)		(2)		(3)
Sp.gr. at 20°C.		0.920		0.894		0.909
Flash point		200°C.		200°C.		215°C.
Pour point		-12°C.		-28°C.		-14°C.
Carbon residue		0.06%		0.04%		0.21%
Acid value	not above	0.05	not above	0.05%	not above	0.05
Sap. value	" "	0.15	" "	0.15	" "	0.15
Tar value (Verteerungszahl)	" "	-	" "	0.10	" "	0.10
Colour		2		2		2-2.5
Visc. at 20°C.		21.5°E.		-		-
" at 50°C.		4°E.		3°E.		6°E.
" at 100°C.		1.46°E.		-		-
Hard asphalt		-		0		0
Ash		-		trace		trace

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Answer. Saturated and super-heated steam cylinder oils of American origin.

Question 2. What changes have been made in quality during the war and for what reason?

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Question 3. What troubles were experienced in the use of these oils?

Answer. Complaints were made because of deposit formation. However, it could be shown in almost all cases that running troubles which occurred were not due to the oil.

Question 4. What are the specifications which cover these types of oil?

Answer.

Sp. gr. at 20°C.	0.964	0.966
Flash point	285°C.	311°C.
Pour point	+ 4°C.	- 2°C.
Hard asphalt	0.28%	below 0.1%
Carbon residue	-	7.57%
Ash	-	0.02%
Colour	dark green	-
Viscosity at 50°C.	66,54°E.	100
" at 100°C.	4.75	6.5

Question 5. What inhibitors and/or additives have been used in these products?

Answer. Inhibitors and additives were not used.

Axle Oils or Greases.

No information.

Railway Diesel Oils.

No information.

C. <u>Cutting Oils</u>.

Question 1. What type of products have been used and what were the different types used for different machining operations on different types of metal, including non-ferrous?

Answer. Two kinds of cutting oil were made :-

(i) Distillates, containing as additive elementary sulphur 0.4%.

(ii) Distillates with the addition of Faktispaste R.M.9, from Deutsche Olwerke Dr. Grandel & Co. Hamburg. Faktispaste R.M.9 is a special product consisting of sulphur, crude rape oil and mineral oil. Details of its manufacture are not known.

These types of oil were generally found satisfactory in metal working (cutting operations). Cutting oils containing Faktispaste R.M.9, had good properties, especially in the case of boring (cf. the opinion given by Kaltenbach and Voigt, Potsdam). We do not know to what extent our cutting oils were used with non-ferrous metals and any troubles encountered are unknown to us.

Cutting oil with flowers of sulphur and Faktispaste as additives were manufactured as follows :-

Composition :

(i) 15.00% Faktispaste R.M.9.
85.00% Spindle oil distillate 2.5°E. at 50°C.

(ii) 0.4% Flowers of sulphur.
99.6% Machine oil distillate 2.8°

The additives were incorporated mainly by heating and mixing.

Data :	(i)	(ii)
Sp. gr. at 20°C.	circa 0.920	circa 0.920
Flash point	" 185°C.	" 185°C.
Acid value	" 0.25	-
Viscosity at 50°C.	" 3°E.	" 3°E.

Question 2. What changes have been made during the war either voluntarily or due to lack of the required raw materials?

Answer. No changes because of lack of suitable materials were necessary during the war. Cutting oil containing Faktispaste R.M.9, was developed during the war as an improvement on the cutting oil types containing elementary sulphur.

Question 3. Were these materials satisfactory or could they have been improved?

Answer. The materials were satisfactory; no work was done on improving them.

Question 4. Were sulphur, phosphorous and chlorine additives used in these products as well as fatty oils?

Answer. Sulphur and sulphur compounds were used together with fatty oils. Phosphorous and chlorine additives were also used.

Question 5. Have substitutes been employed for fatty oils?

Answer. No substitutes for fatty oils were used.

Question 6. Were any troubles encountered in their use?

Answer. As such substitutes were not used no troubles were encountered.

D. Cable Oils.

Question 1. What types of oils were used for both the light and heavy types of cable oil?

Answer. Formerly American Cylinder Oil raffinate, viscosity 50°E./50°C. and cylinder oil distillate 2.8°E./100°C. were employed.

Question 2. To what specifications were they manufactured?

3. What changes in composition had to be made during the war and what troubles were experienced?

4. Were any additions made to increase dielectric properties, to stop hydrogen formation, or to impart special properties?

Answer. No information is available.

E. Marine Oils.

Question 1. What products were used for this purpose and to what specifications were they produced? Were Voltol products used in these oils?

Answer. Before the war, a number of so-called Marine Oils with varying additions of thickened rape oil were manufactured. Similarly the Navy Marine Oil was made with 23% of thickened rape oil. They were used generally for maritime purposes. No Voltol products were included in these oils.

Question 2. What changes have been made in their composition during the war and for what reason?

Answer. During the war rape oil was no longer available in sufficient quantities. Oil for the lubrication of reciprocating steam engines was therefore made sufficiently emulsifiable by using emulsifying agents produced by I.G. Farbenindustrie instead of thickened rape oil.

Emulphor A extra was first used for this purpose and later by order of the Naval Authorities, Emulphor FM (oil soluble). The basic oil distillate or raffinate had a viscosity of 9°E. at 50°C. The addition of Emulphor was 0.2% and of Emulphor A extra, 0.5%.

Question 3. What difficulties were experienced in their use?

Answer. No trouble occurring with our oils is known but oils containing various emulsifying agents were found, to some extent, to be unstable in storage. Emulsifying agents were inclined to separate during storage at low temperatures.

Question 4. Were any additions made to impart special properties?

Answer. No other additions other than those mentioned above were made.

F. Refrigeration Oils.

Question 1. What products and from what sources were used for this application, either for SO_2, CO_2, NH_3 or Freon, compression? To what specifications were they produced and what changes in composition have been necessary during the war and for what reason? What troubles have been experienced in their use and what additions have been used to overcome sludging and/or corrosion?

Answer. Refrigerator oils for CO_2 and NH_3 were supplied before the war coloured yellow and red.

Inspection data :

Specific gravity at 20°C.	0.890
Flash point	176°C.
Pour point	-48°C.
Acid value	0.05
Colour	-2
Viscosity at 20°C.	6.21°E.
" at 50°C.	2.04°E.

During the first war year they were made from Reitbrook spindle oil R.22. We know of no trouble occurring with our refrigerator oils. Additives to improve the sludge and corrosion tests were neither tested nor used.

G. Insulating Oils.

Question 1. What oils were used for this application and to what specification were they produced?

Answer. Basic oils of the following types were employed :

Sp. gr. at 20°C.	0.881	0.868
Flash point	164°C.	156°C.
Pour point	-45°C.	below -40°C.
Colour	-1	whitish
Viscosity at 20°C.	4°E.	5°E.
" at 50°C.	1.78°E.	1.9°E.
" at 100°C.	1.20°E.	1.23°E.
" Poleheight	2.0°E.	2.2°E.

Question 2. What changes in composition have been necessary during the war and for what reason?

Answer. The basic oil was changed during the war because the earlier source of supply was cut off.

Question 3. What troubles have been experienced in their use?

Answer. No trouble is known to have occurred with these products.

Question 4. What additives, e.g. anti-oxidants or metal passifiers have been used?

Answer. Anti-oxidants and other additives were not used but 0.15% Paraflow was used to improve the pour point.

Question 5. Have any non-petroleum products been used for this purpose?

Answer. Non-petroleum products were not used.

H. Specialities.

Question 1. Have any oils been developed for high temperature lubrication?

Answer. No particular oils were developed for high temperature lubrication.

Question 2. Describe the use of wire rope lubricants, their composition etc. Have refinery extracts found application in this field?

Answer. Wire rope oils were not manufactured.

Question 3. What oils have been used as mould lubricants? Describe their use and composition.

Answer. No information.

Question 4. What products have been used as air tool lubricants? Describe their use, changes in composition as outlined in the above question.

Answer. No information.

Question 5. What types of products have been used as general machinery oils and black oils?. Describe their use, composition etc. To what extent have oils been replaced by emulsions in the former case and to what extent have solvent extracts been used in the latter?

Answer. General machine oils consist of blends of auto oils, engine oils using bright stock and other suitable basic materials. Before the war these came from overseas and during the war the bright stock component was decreased to vanishing point. Suitable home produced oils were also used with no apparent bad effect. Black oils were not blended by us. Part of the lubricating oil raffinate was replaced by emulsion lubricating oils.

I. Greases.

Question 1. What types of products have been used for the following purposes?

- (i) Ball and roller bearing greases, for high and low speed, high and low temperature and for heavily loaded applications.
- (ii) General lubrication greases.
- (iii) Block greases for steel mills, paper machinery crushers etc.
- (iv) Greases or oils for open gears.
- (v) Greases for marine applications.
- (vi) Any other types of greases.
- (vii) Extreme pressure greases.

Answer. (i) Our former lubricating grease red (Lubfett rot) as well as transparent grease 1453 were used for ball and roller bearings.

Composition

80.70%	Spindle oil distillate	80.00%	Machine oil distillate
2.00%	Whale oil	4.00%	Whale oil
6.00%	Fish oil fatty acid	6.00%	Fish oil fatty acid
7.00%	Ground nut oil fatty acid	3.00%	Lime
3.00%	Lime	0.20%	Caustic soda lye
0.25%	Caustic soda	0.02%	Dye 1531
0.05%	Red dye	0.78%	Water
1.00%	Water	6.00%	Ground nut oil fatty acid

Inspection data

Ash	3.00% CaO	3.00% CaO
Colour	Red	Dark yellow
Water	1%	Below 1%
Flow point	70°C.	70°C.
Drop point	80°C.	80/85°C.
Consistency	like Vaseline	like Vaseline

(ii) General Lubricant Greases 1451 and 1452.

Composition

80.7%	Machine oil distillate	77.7%
3.00%	Whale oil	5.0%
7.00%	Fish oil fatty acids	8.0%
3.00%	Lime	3.0%
0.20%	Caustic soda lye	0.2%
1.26%	Zinc white	1.26%
0.04%	Dye 1531	0.04%
3.00%	Ground nut oil fatty acid	3.00%
1.80%	Water	1.80%

Inspection data

Ash	ca. 3% CaO	3% CaO
Colour	Light yellow	Light yellow
Water	ca. 2%	ca. 2%
Flow point	70/75°C.	70/75°C.
Drop point	80/85°C.	85/90°C.
Consistency	Ointment like	Ointment like

(iii) Block greases were not made.

(iv) Greases for open gears were not made.

(v) Marine greases (ZdM 20) were first prepared on a crude montan wax basis during the war.

(vi) To the greases previously mentioned may be added the following wagon greases.

Composition

(a)

65%	Machine oil distillate
17%	Rosin oil - Jeserich
18%	Light mother liquor (Mutterlauge)

(b)

88.75%	Spindle oil distillate 2.8°E./20°C.
4.5%	Crude montan wax
3.00%	Final distillate fatty acid
2.00%	Fatty acid
0.60%	Caustic soda
1.00%	Lime
0.10%	Water

Inspection data

Ash	ca. 2% CaO
Colour	natural
Water	traces
Drop point	90/100°C.

(vii) High pressure lubricating greases were not made.

Question 2.	With each of the above give the relevant specification.
Answer.	Because of the shortage of raw materials for greases, all compositions were given up during the war. Greases were based on crude montan wax and fatty material (Fettstoff). Besides being dark in colour such greases were lumpy in structure and not waterproof.
Question 3.	What changes have been made in composition during the war and for what reason?
Answer.	To overcome the above disadvantages, a standardised type of grease was prepared containing an addition of acetic acid and this was found to be satisfactory.

1350 S.P.& Co LTD. 12-8-46 50271

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B.I.O.S.—FINAL REPORT No. 636
ITEM No. 30

MINERALÖLWERKE F. HARMSEN
KIEL—GERMANY
LUBRICANTS

Thomas, W. H., Withers, J. G.

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BRITISH INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

MINERALÖLWERKE F. HARMSEN

KIEL - GERMANY

LUBRICANTS

October 9th - 19th, 1945

Reported by :

W.H. Thomas, British, Ministry of Fuel and Power.

J.G. Withers, British, Ministry of Fuel and Power.

BIOS Trip No. 1248

B.I.O.S. Target No. C30/40

Fuels and Lubricants

British Intelligence Objectives Sub-Committee,
32 Bryanston Square,
London, W.1.

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PERSONNEL OF TEAM.

Dr. W.F. Faragher, U.S. Petroleum Administration for War.
Mr. J.G. Allen, U.S. Petroleum Administration for War.
Major W.H. Thomas, British, Ministry of Fuel and Power.
Mr. J.G. Withers, British, Ministry of Fuel and Power.

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ACKNOWLEDGEMENT.

The German translations involved in the production of this report were made by Mr. K. G. C. Kirkpatrick.

1. SUMMARY.

General information on the products made by Mineraloelwerke F. Harmsen and their methods of manufacture is given. These products were, in the main, greases of various types and torpedo lubricants. Specifications and compositions are included together with any changes necessitated by war conditions.

INTRODUCTION.

The information given in this report was obtained from Herr H. Wegner - Sales Manager of Mineralölwerke F. Harmsen in the course of a visit (B.I.O.S. Trip No. 1248) to the Hamburg area during the period October 1st to 31st 1945.

The object of the contact was to obtain information respecting the types and quality of lubricants manufactured by this firm and to collect samples of typical refined materials.

GENERAL.

The firm of Mineralölwerke F. Harmsen, Kiel with its office and refinery at Kiel-Hassee, Neuenrade 2-10 is equipped for the distillation and refining of light lubricants (20 to 30 tons per day) and for the manufacture of sodium and calcium base greases (6-10 tons per day). The equipment consists of a pipe-still distillation unit, facilities for carrying out acid and clay treatments, filter presses, grease making plant and plant for blending lubricating oils. There is also a plant for the preparation of Invarol grease, the process, which is a patented one, being leased from Dr. Marder of the Berlin Technical High School.

The feed-stock to the refinery is normally Heide crude oil, the light fractions of which are sold as cleaning oil. On occasions, however, a long Floridsdorf (Vienna) distillate supplied by Rhenania Ossag has been processed to yield diesel oil (12-20%), spindle oil (80%) and a residue with a viscosity of 2°E. at 20°C. (2-8%). The spindle oil (pour point -50°C.) is refined with 15 to 20 per cent of acid, neutralised by clay treatment and returned together with other products to Rhenania Ossag.

Much of the finished lubricating oil was sold for Naval purposes and the greases were sold to Rhenania Ossag or to jobbers. At the time of the interrogation, the refinery grease production was allocated entirely to Rhenania Ossag.

The firm of Harmsen produced a limited range of lubricants and hence was not in a position to give answers to questions on fuels or automotive lubricants. Likewise, no information could be obtained with respect to the following:-

Turbine Oils
Drawing Oils
Core Oils
Ink Oils
Cable Oils
Waxes
Railway Oils
Rolling Oils
Rust Preventatives
Textile Oils
Refrigeration Oils
Extracts
Cutting Oils
Quenching Oils
Slushing Oils
Leather Oils
Insulating Oils
Bitumens.

The laboratory facilities at the Kiel refinery were devoted entirely to the control of product quality but it is understood that some development work had been carried out in conjunction with Dr. Marder on the production of Invarol grease. This material was prepared from selected stocks by controlled continuous heating (up to 8 days) without the use of any additives.

4. TORPEDO OILS.

The following are details of the torpedo oils produced during the war at the Harmsen works.

The raw materials used for the separate lubricants were heated to 40/60°C. and then mixed in agitator. mixers or by circulation pumping.

The lubricants LP.151 and 295 III E.N. were submitted to a further refining after preparation. The addition of tricresylphosphate made these oils especially suitable for use as torpedo lubricants, as it imparted to them a specially high E.P. value.

The data given below include formulae for the composition of the individual lubricants but wherever a change in formula became necessary during the war, the new composition is given with the date of manufacture. The basic oil processed was a machine oil raffinate from Heide crude together with a low cold test spindle oil from Polish crude oil. After the use of Polish oil was rendered impossible, an oil supplied by the Navy and designated ZdM.9 was processed in its place, but the origin of this material is unknown to Harmsen.

LP.151: Torpedo Lubricant.

Prepared between December 1940 and December 1941.
Quantity delivered, approx. 50 tons per month.
This lubricant was withdrawn because the tricresylphosphate content caused trouble by its corrosive and destructive action on paint work.

Normal inspection data are :-

Colour	**Golden yellow**
S.G.	**1.13 at 20°C.**
Flash Point	**234°C.**
Viscosity	**10.90°E. at 20°C.** **2.71°E. at 50°C.**
Ash	**0.034% wt.**
Freezing-Point	**Solid at -25°C.** **Liquid at -21°C.**
Acid Value	**0.40**

Its composition as supplied to the Navy was :-

86% Tricresylphosphate
10% Crude rape oil
4% Thickened rape oil.

IP.295 III E.N.: Torpedo Lubricant

Manufactured from January 1942 until the end of the war. Quantity delivered approx. 100 tons per month.

Inspection data:

Appearance	Clear
Colour	Green
Smell	Faint
S.G.	0.933 at 20°C.
Viscosity	12.35°E. at 20°C. 3.15°E. at 50°C.
Poleheight	1.90
Flash Point	163°C.
Acid content:	
(a) mineral acids	Absent
(b) acid value	0.23
Ash	0.0055% wt.
Pour Point	Solid at -43°C.

Compositions were :-

10.2.43	1.2.45
15% Original torpedo oil	15% Original torpedo oil
3% Blown rape oil	3% Blown rape oil
10% Tricresylphosphate	10% Tricresylphosphate
3% Paraflow	72% ZdM.9
38% Spindle oil raffinate 5.2°E. at 20°C.	
31% Machine oil raffinate 6.5°E. at 50°C.	

IP.295 E has an addition of 3% Emulphor X.

E.T.42 Torpedo Lubricant

Manufactured between November 1942 and the end of the war. Quantity delivered - about 10 tons per month.

Inspection data:

S.G.	1.100 at 20°C.
Viscosity	39.40°E. at 20°C. 6.06°E. at 50°C.
Poleheight	2.65
Flash Point	215°C.
Acid Value	0.10
Ash	Below 0.010% wt.
Freezing Point	Solid at -33°C.

Compositions were:

1.11.43	8.2.45
6% Paraflow	2% Paraflow
25% Original torpedo oil	25% Original torpedo oil
29% Machine oil raffinate 6.5°E. at 50°C.	33% Machine oil raffinate 6.5°E. at 50°C.
40% Clophen A 60	40% Clophen

Torpedo Lubricant "Harminol 309"

Manufactured from January 1942 until September 1942. Quantity delivered - about 500 Kg. monthly.

Inspection data:

S.G.	0.901 at 20°C.
Flash Point	219°C.
Viscosity	9.30°E. at 50°C.
Freezing Point	Solid at -30°C.

Compositions were:

5.3.43	8.4.43
96% Machine oil raffinate 8.5°E. at 50°C.	71.0% Machine oil raffinate 8.5°E. at 50°C.
4% Paraflow	25.0% Machine oil raffinate 6.5°E. at 50°C.
	4.0% Paraflow

Resistance Oil (Widerstandsöl) 302°C.

Manufactured between February 1942 and September 1944. Quantity delivered, about one ton monthly.

Inspection data:

S.G.	**1.176 at 20°C.**
Flash Point	**138°C.**
Viscosity	**17.90°E. at 20°C.** **3.84°E. at 50°C.**
Acid Value	**0.125**
Ash	**0.361% wt.**
Freezing Point	**Solid at -55°C.**

Compositions were:

<u>16.7.43</u>	<u>30.6.44</u>
30% Glycol	40% Glycol
70% Glycerogen	60% Glycerogen.

5. <u>GREASES</u>.

Below are given details of greases made by Harmsen during the war and despatched for both civilian and army use. Wherever a change in composition has been made necessary because of raw material difficulties, the new formula is given with the appropriate date.

The manufacture of calcium soap greases was carried out in the autoclave, followed by stirring, whereas the sodium soap greases were prepared in oil heated boiling kettles. The amount produced annually ran to a total of about 2,300 tons of various greases, of which about 20 per cent was reserved for Army needs.

<u>Products Supplied for Army Use.</u>

<u>ZdM 20. Stauffer Grease.</u>

<u>Specification:</u>

Drop Point	80°C. min.
Flow Point	75°C.
Ash	2% max.
Water	2% max.
Consistency	
at -10°C.	0.6
at +20°C.	0.5
Water resistance in stationary sea and distilled water.	No clouding and no change in the grease surface (i.e. no swelling, decomposition or change in colour).
Water stability in agitated sea water.	No oil separation or change in the grease surface.
Oil separation at 35°C.	0.5% max.

Change in consistency figure at -10°C.	**0.8 max.**

Composition:

<u>12.2.43</u>	**<u>1.7.43</u>**
9% Crude montan wax	**16% Synthetic fatty acid**
3% Synthetic fatty acid	**2% Calcium hydroxide**
2% Calcium hydroxide	**82% Spindle oil distillate** **2.5°E. at 20°C.**
3% Caustic soda lye	
83% Spindle oil distillate **2.5°E. at 20°C.**	

<u>ZdM 21. Roller Bearing Grease.</u>

<u>Specification:</u>

Drop Point	150°C. min.
Flow Point	140°C.
Ash	2% max.
Water	0.5% max.
Consistency -15°C.	1.9
+20°C.	0.7
+70°C.	0.6
Oil separation at 75°C.	0.5% max.
Acid Value	0.5% mg. KOH/g. max.
Change in the consistency figure at -15°C.	3 max.
+70°C.	0.5 min.

Composition:

<u>12.2.43</u>	<u>1.10.44</u>
3% Emulsifying wax P.S.	10% Technical tallow
2% Cemon wax	4% I.G. wax "L"
2% I.G. wax O	1% I.G. wax "BJ"
4% Caustic soda lye	1% Caustic potash lye
8% Machine oil raffinate 6.5°E. at 50°C.	2% Caustic soda lye
81% Prepared 295	82% Machine oil raffinate 5.4°E. at 50°C.

<u>Torpedo Roller Bearing Grease L.P.182a</u>

Composition:

<u>8.6.43</u>	<u>1.11.44</u>
6% Technical tallow	6% Technical tallow
3% Cemon wax	5% I.G. wax "S.T."
2% I.G. wax "S"	2% Caustic potash lye
2% Caustic potash lye	2% Paraflow
87% Machine oil raffinate 6.5°E. at 50°C.	83% Machine oil raffinate 4.5°E. at 50°C.

Invarol Grease PX.

Specification:

Drop Point	147°C.
Can be stirred down to	-36°C. when it solidifies
Consistency	0.90 Kg./20°C.

Composition:

30.6.43

60% Shell oil AB.11
30% Nova oil (Schwechat)
10% Synthetic lubricating oil R.2000.

LP.303 Bearing Grease.

Composition:

15.5.43	17.11.44
27% Wax	32% Ozocerite
14% Ozocerite	2% Densodrin
5% Rape oil	5% Vaseline
54% Spindle oil distillate	61% Spindle oil distillate
2.5°E. at 20°C.	5.2°E. at 20°C.

Products Supplied for Civil Use.

No. 1010a. Machine Grease (Dark)

8.1.43	10.11.44
6% Crude montan wax	15% Train oil fatty acid
8% Synthetic fatty acid	2% Calcium hydroxide
2% Hydrated marble lime	83% Machine oil distillate
84% Spindle oil distillate	4.5°E. at 50°C.
2.5°E. at 20°C.	

No. 1011a and 1072. Ball Bearing and Chassis Grease.

4.3.45	5.5.45
11% Technical tallow	10% Technical tallow
1% I.G. wax "O"	5% Train oil fatty acid
2% Synthetic fatty acid	2% Calcium hydroxide
1% Calcium hydroxide	83% Machine oil raffinate
42% Extr. oil	4.5°E. at 50°C.
43% Machine oil distillate	
4.1°E. at 50°C.	

No. 1071a. Special Lubricating Grease (Red)

24.5.43	30.3.45
13% Technical tallow	15% Train oil fatty acid
2% Calcium hydroxide	2% Lime
2% Crude montan wax	83% Machine oil distillate
4% Final distillate fatty acid	4.5°E. at 50°C.
79% Spindle oil raffinate	
2.5°E. at 20°C.	

No. 1021. Special Wagon Grease.

29.3.43	
10% Talc	12% Talc
8% Resin oil	8% Calcium hydroxide
10% Calcium hydroxide	10% Resin oil
72% Black oil	70% Machine oil distillate
	6.5°E. at 50°C.

No. 1022F. Wagon Grease (Dark)

1.2.43	1.6.45
4% Resin oil	10% Resin oil
20% Crude paraffin wax	8% Calcium hydroxide
20% Talc	16% Talc
4% Calcium hydroxide	66% Black oil
52% Black oil	

An addition of 1 per cent graphite was made to Graphited Wagon Grease.

No. 1061. Roller and Ball Bearing Grease.

15.3.45	31.12.44
6% Technical tallow	10% Technical tallow
4% Cemon wax	4% I.G. wax "L"
1% I.G. wax "O"	1% Caustic potash lye
1% Caustic potash lye	4% " soda lye
37% Machine oil distillate	81% Machine oil distillate
4.5°E. at 50°C.	4.5°E. at 50°C.
51% Extr. oil	

No. 1066. Hot Neck Grease.

21.1.43	30.7.44.
2% I.G. wax "S"	10% Technical tallow
2% " " "E"	4% I.G. wax "L"
7% Caustic soda lye	1% " " "BJ"
89% Extract oil 295	1% Caustic potash lye
	3% " soda lye
	81% Spindle oil raffinate 5.2°E. at 20°C.

No. 1111. Special Auto-Transmission Grease.

27.3.43	13.9.43
10% Technical tallow	31% Spindle oil distillate 2.5°E. at 20°C.
1% Caustic soda lye	69% Captured grease (Beutefett)
89% Machine oil distillate 4.5°E. at 50°C.	

No. 1214. Boring and Drawing Grease.

2.3.43	3.3.45
15% Train oil fatty acid	16% Train oil fatty acid
3% Caustic potash lye	5% Caustic potash lye
52% Spindle oil raffinate 5.2°E. at 20°C.	55% Machine oil distillate 4.5°E. at 50°C.
30% Captured grease (Ausbeute)	24% Captured grease (Ausbeute)

No. 1213. Drawing Paste.

14.1.43

43% Soap stock
43% Spindle oil raffinate 5.2°E. at 20°C.
7% Liquid resin (Tallöl)
4% Caustic soda lye
3% " potash lye

No. 1031. Leather Grease (Black)

16.3.43	3.4.45
12% Ozocerite	17% Ozocerite
18% Crude paraffin wax	58% Black oil
70% 754 Raffinate	20% Spindle oil distillate 1.7°E. at 20°C.
	3% Crude paraffin wax
	2% Ceresin.

No. 1150. Milk Grease (Melkfett)

20.1.43	11.7.45
30% Soft wax	60% Vaseline
15% Wax "Elap"	10% Ointment wax
2% Paraffin wax	30% Spindle oil raffinate 5.2°E. at 20°C.
52% White oil	
1% Ceresin	

No. 1215. Emulsion Grease.

26.5.43	15.7.44
5% Crude montan wax	9% Crude montan wax
2% Densodrin	41% Black oil
3% Ozocerite	50% H_2O
7% Spindle oil raffinate 5.2°E. at 20°C.	
33% Black oil	
50% H_2O	

L.P. 196. Protective Grease 40.

21.4.43	3.1.44
40% Vaseline	40% Vaseline
9% Ozocerite	9% Ozocerite
20% Machine oil raffinate 6.5°E. at 50°C.	20% Machine oil raffinate 6.5°E. at 50°C.
31% Spindle oil raffinate 5.2°E. at 20°C.	31% Spindle oil raffinate 5.2°E. at 20°C.

6. SAMPLES.

The following samples of Harmsen products were obtained and are now in the custody of the **Ministry of Fuel and Power.**

Sample	No.	203	- Ball bearing grease	1 lb. tin
"	"	204/73	- Invarol grease	1 lb. tin
"	"	205	- Machine grease	1 lb. tin
"	"	206	- Lubricating grease - red	1 lb. tin
"	"	207	- Torpedo oil L.P. 295 III E.N.	1 litre can
"	"	225/13	- Torpedo oil E.T. 42	20 litre can.

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B.I.O.S. FINAL REPORT No. 598

ITEM No 30

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HAMBURG - GERMANY

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Thomas, W. H., v Withers, J. D.

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON-H.M. STATIONERY OFFICE

OELWERKE JULIUS SCHINDLER G.m.b.H.

HAMBURG - GERMANY

LUBRICANTS

October 2nd - 10th 1945

Reported by:

W.H. Thomas, British, Ministry of Fuel and Power.

J.G. Withers, British, Ministry of Fuel and Power.

B.I.O.S. Target No. 30/231
Fuels and Lubricants

British Intelligence Objectives Sub-Committee
32 Bryanston Square,
London, W.1.

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PERSONNEL OF TEAM.

W.F. Faragher, U.S. Petroleum Administration for War.
J.G. Allen, U.S. Petroleum Administration for War.
W.H. Thomas, British, Ministry of Fuel and Power.
J.G. Withers, British, Ministry of Fuel and Power.

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ACKNOWLEDGEMENT.

The German translations involved in the production of this report were made by Mr. K.G.C. Kirkpatrick.

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1. SUMMARY.

This report gives information regarding the refinery operations of and products produced by the firm of "Oelwerke Julius Schindler" of Hamburg.

General information is provided with respect to the two refineries operated by the firm and as an illustration of normal operation, throughput and yield data for the year 1940 have been included.

The products prepared for sale comprised a wide range of general and specialised lubricants etc. but no fuels or engine lubricants were produced. Detailed information on the products is presented in the form of answers to a comprehensive questionnaire.

Three 4 gallon samples of typical products were obtained; they were :-

Liquid paraffin
Transformer oil and
Turbine oil.

2. INTRODUCTION.

The information given in this report was obtained from Herr K. Albrecht - Manager and Dr. H. Sieveking - Lawyer of "Oelwerke Julius Schindler" in the course of a visit (B.I.O.S. Trip No. 1248) to the Hamburg area during the period October 1st to 31st 1945.

The object of the contact was to obtain information respecting refinery operations and quality of products and to collect samples of typical refined materials.

3. GENERAL.

The firm of Oelwerke Julius Schindler with its Head Office at Hamburg, 36, Hohe Bleichen, 28, was engaged in the manufacture, importation and marketing of lubricants and special products of the white-oil type.

The directors were C.W. Bunte, K. Albrecht and H. von Wunsch and the capital amounted to 2,400,000 marks.

Prior to the war, the control of the firm was from outside Germany the shares being held as follows :-

Pure Oil Co. (U.S.)	49 per cent
Julius Schindler (U.S.)	24 " "
Gewerkschaft Neue Erdölraffinerie (NERAG)	27 " "

Refining operations were carried out at Wilhelmsburg and at Mineralölwerke Peine near Hannover.

The Wilhelmsburg refinery was equipped with a vacuum distillation unit having a finished lubricant throughput of 800 tons (metric) per month and capable of operating on either crude oil or residue therefrom. In addition, plant was available for carrying out normal refining treatments with sulphuric acid and/or clay.

Until hostilities with Russia commenced, Russian oil was processed but subsequently German crude oil (mainly from Hannover) was used.

The Hannover works distilled heavy German crude oil at a throughput of 4,500 tons per month, the main product being 3,000 tons per month of railway axle oil.

Light distillates from both refineries were disposed of through the Z.B. organisation and a high proportion of the finished lubricants were sold through the A.C.V. organisation.

Export of lubricating products to European countries was arranged by the Export Gemeinschaft Schmierstoff und Weissöl which was in effect a combination of the following firms :-

(a) Schliemann
(b) Schindler
(c) Albrecht
(d) Rhenania Ossag.

The firm of Schindler operated no patented processes.

4. REFINERY DATA.

The following is a statement showing the production of the Wilhelmsburg works during the year 1940 (see attached table). This is typical of operations carried out before the war and during the first years of the war on the basis of imported Russian raw materials. As the grade programme was very comprehensive, the throughput and yield figures of various qualities which are distinguished essentially by their colour have been classed in groups. During the year 1940 it was possible to maintain the programme of grades almost completely, because only unimportant quantities were manufactured from German raw materials and Russian stocks were available which were particularly suited to the production programme.

Later on during the war it was necessary to switch over to the processing of Roumanian, Galician and German crude oils and although at first there were no considerable changes in the quantity processed, the change-over to these raw materials made itself felt in a lower yield. When processing German and Roumanian spindle oils instead of Russian solar oils, the yield decreased :-

for transformer oil	from 90 to 70 per cent
for pharmaceutical white oils	from 70 to 50 " "
for technical white oils	from 80 to 70 " "

A raw material corresponding to light Russian engine oil could not be obtained and when processing German and Roumanian heavy engine oils, the yield of medicinal white oil (paraffinum liquidum) dropped from 58 per cent to 30 per cent.

In spite of a certain amount of air-raid damage, production was maintained until June 1944, after which the works were hit several times in air raids. Although the plant was never completely closed down, repairs occupied a considerable part of the time of the personnel and production fell heavily. However, it was possible to put the distillation unit into operation again after a time and to employ the personnel for the manufacture of raw materials for the refinery. At present the latter is operating at an average throughput of 700 tons per month in respect of transformer and white oils, plus 75 tons per month of paraffinum liquidum. This performance is dependent on the necessary raw materials, chemicals and coal being available. The use of the German raw materials now available results in the same difficulties experienced during the war, which on the one hand led to the less favourable yield mentioned above and on the other hand to the impossibility of manufacturing certain special qualities which are urgently needed. Experiments made before the outbreak of war showed that certain American oils are particularly well suited for production purposes. These are the Paratox oils with Saybolt viscosities of 75, 100 and 500 secs. which were intended to be purchased from the Gulf Oil Corporation, Pittsburg. These oils showed even better yields than the Russian oils formerly processed. Accordingly, shortly before the outbreak of war, certain quantities had already been purchased and shipped and it was expected that the difficulties already existing at that time in respect of purchasing Russian oils would be circumvented thereby.

THROUGHPUT AND YIELDS FOR 1940

	Russian solaroil		Light Russian machine oil 3°E./50°C.		Heavy Russian machine oil 6.5°E./50°C.		Total
	tons	per cent	tons	per cent	tons	per cent	tons
Throughput	6.200	-	855	-	1.700	-	8.755
Yields	5.560	69.7	646	75.5	976	57.5	7.181
4 qualities liquid paraffin (medicinal)	150	66.3	150	67	810	58	1.110
11 " pharmac. white oil	1.690	70.8	140	66.4	33	59	1.863
6 " technical " "	660	80	230	79	23	65	913
2 " transformer oil	3.060	91	-	-	-	-	3.060
3 " turbine oil	-	-	125	10	110	91	235
Loss	640	10.3	210	24.5	724	42.5	1.574

5. ANSWERS TO QUESTIONNAIRES.

QUESTIONNAIRE NO. 1

FUELS

No information.

QUESTIONNAIRE NO. 2

LUBRICANTS

A. Aviation
B. Automotive
C. Marine
D. Transmission
E. Hydraulic, Recoil and Buffer Oils
F. Railroad

No information.

G. Industrial

Question 1. What types of oil are used in steam turbine lubrication? Are any troubles experienced with corrosion, emulsification and if so, how are they combated? What additives are used, if any? What is the average life of a steam turbine oil?

Answer. Carefully refined mineral oils of viscosity 3-6.5°E./50°C, which are stable as regards emulsifying and ageing. Hardly any corrosion trouble occurs and emulsion difficulties are rare and when experienced, are caused by circumstances outside the quality of the oil itself, e.g. contamination of the oil charge, excessive ageing or poor treatment of the oil.

Length of life varies considerably, depending primarily on the running conditions, especially temperature relationships in the steam turbine. According to such conditions, the life period varies between 3000 and 60,000 working hours. In the last case, the topping-up quantity becomes so large that it may be said, for all practical purposes, that the charge has been renewed.

Question 2. What types of oils are used for transformers? What electrical tests, if any, are carried out? Are any additives employed?

Answer. Carefully refined mineral oil with a viscosity of about 2.5-6 °E./50°C. which above all must resist ageing. The oil is tested for the break down voltage only in case of need but other test centres examine the oils, for specific resistance or voltage loss. Additives are not used.

Question 3. To what extent are mineral, non-mineral, compounded oils and aqueous emulsions used in quenching? What types are preferred for small and large objects?

Answer. No information is available.

Question 4. What types of cutting oils are used? To what extent are sulphurised and compounded oils used? Are corrosion inhibitors added and if so, what are they?

Answer. Pure or sulphurised mineral oils are used as cutting oils. Compounded oils are not prepared for this purpose, because of fat shortage. No anti-corrosion media are added.

Question 5. What types of slushing oils are used? What additives are employed to give anti-corrosion properties?

6. From what sources are textile oils refined? What research has been done on carcogenicity?

7. What oils are used for clock and instrument lubrication, especially for aviation use? What compounding agents are used?

8. What is "Radiol" and for what uses is it employed?

9. What is the composition of compounded light machine oils?

Answer. No information on Questions 5, 6, 7, 8 and 9 is available.

H. Greases

Question 1. To what extent are barium, lithium and aluminium soap greases used?

Answer. Barium, lithium or aluminium greases were not manufactured.

Question 2. What fats are used for greases and what substitutes have been employed?

3. To what extent are waxes, especially montan wax, used in grease manufacture?

Answer. Synthetic waxes only were used in the manufacture of lubricating greases.

Question 4. What types of greases are used for high temperature applications?

Answer. For high pressure conditions, greases were based on very viscous mineral oils as well as with additives. To date such greases have not been prepared by Oelwerke Schindler.

Question 5. For what reason and to what extent are greases used in shock-absorbers-for-aeroplane landing gear?

Answer. The use of greases in shock absorbers and aeroplane landing gear is not familiar to us.

Question 6. What material is employed for ship launching purposes?

Answer. The following are used for ship launching: soap, wax and lime greases. Several such products were often used together.

I. General

Question 1. To what extent are aqueous emulsions used in industrial lubrication?

2. Has this type of lubricant ever been employed in engines of any type?

Answer. During the war, water-in-oil emulsions were used extensively for bearing lubrication instead of pure mineral-oil distillates or raffinates. Exceptions were machines with circulation-lubrication, those working at or exposed to low temperatures or very delicate machines or machine parts, e.g. fine mechanisms.

Question 3. By what processes is Voltol made?

Answer. Voltol is prepared by submitting pure mineral oils, compounded mineral oils or pure fatty oils to an electrical glow discharge. As far as is known, the only manufacturing plant in Germany for Voltol is that of the Rhenania Ossag at Freital near Dresden.

QUESTIONNAIRE NO. 3

SPECIAL PRODUCTS

A.	Bitumens	No information is available.
B.	Extracts and Polymers	
C.	Waxes	
D.	Rust Preventatives	

E. Cable Oils

Question 1. To what extent are oil filled cables used; what oil specifications are laid down; what are the best sources for oils of this type?

Answer. Oil filled cables have been used to an increasing extent during the last decade. A transformer type of oil is required which, however, must be tested for specific resistance and dielectric losses before and after ageing treatment. It is also often required that, under the influence of high voltage, a minimum of gas formation is caused. Oil from Russian sources proved to be the best but it is believed that Roumanian oil would be suitable.

Question 2. Are any blending agents or additives employed (e.g. resins)?

Answer. Only pure mineral oils were delivered for this purpose, without any blending or additives. Such additives are not used for oil filled cables. They are largely employed for solid cables (Massekabel) and are added in the cable works.

Question 3. To what extent are oil impregnated insulated types used? What oils are used and what specifications have been laid down?

Answer. The use of oil impregnated insulated cables is vastly greater in extent than that of oil filled ones, which, as far as is known, represent only a very small percentage of total production.

Oils with viscosities ranging from that of machine oil to that of the thickest cylinder oils are used for insulated cables. The degree of refining varies considerably as does the type of oil (from asphaltic to paraffinic). Specifications depend largely on the conditions to which the cables will be submitted. In addition, divergent views are expressed as regards the necessary analytical requirements by different experts and cable manufacturers. Generally, it may be said that raffinates, as pure as possible, are preferred because of their good electrical behaviour before and after artificial ageing.

Further, it is true to say that in pre-war years, the strongly paraffinic oils of Continental type were already being replaced by oils with the lowest possible paraffinic content. Here too, in the case of cables for high and very high voltages, the test for stability as regards gas formation has often been employed.

Question 4. What methods of test are used to determine di-electric strength etc. for such oils?

Answer. The following are tested: the specific resistance, using a bridge; dielectric losses, using a condenser; gas evolution, using a small condenser-like testing tube.

F. Coolants and De-Icing Fluids

No information is available.

G. Miscellaneous

No information is available.

QUESTIONNAIRE NO. 4

INDUSTRIAL LUBRICANTS

A. Turbine Oils

Question (a) What type of oil has been used for this purpose and from what raw materials?

(b) What changes have been made in this type of product during the war and for what reason

Question (c) What inhibitors and/or additives have been used in those oils?

(d) What troubles have been experienced in their use?

(e) What length of service has been common without oil change and what limits in deterioration are allowed before an oil change is considered necessary?

(f) What specifications exist for these oils?

Answer (a) Mineral oil raffinates with viscosities of 3-6.5/50 with good ageing properties, prepared by sulphuric acid treatment and 'wet' refining, from American, Russian, Roumanian or German crudes.

(b) During the war changes in the source of the oil occurred and every German oil which was not absolutely waxy was processed. Certain concessions were made as regards ageing and appearance.

(c) Inhibitors and additives were not used.

(d) They were found unnecessary in all more or less normal cases.

(e) The running time before oil change varies considerably, according to the working conditions of the turbine. A general guide is contained in the "Ölbewirtschaftung" of the VDEW, 2nd Edn., where limiting values for acid and saponification values are given. At this point, sludge formation is usually so far advanced as to make a change advisable.

(f) The limiting values in the "Ölbewirtschaftung" for used turbine oil are as follows :-

Acid value	not above 3.0
Saponification value	not above 6.0

B. Railway Lubricants

Steam Cylinder Oils - no information is available.

Axle Oils or Greases.

Question (a) What products have been used for this purpose?

(b) What specifications have been used covering these products?

(c) What changes have been made in these products during the war and for what reason?

(d) What troubles were experienced in the use of these oils and greases?

(e) What additives have been used in these products?

Answer (a) Axle and cylinder oils (Summer and Winter quality) corresponding to German Railway Specifications were used.

(b) The Specifications are as follows :-

AXLE OIL

	Summer oil	Winter oil
S.G. at 20°C.	below 0.950	below 0.950
Flash Pt. (open)	above 160°C.	above 140°C.
Viscosity E/20°C.	40-60	25-50
" E/50°C.	7-10	4.5-8
Hard Asphalt	below 0.2%	below 0.2%
Acid value	" 2.2	" 2.2
Water	" 0.2%	" 0.2%
Cold test	still fluid at -5°C.	still fluid at -20°C.

CYLINDER OIL

	Superheated steam cylinder oil	Wet steam cylinder oil
S.G. at 20°C.	below 0.950	below 0.960
Flash pt. (open)	above 300°C.	above 260°C.
Viscosity E/100°C.	above 5	above 3
Hard Asphalt	below 0.1%	below 0.2%

By appropriate choice of raw material and fraction, these oils were so adjusted as generally to lie just within these limits.

(c) The railway specifications already permitted dark oils to a large extent, so that significant changes in quality probably did not occur.

(d) No difficulties were known to us.

(e) Additives were not used.

Railway Diesel Oils - no information.

C. Cutting Oils

Neat Cutting Oils.

Question (a) What type of products (with formulae) have been used for this work? Show the different types used for different machining operations on different types of metal, including non-ferrous?

(b) Were these made to specification and if so, what?

(c) What changes have been made during the war either voluntarily or due to lack of the required raw materials?

(d) Were these materials satisfactory or could they have been improved?

(e) Were sulphur, phosphorous and chlorine additives used in these products as well as fatty oils?

(f) Have substitutes been employed for fatty oils?

(g) Were any troubles encountered in their use?

Answer. (a) Pure mineral oils of the spindle oil type and mineral oils with up to 1 per cent sulphur. No special information as to the uses of such oils is known.

(b) They were not made to a strict specification.

(c) There were no changes because of the war. However, the consumers used distillate as no more raffinates
(d) were assigned to them. The former were practically as suitable as raffinates.

(e), (f) and (g)

Sulphur was added. Other additives were not used by us.

Soluble Oils

Question (a) Show, with formulae, the changes made in these products during the war.

(b) Were they used to a greater extent than pre-war as a measure of economy?

(c) Did the shortage of petroleum sulphonates greatly affect the properties of these products?

(d) What troubles were experienced in their use?

(e) Were these products made to company or outside specifications: give details of these?

(f) What additives were used to impart outstanding qualities, such as E.P., anti-rust, anti-foaming etc.?

(g) Indicate what operations they were used on and for what metals.

Answer (a) and (b)

Soluble oils were based on naphthene sulphonic acid soaps instead of fatty acid soap and possessed the following approximate composition :-

ca. 15% Sodium naphthene sulphonate
" 80% Spindle oil distillate
" 1% Alcohol
" 4% Water.

(c) Adequate supplies of naphthene sulphonates were available so that the soluble oils did not suffer in this way.

(d) Any difficulties are unknown.

(e) The product was made to our own specification.

(f) and (g)

No additives were used for special properties. They were not necessary.

D. Drawing Oils
E. Rolling Oils
F. Quenching Oils
G. Core Oils
H. Rust Preventatives
I. Slushing Oils
J. Ink Oils
K. Textile Oils
L. Leather Oils

No information is available.

M. Cable Oils

Question (a) What types of oil were used for both the light and heavy types of cable oil?

(b) To what specifications were they manufactured?

(c) What changes in composition had to be made during the war?

(d) What troubles were experienced?

(e) Were any additions made to increase dielectric properties or to stop hydrogen formation?

(f) Were any additions made to impart special properties?

Answer (a) Only light cable oil, corresponding on the whole to transformer oil were supplied.

(b) No specifications are known. Consumers bought according to type.

(c) None were made.

(d) No difficulties are known.

(e) No additions were made to improve electrical properties or to avoid hydrogen formation.

(f) No other additives were used.

N. Marine Oils

No information is available.

O. Refrigeration Oils

Question (a) What products were used for this application, either for SO_2, CO_2, NH_3, Freon etc: and what were their sources?

(b) To what specifications were they produced?

(c) What changes in composition have been necessary during the war and for what reason?

(d) What troubles have been experienced in their use?

(e) What additions have been used to overcome sludging and/or corrosion?

Answer (a) In particular, spindle oils from Russian or Roumanian sources.

(b) Only the cold test was specified.

(c) No alterations were necessary during the war.

(d) None is known.

(e) No additives were used.

P. Insulating Oils

Question (a) What products were used for this application and what were their sources?

(b) To what specification were they produced?

(c) What changes in composition have been necessary during the war and for what reason?

(d) What troubles have been experienced in their use?

(e) What additives, e.g. anti-oxidants or metal passifiers have been used?

(f) Have any non-petroleum products been used for this purpose?

Answer (a) They were made from German, Russian, Roumanian or Polish oils.

(b) To those of the "Verein deutscher Elektrotechniker" and of the "VIEW" for insulating oils.

(c) Towards the end of the war, specifications were relaxed as regards cold test and ageing behaviour so that the degree of refining was not so high and the losses incurred were decreased.

(d) No difficulties are known.

(e) Additives were not used.

(f) Apart from unsuccessful trials, nothing is known of non-petroleum products being used as insulating oils.

Q. Specialities }
R. Greases } No information is available.

6. SAMPLES.

The following 4 gallon samples of Schindler products were obtained and are now in the custody of the **Ministry of Fuel and Power, London.**

Sample No. 124/84m.	White Liquid Paraffin
" " 125/84n.	Transformer Oil
" " 126/84p.	Turbine Oil.

50

Copy No. 1

B.I.O.S.—FINAL REPORT No. 637

ITEM No. 30

OLEX DEUTSCHE BENZIN UND PETROLEUM GESELLSCHAFT

FUELS AND DISTRIBUTION

Thomas, W. H., Withers, J. D.

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SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

OLEX DEUTSCHE BENZIN UND PETROLEUM GESELLSCHAFT

FUELS AND DISTRIBUTION

October 13th 1945

Reported by:

W.H. Thomas, British, Ministry of Fuel and Power.
J.G. Withers, British, Ministry of Fuel and Power.

BIOS Trip No. 1248

B.I.O.S. Target No. C30/279
Fuels and Lubricants

British Intelligence Objectives Sub-Committee
32 Bryanston Square,
London, W.1.

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PERSONNEL OF INTERROGATING TEAM.

Major W.H. Thomas, British, Ministry of Fuel and Power.

Mr. J.G. Withers, British, Ministry of Fuel and Power.

- - - - oOo - - - -

ACKNOWLEDGEMENT.

German translations involved in the preparation of this report were made by Mr. K.G.C. Kirkpatrick.

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1. SUMMARY.

This report gives information on the distribution and quality of fuels handled by the Olex Deutsche Benzin und Petroleum Gesellschaft and as a corrollary, a chart has been provided which indicates the manner in which the German government controlled the production and refining of petroleum.

The report also includes details of road tests carried out by the Olex company on alcohol fuels.

2. INTRODUCTION.

The information given in this report was obtained from Herr Gubish - Manager of Olex for the whole of Germany and from his technical assistant Dr. ing. W. Charpentier, in the course of a visit (B.I.O.S. Trip No. 1248) to the Hamburg area during the period October 1st - 31st 1945.

The purposes of the interrogation, which took place in Shell Haus, Hamburg, on October 13th, were :-

(a) to obtain information on the quality of fuels handled by the Olex company

(b) to ascertain the results of any road tests of fuels which may have been carried out

and (c) to determine the role executed by the Olex company in the German oil distribution organisation.

3. GENERAL.

The Olex Deutsche Benzin und Petroleum Gesellschaft was a marketing subsidiary of the Anglo-Iranian Oil Company prior to the war but in September 1939, its gasoline and diesel oil business was transferred to the Z.B. organisation. Later as from August 1st 1944, the Olex lubricating oil sales department was absorbed into the A.S.V. organisation in which Olex was represented by W. Koch - Manager of the Berlin Sales Branch. Olex however was not compelled to become a member of the Wirtschaftsgruppe Kraftstoffindustrie nor to subscribe to the Kontinental Oel A.G.

The war-time employement of the Olex company was the operation of its own tankage and the maintenance and repair of plant, pumps, pipelines etc. All types of petroleum products except asphalt were handled and were received from Z.B. and from German refineries, the latter embracing those of Roumania during the early stages of the war. Deliveries were made to civilian organisations (including Luft Hansa) according to Z.B. instructions which also applied to the Olex distribution system. Aviation fuel was not handled.

During the war, Herr Gubish continued with his control of the Olex operations and Dr. Charpentier was employed by the Z.B. (Zentral Büro) but his salary was paid by Olex.

Dr. Manasser one of the directors was fully occupied with administrative duties in the Berlin Head Office while Dr. Brandt, Herr Oechner, Herr J. Kramer and Herr Friedrich formed the directorate of the engineering staff. Dr. Deval was in charge of the Berlin laboratory.

4. FUEL SPECIFICATIONS.

Herr Gubish supplied details of the following fuel specifications :-

A. Aviation Fuel EI
B. Aviation Fuel EII
C. Aviation Fuel JII
D. Carburettor Fuel for the Army (Summer 1945)
E. Diesel Fuel for the Army (Summer 1945)
F. Diesel Fuel for the Navy.

They are reproduced below and are indicative of the reduction in quality brought about by loss of supplies and of refining capacity at the closing stages of the war.

A. Delivery Specification for Aviation Fuel EI

Appearance and Colour	- Clear, free from undissolved water and acid. Shall contain no solid impurities.
Specific Gravity at 15°C.	- 0.730/0.770
Boiling range	- I.B.P. between 30° and 50°C. Up to 75°C. 10% by vol., min. " " 100°C. 30% " " " " " 220°C. 95% " " "
Evaporation residue	- 50 mg./100 ml. max.
Corrosion test	- No grey or black pitting or spots in the copper strip test.
Separation	- No separation shall occur at temperatures above - 30°C.
Cloud point	- Cloud point of the fuel, cooled to the crystallisation point, shall not lie above - 30°C.
Lower calorific value	- 9,600 cals./Kg. min.
Water soluble components	- Absent.

B. Delivery Specification for Aviation Fuel EII

Appearance and Colour	- Clear, free from undissolved water. Shall contain no solid impurities.
Specific gravity at 15°C	- 0.820/0.860
Viscosity at +20°C.	- 1.0°E. min.
" " -25°C.	- 1.5°E. max.
Boiling range	- I.B.P., 40-70°C. - Up to 100°C., 10% by vol. min. F.B.P., below 360°C.
Corrosion test	- Copper strip - negative Zinc strip - 4 mg., max.
Melting point	- below - 30°C.
Separation	- No separation shall take place at temperatures above - 30°C.
Lower calorific value	- 9,600 cals./kg. min.
Phenol content	- 0.5% by vol. max.
Aromatics	- 25% by vol. max.
Water soluble components	- Absent
Conradson test	- 0.05% by weight, max.
Miscibility with aviation fuel EI	- The aviation fuel EII, must be miscible in all proportions with Aviation Fuel EI in the temperature range from - 30°C. to + 50°C.

C. Delivery Specification for Aviation Fuel JII

Specific gravity	- At least 0.764 at 15°C.
Vapour pressure @ 37.8°C	- 0.3 atms. max.
Viscosity at + 20°C.	- 1.0°E., min.
" " - 20°C.	- 5.0°E., max.
Filterability at -25°C.	- 120 secs. max.
Pour Point	- -25°C. min.
Sulphur content	- 3.0% by weight, max.
Phenol content	- 1.0% by vol., max.
Aromatic content	- 35% by vol., max.
Conradson test	- 1.5% by weight, max.
Ash	- 0.5% by weight, max.
Lower calorific value	- 9,000 cals./kg. min.
Zinc corrosion	- 4 mg. max.
Copper corrosion	- Negative.

D. Specification for Carburettor Fuel for Delivery to the Army, Summer 1945 (26.3.45)

Front region	- East, West and South.
Time of delivery	- From 1.3.45. *
General	- The fuels shall be clear, free from undissolved water and solid impurities and shall not attack copper.
Colour	- Red.
Density at 15°C.	- 0.710 - 0.820
Octane No. (Motor)	- 72(1)
Boiling Range:	
Distillate to 75°C.	- 30% by vol., max.
" " 100°C.	- 20% by vol., min.
" " 225°C.	- 95% by vol., min.
Vapour pressure at 40°C. - Reid	- 0.20 - 0.65 kg./cm.² (see footnote (2))
Evaporation residue	- Not above 10 mg. per 100 ml. (220°C.)
Cold test	- Clear and free from crystals down to - 10°C.(3)
Calorific value	- Above 7,500 cals./per litre.
Sulphur content	- Not above 0.8% by weight
Lead content	- 0.08% by vol. T.E.L.

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(1) Minimum Octane Number - 71.5.

(2) The vapour pressure may fall to 0.15 kg. per cm.² if the amount distilling over up to 100°C., is at least 30% by vol. (including distillation loss).

(3) A cloud point brought about by dissolved water must be noted on the inspection report.

* The winter grade can be delivered by 15.4.45, according to the transport position.

E. Specification for Diesel Fuel for Delivery to the Army, Summer 1945 (26.3.45)

Front region	- East, West and South
Time of delivery	- From 1.3.45
General	- Diesel fuel must be free from solid impurities
Density at 15°C.	- 0.800 - 0.900
Engler Viscosity at 20°C. (Vogel-Ossag)	- 1.1 - 2.0°E.
Pour Point	- Not above - 10°C.
Filterability	- 200 ml. in not over 60 secs. at -5°C.
Flash Point (P.M.)	- Not below 21°C.
Acid value	- Not above 0.4
Zinc corrosion	- Not more than 4.0 mg. loss in weight
Sulphur content	- Not above 1.0% by weight
Lower calorific value	- Not below 9,900 calories/kg.
Ignition value	- Not below 40 Cetane Number
Water content	- Not above 0.5% by weight
Ash content	- Not above 0.05% by weight
Carbon residue	- Not above 2.0% by weight, coke and hard asphalt (Hagemann-Hammerich), or not above 0.5% by weight in the Conradson test.
Boiling range	- At least 80% by vol. up to 360°C.
Miscibility	- All Diesel fuels must be mutually miscible.

F. Specification for Diesel Fuel for Delivery to the Navy (5.7.44)

	Diesel Fuels	Fuel Oil 40
Appearance	Clear, Ostwald colour 4-5 min.	
Mechanical impurities	Must be absent	
Density at 20°C.	0.84 - 0.87(1)	not above 0.88
" " 15°C.	0.839 - 0.877	" " 0.887
Viscosity at 20°C. (Vogel-Ossag)	1.2 to 2.6°E.	
Cloud point	Must flow and show no signs of separation at 0°C.	
Flash point PM	Not below 55°C.	Not below 40°C.
DVM	" " 70°C.	" " 55°C.
Acid value	Not over 1.5	
Sulphur content	Not over 1.0% by weight	
Lower calorific value	Not below 9,900 cals./kg.	
Ignition value (2)	Not below 35 cetane number	
Water content	Not over 0.5% by weight	
Ash content	Not over 0.5% by weight	
Carbon residue (Conradson) (2)	Not over 0.5% by weight.	
Mineral acids	Must be absent	
Boiling range (2)	Up to 350°C., at least 70% vol.	
Emulsification properties	Negative	-
Water colouration test	Water-white	-

(1) If in exceptional cases, the specification for the specific gravity cannot be complied with, then deliveries may be made which are within the limits for fuel oil 40, but only to Kiel or Gotenhafen.

(2) The following modifications are to be made to the specifications with immediate effect.

Cloud Point	Must flow without deposit at + 10°C., except for deliveries to Norway, where + 5°C. is specified.

Ignition value	Not below 32 Cetane Number
Carbon Residue (Conradson)	Not above 0.8% by weight
Boiling range	At least 60% by vol. up to 350 °C.

These relaxations have been caused by the present fuel position. Since they can lead to engine trouble, the previous specification will come into force again as soon as the fuel position improves.

5. ROAD TESTS ON ALCOHOL FUEL.

As a result of the interrogation of Herr Gubish and Dr. Charpentier with regard to road tests on motor fuels, it would appear that the only tests of which they had knowledge were carried out on alcohol fuel.

The purpose of the tests was to ascertain whether normally equipped automotive vehicles could run without trouble on fuels with an alcohol content of up to 60 per cent by weight, without any considerable structural changes. It was also desired to find out what the effect of such fuels would be on performance, consumption and starting behaviour. The tests were carried out using a Mercedes Benz Pkw, type 170V, (319133 engine) 1940 model which had covered a distance of about 15,000 Km. before the commencement of the trials :-

Operating details were as follows :-

Carburettor - Solex (with starting carburettor)
Main jet - 112.5 x 51
Light running jet - 0.50
Air filter - 23.5 mm. diameter.

No changes were made in the jet adjustment during the course of the experiments.

Ignition was adjusted, using benzine without alcohol, so that the commencement of ignition took place at the top dead centre. With alcohol fuel, the ignition point was 5° before top dead centre. (Measured at the distributor head).

The following fuels were used :-

1. Fuel benzine, consisting of mineral-oil basic benzine from the Trzebinia Refinery, + 0.035 vol. % T.E.L.

<u>Inspection data</u> :-

Specific gravity at 15 °C.	0.769
Octane No. (Motor method)	71.0
Reid Vapour Pressure at 40 °C.	0.21 atm.
Evaporation Residue (110 °C.)	5 mg./100 ml.
Boiling range, up to 75 °C.	3.0% by vol.
" " " " 100 °C.	10.0 "
" " " " 150 °C.	56.0 "
" " " " 200 °C.	97.0 "
F.B.P.	211 °C.

2. 40 per cent by weight of the above benzine + 60 per cent by weight of alcohol fuel :-

<u>Inspection data</u> :-

Specific gravity at 15 °C.	0.786
Octane No. (Motor method)	89
Reid vapour pressure at 40 °C.	0.24 atm.
Evaporation residue at 110 °C.	3 mg.
Boiling range up to 75 °C.	20% by vol.
" " " " 100 °C.	80% "
" " " " 150 °C.	84% "
F.B.P.	194 °C.

Results.

(a) Starting experiments.

The prolonged cold period in December 1944 to January 1945 made it possible to carry out extensive starting trials with these fuels at various temperatures. It was found that the benzine consisting of a leaded basic benzine from the Trzebinia Refinery, had a relatively poor starting behaviour. It was shown quite conclusively, that on leaving the vehicle over night in the open, good starting was only possible at temperatures of about +5 °C. and over. At temperatures between 0 °C. and circa. +5 °C., the starter had to be used for about 2 mins., with small intermediate pauses before the engine started. In every case the warming up time was very long. It took about 10 mins. from the time of cutting out the starting-carburettor before the vehicle could be put into fourth gear without difficulty. At temperatures even only a few degrees below 0 °C., it was no longer possible to start the engine after standing overnight, using ordinary methods. The engine only started when benzine with a better boiling range was injected into the inlet tube and the combustion chamber. At still lower temperatures this procedure had to be repeated several times before the engine continued to run without stopping. The warming-up time was correspondingly longer in such cases. All the starting trials described were carried out with the bonnet closed.

Those results illustrate the use of fuels with a small content of components boiling up to 100 °C.

The starting behaviour of the fuel consisting of 40 per cent by weight of Trzebinia benzine and 60 per cent by weight of alcohol fuel was considerably worse. It was only possible to start the engine by the normal method (starting carburettor) at temperatures down to +10 °C. At temperatures between +5° and +10 °C., the engine started with the usual methods only in a few cases and at still lower temperatures, benzine with a good boiling range had to be injected into the inlet tube and the cylinders. At temperatures below 0 °C. the injection had to be repeated several times before the engine continued running. Warming up time, however, was no longer than that found when using the neat benzine, except that it was necessary to take care that, during running the bonnet was left slightly open.

Further to investigate starting behaviour, a mixture consisting of 20 per cent by weight Trzebinia basic benzine + 20 per cent by weight of benzol + 60 per cent by weight of fuel alcohol and a mixture, of 20 per cent by weight of Trzebinia basic benzine + 40 per cent by weight of benzol + 40 per cent by weight of fuel alcohol, were used for comparison purposes.

The first of these two blends exhibited starting properties which were very little better than those of the initial Trzebinia benzine 40/60 fuel alcohol blend while the second blend behaved, in this respect, noticeably better than the fuel benzine.

The starting properties of the fuel benzine were also considerably surpassed by a 60/40 Trzebinia basic benzine/benzol blend (Gembo). Thus benzol can be used as a starting improver for basic benzines with poor 75-100 °C. points. Further it is seen from these trials, that the minimum vapour pressure of 0.2 atm. is not sufficient in itself to guarantee satisfactory starting properties. The important factor in this case is the volume distilling up to 100 °C. but the 75 °C. point apparently does not play an important part, as it is but little improved by the addition of benzol.

During the course of the trials, starting benzine in the form of basic benzine with a good boiling range was filled into the winter auxiliary tank. When the vehicle was stationary for any long period, the engine continued running with the benzine shut off until the carburettor was emptied. Starting was then carried out using the basic benzine in the auxiliary tank..

(b) Performance.

The following results refer exclusively to running with a well warmed engine. Since trouble-free operation could not be obtained with normal ignition adjustment using a blend of 40 per cent by weight of Trzebinia basic benzine and 60 per cent by weight of alcohol fuel, especially during acceleration and while climbing, the ignition was advanced for this fuel to 5° before top dead centre, measured at the distributor head.

The performance given by fuel benzine was normal. Maximum velocity on the level (Autobahn) was about 105 Km./hour but with the alcohol mixture, a maximum velocity of only 85-90 Km./hour could be reached under similar conditions. The loss of performance in climbing was still greater, amounting to about 25 per cent. Nevertheless, the engine ran quietly and without trouble in all gears with the alcohol blend. In spite of the performance loss, the vehicle could be run on the Autobahn in very hilly country over long distances at average speed of 75/80 Km./hour without any trouble. This is an average only little less than that obtained with fuel benzine. Considerable differences between the average velocities attained with fuel benzine and alcohol blend are shown however on ordinary roads in mountainous country where there are many turns. As the momentum of the vehicle can only be utilised to a small extent or not at all in overcoming difficult stretches in such country, it is obvious that gear changing must be more frequently employed with the alcohol blend than when using fuel benzine.

It may also be mentioned that no difficulties were encountered when the vehicle was travelling with a fully laden single-axle trailer (circa. 250/kg. useful load) over a normal route.

(c) Consumption.

According to load, average velocity, nature of country and road surface, the consumption of fuel benzine (leaded Trzebinia basic benzine) varied between 9.5-11.0 litres/100 km. The consumption, using the alcohol blend under similar conditions, was from 12.5 to 14.0 litres/ 100 kg. Thus the increase in consumption when using the alcohol blend, as compared with fuel benzine, amounts to 27 - 30 per cent. The difference in calorific value between fuel benzine and alcohol blend is about 17 per cent, the fuel benzine having a calorific value of 8,000 cals./ litre and the alcohol blend one of 6,860 cals./litre. It can thus be seen that the difference in calorific value of about 17 per cent corresponds to a change in consumption of 27/30 per cent and may doubtless be explained by the falling off in performance with the alcohol blend, which necessitates more frequent and longer running in the lower gears in all types of country. The possibility of offsetting the increased consumption due to the difference in calorific value by changing the carburettor adjustment has not yet been examined.

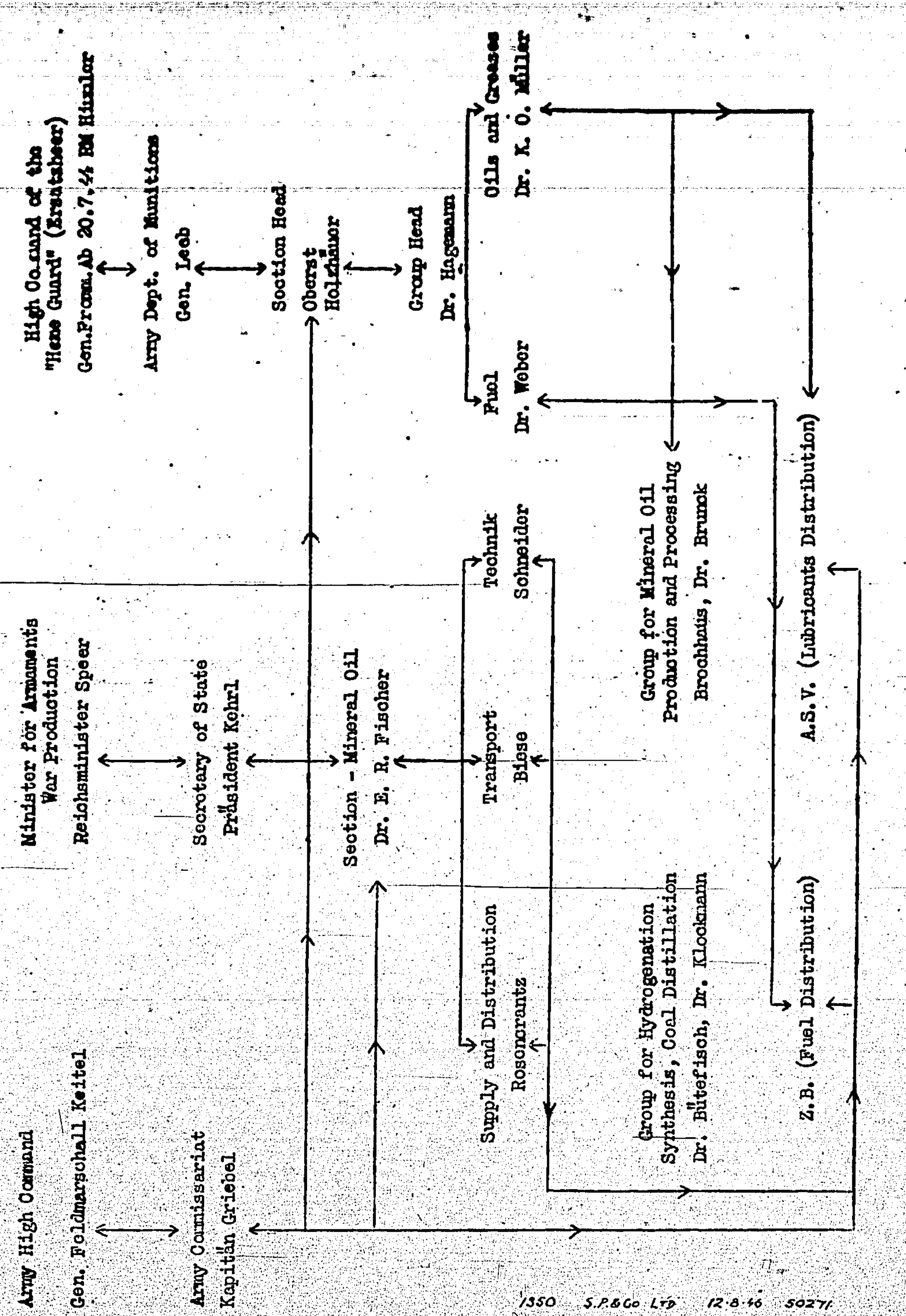
Conclusions.

From the trials described above, it can be concluded that a spirit blend consisting of 40 per cent by weight of benzine and 60 per cent by weight of alcohol fuel can be used in a Pkw Mercedes Benz, 170V, without changing the carburettor adjustment, when ignition is advanced by 5° and equipment is fitted to enable starting to be carried out with a benzine of good boiling range, free from alcohol. Allowance must be made for an increase in fuel consumption of 27/30 per cent.

6. DISTRIBUTION ORGANISATION.

During the discussion with Herr Gubish on the War time activities of the Olex organisation, information was obtained with respect to the manner in which control of oil production and refining was effected by the German government.

This information is detailed below in the form of an organisation chart which will be found to be self explanatory.



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FIAT FINAL REPORT 859

I. CONTINUOUS CHILLING AND COOLING OF CALCIUM CARBIDE
II. ACETYLENE GENERATION BY DRY TYPE GENERATORS
III. PURIFICATION AND DRYING OF ACETYLENE FOR CHEMICAL USE

Alexander, W. E.

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FIELD INFORMATION AGENCY, TECHNICAL

OFFICE OF MILITARY GOVERNMENT FOR GERMANY (US)

FIAT FINAL REPORT NO. 859 23 July 1946

I. CONTINUOUS CHILLING AND COOLING OF CALCIUM CARBIDE
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BY

W. E. ALEXANDER

TECHNICAL INDUSTRIAL INTELLIGENCE BRANCH

U.S. DEPARTMENT OF COMMERCE

THIS REPORT IS ISSUED WITH THE WARNING THAT IF THE SUBJECT MATTER SHOULD BE PROTECTED BY U.S. PATENTS OR PATENT APPLICATION, THIS PUBLICATION CANNOT BE HELD TO GIVE ANY PROTECTION AGAINST ACTION FOR INFRINGEMENT.

FIELD INFORMATION AGENCY, TECHNICAL

ABSTRACT

Part I. Reviews the details of the continuous chilling and cooling of calcium carbide in a single rotary unit as practiced at Ludwigshafen.
Part II. Reviews the details of two dry type acetylene generation systems used in Germany; a third type has previously been reported in detail (see Reference 2, Appendix 3).
Part III. Reviews various methods used for purification and drying of acetylene for chemical use.

PERSONNEL OF MISSION

Mr. W. E. Alexander, TIIB

Dr. E. N. Rosenquist, TIIB

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INTRODUCTION

Objective:

The purpose of these investigations was to obtain more details on methods of handling calcium carbide and generating and purifying acetylene than was given in previous reports on these subjects. (See Bibliography, Appendix 3 for list of previous reports).

Evaluation:

The method described for continuous chilling and cooling of calcium carbide is unique and new. The German dry type acetylene generators are quite different from those known in the U.S.A., especially the vertical type unit. Processes for acetylene purification include some new methods; the presence of divinyl sulfide in acetylene has not been previously reported in technical literature.

Guide to the Reader:

This report has been divided into three parts in order to distinguish the separate phases of chemical acetylene preparation from calcium carbide and yet keep these closely related subjects within a single report. Part I describes the method used for continuously chilling and cooling calcium carbide. Part II describes two designs of dry type acetylene generators used in Germany. Part III gives detailed descriptions of various purification and drying processes for acetylene as used at the various plants investigated. Appendix 4 includes diagrammatic flow sheets for the more important processes discussed as well as details of some of the principal equipment referred to in the text of the report.

1. CONTINUOUS CHILLING AND COOLING OF CALCIUM CARBIDE

At the Ludwigshafen plant of I.G. Farbenindustrie, calcium carbide is tapped continuously from a furnace and is fed directly to a rotary carbide chilling and cooling unit which discharges the product in lumps, most of which do not exceed 80 mm. in size. The ratio of material smaller than 6 mm. is 1-2%.

The rotary unit is 2 m. diam. x 42 m. long, including a copper feed section which is a cone frustum 2.5 m. long and having a large diameter of 2 m. and a smaller diameter of ca. 1.7 m. Molten carbide is fed into the smaller diameter opening, and the larger diameter opening is flange connected to the 2 m. diameter steel section. The 39.5 m. long steel section has a shell thickness of 8 mm. and the copper head section is 20 mm. thick. The unit is rotated at 40 r.p.m. by a 58 k.w. motor. The opening in the copper head section is ca. 1.7 m. diameter. The first half of the steel section is bare, but the last half is equipped with sectional lifting baffles 30 cm. long x 60 mm. high. These baffles are staggered in a spiral, one baffle being located 50 cm. around the periphery from the preceding one. They are also spaced 50 to 60 mm. apart along the longitudinal axis direction; this permits the fines to pass through with a minimum of relifting and consequent breakage. There are six baffles around the circumference through any one cross section, or six spirals of these short baffles along the last half of the rotary. The unit has a slope toward the discharge end of 1.5%.

At the discharge end of the rotary, the diameter is suddenly contracted to about 1 meter, to which is directly attached a smaller drum 1 m. diam. x ca. 5 m. long. Inside this shell are one or more rotary screens for preliminary sizing of the product. Special lifting flights in the end of the 2 m. diameter section transfer the cooled carbide to the smaller diameter rotary screen section. By proper arrangement of screens, the various desired sizes of product are discharged to separate chutes and are then conveyed to use points.

The unit is cooled by directing large volumes of cooling water onto the outer surfaces of the shell at four points around the circumference of the drum. Cooling water used is from two sources. About 70 to 80 cbm./hr. of cooling water from the cooling tower sump is first used for cooling the carbide furnace electrode jackets; it is then discharged into two overflow type troughs running the full length of the 2 m. diam. steel section of the cooler. The troughs are located above the drums and slightly off the vertical center plane of the drum. The water overflows from the troughs and falls onto the drum. On either side of the drum near the bottom is a perforated pipe line running the full length of the drum, including about 2 meters of the 2.5 m. long copper head section. From these pipes about 70-80 cbm./ hr. of water directly from the spray tower sump is sprayed directly

onto the drum; two short spray lines also discharge a portion of this water onto the top section of the copper head. All water is collected in a concrete sump running the full length of the unit and is pumped to the cooling towers for recycle. Steam condensate is used to as great an extent as possible as make-up water for this system in order to minimize salting out and consequent scaling of the rotary surface since considerable evaporation occurs, especially at the hotter feed end. The copper head section is provided with a light gauge metal guard collar to prevent water from splashing onto the incoming molten carbide stream or into the open feed end of the unit.

The size distribution of the discharge can be varied by changing the number of baffles in the cooler. The only analysis claimed to be available from their records was one made in 1943, at which time the unit contained about 10% more baffles than at present. This analysis is as follows:

Screen Opening in mm.	% Retained on Screen
30	13.6
25	7.0
20	21.0
15	28.7
10	17.9
6	10.2
Fines	1.6

When starting the cooler on carbide from a newly started furnace, the first (ca.) 15 tons of carbide discharged from the furnace is lower grade than the normal. This material sticks to the copper section and eventually builds up into a solid layer about two inches thick; this material will not break loose of its own accord. When the layer reaches this thickness, nitrogen is purged into the head section and the operators then beat on the outside of the shell with sledge hammers until the frozen layer is broken loose. The carbide feed is then resumed, the nitrogen purge is stopped, and the operation then proceeds without further difficulty from sticking.

Ferrosilicon contained in the carbide alloys with the copper head section and eventually results in a hole forming through the copper. When this occurs, water is admitted into the cylinder where it contacts the hot carbide; an explosion usually results. However, the explosions are said to be not serious enough to damage equipment or injure personnel. When this occurs, the unit is shut down and a new copper head section is installed (one spare new unit is kept on hand at all times). It is claimed that the unit shut-down time for changing the copper head section amounts to only two hours. On the average, a new copper cap is required for every 140,000 tons of carbide produced. The last unit replaced was in service from October, 1945 to May, 1946, during which time the continuous production rate for the unit was 4,000 to 5,000 tons per month.

Following is a list of drawing numbers with titles for a group of copies which were examined. It is claimed that the originals to these drawings were evacuated to Heidelberg by U.S. troops which originally entered the Ludwigshafen plant. These are recorded here in case these drawings are included in those which have now been microfilmed.

Nr.220/7 TB 0019	) Anordrung der Rohstoffbeschiekung Bunker, Schräg u.) Pendelschunen am Ca Ofen (Buna-Lu)
Nr.T 0709/33	Hochstrombahn
Nr.T 0310a	Ofenhaus - Querschnitt
Nr.T 0459	Ofenschaltwarte Leuchtschaltbild f. 2 Ofen

II. ACETYLENE GENERATION BY DRY TYPE GENERATORS

A. General

German plants employing dry type acetylene generators use one of two types of generating units. The most widely used type is a large vertical unit quite similar in form to the well known Herreshof oven commonly used in the United States for roasting of pyrites ores. The second type is a horizontal rotary unit inside of which is a rotary screen onto which the carbide is fed. These units are described in detail in their respective sections of this report.

The user of either type claims superior performance for that type as compared with the other. Unfortunately, no one man was encountered who is experienced in the operation of both. There is, however, one difference which appears to indicate better performance from the vertical "Herreshof oven" type. This unit discharges lime which is free of unreacted carbide and of acetylene. The horizontal rotary type discharges lime containing 1-2% of unreacted carbide which must be separated from the lime and recycled to the generator. This requires additional auxiliary equipment not required for the vertical type unit.

Another comparison which favors the vertical type generator is the operating cost. Rough comparisons of operating power per ton of carbide reacted reveals that the vertical type consumes on the order of 2-3 k.w. per ton of carbide whereas the horizontal type requires about 12-15 k.w. per ton of carbide reacted. This is quite understandable since the heavy shell and screen of the horizontal unit must be rotated while only the shaft and blades of the vertical unit are rotated. Both units must move the carbide-lime mixture, but the vertical unit has the aid of gravity in moving the material through the unit. The auxiliary equipment on the horizontal equipment accounts for about 45% of the total power load.

The horizontal unit can satisfactorily handle any reasonable size distribution of carbide feed, that is, material ranging from dust to 80 mm. particle size. However, for any size distribution, this unit appears to be characterized by its failure to react more than 99% of the carbide per pass. The ability of the vertical units to utilize various particle size carbide is controversial. At Burghausen the carbide fed to the unit is previously ground to a small maximum particle size, (not larger than 2 mm.) but at Knapsack it is claimed that the vertical unit satisfactorily reacts a carbide feed containing particles up to 10 cm. size. It is apparent that the vertical unit should give better performance when using a finely ground carbide feed.

Both types of unit operate at about the same acetylene outlet temperature. The horizontal unit has a gas outlet temperature of 100°C., and that for the vertical unit is 90-95°C.

Water is introduced into the horizontal unit through six sprays located equidistantly along the longitudinal axis. The vertical units have all the water introduced only onto the top tray of its 10 or 11 trays. When first used, the vertical units were designed for introduction of water on several of the plates lower in the unit with correspondingly less water going to the top tray. However, it was found that by putting water only on the top tray, the organic sulfides content of the generated gas was appreciably decreased. Such selective reaction zone cannot be chosen in the horizontal type unit.

Building space requirements are vastly different for the two types of generator. It is roughly estimated that the vertical requirements for equivalent capacity are about equal, but horizontal floor space for the horizontal units is about four times that required for the vertical unit.

It is difficult to compare plant cleanliness under existing conditions since war damage and availability of needed repair materials must be considered in such opinions. It was observed, however, that the Burghausen generation area was the cleanest; Ludwigshafen was somewhat dirtier and the odor of acetylene was quite pronounced; the Knapsack plant was much dirtier than either of the other plants inspected, but "housekeeping" in general seemed to be poor at this plant and equipment in poor condition.

B. Horizontal Rotary Generator

The Ludwigshafen acetylene generation plant consists of two horizontal rotary-type dry generation systems, each having a rated capacity of 2,000 cbm/hr. of acetylene; experience has shown that the units have a sustained production capacity of more nearly 1,800 cbm./hr. Following is a description of the units and the operation procedure used. A diagrammatic flow sheet of this system is presented as Figure I, Appendix 4, to which item number references in the following text apply.

Calcium carbide discharged from the continuous chill and cooler described in Part I of this report is conveyed by a housed inclined belt conveyor to the acetylene generation plant and is discharged into a steel bunker, Item 1, having a capacity of 90 tons. This bunker is continuously purged with a low flow rate of nitrogen, the excess passing out the top of the bunker into the inclined conveyor housing. The bunker has a double triangular bottom, each equipped with screw feeders for discharging carbide, as required, to either of the two generation units. The feeders discharge the carbide into screw conveyors which then deliver it to a bucket elevator, Item 2.

The bucket elevator is continuously purged by a small stream of nitrogen. The carbide is delivered to a small steel bunker, Item 3, having a capacity of two tons. This bunker

is equipped with level controls which operate the feeders on the bottom of the large bunker, Item 1. The contents of this bunker are periodically dropped to an identical bunker, Item 4, through a quick opening, air operated slide gate valve. When the lower bunker is empty, the valve operates to refill it from the upper bunker, after which the valve closes and the top bunker is refilled from the large storage bunker. Both small bunkers, Items 3 and 4, are continuously purged with nitrogen. The lower bunker is equipped with a bin feeder which continuously discharges the carbide into the feed screw of the generator, Item 5. A variable speed drive is used for regulation of the carbide feed rate.

The feed screw of the generator is a screw feeder about 50 cm. diameter x 5 meters long. It is connected to the rotating drum by a sealed slip ring. The feed is at the opposite end, and at about the mid-point is a large vertical nozzle connected to the bottom of the scrubbing tower, Item 8, through which the generated acetylene leaves the unit.

The carbide is discharged from the feed screw into the rotary drum generator, Item 6, inside the screen section. This unit is 2.5 m. diam. x 9 m. long and contains an inner screen cylinder 2 m. diam. x 9 m. long. The screen is an assembly of steel plates having open slots 2 mm. wide x 6 mm. long. In the annular space between the outer solid shell and the screen is a spiral ribbon welded to the outer shell; this moves the material which passes through the screen to the discharge end of the unit. Inside the unit, along its longitudinal axis, are six equidistantly spaced spray nozzles for introducing water onto the carbide. At each spray nozzle location there is also a thermocouple entering the unit through the shell and extending into the unit about one meter, or into the gas space. Water flow rates to each of the six spray nozzles are individually adjustable to provide desired temperature readings on each of the six thermocouples. Control is maintained to give approximately the following temperature readings in order from carbide feed end (acetylene discharge end) to the lime discharge end: 95°C., 100°C., 102°C., 105°C., 105-110°C. and 105-110°C. Total water requirement is said to be 1.3 kg. water per kg. of carbide fed to the unit. The drum is rotated at 1.5 r.p.m. by a 58 k.w. motor; the feed screw is driven by a 7.5 k.w. motor. The entire unit is insulated with 10 cm. of glass wool insulation encased in light gauge metal sections. As the carbide-water reaction proceeds, larger lumps of carbide disintergrate to smaller pieces and subsequently is converted to hydrated lime. The lime, along with some small particles of unreacted carbide, pass through the screen into the annular space and are conveyed to the discharge end hood. The unit operated under a pressure of about 200 mm. water column. The end hood is sealed against this pressure through a water seal vent tank in such a manner that if the pressure exceeds 200 mm. of water, the seal is blown and pressure is relieved to the atmosphere. The bottom of the discharge end hood is kept sealed by retaining some lime in the bottom of the hopper at all times. The lime is continuously discharged by two screw feeders, Item 7, to a bucket elevator, Item 13.

Acetylene leaves the generator in countercurrent flow to the incoming carbide through the screw feeder, Item 5. It passes to the bottom of a triple section scrubbing tower, Item 8, where it is freed of entrained lime particles before being sent to the purification plant. See Figure 2, Appendix 4, for details of this scrubber. This tower is 3 m. diam. x 15 m. high, and has three compartments each 5 meters high; the unit has a conic bottom (not included in the 15 meter height given above). The top compartment is packed with a 3 meter thick bed of iron Raschig rings; the two lower compartments are similarly packed with porcelain Raschig rings. Each section is equipped with spray nozzles for evenly distributing the scrubbing fluid onto the Raschig ring bed. The acetylene is scrubbed in the lower section with lime saturated water recycled as supernatant liquor from catch tanks Items 9 and 10 by a pump, Item 11. The 1,800 cbm./hr. of gas is scrubbed with lime water at a rate of 80 cbm./hr. The solution is combined with the effluent solutions leaving the two upper compartments and flows to the previously mentioned catch tanks, Items 9 and 10.

The gas passes upward into the middle compartment where it is again scrubbed, this time with 10 cbm./hr. of water containing 1.0 to 1.3 kg. of chlorine per cbm. of water. It is claimed that the only purpose of using chlorine water is for keeping the Raschig ring packing freed of lime which would otherwise deposit; it is claimed that this chlorine is not consumed in purification of the gas, i.e. in forming PCl_3, PCl_5, $SiCl_4$, etc. The effluent from this completely rubber covered section is combined with the effluent from the top section and is sent to the conic bottom section of the tower.

The gas is scrubbed in the top section with fresh water, the flow rate being controlled to give a gas outlet temperature of 18°C. The water requirement is 20-30 cbm./hr. The gas then passes to a small gas holder from which it is pumped by a centrifugal blower to the purification plant.

The lime discharged from the end of the rotary drum contains 1-2% unreacted carbide. If this lime is to be bagged and sold for either agricultural or construction (mortar lime) purposes, it is elevated by a bucket elevator, Item 13, and is discharged into a 2 m. diam. "Whizzer" type separator, Item 14, where a primary separation of lime and carbide is made. The lime fraction passes to a bucket elevator, Item 18. The heavier fraction containing the carbide is sent to a second "Whizzer" type separator, Item 15, which is identical to Item 14. Here the final separation is made between lime and carbide. The lime is discharged to a bucket elevator, Item 18, where it combines with that from Item 14, and is sent to a receiver, Item 19. The lime feeds from this receiver into a Fuller pumping system, Item 20, which transfers the lime to the bagging house. This lime contains about 5-8% free moisture.

The separated carbide fraction leaving Item 15 is elevated by a bucket elevator, Item 16, to a screw conveyor, Item 17. This conveyor at present delivers the carbide to a small wet type generator where the acetylene values are recovered and the lime sludge is wasted. However, this material can also be recycled to the dry generator carbide inlet, as is shown on Figure 1.

If it is not desired to recover the lime from the dry generator for sales, provision is made for discharging the lime directly from the first screw feeder on the end housing hopper (Item 7) to a sluiceway. High velocity water flowing through the sluiceway carries the lime to a large basin where some lime settles out, later to be dissolved by throughflowing water, and the basin overflow is discharged to the Rhine river.

The combined effluents from the three compartments of the gas scrubber, Item 8, overflow from the conic bottom section to catch tanks, Items 9 and 10, where undissolved lime settles to the bottom of tank, Item 10, from which it overflows. Supernatant solution is withdrawn from a side outlet of the second tank and is recirculated to the scrubbing tower's lower compartment by a circulation pump, Item 11. The lime solution containing suspended lime which overflows from tank, Item 10, is sent to a series of degassers, Item 12, where dissolved acetylene is driven off and is returned to the acetylene stream. The solution is discharged to the sluiceway previously mentioned.

C. Vertical Type Generator

The vertical "Herreshof oven" type acetylene generator is in use at both the Wacker Works in Burghausen and at A.G. für Stickstoffdünger near Knapsack. The two plants are essentially the same, so one description of the process and equipment is sufficient. There is one difference in the generators used at these two plants; the Burghausen generators discharge the acetylene from the top of the unit, whereas those at Knapsack have the gas discharge located below the "shelf" section.

The Burghausen plant appears to be more modern than that at Knapsack, and operations are cleaner and smoother. The description which follows will therefore be based upon data obtained at Burghausen. Details of the Knapsack generator are presented as Figure 3, Appendix 4.

Finely ground calcium carbide is fed from double bunkers similar to those used at Ludwigshafen (see Section B, Part II) by a screw feeder which discharges the carbide onto the top shelf of the generator at a rate of 10 tons per hour. About 11 cbm. of water is sprayed onto the top shelf through four spray nozzles. Acetylene leaves the top of the unit at 95°C. and at a pressure of ca. 95 cm. water column. The solid material is then scraped from the top plate and falls to the plate below, etc., until a series of ten plates are passed. The shaft to which the scrapers are attached is rotated at a speed of 12 r.p.m. by a 17 k.w. motor. The solid material drops from the bottom shelf into a deep hopper which is kept at a predetermined working level in order to furnish a suitable seal for the acetylene pressure. The lime is removed by a rotating table type feeder and is conveyed to use points. The discharged lime is tested periodically by immersing a grab sample in water and watching for gas generation. If any gas is evolved, the water feed rate is increased. If the lime is discharged with more than 7% free water, it is not free flowing and the water rate must be decreased accordingly. Figure 3, Appendix 4, shows the details of a unit similar to the one at Burghausen; no further description is required since dimensions, etc., are shown on the sketch.

The acetylene leaving the generator enters near the bottom of an empty steel tower 1.75 m. diam. x 10.6 m. high where it is contacted with water for removal of entrained lime. About 40 cbm/hr. of lime water is circulated through the tower and is discharged near the top of the unit by a solid cone spray nozzle. Around the periphery of the column near the top are four spray nozzles facing into the center of the column. 10-15 cbm./hr. of fresh water is introduced through these nozzles. The acetylene leaves the top of this tower at about

87°C. and passes to the next item. About 10-15 cbm./hr. of lime water overflows from the bottom of the tower and is sent either to a "lime pond" or to their dichloroethane plant for use in neutralization. The lime water temperature in the bottom of the tower is 90°C.

The acetylene leaves the top of the first scrubber at ca. 87°C. and at 70 cm. water column pressure, and enters near the bottom of a Raschig ring packed steel tower 1.4 m. diam. x 10.3 m. high. This tower serves to further clean the gas and to cool it. The gas is contacted with cooled recirculated water which is pumped over the packing at a rate of 40 cbm./hr. The water is at 85°C. in the bottom of the tower, but is cooled by a heat exchanger in the circulation line to 30°C. The cooling of the gas results in some condensation of water, which is dropped out in this tower; the excess water overflows from the bottom section of the tower and goes to the lime water vessel at the first tower and thence to the "lime-pond"

The acetylene leaving the top of the Raschig ring packed tower then flows through a "safety bottle" (water seal to prevent flash-back in case of ignition), after which the pressure is ca. 60 cm. water column. The flow rate of gas entering the gas holder is given as ca. 3,000 cbm./hr.

III. PURIFICATION AND DRYING OF ACETYLENE FOR CHEMICAL USE

A. General

Each plant included in this investigation employs a slightly different acetylene purification process; only one process for drying acetylene to very low moisture content was encountered. In all cases except at Höchst, the acetylene being purified is that coming from dry type generators; Höchst uses 40 year-old wet type generators.

The most elaborate purification system is used at Ludwigshafen, where the principal end uses are for reaction with formaldehyde to give butinediol, for Buna synthetic rubber by Reppe synthesis method, and for hydrogenation to ethylene for use as an intermediate chemical. At present a large ratio is used for vinyl compounds, since the Buna program is nil. The most simple process for acetylene purification investigated is that used at Höchst, where the end use is for vinyl acetate monomer production and for welding purposes. Acetylene generated at Knapsack is used principally for acetaldehyde production. The end uses of acetylene at Burghausen are acetaldehyde, vinyl acetate monomer, vinyl chloride monomer, vinyl alcohol monomers; that used for vinyl chloride monomer production receives purification within the vinyl chloride production facilities only.

Following are detailed descriptions of each of the processes investigated. The degree of purification is given for each process where sufficient quantitative data were made available.

B. Ludwigshafen Process

A diagrammatic flow sheet of the acetylene purification plant at Ludwigshafen is presented as Figure 4, Appendix 4. Item numbers referred to in the following text refer to the encircled numbers adjacent to equipment items shown on the flow sheet.

The Ludwigshafen plant has a capacity for purifying 600 to 700 tons of acetylene per month. Raw materials required per month are reported as follows: 500 kg. of 96% H_2SO_4, 12,000 kg. of chlorine, and 9,000 to 10,000 kg. of 100% NaOH.

Impure acetylene from the generation plant (see Section II, Part B) enters the purification plant at a rate of 1,800 cbm./hr. The gas first enters near the bottom of an acid-proof brick lined tower (Item 1) 1.8 m. diam x 11 m. high, packed with porcelain Raschig rings. Sulfuric acid solution is circulated over the packing at a rate of 25 cbm./hr. by a pump, Item 2. When fresh, the sulfuric acid solution contains 4% H_2SO_4, but as it becomes spent, the H_2SO_4 concentration increases to 7% and the acid becomes badly discolored and fouled with resinous reaction products. It is then sent to waste and replaced with fresh acid from a 10 cbm. acid-proof brick lined storage tank, Item 3.

The gas leaves the top of the first tower and enters the top of a second tower, Item 4, identical to Item 1 tower. Here the gas is concurrently scrubbed with fresh chlorine-water solution containing about 1.3 gms. Cl_2/liter which is fed to the top of the tower from a 20 cbm. acid-proof brick lined storage tank, Item 6, via a pump, Item 7. The rate is controlled in accordance with quantitive tests made on the gas leaving the next tower, Item 9, so that a minimum of free chlorine remains in the gas. The chlorine water which collects in the bottom of the tower is circulated by Item 5 pump to the top of the tower at a rate of 20-25 cbm./hr. The excess chlorine water, equivalent to the inflow rate of fresh chlorine water, overflows from the bottom section of this tower to a pump, Item 8, which sends it to the top of the next tower, Item 9. The gas leaving the bottom of the second tower, Item 4, next enters near the bottom of a third tower, also identical in construction to Item 1 tower. Here the gas is countercurrently scrubbed with the partially spent chlorine-water solution which leaves Item 4 tower. The solution is circulated over the tower at a rate of 20-25 cbm./hr. and the excess is overflowed to waste.

The gas leaving the top of the third tower enters near the bottom of another tower, Item 10, which is acid-proof brick lined steel and is 1.8 m. diam. x 11 m. high and is packed with Raschig rings. In this tower the gas is contacted by 7% NaOH solution which is circulated over the packing at a rate of 10-15 cbm./hr. by a circulation pump, Item 11. Fresh NaOH solution is continuously introduced into the top of the tower from a 20 cbm. storage tank, Item 12, by a feed pump, Item 13. The feed rate is at about 100-200 liters per hour of 7-10% NaOH solution. An equal volume of solution is continuously withdrawn from the tower and is sent to waste. The gas leaves the top of this tower and enters near the bottom of an entrainment separation column, Item 14, which is a steel tower, 1.8 m. diam. x 7 m. high and is packed with lump coke. Entrainment is sent to waste.

The gas then is sent to a series of activated carbon packed towers, Item 15, where it passes through two of the three towers in series; the third is on a reactivation cycle. These towers are 3 m. diam. x 3 m. high, and have a centered bed of 2 meters height of 3 to 6 mm. activated carbon (ca. 7,000 kg.) When the first tower of the "on stream" series begins to permit passage of impurities as tested qualitatively, it is removed from the stream. The second tower is then made the first tower of the series, and the newly reactivated tower becomes the second tower of the series. The tower removed from the gas stream is then put on a regeneration cycle. One tower will purify from 30,000 to 40,000 cbm. of gas before requiring reactivation. The gas is then sent to the various use points.

The activated carbon is regenerated by superheated steam at above 110°C. and at about atmospheric pressure. Steam is passed through the carbon bed until all four thermocouples located in a vertical plane in the carbon bed read 110°C. The steam is condensed and is passed through an acetylene stripper, Item 19, before being discharged to waste. The carbon is then dried by circulating acetylene through the bed by means of a blower, Item 16, at a rate of 400 cbm./hr. The gas is heated by a steam heated tubular heat exchanger, Item 17, to 110-120°C., and is then passed over the carbon bed. The gas then passes through a finned type cooler where condensate is removed and sent to the acetylene stripper, Item 19. A total of only about 150-200 liters of condensate is removed for the cycle. After all water has been removed (no further condensation from the gas), the heater is then by-passed and the carbon is cooled by continuing circulation of the gas alternately through the carbon bed and through the cooler. Circulation is continued until the four thermocouples in the carbon bed all read 40°C.

If it is desirable to have the acetylene gas absolutely free of chlorine, such as for use in acetone synthesis, the gas should be passed through a bed of zinc oxide following the activated carbon towers.

Some available analyses gave the following impurities contents of the acetylene at three points in the process. These figures are not necessarily from concurrent analyses at the three points, but rather are said to be somewhat representative of analyses made at these points.

Impurity	Impurity Content in Milligrams per Cubic Meter		
	In Feed Acetylene	Before Activated Carbon Towers	After Activated Carbon Towers
Sulfur	1,388	133	30
Phosphorus	372	29	8.5
Chlorine	-	400	83 x

x It is claimed that the final chlorine content should not exceed 15 mgms./cbm. and is normally 7-10 mgms./cbm. It is claimed that the figure shown in the tabulation above (83 mgms./cbm.) is one from an erroneous analysis.

C. Knapsack Process

The process at Knapsack is quite similar to that used at Ludwigshafen except that no activated carbon is used. There are insufficient differences to warrant any separate report on the Knapsack operations.

One interesting disclosure revealed during the interrogation of Dr. Lösch was his claimed discovery during the war of the presence of divinyl sulfide in acetylene generated from calcium carbide. Dr. Lösch claims that this compound is present to the extent of 400-500 milligrams per cubic meter of generated acetylene, and that the combination sulfuric acid-chlorine-water treatment completely removes this compound from the gas.

Following are some typical approximate impurity contents of acetylene before and after purification treatment as reported at Knapsack:

Impurity	Impurity Content in Milligrams per Cubic Meter	
	Before Purification	After Purification
Divinyl Sulfide	400 - 500	0.0
PH_3	500	30-40
H_2S	200	10-20
NH_3	300	0.0

D. Burghausen Process

The acetylene purified at Burghausen by the process described below is used only where the acetylene must not be very dry. Acetylene required to be moisture free (for vinyl chloride monomer production) is dried before purification (See Section F. for drying process details) and is purified within the vinyl chloride equipment by treatment with gaseous chlorine. The acetylene purified by the process described in the following text is used in the production of acetaldehyde, vinyl acetate and vinyl alcohols.

Chlorine and water are brought together in such ratios as to give the 15-20 cbm./hr. of water a chlorine content of 1.2 gms. per liter. This is accomplished in a stoneware tower 1 m. diam. x 6 m. high filled with 35 mm. porcelain Raschig rings. Water enters at the top and chlorine gas enters at the bottom of the tower. The effluent from this tower flows to a rubber lined steel tank, 3 m. diam. x 4 m. high which is equipped with a rubber covered paddle type agitator rotating at 20 r.p.m. It is claimed that the agitation increases the solubility of chlorine in the water. This solution is continuously pumped by a stoneware pump to a feed tank in the top of the purification building; excess solution overflows from the feed tank and is returned to the larger agitated tank.

Acetylene from the vertical dry type generators is metered to the purification plant from a gas holder at a rate of ca. 2,500 cbm./hr. and at a pressure of 550 mm. water column. This gas enters at the top of a rubber lined steel tower 1.8 m. diam. x 8 m. high. The tower is filled with 35 mm. Raschig rings. Also entering the top of the tower is a stream of fresh chlorine water from the feed tank previously described; the rate is 15-20 cbm./hr., as required. Solution collected in the bottom of the tower is circulated back to the top of the tower by a stoneware pump at a rate of 20 cbm./hr. Excess solution, equivalent to the rate of fresh chlorine-water input, overflows from the bottom of the tower and is sent to waste. Efforts were made to recover acetylene from this effluent solution, but were abandoned as being unsuccessful. This tower operates at 5-25°C., and at a pressure of ca. 450 mm. water column. The gas leaving the chlorine-water scrubber is periodically tested with wetted test papers containing $AgNO_3$, KI and lead acetate, as qualitative tests for PH3, excess Cl_2 and H_2S respectively. Adjustments to chlorine water feed rate are manually made accordingly by the operator.

The gas next enters near the bottom of a steel tower 1.8 m. diam x 8 m. high, packed with 35 mm. Raschig rings. Here the gas is countercurrently scrubbed with 300 liters per hour of 3% NaOH solution from a feed tank containing the previously prepared solution. Also introduced to the top of the tower is 20 cbm./hr. of solution circulated from the bottom of the tower by a circulation pump. The excess solution, equivalent to the inflow rate of fresh NaOH solution, overflows from the bottom of the tower and is wasted.

The gas then passes to a rubber lined entrainment separator ca. 2 m. diam. x 3 m. high where entrained liquids are dropped from the gas stream and sent to waste. The gas is then delivered to the various use points.

No tests other than the qualitative tests mentioned above are made on the gas; hence, no quantitative data were available.

E. Hochst Process

Acetylene is purified at the Höchst plant only for use in vinyl acetate monomer production and for welding purposes. The process and plant are very simple and appear to give an adequate degree of purification for the end uses intended.

Acetylene generated in a 40 year-old wet type

generation plant is pumped from a gas holder and is metered to the purification plant at a rate of ca. 350 cbm./hr. and enters the bottom of a rubber lined steel tower 1 m. diam. x 8 m. high, packed with Raschig rings. The gas is countercurrently scrubbed with a sulfuric acid solution containing sodium dichromate. The solution is circulated over the packing from a rubber lined catch tank by a pump at a rate of ca. 10 cbm./hr. This solution is in a closed circuit and is circulated until it becomes spent as measured by its oxidation power. It is solution which was transferred to this tower from the second tower of the series, which receives all the fresh solution. The second tower is identical to the first and the gas leaving the top of the first tower enters near the bottom of the second. The scrubbing solution is, when fresh, of the following composition: 720 kg. of 95% H_2SO_4, 62 kg. of $Na_2Cr_2O_7$, and 700 kg. of water. This solution is considered to be spent when the chromic acid concentration reaches 10-20 gms. per liter; it is then withdrawn from the first tower recirculation system and is sent to another department where it is electrolytically oxidized to restore the original chromic acid concentration. It is said that the acid becomes darkened, but does not accumulate any solid "sludges", and can be recycled indefinitely without being wasted. Apparently replacement of mechanical losses, etc., maintains a satisfactory balance on the collected impurities. It is also claimed that no concentration change occurs which would require either addition or evaporation of water.

The gas then passes to two brine cooled tubular heat exchangers for removal of moisture. The gas passes through these units in series, and is cooled to minus 12 to 14°C. Condensate is sent to waste.

The Höchst operation formerly next sent the gas to units containing a "Kieselguhr mass" made from 140 kg. kieselguhr, 66 kg. of 95 H_2SO_4 and 70 kg. of $Na_2Cr_2O_7$. This was for further purification and drying. However, this has recently been abandoned since the acid or dichromate values could not be recovered, and the gas is sent from the coolers directly to one of two solid NaOH packed towers. These towers are 1.5 m. diam. x 4.5 m. high and are constructed from steel. The gas is freed of any acidic constituents such as SO_2 and CO_2 and is further dried in this tower. The gas is then sent to the use points.

The purified gas is checked hourly by qualitative methods only. Papers saturated with silver nitrate and lead acetate are used to prove the absence of PH_3 and H_2S respectively.

metered to the purification plant from a gas holder at a rate of ca. 2,500 cbm./hr. and at a pressure of 550 mm. water column. This gas enters at the top of a rubber lined steel tower 1.8 m. diam. x 8 m. high. The tower is filled with 35 mm. Raschig rings. Also entering the top of the tower is a stream of fresh chlorine water from the feed tank previously described; the rate is 15-20 cbm./hr., as required. Solution collected in the bottom of the tower is circulated back to the top of the tower by a stoneware pump at a rate of 20 cbm./hr. Excess solution, equivalent to the rate of fresh chlorine-water input, overflows from the bottom of the tower and is sent to waste. Efforts were made to recover acetylene from this effluent solution, but were abandoned as being unsuccessful. This tower operates at 5-25°C., and at a pressure of ca. 450 mm. water column. The gas leaving the chlorine-water scrubber is periodically tested with wetted test papers containing $AgNO_3$, KI and lead acetate, as qualitative tests for PH3, excess Cl_2 and H_2S respectively. Adjustments to chlorine water feed rate are manually made accordingly by the operator.

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The purified gas is checked hourly by qualitative methods only. Papers saturated with silver nitrate and lead acetate are used to prove the absence of PH_3 and H_2S respectively.

Following are some analytical data on the gas before and after purification; these data do not represent analyses of samples taken concurrently:

Component	Before Purification	After Purification
Acetylene	99.5%	99.4%
CO_2	0.034%	0.07%
CO	0.03%	0.02%
PH_3	0.054%	0.0001%
H_2S	0.003%	0.001%
H_2	0.4%	0.5%

Moisture content of gas, on wet basis, is ca. 0.02%

F. Burghausen Drying Processes

1. Drying for Vinyl Acetate Monomer Manufacture

A very simple drying system is employed at Burghausen for acetylene used in the production of vinyl acetate monomer. There is probably some further purification in the second step of the process.

Acetylene previously purified by the chlorine-water process described in Section D above is brought to the vinyl acetate plant from a wet type gas holder at a rate of ca. 80 cbm./hr. The gas then passes through three steel tanks packed with calcium carbide, each tank holding 30 kg. of the 1 x 2 inch sized lumps. The gas may be directed through these drying tanks either in series or in parallel. The exit gas is said to contain 5 gms. of water per cubic meter.

The acetylene is then sent through three "kieselguhr mass" units in parallel. These units are packed with three trays each containing a 10 cm. deep bed of the kieselguhr mass, having a composition of 20 parts kieselguhr, 14 parts $Na_2Cr_2O_7$, 13 parts 95% H_2SO_4, and 20 parts water. Two of the units are about 1 m. x 1 m. x 2 m. high, and the third unit is about 2 m. x 2 m. x 2 m. high. The smaller units require ca 120 kgs. each of the mass, and the larger unit requires 360 kg. The life of the mass is about two weeks, after which it is wasted. The mass is considered to be "spent" when the gas leaving that unit discolors either silver nitrate or lead acetate solution saturated test paper. The gas is then sent on to the vinyl acetate facilities.

2. Drying for Vinyl Chloride Monomer Manufacture

The acetylene used for the manufacture of vinyl chloride monomer must be practically free of moisture because of the HCl present in the vinyl chloride synthesis. At Burghausen, the acetylene is dried previous to any purification and is delivered to the vinyl chloride plant containing less than 0.2 gms. of water per cubic meter of gas.

Following is a detailed description of the process and equipment employed at Burghausen. Item numbers refer to the encircled numbers adjacent to equipment items on the diagrammatic flow sheet presented as Figure 5, Appendix 4.

Acetylene from the generation plant gas holder is pumped to the purification plant by a Roots-Connersville type blower, Item 1, at a rate of 600 cbm./hr. and at a pressure of one atmosphere gauge. The gas enters near the bottom of a rubber lined steel tower, Item 2, 1 m. diam. x 8 m. high which is packed with Raschig rings. A circulation pump, Item 3, circulates 1 cbm./hr. of 20% sulfuric acid over the packing for drying of the gas and for neutralization of ammonia. The fresh solution contains 20% H_2SO_4 and is used until the free H_2SO_4 content reaches 1%, at which time it is discharged and replaced with a fresh batch of acid. If ammonium sulfate starts crystallizing from solution, water is added to keep the salt in solution. The spent solution is crystallized for recovery of the ammonium sulfate.

The gas then enters the bottom of a second tower, Item 4, which is a steel tower 1 m. diam. x 8 m. high and filled with Raschig rings. A circulation pump, Item 5, circulates 3 cbm./hr. of a solution containing 350 gms. NaOH per liter. It is claimed that the NaOH concentration remains about constant in this tower and that the solution is considered to be "spent" only when it becomes badly discolored and accumulates appreciable insoluble solids. The function is claimed to be for neutralization of acidic constituents such as H_2SO_4 from the first tower. It is said to be necessary to change the solution in this tower only about once every three months. The spent solution is wasted.

The gas next enters the bottom of a third tower, Item 6, which is a steel tower 1 m. diam. x 8 m. high and is packed with Raschig rings. Here the gas is contacted with two streams of drying solution which is a 5% trichloroethylene solution of methylpentandiol (2,4). A stream of the solution previously cooled to minus 5°C. by a brine cooled tubular heat exchanger, Item 9, is introduced into the top of the tower. A second stream of the solution cooled only by a water cooled heat exchanger, Item 13, is introduced into the column about 2.5 meters down from the top. This fraction of the solution is at a rate of 3 cbm./hr. and is at 20°C. The gas leaves the top of the tower at about minus 5°C., and passes to a brine cooled heat exchanger, Item 8, where it is further cooled to minus 18°C. Condensed trichlorethylene solution is returned to the column. The gas is then sent to the vinyl chloride plant; it contains 40-50 gms. per cubic meter of trichlorethylene and less than 0.2 gms. of water per cubic meter.

The trichlorethylene solution, containing the absorbed water is collected in the bottom of the tower, Item 6, and then passes out through a liquid level controlled valve to a gas separator, Item 10. This unit operates at atmospheric pressure and hence gas is released as it enters the tower. This unit is steel and is 50 cm. diam. x 3.3 m. high and has an expanded top section 1.2 m. diam. x 1.2 m. high. The liquid leaving the third column enters the top of this tower and flows out the bottom to the next unit. Gas leaving from the top of the unit is sent to the suction side of the feed blower, Item 1.

The liquid trichlorethylene solution leaving the bottom of the gas separator next enters a fractionation and stripping column, Item 11. This is a steel column having a 1 cbm. reboiler pot, a stripping section 1.2 m. diam x 6.5 m. high, and a fractionation section 0.8 m. diam x 4 m. high. The reboiler has two heating coils having a total surface of 5 sq.m. Liquid level in the reboiler is maintained at 0.5 cbm. On top of the fractionation section is a vertical tubular dephlegmator ca. 0.8 m. diam. x 1.5 m. high. Feed to the column is at the change of diameter. The unit is packed with Raschig rings. The reboiler contents are maintained at 91°C., and vapors leave the water cooled dephlegmator at ca. 20-30°C. The bottoms are the regenerated trichlorethylene solution of methylpentandiol, free of water. These are continuously withdrawn by a pump, Item 12, which sends ca. 4 cbm./hr. through a water cooled tubular heat exchanger, Item 13, and thence back to the absorption column, Item 6. Gas and vapors leaving the dephlegmator next pass to a brine cooled tubular heat exchanger, Item 15, where evaporated trichlorethylene and water mixture is condensed. Condensate is dropped from the gas stream in a separator; gas is returned to the suction side of the feed blower, Item 1. The condensate is a two phase mixture of water and trichlorethylene

which separates into two layers in the separator. The water phase is sent to waste, and the trichlorethylene phase is returned to the column, Item 11

APPENDIX I

LIST OF GERMAN PERSONNEL INTERVIEWED

Name	Position	Location
Dr. Norbert Platzer	Asst. to Acting Works Director	Burghausen
Dr. Schwarzbauer	Dept. Head	Burghausen
Dr. E. Schaeffer	Dept. Head	Burghausen
Dr. Konig	Dept. Head	Ludwigshafen
Ing. Heck	Dept. Head	Ludwigshafen
Dr. Josef Lösch	Dept. Head	Knapsack
Dr. Steffen	Dept. Head	Höchst

APPENDIX 2

LIST OF TARGETS VISITED

Dr. Alexander Wacker, Gesellschaft für Elektrochemische Industrie, G.m.b.h.	Burghausen, Kreis Alt Ötting
I.G. Farbenindustrie Aktiengesellschaft	Ludwigshafen am Rhein
A.G. für Stickstoffdünger Köln, Werke Knapsack	Knapsack, Kreis Köln
I.G. Farbenindustrie, Aktiengesellschaft, Frankfurt/Main, Höchst Werke.	Höchst/Main

APPENDIX 3

BIBLIOGRAPHY

Following is a list of reports used as references previous to the field work of this investigation.

1. "Preparation of Acetic Anhydride from Acetylene at Alexander Wacker Company, Burghausen", by L.H. Smith, S.P. Hoff, J.B. Guig, D.B. Wicker and J.J. Schilthuis; FIAT Final Report No. 12, p.1.

2. "Acetylene Generator Designs in Germany 1945", by F.E. King; FIAT Final Report No. 511.

3. "Chemical Installations in the Cologne Area", by Paul V. Seydel; CIOS Report No. 22 - XVIII - I, pp. 15 and 16.

4. "German Carbide, Cyanamide and Cyanide Industry", by W.G. McBurney, G.W. Sinclair and H.S. Sutherland; CIOS Report No. 22 - XXVII - 92, pp. 5, 11, 12 and 15.

5. "Miscellaneous Chemicals I.G. Farbenindustrie A.G. Ludwigshafen and Oppau", by I.G. Kern, R.L. Murray and R.W. Sudhoff; CIOS Report No. 22 - XXVII - 85, pp. 36, 37.

6. "I.G. Farbenindustrie Synthetic Rubber Plant Ludwigshafen", by Russel Hopkinson, W.C. Davey, P.D. Patterson, C.C. Mourad, E.T. Handley and Earl Glenn; CIOS Report No. 22 - XXII - 7, pp. 23, 24.

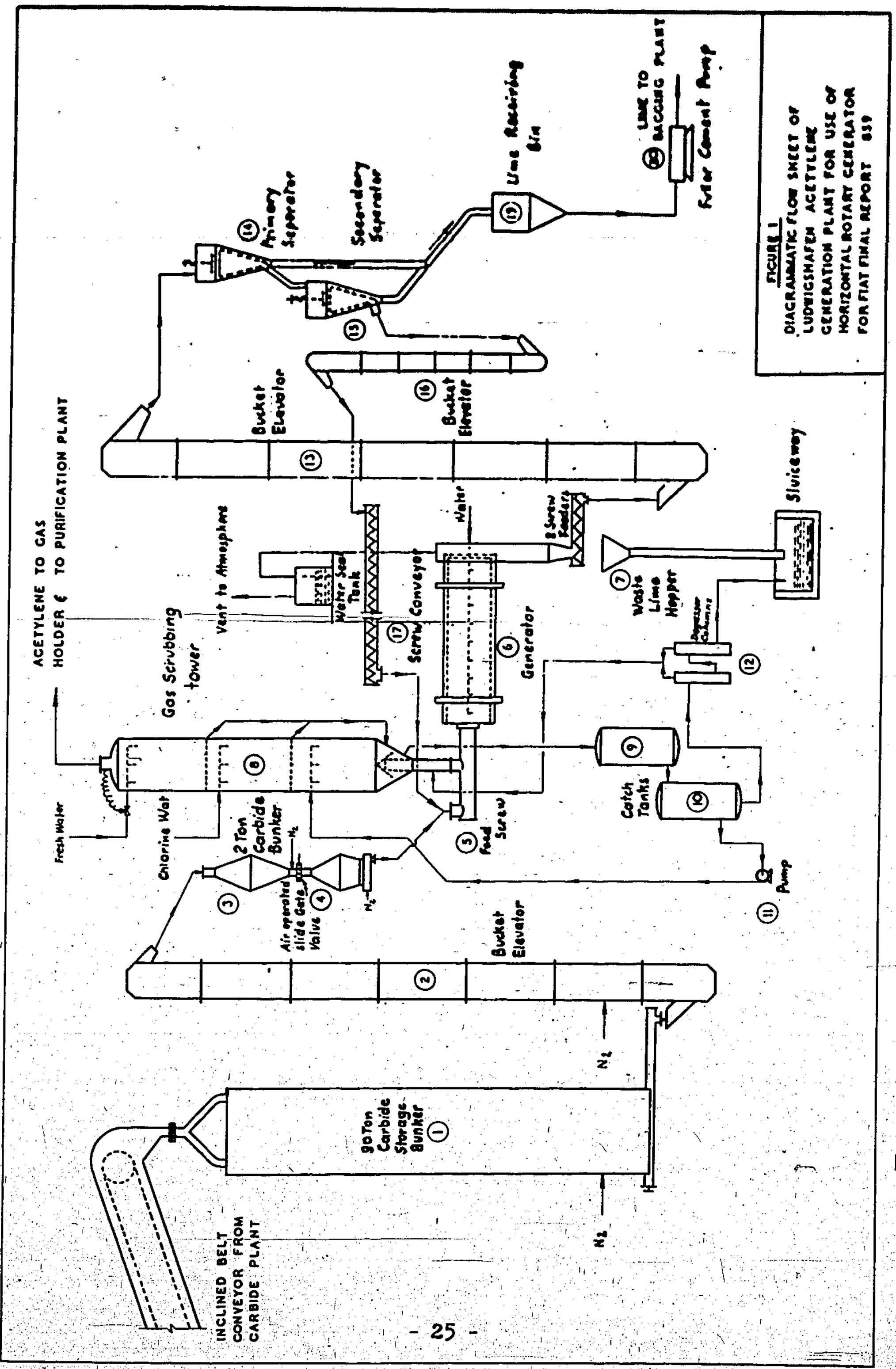
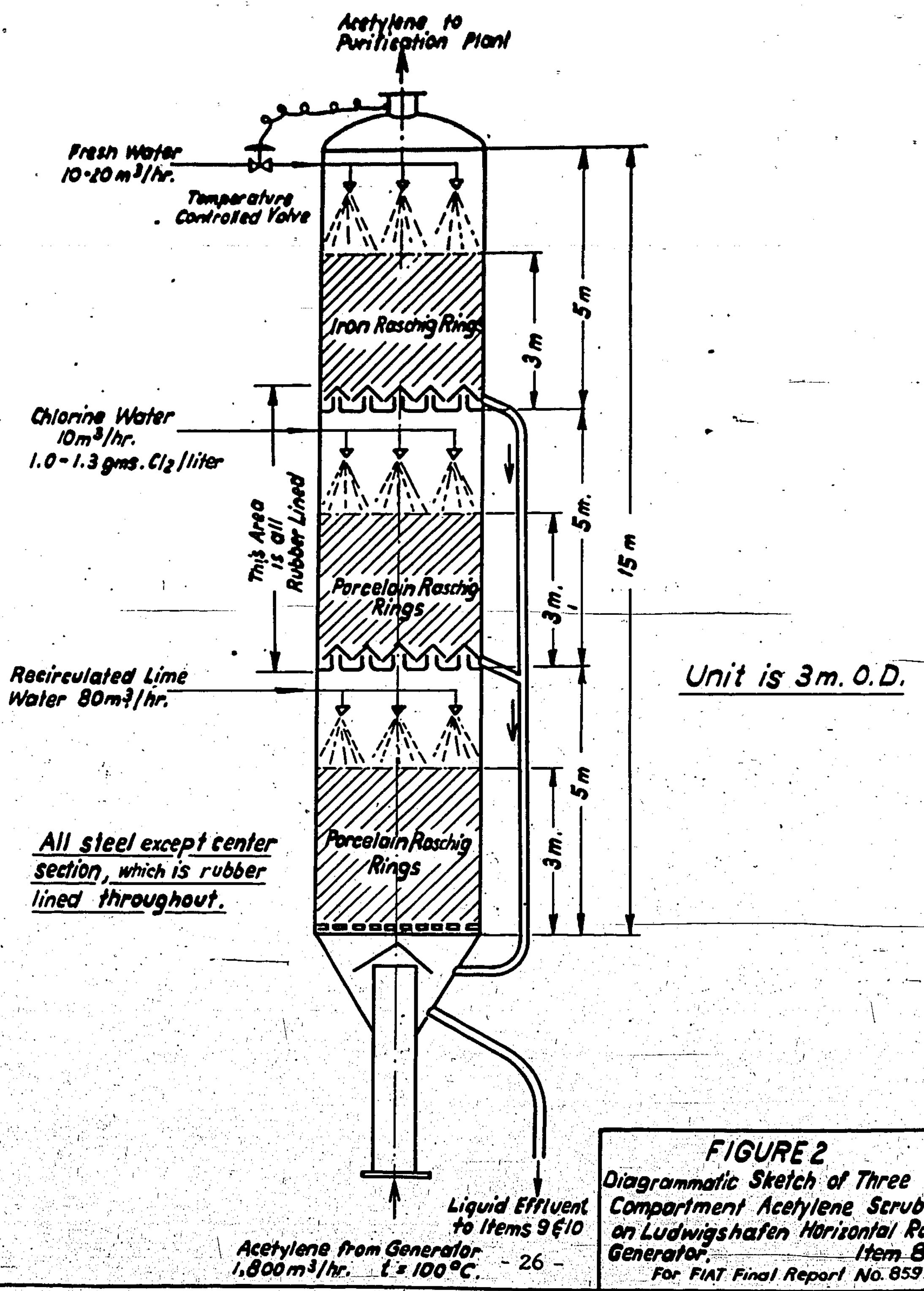


FIGURE 1
DIAGRAMMATIC FLOW SHEET OF LUDWIGSHAFEN ACETYLENE GENERATION PLANT FOR USE OF HORIZONTAL ROTARY GENERATOR FOR FIAT FINAL REPORT 839



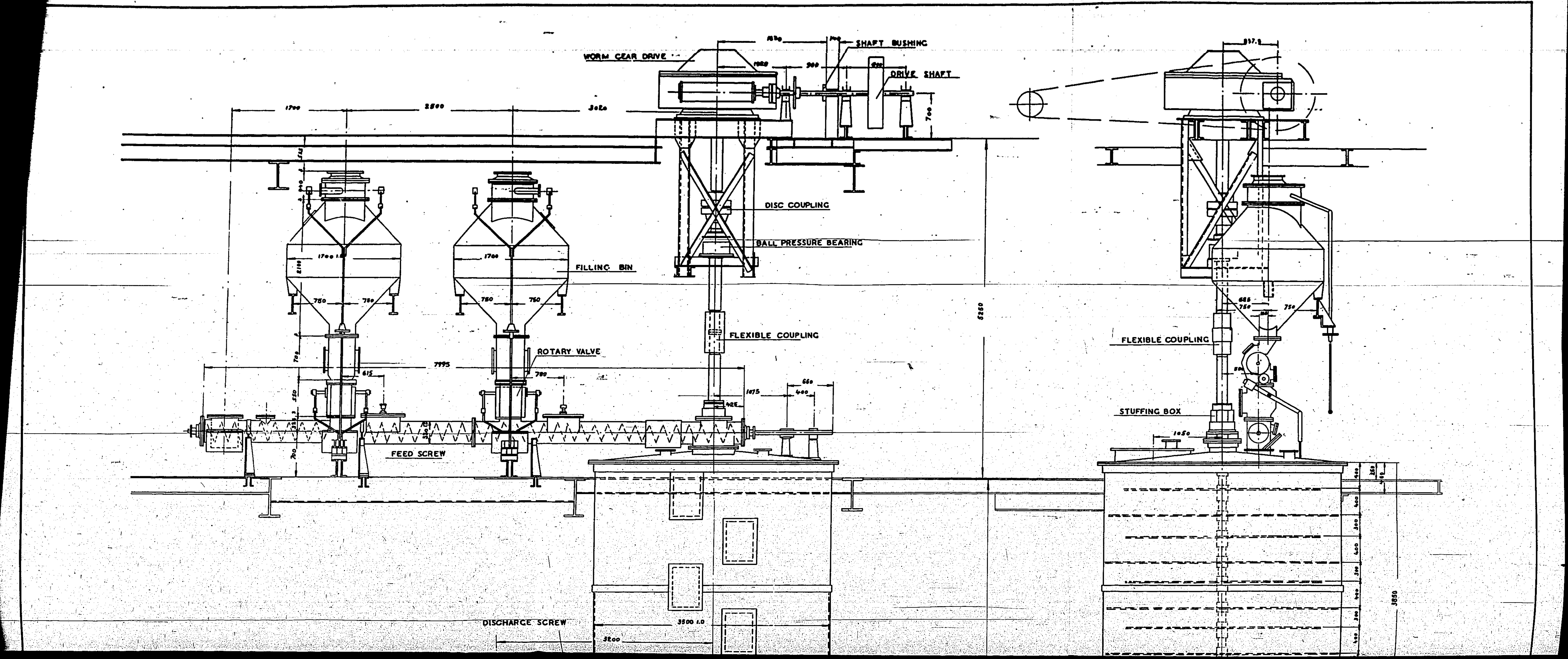
Unit is 3m. O.D.

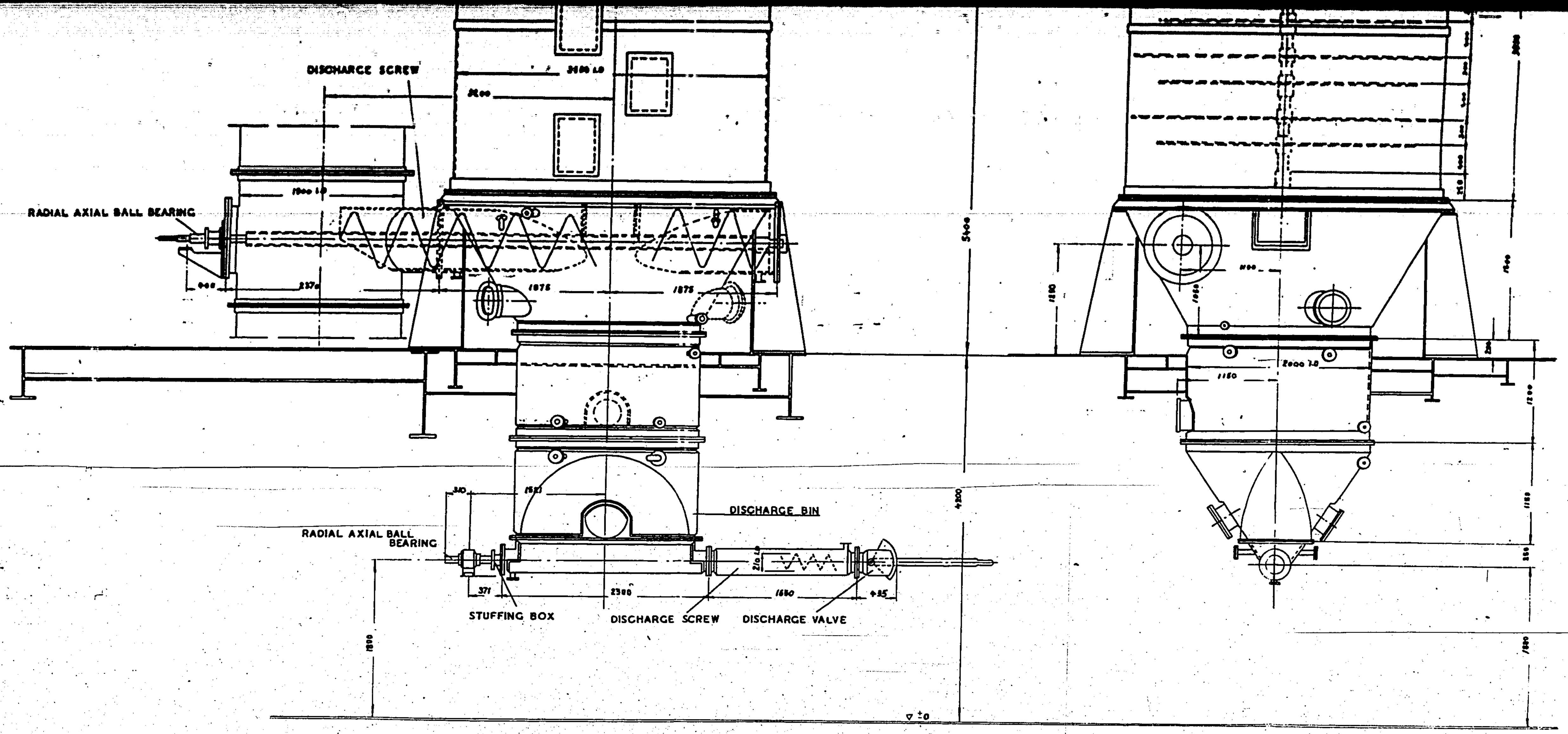
All steel except center section, which is rubber lined throughout.

FIGURE 2

Diagrammatic Sketch of Three Compartment Acetylene Scrubber on Ludwigshafen Horizontal Rotary Generator. Item 8.

For FIAT Final Report No. 859


WORM GEAR DRIVE
SHAFT BUSHING
DRIVE SHAFT
DISC COUPLING
BALL PRESSURE BEARING
FILLING BIN
FLEXIBLE COUPLING
ROTARY VALVE
FEED SCREW
STUFFING BOX
FLEXIBLE COUPLING
DISCHARGE SCREW
1700
2500
7995
5260
3850
1700
750
750
700
1050
3500 I.D.
3200



ALL DIMENSIONS IN MILLIMETERS

FIGURE 3

DETAIL DRAWING OF KNAPSACK VERTICAL DRY TYPE ACETYLENE GENERATOR

(COPIED FROM KNAPSACK DWG No. BI 225)

FOR FIAT FINAL REPORT No. 859

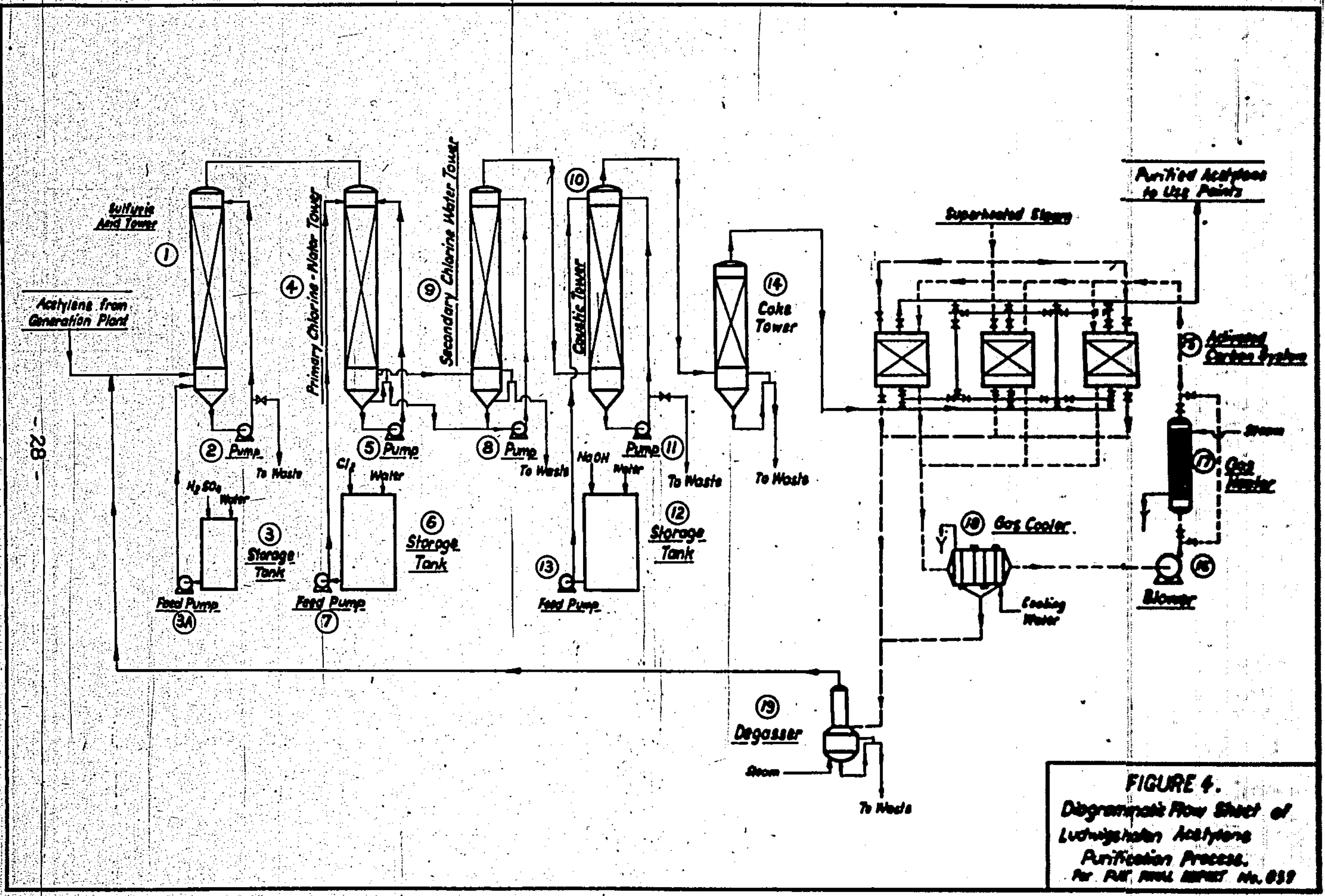


FIGURE 4.
Diagrammatic Flow Sheet of
Ludwigshafen Acetylene
Purification Process.
Per FIAT FINAL REPORT No. 839

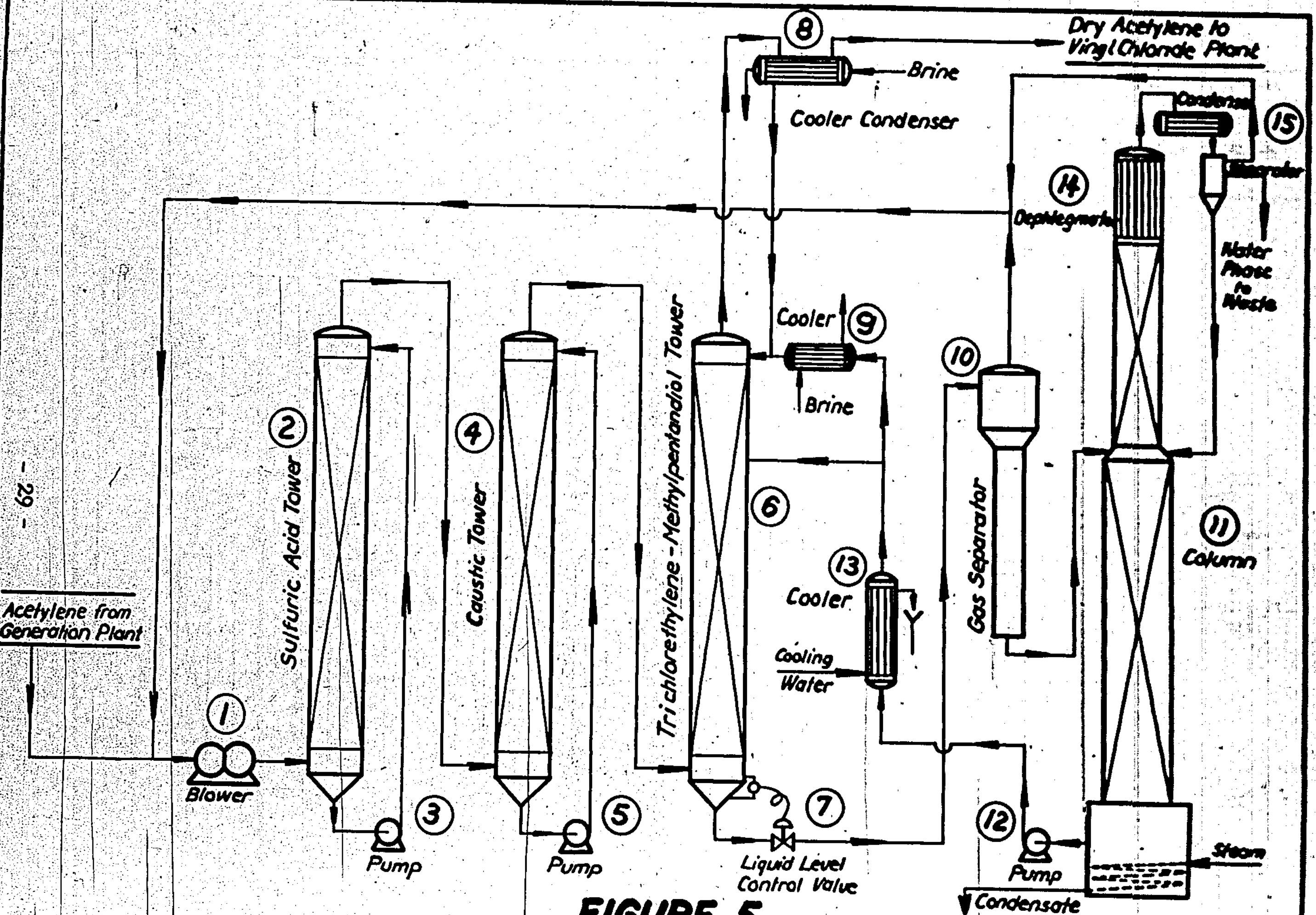


FIGURE 5

Diagramatic Flow Sheet of Burghausen Acetylene Drying Process.

For FIAT Final Report No. 859

Copy 1

FIAT FINAL REPORT NO. 905

DETERMINATION OF SUITABILITY OF PARAFFIN MIXTURES FOR CONVERSION TO FATTY ACIDS BY CATALYTIC OXIDATION

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FIAT FINAL REPORT NO. 905

SEPTEMBER 3, 1946

DETERMINATION OF SUITABILITY OF PARAFFIN MIXTURES FOR CONVERSION TO FATTY ACIDS BY CATALYTIC OXIDATION

BY

H. S. COITH and F. O. ROBITSCHEK
TECHNICAL INDUSTRIAL INTELLIGENCE DIVISION
U. S. DEPARTMENT OF COMMERCE

THIS REPORT IS ISSUED WITH THE WARNING THAT IF THE SUBJECT MATTER SHOULD BE PROTECTED BY U. S. PATENTS OR PATENT APPLICATION, THIS PUBLICATION CANNOT BE HELD TO GIVE ANY PROTECTION AGAINST ACTION FOR INFRINGEMENT.

FIELD INFORMATION AGENCY, TECHNICAL

ABSTRACT

Since the successful carrying out of the catalytic oxidation of paraffins to fatty acids is so dependent on the characteristics of the raw material, small scale tests for evaluating proposed raw materials are important. A description of two such tests is given. By way of showing how the tests compare with the actual production procedure, the latter is briefly reviewed.

TABLE OF CONTENTS

INTRODUCTION

Objective:

The original purpose of this study was to review the German practice for converting paraffins to fatty acids. Previous investigations had shown the method, as carried out on a production scale, to be too expensive to be of interest under conditions normally prevailing in the United States. It was hoped, however, that during the war years further laboratory research, which still had not been applied to plant operation, might have resulted in developments which could give some promise of making the process of interest. With this in mind, the present trip was made to the research laboratories of Henkel & Co. in Düsseldorf, where research for the Deutsche Fettsäurewerke is carried out.

Evaluation:

No new developments were uncovered which made the situation different than previous investigators had reported it. There were found however, two small-scale techniques for evaluating raw materials for paraffin oxidation which may prove of interest to future research workers carrying out further laboratory studies on the problem.

Guide to the Reader:

Several references to material on the production and uses of the synthetic fatty acids are given in Appendix No. 3.

BRIEF REVIEW OF THE PRODUCTION OF SYNTHETIC FATTY ACIDS THROUGH PARAFFIN OXIDATION

In its essentials, the process of oxidizing paraffins to fatty acids consists in blowing air through the heated paraffins, using potassium permanganate as a catalyst. The procedure is not to be confused with the Oxo process for fatty acids, which consists in treating unsaturated paraffins with a mixture of carbon monoxide and hydrogen to form an aldehyde, and subsequently oxidizing the latter, to a fatty acid.

The steps in the paraffin oxidation process are as follows:

(1) A paraffin mixture boiling between 320° C. and 450° C. (Fischer Tropsch Gatsch) is oxidized by blowing air through it in the presence of permanganate which serves as a catalyst. The oxidation is allowed to progress until about one third of the paraffin is converted to fatty acid.

(2) The resultant mixture of fatty acids and unreacted paraffins is given a counter current water-wash which removes water soluble fatty acids.

(3) The paraffin-fatty acid mixture from the water washing is saponified with sodium carbonate and sodium hydroxide.

(4) The resulting paraffin-soap mixture is autoclaved, during which some of the unreacted paraffin is separated out by gravity. This is returned to the original oxidation vessel.

(5) The paraffin-soap mixture from the above step is steam distilled to remove the remaining paraffin, which is also returned to the original oxidizing vessel.

(6) The residual soap from the above process is acidified with sulphuric acid.

(7) The liberated fatty acids are given a counter current water-wash.

(8) The washed fatty acids are fractionally distilled.

Three plants were actually engaged in converting paraffin to fatty acids by this method. They were the I. G. plants at Oppau and at Heydebreck, each with a capacity of 20,000 tons per year, and the Deutsche Fettsäurewerke at Witten with a capacity of 40,000 tons per year.

A tabulation of the results of a year's operation of the Deutsche Fettsäurewerke, at Witten is given below:

Yields obtained in the Witten Operation from working up 40,000 tons of Gatsch per year

	Amount from various steps expressed in tons.	Yield in tons expressed in 100% fatty acid or residual paraffin.	Percentage yield by weight based on 100 parts of gatsch *	Yield based on carbon content.
Crude fatty acid	31,000	31,000	78 —80	66
"Cooler oil" **	2,000	2,000	5 — 8	4
"Cooler water" **	11,000	3,500	(28) 6	5
Oxidate wash water	8,000	1,200	(20) 3	4
Acid waste water	100,000	3,000	(250) 7.5	6
Exhaust air	180 millions m³			10
Loss				5
				100
Fatty Acid Distilling				
First cut (C_4—C_9)	5,400		13.5	10.2
Main cut (C_{10}—C_{20})	22,200		55.5	48.0
Heavy cut (Over C_{20})	2,000		5.0	
Still residue	3,000		7.5	7.4
Total	32,600			

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* Percentages, of course, total over 100 because of weight gain resulting from oxidation.

** Separated by condensation from the exhaust air leaving the oxidator.

TESTING GATSCH FOR THE FATTY ACID PROCESS

Of prime importance to the success of paraffin oxidation is the quality of the hydrocarbon used as a starting material. The only tests which have been devised for evaluating such a raw material are actual small scale oxidation tests. An outline of the two finally adopted is given below.

I. One apparatus for such a test, capable of being used with 2 kgs. of raw material appears as Appendix 4. In this test the steps of the actual plant procedure, tabulated on page 6, are reproduced on a miniature scale. The procedure is as follows:

A. Four grams of potassium permanganate are dissolved in a small quantity of water and this solution is slowly added, while stirring, to a beaker containing 200 grams of gatsch at a temperature of 120° C. As soon as the water is evaporated off, the temperature is slowly raised to 150° C. The contents of the beaker are then added to 1,800 grams of gatsch previously introduced into the reaction chamber of the apparatus.

The temperature of the mass in the reaction chamber is then brought to 105—110° C. and air is passed through at a rate of 100 to 150 liters per hour (50 to 75 liters per kilogram). The blowing of air through the mass is continued until a saponification value of about 140 is reached, or until the rise in saponification value ceases. (The foregoing air blowing duplicates step (1) of the plant operation.)

The material condensed and collected from the two condensers separates into two layers and corresponds to the "cooler oil" and "cooler water" of the full scale process.

The material remaining in the oxidation vessel, designated as "crude oxidate", is water washed in a separatory funnel and yields "washed oxidate" and "oxidate wash water". This procedure corresponds to step (2) of the plant procedure as outlined.

The "washed oxidate" is then saponified with the calculated quantity of sodium hydroxide (37% NaOH solution), and the material is again allowed to separate into two layers. The upper or oily layer corresponds to the "mechanical gatsch" of the larger scale process and the lower or watery layer to the "crude soap". This procedure is analagous to steps (3) and (4) outlined on page 2.

The "crude soap" is hot extracted eight times with benzine, the latter being subsequently evaporated off and yielding a benzine extracted "return gatsch". This corresponds to step (5) on page 2. In plant practice the gatsch is separated by distillation instead of extraction and is known as "distillation gatsch".

The benzine washed "crude soap" is acidified with sulphuric acid to liberate the fatty acid (analagous to step 6) which is subseqently water-washed (step 7).

The washed fatty acids are then distilled (corresponding to step 8) at a pressure of 4 mm., the distillate being separated into "light cut" and "middle cut". The portion, which would in a large-scale operation be taken off as a "heavy cut" is allowed to remain in the distillation flask with the residue. An example of data from a run in this apparatus appears on the following sheet:—

Example of an Oxidation Test on Fischer-Tropsch Gatsch in the 2 Kilogram Apparatus.

Charge	**2 kg gatsch *(fresh, no return gatsch)* 4 gr potassium permanganate**		
Oxidation temperature	**108° C.**		
Oxidation time	**40 hours**		
Blowing air per hour	**140 liters (70 l per kg)**		
Cooler oil	**80 gr.**		
Cooler water	**300 gr.**		
Crude Oxidate	**1,825 gr.**	**Acid Val. 88**	**Sap. Val. 173**
Water washed crude Oxidate	**1,710 gr.**	**Acid. Val. 77**	**Sap. Val. 157**
Saponified with 37% NaOH	**515 gr.**		
Mechanically separated return gatsch	**590 gr.**		
Benzine extracted return gatsch	**295 gr.**		
Washed crude fatty acid.	**795 gr.**		
First cut distilled fatty acid at 4 mm. pressure boiling up to 140° C.	**81 gr.**		**Sap. Val. 430**
Main cut distilled fatty acid at 4 mm. pressure boiling up to 240° C.	**410 gr.**		**Sap. Val. 234**
High molecular weight fatty acids and still residue	**300 gr.**		

In the example given above, the oxidation was allowed to proceed farther than normal. Usually the reaction is stopped when the crude oxidate has a saponification value between 135 and 140 (15—25 hours). Because of this over-oxidation there is more "cooler water" than normally (300 against a usual figure of 230—250). Another consequence of this over oxidation is that the resulting high molecular weight acids, which in this case are essentially oxyacids, are proportionately increased over normal operation at the expense of the "main fraction".

B. It is, of course, impossible to set limit for experimental runs on this apparatus which will show whether or not a given raw material is suitable to use on a plant run because economic conditions may be such at one time as to justify running material which under other conditions would not be practicable. A good Fischer-Tropsch gatsch when run as outlined above will give from 400—500 grams of "maincut".

The data from an experiment on the 2 kg. scale is not fully comparable to that of large scale operations. For one thing, in the larger, less air is used. Also of particular importance is the fact that in a small mass, the separation by distillation of un-saponifiable material from the crude soap does not proceed very smoothly, therefore, the separation is carried out by means of a thorough extraction with benzine. Under such conditions the conversion of the oxyacids into unsaturated acids does not take place. For this reason the yield of fatty acids is somewhat smaller than on a large scale operation.

II. An even smaller experimental oxidation unit, capable of being used for a charge of only 100 g., is shown in Appendix 5. As in the apparatus just previously discussed, this small unit is charged with gatsch containing 0.2% of potassium permanganate in water solution. It is then brought to 108° C. by immersion in an oil bath and the oxidation is carried out by air blowing (7—10 liters per hour) until a saponification value of around 140 is reached. The oxidized mass is then water-washed and a saponification value is run on it.

This small scale apparatus is useful only for determining, in a qualitative manner, whether a given lot of gatsch is suitable for oxidation. It does not give quantitive results which can be correlated with the results of large scale operations.

APPENDIX 1
LIST OF GERMAN PERSONNEL INTERVIEWED

Name	**Position**	**Location**
Dr. Bruno Blaser	**Director of Research**	**Henkel & Co. Düsseldorf**
Dr. Wolfgang Guendel	**In charge of research on Paraffin Oxidation**	**Henkel & Co. Düsseldorf**

APPENDIX 2
GERMAN TARGET VISITED

Name	Location
Research Department of Henkel and Company	Düsseldorf

APPENDIX 3
BIBLIOGRAPHY

Copies of the reports listed below were transmitted to Washington, D. C. Inquiries should be addressed to:

Office of Publication Board,
U. S. Department of Commerce.
Washington 25, D. C.

1. Related Reports Published by Allied Intelligence Agencies:

CIOS Item No. 22, File No. XXVI—50-
Production of Synthetic Fatty Acids and Edible Fats
Deutsche Fettsäurewerke and Markische Seifenfabrik, WITTEN (Ruhr), Germany, by E.L. Baldeschieler, U. S., located at FIAT Records Branch, File No. TP. 200/P 941.

FIAT Final Report No. 407
Deutsche Fettsäurewerke, WITTEN (Ruhr),
by K. S. Markley and W. H. Goss,
located at Records Branch, File No. TX. 341/P 480.

BIOS Final Report No. 86
Same title as above by same authors, but with somewhat different type of information, located at FIAT Records Branch, File No. TJ. 1077/ P 27.
CIOS Evaluation Report No. 12
I. G.Production of Synthetic Fatty Acids,
by L. G. Bellamy and K. T. Millson, located at
FIAT Records Branch, File No. TP. 200/1083.

Form FB 4

Name of Target: Chemische Werke Huels G.m.b.b.H.

Type of Organization: Chemische Werke Huels G.m.b.H. Gas - Abteilung

Land: Westfalen

Kreis: Recklinghausen

City: Huels

Street:

Roll Number: CC-264

Team: CC

Date: ~~9. April 1947~~ 9 April 1947

AGL (G) 12-[illegible]

Chemische Werke Hüls G.m.b.H.   Marl Kr. Recklinghausen
Gas - Abteilung

Betriebsbeschreibung
der Lichtbogenanlage mit Rußabscheidung und -aufarbeitung

A) Lichtbogen

Zur Herstellung des Lichtbogenofengases stehen insgesamt 15
Ofengruppen mit je 2 Flammbogenöfen zur Verfügung. 14 Gruppen
sind mit Gleichrichtern von der Firma BBC und eine Gruppe mit
einer Gleichrichteranlage von Firma SSW ausgerüstet. Die Flam-
rohre und Elektroden der Öfen sind indirekt gekühlt. Die Zün-
dung erfolgt mittels einer preßluftgesteuerten Zündstange.
Beim Zünden wird bereits etwas Gas dem Ofen zugeführt, das
nach dem Zünden auf die volle Menge ausgesteuert wird
(=28oo Nm³ Mischgas/h). Das Mischgas bestand aus einem Gemisch
von entweder Hydrierabgasen oder Bentheimer Erdgas einerseits
und den Restkohlenwasserstoffen der H_2- und C_2H_4 - Apparate
der Gaszerlegung andererseits. Folgende Zusammensetzungen
von Mischgas seien hier aufgeführt:

a) mit Hy - Abgas (20.I.44)		b) mit Bentheimer Erdgas (12.III.45)	
CO_2	0,2 %	CO_2	0,3 %
C_2H_4	2,7 %	C_2H_4	1,5 %
Olefine	3,2 %	Olefine	1,4 %
O_2	0,2 %	O_2	0,3 %
CO	1,4 %	CO	3,0 %
H_2	10,9 %	H_2	2,5 %
CH_4+Hom.	74,5 %	CH_4+Hom.	80,2 %
N_2	6,9 %	N_2	10,8 %

Für die Umsetzung der oben erwähnten Gasmengen werden rund 7000 kW (wechselstromseitig gemessen) verbraucht. Dabei entstehen 4200 Nm³ Ofengas/h folgender Zusammensetzung:

a) mit Hy - Abgas (20.I.44)		b) mit Erdgas (12.III.45).	
CO_2	0,0 %	CO_2	0,0 %
C_2H_2.............	16,2 %	C_2H_2	13,3 %
Olefine	3,6 %	Olefine	0,9 % (-1,2 %)
O_2	0,2 %	O_2	0,2 %
CO	1,0 %	CO	2,9 %
H_2	50,5 %	H_2	46,0 %
CH_4+Hom.	25,1 %	CH_4+Hom.	27,8 %
N_2	3,4 %	N_2	8,9 %

Außerdem enthält das Ofengas noch HCN:	a)	1-3 gr/m^3 Gas
	b)	2-5 gr/m^3 Gas
ferner Naphtalin:	a)	1-3 gr/m^3 Gas,
	b)	0,12-2,0 gr/m^3 Gas
Benzol:	a)	1-6 gr/m^3 Gas
	b)	1,5-8,8 gr/m^3 Gas (spektrographisch)
Diacetylen:		15-30 gr/m^3 Gas (spektrographisch)
geringe Mengen Schwefelwasserstoff und Ruß	a)	20-25 gr/m^3 Gas
	b)	11 gr/m^3 Gas.

Zum Einfrieren des Gleichgewichtes werden am unteren Ende des Flammrohres ca. 4 m^3 Trinkwasser/h eingespritzt. Die Flammrohre sind 1000 mm lang und haben eine lichte Weite von 95 mm bei einer Wandstärke von 9 mm. Das zur Herstellung der Flammrohre verwendete Material ist Eisen. Die Elektroden sind aus Kupfer.

Sicherheitsmaßnahmen: Mechanische Verriegelung der Türen zu den Ofenschutzkäfigen, automatische Abschaltung bei Störungen durch Relais, insbesondere Schnellauslösung der Leistungsschalter, zentrale Abschaltung der gesamten Anlage vom Kraftwerk und vom Betrieb bei Kühlwasserstörungen.

Maßnahmen zur Verhütung von Bombenschäden: Splitterschutz durch trockengemauerte Ziegelsteine zwischen je 2 Schaltpulten und 2 Gleichrichtern. Mauern etwa 2 m hoch. Längs der Außenwand des Gebäudes ist zum Schutz der Hochspannungstrafos eine Splitterschutzmauer, ebenfalls eine solche unter den Ofenbühnen zum Schutz der Hilfstrafos hochgezogen. Das D ach der Trafoboxen ist durch Auflage einer Ziegelsteinschicht verstärkt. Mittelteil des Nordteils des Gebäudes aus Eisenbeton zum Schutz der Hilfstrafos und der zentralen S teueranlagen.

In der Halle sind verschiedene 2-Mann-Bunker für die Notbelegschaft aufgestellt.

B) <u>Rußabscheidung</u>

Das die Lichtbogenöfen verlassende, durch die direkte Wassereinspritzung auf etwa 150° C abgekühlte Ofengas passiert zwei hintereinander geschaltete Zyklonpaare (obere Durchmesser ca. 2 m) in denen etwa 60 - 70 % des im Gase enthaltenen Rußes abgeschieden worden. Anschließend geht das Gas durch eine Wasserwäsche, wo es auf 35°-40° C abgekühlt wird. Diese Wasserwäsche besteht für jede Gruppe aus einem Einspritzkühler von 1400 mm Durchmesser und 8,5 Länge. Das Wasser wird in 5 Etagen à 4 Düsen eingespritzt. Dabei werden etwa 10 - 20 % Ruß, bezogen auf den Anfangsgehalt, abgeschieden. Zur Feinreinigung von Ruß wird das Gas anschließend durch Schlauchfilter gebracht, die aus 8 Zylindern mit je 15 Schläuchen bestehen. Ein Zylinder hat einen Durchmesser von 1100 mm und eine Höhe von 5315 mm. Ein Schlauch hat einen Durchmesser von 200 mm und eine Länge von 3500. In den Schlauchfilteranlagen wird der Rest des noch im Gase verbliebenen Rußes abgeschieden. Die Schlauchfilter sind indirekt beheizt um Kondensation zu vermeiden. Die Lebensdauer der aus Kunstseide hergestellten Filterschläuche beträgt etwa 500 Stunden.

Besondere Luftschutzmaßnahmen konnten wegen der Anordnung der Geräte nicht getroffen werden.

c) Rußaufarbeitung

Die Gewinnung des Rußes wird in zwei Betrieben durchgeführt. Die anfallenden Produkte tragen die Bezeichnung T-Ruß und N-Ruß.

1) Trockenrußaufarbeitung (T-Ruß)

Der in den Zyklonen der Rußabscheidung hinter den Lichtbögen abgeschiedene Ruß wird mittels Transportschnecken und Becherwerken auf Sammelbunker gefördert und von da pneumatisch über einen Schwerkraftsichter den Hauptbunkern der Aufarbeitung zugeführt. Es stehen zwei Förderleitungen á 300 mm Durchmesser mit je einem Windsichter (h = ca. 10 m u. ø = 400 mm und 600 mm) zur Verfügung. Die Aufarbeitung erfolgt in 26 Granuliertrommeln. Diese bestehen aus feststehenden Gehäusen von 4,4 m Länge und 0,8 m Durchmesser, in denen je eine Welle mit wassergekühlten Schlägern (4 Reihen á 20 Schläger von 300 mm Länge) mit Drehzahl von ca. 830/Min. umläuft.

Der Ruß wird hierbei von einem Schüttgewicht von 50 gr/l auf ca. 150 gr/l verdichtet. Über Austragsschnecken wird der Ruß nach Wägen in Papiertüten verpackt, der in dem Schwerkraftsichter anfallende Grit (6 - 10% des Gesamtanfalls) wird pneumatisch zur Kesselfeuerung abtransportiert.

Die anfallende Menge an verkaufsfähigem Ruß beträgt bei Hy-Gas und 10 Gruppen ca. 400 - 450 moto, bei Erdgas und 10 Gruppen ca. 200 moto.

2) Naßrußaufarbeitung (N-Ruß)

Bei der Beschreibung der Rußabscheidung wurden der hinter den Zyklonen geschaltete Gaskühler und die Schlauchfilter erwähnt. Aus letzteren wird der abgeklopfte Ruß mittels einer Wasserspülung mit dem aus dem Gaskühler abfließenden Schlamm vereinigt und den Klärbecken zugeführt. Es sind insgesamt drei Klärbecken vorhanden (d = 15 m; h = 2,5 m; v ca. 450 m³). Der abgeschiedene Ruß wird in Schlammform von den Klärbecken über Schurren den Schlammpumpen zugeführt, die diesen auf Saugzellenfilter drücken. Von diesen stehen insgesamt 5 zur Verfügung (d = 1500 mm; l = 1000 mm; Drehzahl 0,333/Min.)

Hinter den Filtern sind je zwei mit Niederdruckdampf beheizte Tellertrockner, in denen der Ruß 9 bezw. 11 Etagen auf ca. 1 % Wassergehalt getrocknet wird. Anschließend wird der Ruß über Absackwagen in Papiertüten verpackt. Sein Schüttgewicht beträgt etwa 130 gr/l. Die anfallende Menge an verkaufsfähigem N-Ruß beträgt bei 10 Gruppen und Hy-Gas etwa 200 moto, bei Erdgas etwa 100 - 120 moto. Der Unterschied zwischen beiden Typen liegt in dem Gehalten an extrahierbaren organischen Substanzen. Es enthält:

	Hy - Gas	Erdgas
T - Ruß	3 - 5 %	2 - 4 %
N - Ruß	14 - 18 %	9 - 11 %

Bei Hygas liegt das Produktionsverhältnis T : N ca. 70% : 30%, bei Erdgas T : N ca. 60% : 40%.

Sicherheitsmaßnahmen: Zwischenlagerung des heißen, getrockneten N-Rußes zwecks eingehender Überwachung gegen Selbstentzündung.

Luftschutz: Die Apparate können wegen technischer Schwierigkeiten nicht besonders geschützt werden.

4

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Betriebsbeschreibung
der Niederdruckgasreinigung

In diesem an die Rußabscheidung angeschlossenen Betrieb wird das
Gas von höher siedenden Verbindungen der aliphatischen und aromati-
schen Reihe durch Ölwäsche, ferner von Blausäure durch eine Wasser-
wäsche und von H_2S durch eine Lux-Masse-Anlage befreit.

Das Gas wird nach der Rußabscheidung in zwei Einspritzkühlern von
je 3 m³ Durchmesser und etwa 10 m Höhe, durch Frischwasser möglichst
tief abgekühlt (20 - 25° C) und dann in nach dem System Walter
Feld arbeitenden rotierenden Waschern mit einem aliphatischen
Waschöl (Siedebereich 200° - 300° C) bei Normaldruck und Außen-
temperatur gereinigt. Es stehen vier Feldwascher à 20,000 m³
Gas Stundenleistung zur Verfügung (d = 3500 mm, 11 Stufen,
14 200 mm = h). Die Ölaufgabe erfolgt in der Weise, daß das Frisch-
öl (ca. 1 m³/h und Wascher) der obersten Stufe und Kreislauföl
(ca. 10 m³) der 8. Stufe zugeführt werden. Das den Wascher verlassen-
de Öl wird in der Weise regeneriert, daß es bei 600 mm Vak. ausge-
gast und 30 % des Gesamtumlaufs (ca. 6 m³ - 8 m³/h) einer Wasser-
dampfbehandlung im sog. Schnattertopf unterworfen werden, von
denen anschließend ein Teil destilliert wird. Zur Ausgasung stehen
3 mit Raschigringen gefüllte vierschüssige Türme von 800 mm Durch-
messer und 31 m Höhe zur Verfügung. 3 Schüsse der Türme sind ge-
füllt, der oberste ist leer. Zur Ausgasung des gesamten Öls werden
zwei Türme gefahren und jedem Turm 100 - 200 m³ Blasegas/h zuge-
führt. Das Vakuum wird mittels Elmopumpen erzeugt und das die
Türme verlassende Gas zur Kesselfeuerung verwendet.

Die Wasserdampfbehandlung erfolgt in einer dampfbeheizten Blase
in die direkter Dampf von unten in das Öl eingeführt wird (d =
1900 mm, h = 5320 mm). Das die Blase verlassende Öl wird durch
Mischen mit Wasser direkt gekühlt (ca. 50° C), von ausgefallenem
Polymerisat abfiltriert, das Wasser vom Öl in einem Trenngefäß
geschieden und das Öl z.T. der Destillation (3 - 5 m³/h) und zum
restlichen Teil in den Kreislauf wieder zurückgeführt. /regeneriert.
In der Destillation werden die oben erwähnten Ölmengen vollkommen /
Es stehen vier mit 200 ata Dampf beheizte Blasen zur Verfügung.
Die Leistung jeder Blase beträgt etwa 1 - 1,2 m³ Reinöl/h. Wenn mit
aromatischem Öl gefahren wird, fällt die Wasserdampfbehandlung weg
und die Regenerierung erfolgt nur durch Ausgasen und Destillation.

Nachdem das Gas die Feldwascher passiert hat, wird es mit drei
zur Verfügung stehenden Turbogebläsen durch die Wasserwäsche und
Schwefelreinigung gedrückt. Für die Wasserwäsche stehen drei
Raschigtürme zur Verfügung, zwei mit Durchm. von 3500 mm und einer
mit 4000 mm. Die Höhen betragen ca. 16 m. Die Raschigringe liegen
in zwei Schüssen, die Gesamtfüllräume betragen 120 m³ und 160 m³.
Das Waschwasser wird nach Ablauf von den Türmen in der Rußabschei-
dung zum Berieseln der hinter den Zyklonen vorhandenen Gaskühler
weitergeleitet, fließt von da zu den Klärbecken, von dort zu Vakuum-
ausgasern, anschließend über ein Rückkühlwerk und kehrt, den Kreis-
lauf schließend, wieder zu den Wasserwaschern zurück. (S.Schema!)
Zur Ausgasung des Wassers stehen zwei Türme mit d = 3,5 m und
h = 31 m zur Verfügung, die in je vier Schüsse mit einem Gesamt-

Füllraum von 290 m³/Turm gefüllt sind. (Der dritte Ausgaser ist
in Montage) Das Vakuum wird mittels Elmopumpen (3 Stück) erzeugt,
von denen je eine 3000 eff. m³ bei einem absoluten Druck von ca.
160 mm Hg ansaugt. Beim Betrieb von 10 Gruppen werden 20 - 30 to's
Dampf (direkt) in die Ausgaser unten eingeblasen. Die abziehenden
Brüden werden in drei hintereinandergeschalteten Kondensatoren
niedergeschlagen und das Restgas von den Elmos angesaugt. Es ent-
hält etwa 10 % - 15 % Vol. Blausäure. Die Gewinnung der Blausäure
ist noch im Versuchsstadium. Sie wurde zuletzt so betrieben, daß
die Elmopumpe gewissermaßen als Wascher für die blausäurehaltigen
Brüden diente und von ihrem Sperrwasser (3 - 5 m³/h/Elmo) etwa
1 m³ auf Blausäure aufdestilliert wurde. Das Elmowasser enthielt
etwa 0,5 - 1,5 % HCN.

Nach Passieren der Wasserwäsche geht das Gas durch die Schwefel-
reinigungsanlage. Diese besteht aus 6 Behältern mit Durchmessern von
6 m und Höhen von 7 m. 12 Horden sind mit Luxmasse gefüllt à ca.
250 mm Schütthöhe. Der Gasweg wird von der Anlage geteilt, das Gas
passiert dann je zwei hintereinandergeschaltete Reiniger. Anschlie-
ßend wird es wieder vereinigt und der Gaswäsche zugeführt. (Gaso-
meter liegt im Nebenschluß.)

Korrosionen wurden bisher nur an den eisernen Raschigringen der
vorerwähnten Wasserausgaser festgestellt, die auf den geringen HCN-
Gehalt im Wasser zurückzuführen sind. In den untersten Schüssen
der Ausgaser wurden deshalb die Eisenringe durch Steinzeugringe
ersetzt.

Sicherheitsmaßnahmen: Sämtliche Gasleitungen und die gasführenden
Apparate sind durch Wassertauchungen gesichert. Im Katastrophenfall
erfolgt die Stillegung des Betriebes durch ein zentrales Abstell-
signal. Die für die Belegschaft vorgesehenen Gasmasken sind mit HCN-
und Universaleinsätzen versehen. Dem Betrieb steht außer den übli-
chen Handfeuerlöschgeräten ein CO_2- und ein Schaumlöschwagen zur
Verfügung.

Luftschutzmaßnahmen: Die Gebläse und Elektromotoren sind durch lose
geschichtete Splitterschutzmauern aus Ziegelstein geschützt. Im Luft-
gefahrenfalle wird der Betrieb, wie alle anderen Betriebe des Werkes,
von der sogenannten Notbelegschaft bedient. Diese setzt sich aus
einer geringstnotwendigen Anzahl von Arbeitern zusammen, mit denen
man die Produktion soeben noch aufrecht erhalten kann. Der Rest
der Belegschaft sucht die Bunker auf, für die Notbelegschaft sind
mehrere 2-Mann-Bunker aufgestellt.

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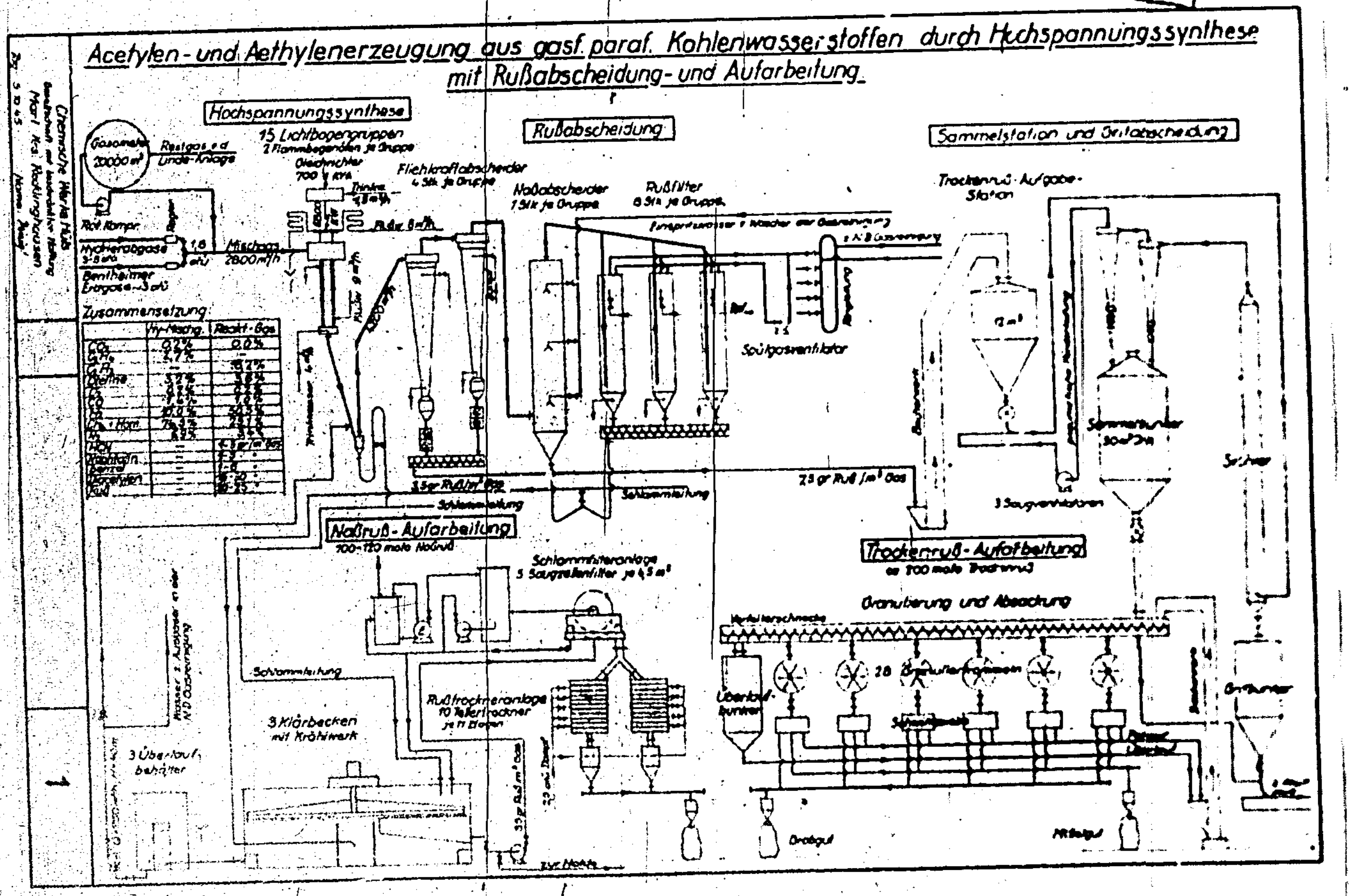
MICROFILMED by FIAT

Acetylen- und Aethylenerzeugung aus gasf. paraf. Kohlenwasserstoffen durch Hochspannungssynthese mit Rußabscheidung- und Aufarbeitung.



Zusammensetzung:

	Hy-Mischg.	Reakt.-Gas
CO_2	0,2%	0,0%
C_2H_6	2,7%	–
C_2H_2	–	[illegible]
Olefine	3,2%	[illegible]
O_2	[illegible]	[illegible]
CO	1,6%	1,6%
H_2	10,9%	[illegible]
CH_4 + Hom.	[illegible]	[illegible]
N_2	[illegible]	[illegible]
HCN	–	[illegible] gr/m³ Gas
Naphthalin	–	[illegible]
Benzol	–	[illegible]
Diacetylen	–	[illegible]
Ruß	–	10-25 "

Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Krs. Recklinghausen
Dat. 3.10.45 Name: [illegible]

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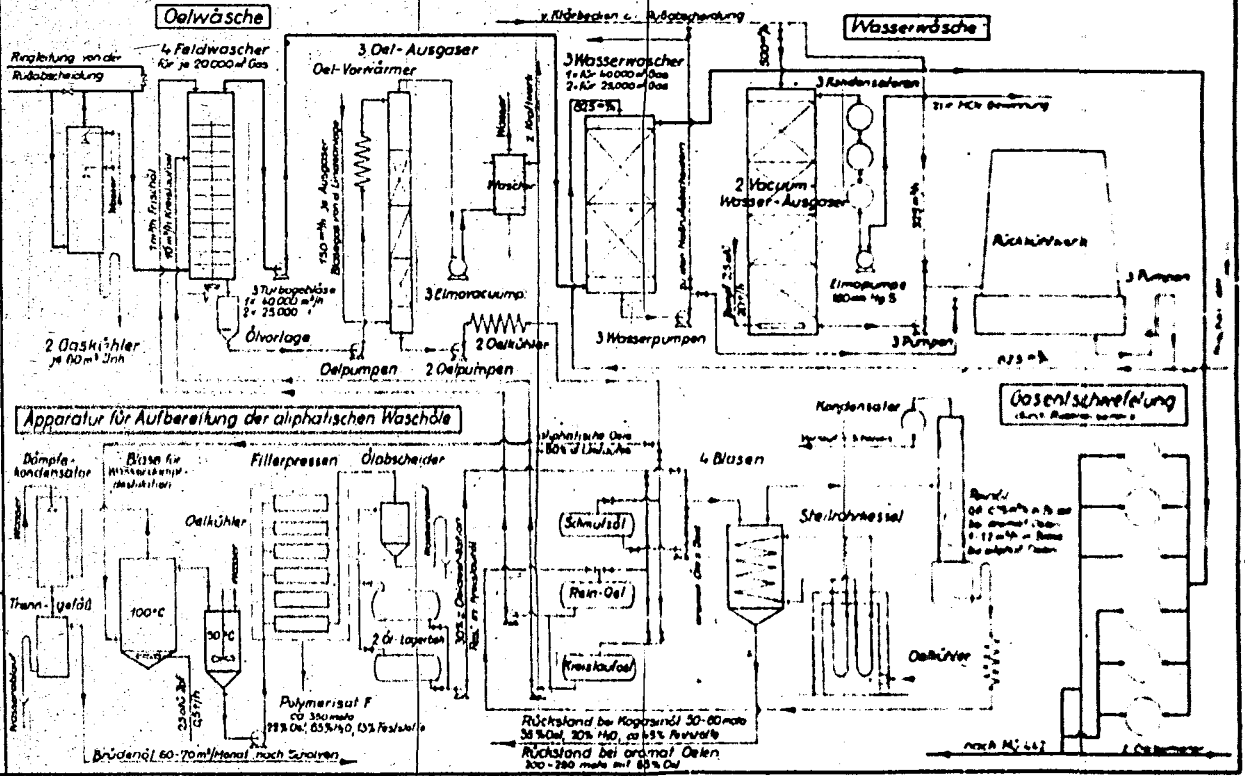
Reinigung der Lichtbogengase von naphtalinhaltigen Bestandteilen, Auswaschung der Blausäure und Entschwefelung im Niederdruck.



Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Krs. Recklinghausen
Tag 6.10.1945 Name Helsig

1a

Betriebsbeschreibung der Gaswäsche [illegible] 442

Ofengaswaschen. Vorgereinigtes Lichtbogengas (= Ofengas) mit einem Acetylengehalt von ungefähr 13 %, dessen Vorreinigung in der Niederdruckgasreinigung erfolgt, kommt in einer Rohrleitung mit einem Gasometer im Nebenschluß zu den 6 Ofengaskompressoren (1200 kW, Förderleistung 13 000 m³/h). Das Ofengas wird in vier Stufen von Gasometerdruck auf 18 atü komprimiert. Hinter jeder Stufe der Kompressoren ist ein Wasserkühler eingeschaltet; bei zwei Kompressoren sind die Kühler der vierten Stufe zurzeit versuchsweise weggelassen.

Von den Kompressoren gelangt dann das Gas in einer Sammelleitung zu den 6 Ofengaswäschen. Jede Wäsche bildet ein für sich abgeschlossenes Aggregat von einem Wascher, vier Entspannungstürmen und einem Ausblaseturm. In dem Wascher (Siebbodenwascher mit 66 Böden) wird unter 19 ata Betriebsdruck das Acetylen aus dem Ofengas ausgewaschen. Hinter jedem Wascher ist ein Abscheider geschaltet, um mitgerissenes Wasser wieder abscheiden zu können, außerdem befindet sich in der Sammelleitung hinter dem Wascher noch ein Abscheider. Das acetylenfrei gewaschene Gas (= Rohgas) wird in der Linde-Anlage (Bau 460) weiter zerlegt.

Gewaschen wird das Ofengas mit Kreislaufwasser, dem zur Ergänzung Flußwasser zugeschaltet wird. Das Wasser wird mittels einer Sulzerpumpe (22 atü Betriebsdruck) oben auf den Wascher aufgegeben und läuft im Gegenstrom zum Gas über die Siebböden. Vom Sumpf des Waschers gelangt das Gas zur Entspannungsturbine, die mit der Sulzerpumpe gekoppelt ist und die einen Teil der Pumpenenergie liefert, und von hier aus dem ersten Enspannungsturm E_1. Das Waschwasser wird hier auf etwa 2,7 ata entspannt. Oben am Entspannungsturm tritt ein Gas mit etwa 45 % C_2H_2 aus, das für eine direkte Verarbeitung noch zu verdünnt ist und das daher wieder in die Ofengasleitung vor die Kompressoren zurückgeführt wird. Dadurch komprimieren die Ofengaskompressoren ein etwa 15 % Acetylen, während der Acetylengehalt des Ofengases nur etwa 13 % beträgt. Aus dem Entspannungsturm E_1 gelangt das Wasser über eine Schleife zum Entspannungsturm E_2 und wird hier auf 1,1 ata entspannt. Hierbei wird etwa 92 %iges Acetylen frei. Das Wasser gelangt dann weiter über eine Schleife zum Entspannungsturm E_3 (0,15 ata), sowie über eine weitere Schleife zum Entspannungsturm E_4 (0,05 ata). Aus den beiden Entspannungstürmen E_3 und E_4 wird das Gas mittels Vakuumpumpen abgesaugt und in zwei bezw. drei Stufen auf Gasometerdruck gebracht und zusammen mit dem Gas aus E_2 zum Rohacetylenbehälter gegeben (Rohacetylen mit etwa 96 % C_2H_2). Das Wasser aus E_4 gelangt dann über eine Limaxpumpe zum Ausblaseturm R, wo es mit Restgas ausgeblasen werden kann, das dann dem Lichtbogen wieder zugeführt wird. Vom R-Turm läuft das Wasser dann in einer Schleife zum Demag-Belüftungsturm, wo das restliche noch im Wasser gelöste Acetylen mit Luft ausgeblasen wird. Vom Belüftungsturm schließlich kehrt das Wasser im Kreislauf wieder zum Wascher zurück.

Ölwaschen. Das im Bau anfallende 96 %ige Rohacetylen enthält höhere Acetylene, die in einer Ölwäsche (Bau 442) ausgewaschen oder in einer Tiefkühlanlage (Bau 462) ausgeschieden werden können. In beiden Fällen wird das Rohacetylen – in 462 mit Hilfe von Elmo – aus dem Gasometer angesaugt und entweder zu den Boxen in 462 oder zu den Ölwäschen in 442 gefördert. In den drucklos gefahrenen

Ölwäschen wurden Erdölfraktionen, Kogasin und zuletzt Steinkohlendestillate als Waschöl verwandt. Auf die Wäscher (30 Glockenböden) wird mittels Pumpen oben das Waschöl aufgegeben, das aus dem im Gegenstrom ankommenden Rohacetylen die höheren Acetylene auswäscht. Das so erhaltene Waschöl wird dann anschließend in Ausbläsern wieder regeneriert. Ausgeblasen wird mit den im Lichtbogen nicht umgesetzten Kohlenwasserstoffen, die in der Linde-Anlage (Bau 460) als Restgas wieder zurückgewonnen werden. Jede Ölwäsche bildet mit den beiden dazugehörigen Ausbläsern ein abgeschlossenes Aggregat. Insgesamt sind 3 solcher Aggregate in 442 vorhanden.

Aus dem Sumpf der Ölwäsche wird mittels einer weiteren Pumpe das Öl angesaugt und gelangt zu einem aus zwei Teilen bestehenden Ausgaser. Der obere Teil (10 Glockenböden) dient als Acetylenausgaser, dabei wird mit kleinen Mengen Restgas ausgeblasen und das dabei gewonnene etwa 50 %ige Acetylen wird wie das Gas aus E_1 über die Ofengasleitung wieder vor die Kompressoren gefahren. Der untere Teil ([illegible] Glockenböden) dient als Diacetylenausgaser. Dort werden mit einer großen Restgasmenge die leichter löslichen höheren Homologen des Acetylens u.a. auch das Diacetylen, ausgeblasen. Das dabei anfallende Gas geht zum Restgasbehälter und wird dann später im Lichtbogen von neuem zerlegt.

Koksgaswäsche. Ein Kompressor (13 000 m³/h 630 kW) komprimiert in einer Stufe das von der Zeche Auguste Victoria kommende Koksofengas von rund 7 atü auf 18 atü. (Ein zweiter Kompressor steht in Reserve) und gibt das Gas auf einen Wasserwascher, derselben Bauart wie die Ofengaswascher. In dem Wascher wird die Kohlensäure ausgewaschen. Das oben austretende Gas gelangt über zwei Abscheider zum Koksgaszerlegungsapparat im Bau 460. Das zum Auswaschen verwandte Wasser wird aus einer Sammelleitung des Belüftungsturmes über eine Sulzerpumpe genau wie bei den Ofengaswaschern oben aufgegeben und gelangt vom Wascher aus über eine Schleife zum Belüftungsturm. Das im Entspannungsturm der Koksgaswäsche frei werdende Gas mit etwa 7 % Olefine und 15 % CO_2 kann ohne Kompressoren der Saugleitung des Koksgaskompressors nicht wieder zugeführt werden und wird daher vor die Ofengaskompressoren gefahren und dort den Ofengaswäschen zugeführt. Am Belüftungsturm wird dann die im Wasser gelöste Kohlensäure wieder ausgetrieben und das Wasser wird erneut den Waschern wieder zugeführt.

Komprimierte Gase

1) Mischgasregulierstation
Vom H_2S mittels einer Wäsche befreites Erdgas wird von 3 atü auf 1,8 atü entspannt, ferner besteht noch eine Möglichkeit, Hy-Gas von den Hydrierwerken, das mit 3 – 5 Betriebsdruck ankommt über einen Askania-Regler auf 1,8 atü zu entspannen.

   Außerdem wird das Restgas aus dem Restgasbehälter mittels Elmos oder Rotationskompressoren auf 1,8 atü komprimiert. Das Gemisch all dieser Kohlenwasserstoffe (= Mischgas) gelangt zur Lichtbogenanlage um dort in Acetylen, Äthylen, Wasserstoff, Ruß usw. zerlegt zu werden.

2) Wasserstoff-Kompression
Wasserstoff fällt an
   a) in Bu 460 aus der Gaszerlegungsapparatur mit einem Druck bis zu 15 atü,
   b) der Chlorelektrolyse Bu 522 mit Gasometerdruck.

   Der Elektrolyt-Wasserstoff aus 522 wird in 2 Kompressoren

(3-stufig) auf 15 atü komprimiert und dann gemeinsam mit dem aus
HU 460 anfallenden Wasserstoff mit 5 Kompressoren (3-stufig) auf
325 atü komprimiert (normalerweise mit 300 atü gefahren). Der
Hochdruck-Wasserstoff geht dann zur Aldol-Hydrierung 258 bzw. zur
Flaschen - Abfüllstation.

3) Stickstoff-Kompression
Stickstoff aus HU 460 (drucklos aus dem im Hauptschluß liegenden
Gasometer kommend) wird mittels Rotations-Kompressoren (2-stufig)
auf 5 atü komprimiert und über Filter und Windkessel in das Werks-
Stickstoffnetz gegeben. Ein Teil dieses Stickstoffs kann mit 2
Hochdruck-Kompressoren (3-stufig) auf 325 atü komprimiert werden.

4) Druckluft-Kompression.
Vorhanden ist ein 2-stufiger Rotations-Kompressor. Die Luft wird
angesaugt aus einer Luftansaugleitung über dem Apparategerüst von
Bu 442x (über ein Staubfilter). Die auf 6 atü komprimierte Luft
wird über Ölfilter und Windkessel in das Werks-Druckluftnetz ab-
gegeben.

5) Sauerstoff-Acetylen-Kompression
Gasförmiger Sauerstoff aus HU 460 wird über einen Gasometer ange-
saugt, mittels 3-stufiger Elmopumpe auf 4,5 atü komprimiert und
in das Werks-Sauerstoffnetz abgegeben. Reinacetylen aus dem Rein-
acetylenbehälter im Nebenschluß kommend wird mittels einstufiger
Elmo auf 0,6 atü komprimiert und in das Werks-Schweiß-Acetylen-
Netz abgegeben. Der Sauerstoff gelangt über einen Windkessel in
das entsprechende Werksnetz. Beim Schweiß-Acetylen wird der Netz-
druck mittels Reglers gehalten.

Nachhydrierung

Das in 460 anfallende Rohäthylen enthält noch einige Zehntel Pro-
zent Acetylen, die später nach der Konzentration im Äthylenapparat
etwa 1 % des Rein-Äthylens ausmachen. Dieses Acetylen wird in der
Nachhydrierung zu Äthylen aufhydriert. Außerdem besteht noch die
Möglichkeit Acetylen in Mengen bis zu 60 m³/h hinzuzusetzen, so daß
ein etwa 1 %iges Acetylen in der Nachhydrierung verarbeitet werden
kann. Als Kontakt wird Kieselgel mit 0,15 g Pd/l verwendt. Der
Hydrierofen wird von oben nach unten durchfahren, damit der Kontakt
nicht aufgewirbelt wird. Das in einem Rieselkühler gekühlte Gas
geht dann zum Rohäthylenbehälter zusammen mit dem Rohäthylen aus der
Acetylenhydrierung (Bau 662).

Korrosion:

Korrosionserscheinungen traten in der Koksgaswäsche auf. Folgende
Maßnahmen sind als Schutz gegen Korrosion getroffen: Der Wascher-
sumpf ist ausgemauert, ausgemauert ist ferner der Entspannungs-
turm, während der Deckel und die Brause dieses Entspannungsturmes
phenytalisiert worden sind. Die Leitungsstücke vor und hinter den
Entspannungstürmen sind versuchsweise gummiert oder phenytalisiert.

Sicherheitsmaßnahmen:

Um ein schnelles und einheitliches Abstellen der Bauten im Gefahren-
fälle zu gewährleisten, können die elektrisch betriebenen Aggregate
zentral vom Kraftwerk abgeschaltet werden. Die Acetylenbehälter
werden bei diesen Abstellungen geschlossen, ebenso die Ferngaslei-
tungen am Werkseingang. Um Undichtigkeiten im Vakuumsystem bemerken
zu können, wird der Sauerstoffgehalt im Acetylen durch einen Sauer-
stoffschreiber überwacht. Vermeidung von Acetylenzerfall durch
Wassereinspritzungen an der Ofengasdruckleitung.

II

Luftschutzanlagen:

Splitterschutzmauern zwischen sämtlichen Maschinen und zum Teil vor den Bedienungsständen. 2-Mann-Bunker für die Notbelegschaft.

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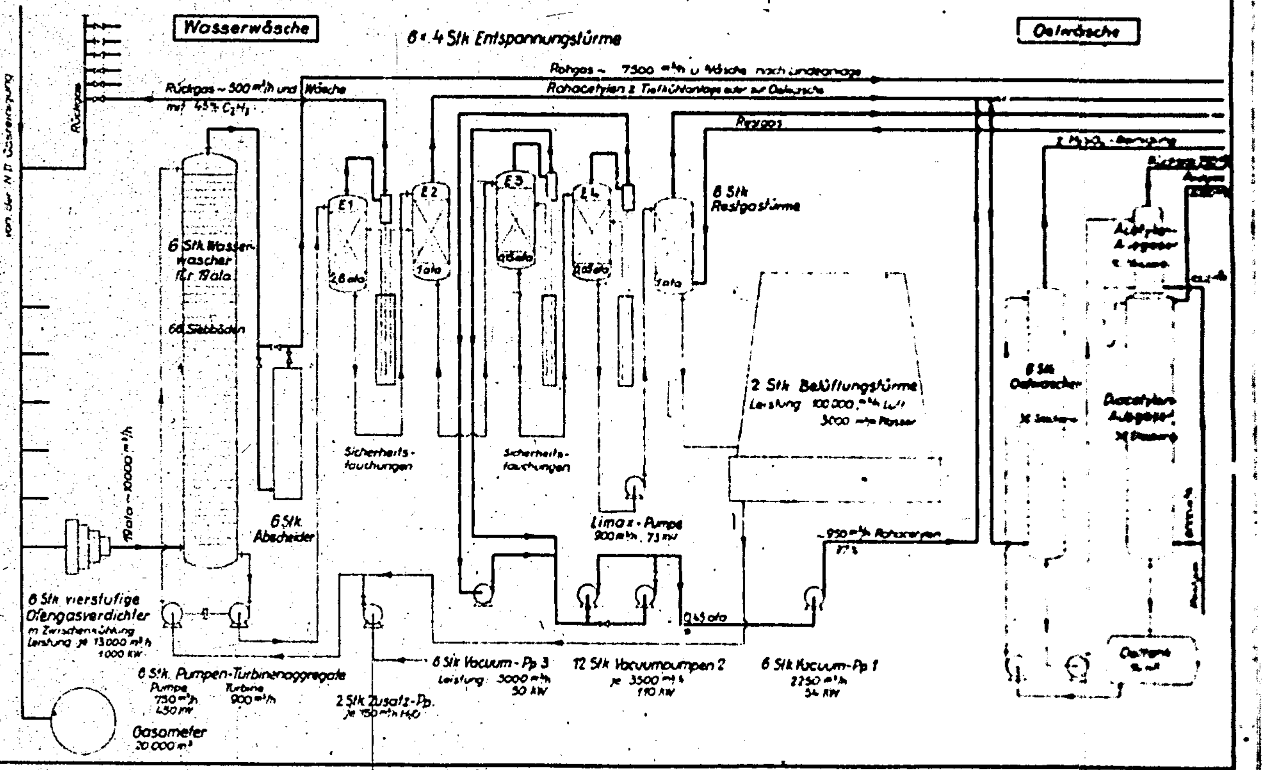
225

Gewinnung von Rohacetylen durch Druckwasserwäsche mit nachgeschalteter Oelwäsche zur Auswaschung von höheren Acetylenen.



Chemische Werke Hüls
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Marl, Krs. Recklinghausen
Tag 9.10.1945 Name [illegible]

2

Beschreibung der Tiefkühlanlage H 452

Der Zweck der Tiefkühlanlage H 452 ist das Reinigen des aus der Wasserwäsche zu uns kommenden Acetylens. Dieses enthält als Verunreinigung eine erhebliche Menge Kohlenwasserstoffe, vor allem Acetylenkohlenwasserstoffe mit einer C-Zahl größer als 2, deren Entfernung vor der Aldehydsynthese ist. Die Zusammensetzung des Rohacetylens ist etwa folgende: erforderlich

C_2H_2	90 %	)
C_4H_2	3 %	)
C_2H_4	1,5 %	) 96 %
C_3H_4	1,5 %	)
C -C K. (C_6H_6)	1 %	
KW + N_2	2 %	
CO_2	1 %	

Das austretende Reinacetylen ist dagegen wie folgt zusammengesetzt:

C_2H_2	97 %
CO_2	1 %
KW + N_2	2 %

Die anschließende Laugewäsche befreit das Gas noch vom CO_2 und ein 98 %iges Reinacetylen verläßt die Anlage.

Das Rohacetylen wird von Wasserringpumpen aus dem Rohacetylengasometer angesaugt und mit einem Druck von 1,3 höchstens 1,4 atü in die Tiefkühlapparate gedrückt. Es sind vier Pumpen und drei Apparate vorhanden, ein vierter Apparat befindet sich imBau. Der älteste der Apparate, Nr. 1, weist eine von den anderen beiden etwas abweichende Bauart auf.

Nach der Kompression passiert das Rohacetylen zunächst einen Rieselkühler, in denen durch herabrieselndes Wasser von 40° C auf 10–20° C abgekühlt wird. Mit dieser Temperatur tritt es in den eigentlichen Apparat, der sich in einer Betonkammer befindet, ein und passiert zuerst einen der beiden umschaltbaren Gegenströmer, in dem es seine Wärme an ein Eis-Benzol-Kohlenwasserstoffgemisch abgibt, und sich auf 0° abkühlt. Das Gemisch taut dabei und läuft als Gegenströmerkondensat zur Kondensataufarbeitung, in der es durch Behandlung mit stark verdünnter Natronlauge bei 60° von den polymerisationsfreudigsten Stoffen, vor allem dem Äthyldiacetylen befreit und durch nachfolgende Wasserdampfdestillation in das sogenannte Rohlibozol, einem Gemisch von 80 % Benzol, 10 % Toluol, 6 % Phenylacetylen und 4 % höherer Acetylene z.T. noch unbekannter Zusammensetzung überführt wird. Die Behandlung mit Natronlauge erfolgt in 2 Rührbehältern in wechselweisem Betrieb, indem jeweils einer der Behälter sich mit dem anfallenden Kondensat füllt, während aus dem anderen das Rohlibozol abdestilliert wird. Das Rohlibozol fließt in einen Tank und kann von da aus in eine Wasserdampfdestillationskolonne gepumpt werden, in welcher es in ein Benzol-Toluol-Gemisch und Phenylacetylen zerlegt wird. Der täglich Anfall an Rohlibozol beträgt 100–500 l/Apparat. Ein Teil der anfallenden Flüssigkeit wird dem ablaufenden Rohkondensat zusammen mit Wasser zwecks Kühlung und Verdünnung zugesetzt.

Im zweiten Gegenströmer gibt das Rohacetylen einen weiteren Teil seiner Wärme an das den Tiefkühler mit −60° verlassende Reinacetylen ab, wobei es sich auf −15° abkühlt und den größtenTeil seines Wassers, sowie einen Teil des [illegible] [illegible]lert. Diese Stoffe frieren aus und werden in der darauffolgenden Betriebsperiode vom frischen Acetylen gelöst. Der Wechsel der Gegenströmer erfolgt alle 2 Stunden.

Vom Gegenströmer tritt das Gas in den Vorkühler 1 über, der mit unter 0,5 ata siedendem Ammoniak gekühlt wird. Auf dem Wege zwischen Gegenströmer und Vorkühler erfolgt die Eindüsung von Methanol in einer Menge von 30–40 l/h. Das Methanol hat die Aufgabe, eine Abscheidung von Eis im Vorkühler zu verhindern. Dadurch wird es ermöglicht, die Kühler dauernd in Betrieb zu lassen. Zusammen mit dem Methanol und dem Rest des Wassers verläßt das Vorkühlerkondensat den Sumpf des Kühlers um in einer auf -40° gehaltenen Leitung in den Kondensatvergaser einzurinnen. Seine Zusammensetzung ist etwa folgende:

$CH_3OH + H_2O$	30 %
Benzol	25 %
$C_4H_2 \cdot C_4H_4$	37 %
höhere Acetylene (C_5–C_6)	8 %

Anfall = 80 g/m³ Rohacetylen.

Vom Vorkühler 1, den das Acetylen mit -35° C verläßt, tritt es in den Vorkühler 2 ein und wird mit unter 0,3 ata siedendem Ammoniak auf -55° abgekühlt. Zwischen den beiden Kühlern wird nochmals Methanol eingedüst, jedoch in erheblich geringerer Menge.

An den Vorkühler 2 ist der Tiefkühler angeschlossen. Im unteren Teil dieses Apparates befindet sich ein Röhrenbündel, welches von dem oberen Teil des Apparates auf -73° gekühlten Reinacetylen durchströmt wird. Der obere Teil enthält ein Rohrschlangensystem, in welchem sich unter 1,8 atü bei -85° C siedendes Äthylen befindet. In diesem Teil erreicht das Acetylen seine tiefste Temperatur von -73° und passiert dann als Reinacetylen die Gegenstromwärmeaustauscher im Tiefkühler und Gegenströmer. Das im Tiefkühler anfallende Kondensat verläßt den Sumpf dieses Apparates mit -60° C und vereinigt sich mit dem im Vorkühler 2 anfallenden Kondensat. Die Flüssigkeiten passieren die Anwärmung, eine Rohrschlange, die indirekt mit gasförmigem NH_3 auf -40° gehalten wird. Das Kondensat fließt mit dieser Temperatur in den Ausgaser.
Es hat dabei folgende Zusammensetzung:

Acetylen	3 %
Methylacetylen	22 %
Vinylacetylen	13 %
Diacetylen	48 %
höhere Acetylene	9 %
sonstige KW	5 %

Im Kondensatvergaser werden nun das Vor- und Tiefkühlkondensat mit Gasöl vermischt und auf den Kopf des Apparates gepumpt. Das Gemisch läuft über eine Raschigringschicht wieder in den Sumpf zurück, wobei es durch entgegenströmende Restgas von den Acetylenen befreit wird. Ein Teil des Kondensats polymerisiert allerdings im Öl, von dem ein Teil ständig über den Filter geht. Im Apparat wird täglich 0,5–1 m³ Frischöl benötigt.

Sicherheitsmaßnahmen:

Sämtliche Gasführenden Leitungen sind einzeln getaucht. Zentrale Abschaltung der Kompressoren. Die Apparaturen sind in Einzelboxen mit 1 m-Stein-Eisenbetonwänden untergebracht. Die Bedienung erfolgt außerhalb dieser Boxe von einem zentralen Bedienungsstand aus.
Luftschutzmaßnahmen: Splitterschutzwände zwischen den Maschinen. 2-Mann-Bunker für die Notbelegschaft.

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15

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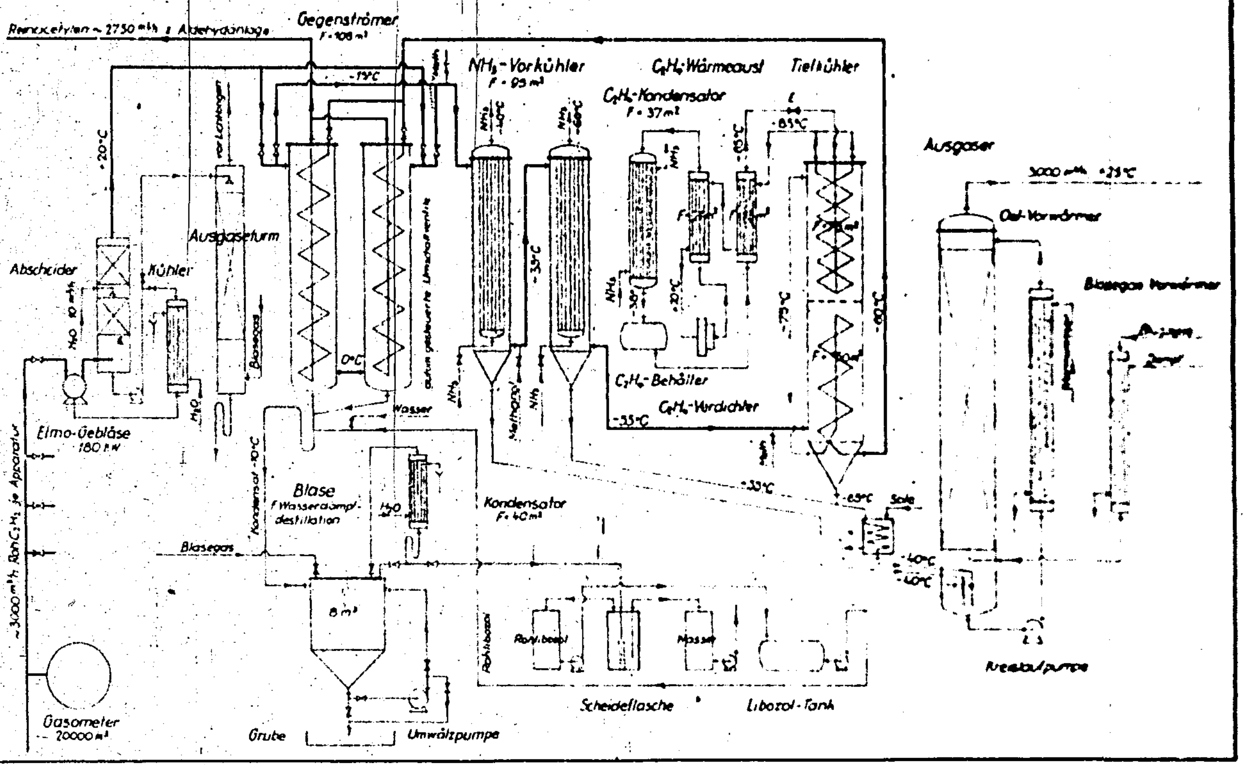
Verfahren zur Ausscheidung von höheren Acetylenen durch Tiefkühlung



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Marl, Krs. Recklinghausen
Tag: 10.10.1945 Name: Henig

3

16

Betriebs[illegible]lung
der Gaszerlegung (Linde-Anlage) Bau 460

In der Linde-Anlage wird das vom Lichtbogen kommende und in der Gas-Wäsche von Acetylen befreite Rohgas durch Tiefkühlung nach Verfahren von Linde in seine Bestandteile zerlegt und z.T. weiter verarbeitet. Im einzelnen gliedert sich der Betrieb der Gaszerlegung folgendermaßen:

1) Gaszerlegung zur Erzeugung von CO-freiem Wasserstoff

Zerlegung von Roh- bezw. Koksofengas in den Wasserstoffapparaten durch fraktionierte Kondensation in Wasserstoff, Rohäthylen, Restgas und CO-Fraktion (5 Wasserstoff- und 2 Koksofengasapparate).

2) Äthylenzerlegung

Gewinnung von Reinäthylen durch fraktionierte Destillation der Rohäthylenfraktion der Wasserstoffapparate in 4 Äthylenapparaten.

3) Luftzerlegung

Trennung von Luft in Sauerstoff und Stickstoff. Verwendung von Stickstoff als Hilfskältekreislauf bei den Wasserstoffapparaten (4Luftzerleger).

4) Flaschenabfüllstation

Technische Abfüllung von Sauerstoff, Wasserstoff, Stickstoff, Äthylen, Methan und Luft in Stahlflaschen.

Zu 1) Das vom Lichtbogen kommende Ofengas wird in der Gaswäsche auf 18 atü verdichtet und durch Wasserwaschung von Acetylen befreit. Dieses "Rohgas" tritt mit 16,5 atü in die Wasserstoffapparate der Linde-Anlage ein. In der Vorapparatur wird es in wechselbaren Gegenströmern mit Zerlegungsprodukten und anschließend in Ammoniakvorkühlern auf -50° vorgekühlt. Das Rohgas tritt dann in die eigentliche Zerlegungsapparatur ein und wird im Gegenstrom zu Zerlegungsprodukten fortschreitend heruntergekühlt, wobei die einzelnen Fraktionen nacheinander kondensieren (C_3 und höhere Kohlenwasserstoffe zwischen warmen und kalten Ast, C_2-Kohlenwasserstoffe im Sumpf des Äthylenverflüssigers 3).
Zur weiteren Kühlung ist ein zusätzlicher Stickstoff-Kältekreislauf eingeschaltet, auf den unten näher eingegangen wird. Im Stickstoffverdampfer wird das im Rohgas enthaltene Methan verflüssigt und das Gas in den Sumpf der Trennsäule 6 eingeleitet. Hier findet eine Schlußreinigung des Wasserstoffs von restlichem Kohlenoxyd und Stickstoff statt, wobei in der Fraktionskolonne mit flüssigem Stickstoff gewaschen wird und der Kondensator am Kopf der Säule mit im Vakuum siedendem Stickstoff auf -212° heruntergekühlt wird. Der reine Wasserstoff wird am unteren Teil der Säule abgezogen und mit 15 atü im Gegenstrom zum einziehenden Rohgas abgeführt.

Im Stickstoffkältekreislauf wird 99,5%iger Stickstoff in 5-stufigen Stickstoffkreislaufkompressoren auf 200 atü verdichtet, durch Vorkühler auf -45° vorgekühlt, im Gegenstrom auf -140° und durch Entspannung in mehreren Stufen (Mitteldruckstickstoff 25 atü, entspannter Stickstoff 2,5 atü und Vakuum-Stickstoff 0,2 ata) auf -212° gekühlt. Das Vakuum im Vakuum-N_2-Teil wird durch eine 2-stufige Vakuumpumpe erzeugt.

Die analytische Zusammensetzung und mengenmäßige Aufteilung der Fraktionen sind aus der anliegenden Tabelle ersichtlich.

Zu 2) Die in den Wasserstoffapparaten anfallende Rohäthylenfraktion wird in den Äthylenzerlegern auf Reinäthylen von ca. 95 % verarbeitet. Bei Verarbeitung von Erdgas in den Wasserstoffapparaten fällt eine Rohäthylenfraktion mit 6 % und in den Koksgasapparaten mit 19 % an. Durch Zumischen von Hydrier- oder sonstigem höherprozentigen Äthylen ergibt sich daraus ein Rohäthylengemisch mit 15 - 25 % Olefinen.

Das Rohäthylen wird in einem 3-stufigen Kompressor (4000 m³/h) auf 30 atü verdichtet und in der Vorapparatur auf -35° vorgekühlt. Im Kälteaustausch mit Zerlegungsprodukten wird das Rohäthylen auf -55° gekühlt und in der Heizschlange im Sumpf der Säule 1 teilweise verflüssigt, wobei gleichzeitig eine Roh-Fraktionierung von Kondensationsprodukten der Säule 1 eintritt. In der Säule 1 (Methansäule) findet eine Abtrennung der C_1- von den C_2-Kohlenwasserstoffen statt, während in Säule 2 die C_2-Kohlenwasserstoffe Äthylen und Äthan getrennt werden. Durch weiteren Kälteaustausch und Entspannung von 30 auf 1 atü wird nach Eintritt in die Methansäule bei 140° das Methan von den höheren Kohlenwasserstoffen gasförmig am Kopf der Säule 1 abgetrennt. Das flüssige Gemisch der C_2-Kohlenwasserstoffe und höheren Homologen wird in Säule 2 (Betriebsdruck 0,6 atü) geführt, wo das Reinäthylen mit ca. 95 % am Kopf der Säule austritt, während aus dem Sumpf (-68°) die restlichen Kohlenwasserstoffe (vorwiegend Äthan und Propylen) abgezogen werden. Die Fraktionierung der Äthylensäule wird durch Waschung mit Reinäthylen bewirkt, das in einem besonderen Kreislauf (Kompression in Rein-C_2H_4-Kompressoren auf 8 atü) geführt wird (Rücklaufverhältnis 1 : 3). Die Verflüssigung des Kreislaufäthylens erfolgt durch Kälteaustausch und Entspannung. Die anfallenden Restkohlenwasserstoffe werden mit den Restkohlenwasserstoffen der Wasserstoffapparate vereinigt und in den Fabrikationsprozess (Lichtbogen) zurückgeführt.

Zu 3) Luft wird je nach Windrichtung von der Ost- oder Westseite des Werkes angesaugt und in Luftkompressoren (2000 m³/h) auf 140 atü verdichtet. Zwischen der ersten und zweiten Verdichtungsstufe wird die Kohlensäure mit 10%iger Natronlauge ausgewaschen. Von der verdichteten Luft, die in der Vorapparatur in wechselbaren Gegenströmen und NH_3-Vorkühlern auf -45° vorgekühlt wird, wird ein Teilstrom über eine Expansionsmaschine geführt und auf 4,5 atü entspannt. Die entspannte Luft, die sich bei der Entspannung auf -140° abkühlt, tritt unmittelbar in den Sumpf der unteren Säule (Drucksäule) ein. Die restliche Hochdruckluft wird in mittelbarem Austausch im Gegenstrom mit Zerlegungsprodukten vorgekühlt und ebenfalls auf 4,5 atü entspannt.

Die Zerlegungssäule ~~besteht aus 2 übereinander~~geordneten Säulen. Die untere Säule wird bei 4,5 atü und die obere Säule bei 0,5 atü betrieben. In der unteren Säule findet eine teilweise Zerlegung der Luft in ihre Bestandteile statt. Dabei wird der anfallende Stickstoff teilweise flüssig abgezogen und nach Entspannung auf 0,5 atü am Kopf der oberen Säule als Waschflüssigkeit aufgegeben, während eine andere Teilmenge gasförmig am Kopf des Kondensators entnommen, im Zusatzkondensator verflüssigt und nach Entspannung ebenfalls als Waschflüssigkeit auf die obere Säule gegeben wird. Die im Sumpf der Drucksäule anfallende Flüssigkeit mit ungefähr gleichen Teilen Sauerstoff und Stickstoff wird im Verflüssigungsgegenströmer z.T. verdampft und in den mittleren Teil der oberen Säule eingeführt. Nach Rektifikation in der oberen Säule wird der flüssige Sauerstoff (330 m^3/h) am Boden des Kondensators abgezogen und über Zusatzkondensator, Acetylenabscheider und Verflüssigungsgegenströmer in die Tankanlage geleitet, wo er flüssig in Kugelbehältern drucklos gespeichert wird. Der Stickstoff wird am Kopf der oberen Säule gasförmig abgezogen. Eine Teilmenge durch N_2 verunreinigter O_2 ("O_2 unrein" mit 20 - 30 % O_2) wird ständig im mittleren Teil der oberen Säule abgeleitet.

Der in der Luftzerlegung erzeugte Stickstoff wird z. T. in der Linde-Anlage verwendet und zwar für den Stickstoffkreislauf der Wasserstoffapparate und zum Tauen sämtlicher Zerlegungsapparate.

Die für die gesamte Gaszerlegung erforderliche Ammoniakkälte wird in einem besonderen Ammoniaksystem erzeugt (5 NH_3-Kompressoren für Temp. bis -40°, 2 Zusatzkompressoren für Temp. bis -55°, Abscheider-, Sammel- und Verteilerflaschen).

Zu 4) In der angegliederten Flaschenabfüllstation wird vorwiegend Sauerstoff- und Wasserstoff in Stahlflaschen und Gastankwagen abgefüllt. (Tägliche Abfülleistung 18 000 m^3 Wasserstoff und 5 000 m^3 Sauerstoff). Weiterhin können Stickstoff, Methan, Äthylen und Preßluft abgefüllt werden.

Sicherheitsmaßnahmen:

Verzinnung der Apparate gegen Acetylenschäden.
Verhütung von Acetylenexplosionen in der Luftzerlegung durch Acetylenabscheider, Syndikagenabsorber und durch die beiden außerhalb gelegenen Luftansaugestationen.

Der Betrieb kann zentral abgeschaltet werden. Die Abschaltung kann an 2 Stellen im Betrieb betätigt werden.

Luftschutzmaßnahmen:

Schutz sämtlicher Maschinen und Bedienungsstände durch Splitterschutzwände. Aufstellung von 2-Mann-Bunkern für die Notbelegschaft.

19

Gasbilanzen

Zerlegung von Rohgas in einem Wasserstoffapparat

bei Lichtbogenbetrieb mit Erdgas
Mengen in Nm³/h bei 0° u. 760 mm

	1) Rohgas		2) Roh-C_2H_4		3) Restgas		4) CO-Fraktion		5) Wasserstoff	
	%	m^3	%	m^3	%	m^3	%	m^3	%	m^3
CO_2	0,01	1	0,1	1	0	0	0	0	0	0
C_2H_2	0,14	9	0,2	2	0	0	0	0	0	0
Olefine	0,9	83	6,0	67	0,6	16	0	0	0	0
CO	2,0	184	1,5	17	4,0	117	11,0	50	0	0
H_2	55,0	5063	2,5	28	5,2	138	5,5	25	97,0	4872
K.W.	33,6	3080	82,0	910	80,0	2140	6,7	30	0	0
N_2	8,0	735	9,0	100	10,0	268	71,5	322	2,9	145
O_2	0,3	37	0,3	3	0,3	8	0,5	23	0,1	3
	100	9200	100	1110	100	2680	100	450	100	5017

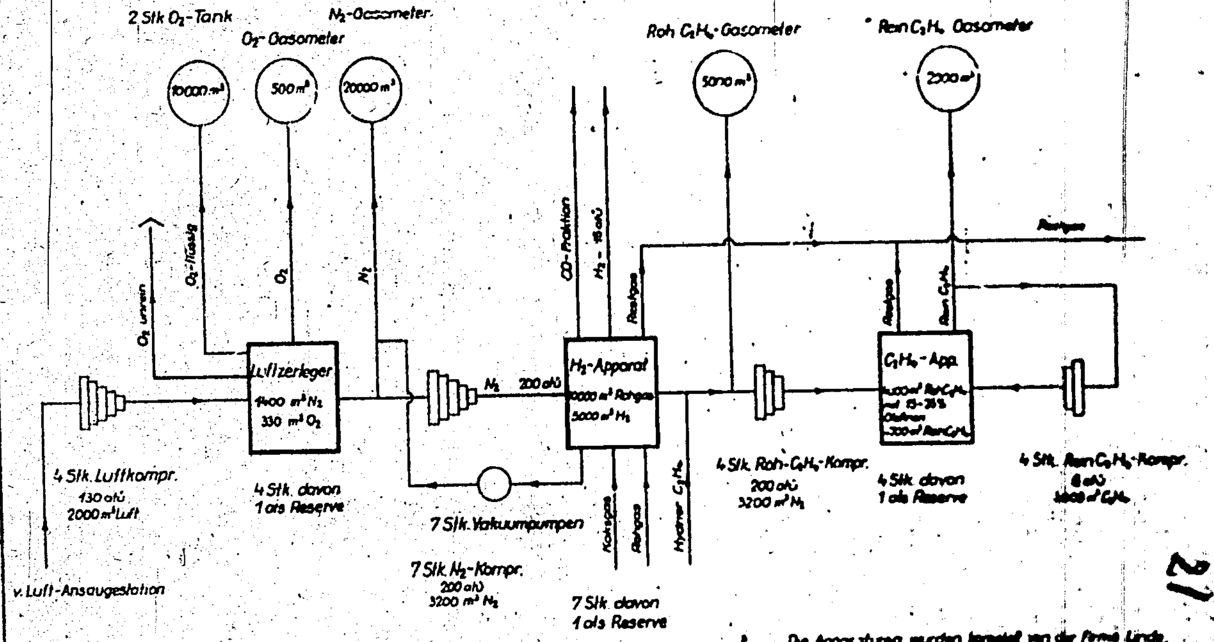
Zerlegung von Koksgas in einem Koksgasapparat

	1) Rohgas		2) Roh-C_2H_4		3) Restgas		4) CO-Fraktion		5) Wasserstoff	
	%	m^3	%	m^3	%	m^3	%	m^3	%	m^3
CO	0,01	1	0	1	0	0	0	0	0	0
C_2H_2	0,04	1	0	1	0	0	0	0	0	0
Olefine	1,0	92	19,0	86	0,03	6	0	0	0	0
CO	4,0	368	2,5	11	3,0	67	17,3	290	0	0
H_2	53,0	4880	1,0	5	4,0	89	3,3	56	97,0	4730
K.W.	26,0	2390	70,0	326	86,0	1920	8,9	150	0	0
N_2	15,0	1380	7,0	30	6,0	132	69,7	1177	2,9	148
O_2	0,3	25	0,3	1	0,3	7	0,8	14	0,1	5
	100	9200	100	450	100	2220	100	1680	100	4880

Gasbilanz für einen Äthylenapparat
Mengen in m³/h bei 0°, 760 mm

	1) Rohäthylengemisch vor C_2H_4-Apparat		2) Reinäthylen		3) CH_4		4) C_2H_6		3) + 4) Restgas	
	%	m³	%	m³	%	m³	%	m³	%	m³
C_3H_6 C_4H_8	3,0	120	-	-	-	-	26,7	120	3,7	120
C_2H_6	7,5	300	2,6	19	0,1	3	62,0	278	8,8	28
C_2H_2 + CO_2	0,3	12	1,6	12	-	-	-	-	-	-
C_2H_4	20,0	800	95,0	740	0,3	9	11,3	51	1,8	60
CH_4	63,6	2644	0,8	5	91,5	2539	-	-	78,8	2539
$CO+H_2+H_2$	5,6	224	-	-	8,1	224	-	-	6,9	224
	100	4000	100	776	100	2775	100	449	100	3224

Gaszerlegung nach dem Verfahren von Linde.



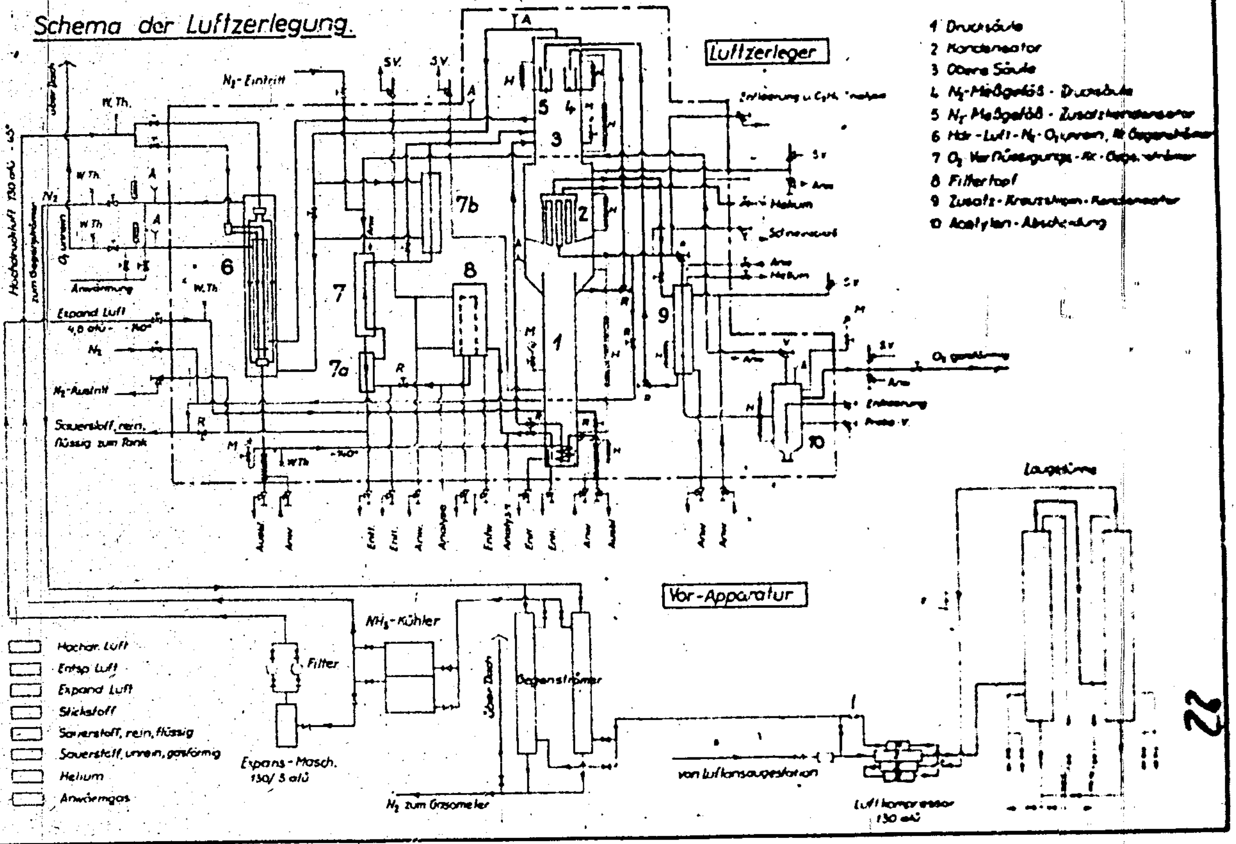
Die Apparaturen wurden komplett von der Firma Linde, Höllriegelskreuth bei München geliefert.
Einzelheiten über die Ausführung sind unbekannt.

Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl, Krs. Recklinghausen
Tag 10.10.1945 Name Hmwg

4

22

Schema der Luftzerlegung.

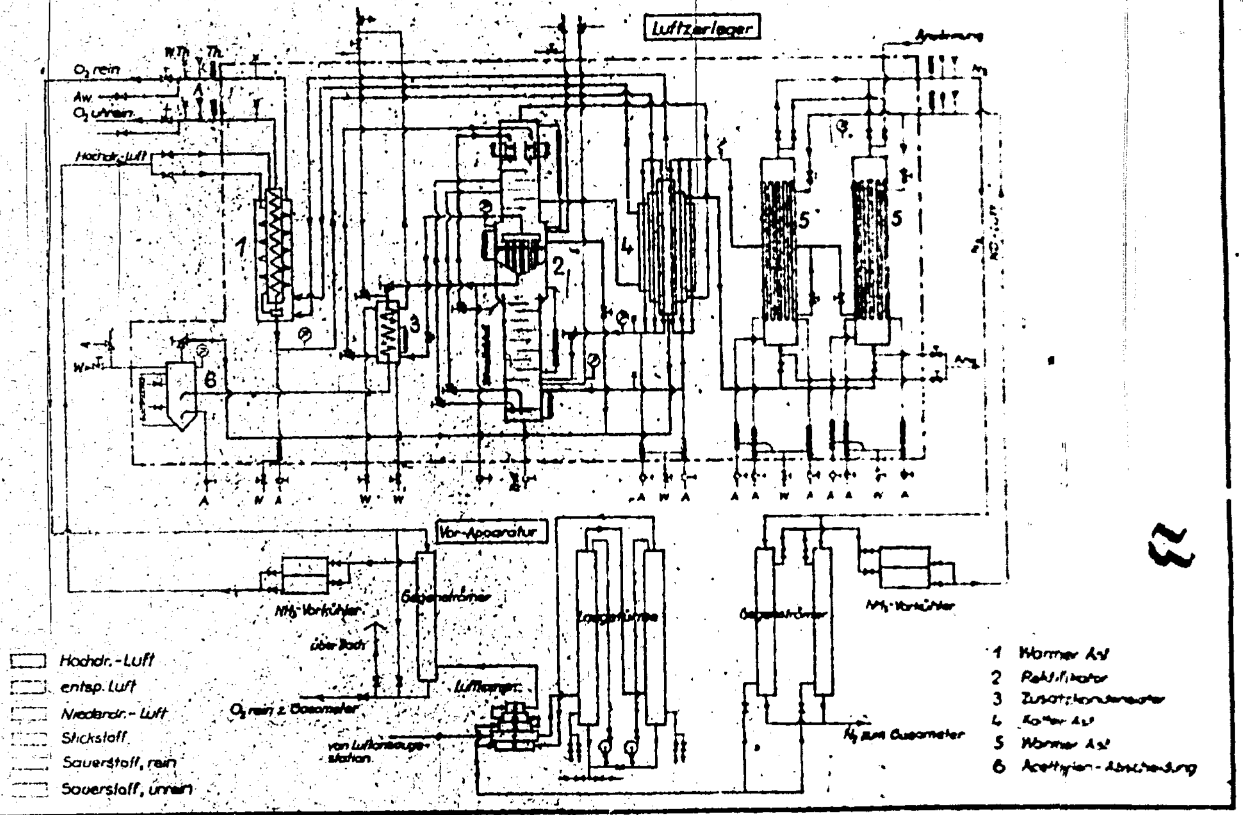


Chemische Werke Hüls
Marl, Krs. Recklinghausen
Tag 23.3.1946 Name Herzog

4a

23

Schema der Luftzerlegung

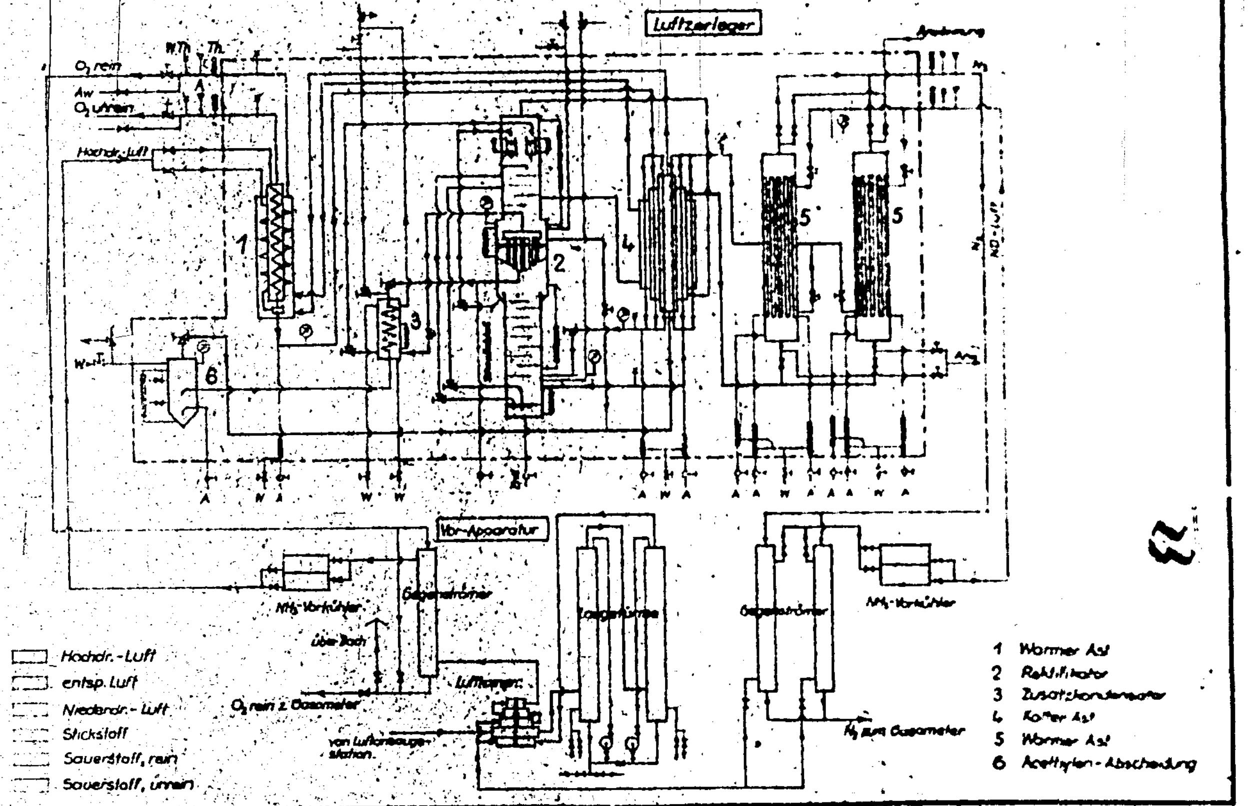


Chemische Werke Hüls
Marl, Krs. Recklinghausen
Zg. 3.6.1946 Name Heisig

4b

23

Schema der Luftzerlegung



Chemische Werke Hüls
Marl, Krs. Recklinghausen
Tag 3.6.1946 Name Heisig

4b

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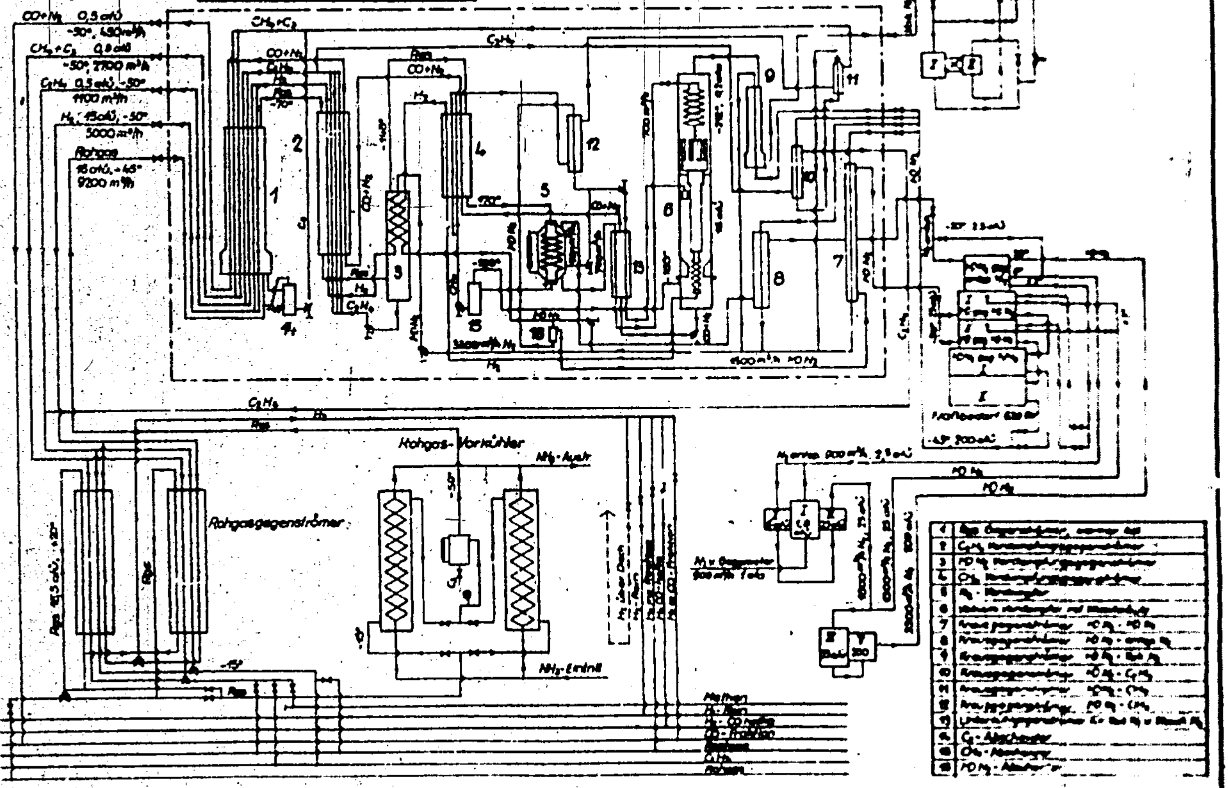
24

Schema der Wasserstoffboxen.



Chemische Werke Hüls
Marl, Krs. Recklinghausen
Tag: 27.4.1946 Name: Heisky

4c

85

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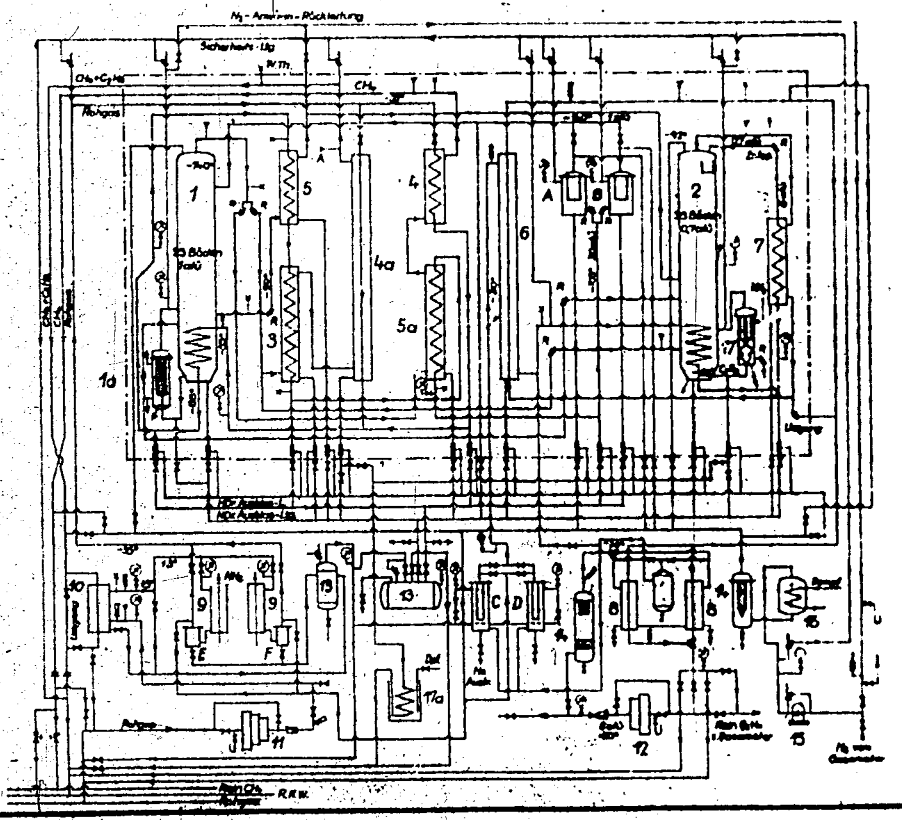
Schema der Aethylen-Anlage



1 Methan-Säule
1a Zusatzverdampfer
2 Aethylen-Säule
3 Rohgas-Unterkühlungs-Gegenströmer
4 Rohgas-Methan-Gegenströmer
4a Rohgas-Äthan-Warmer Ast
5 C_2H_4+C_2H_6 Verdampf.-Gegenströmer
5a Rohgas-Äthan-Kalter Ast
6 C_2H_4-Gegenströmer
7 C_2H_4-Unterkühlungs-Gegenströmer
8 C_2H_4-Gegenstromkühler
9 Rohgas-NH_3-Vorkühler
10 Rohgas-Methan-Gegenstromkühler
11 Rohgas-Kompressor 4000 m³/h
12 Aethylen-Kompressor 3810 m³/h
13 Ausblase-Topf
14 Oelabscheider
15 H_2-Gebläse
16 Anwärmer
17 C_2H_6-Verdampfer
17a C_2H_4-Verdampfer
A Rohgas-Filter
B Rohgas-Filter
C Aethylen-Filter
D Aethylen-Filter
E Rohgas-Filter
F Rohgas-Filter

- ▭ Rohgas
- ▭ C_2H_4-Niederdruck
- ▭ C_2H_4 entspannt
- ▭ CH_4
- ▭ C_2H_6 (Restgas)
- ▭ NH_3
- ▭ C_2H_4+C_2H_6
- ▭ Anwärm-H_2
- ▭ Sicherheits-Leitung
- ▭ Ausblase-Leitung

Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Krs. Recklinghausen
Tag 8.7.1946 Name Heisig

Maßst.

Urheberrechtsschutz nach DIN 34

4d

1900. 8. 42. — Passe hier abschneiden — Carl Schleicher & Schüll, Düren

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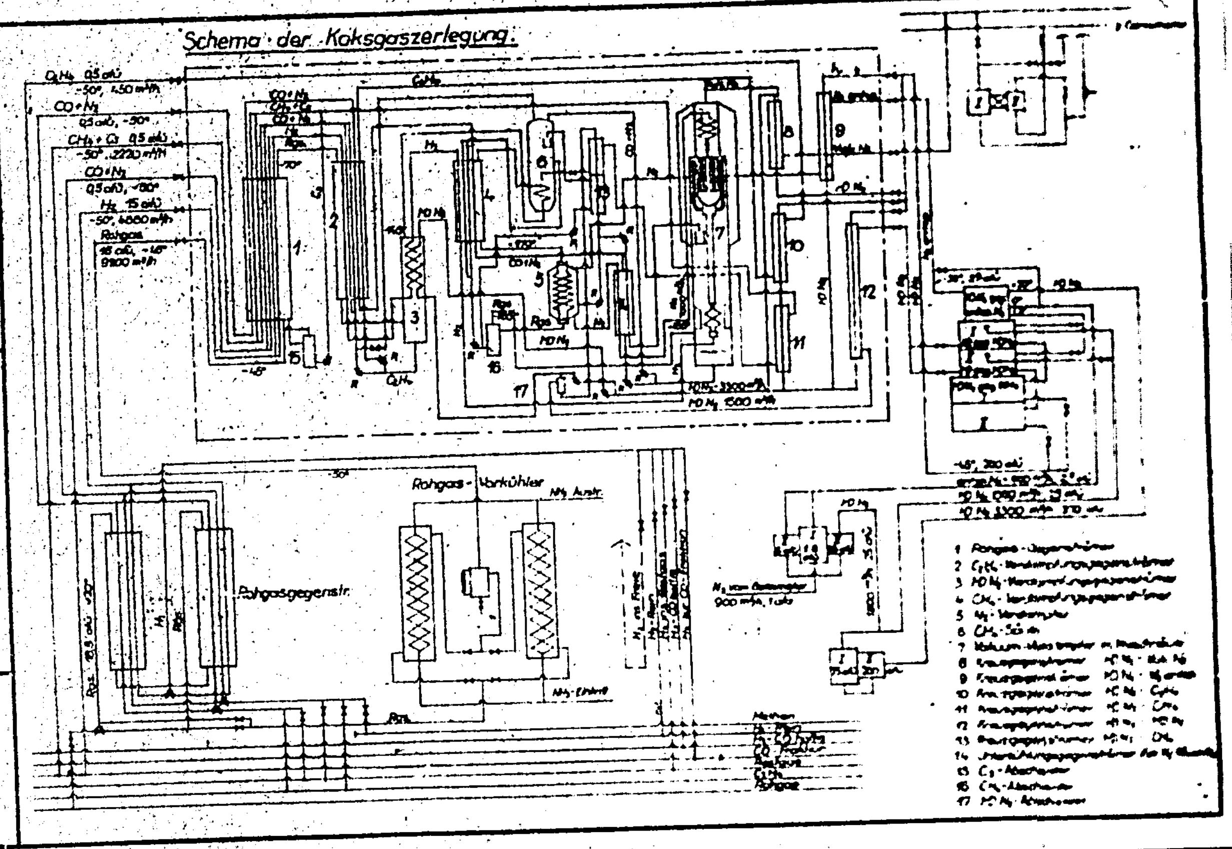
76

Schema der Koksgaszerlegung.



Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Krs. Recklinghausen
Tag 13. 7. 1945 Name Heuig

Maßst.

Urheberrechtsschutz nach DIN 34

4e

Bau .... Betrieb .... Kom. ....

Betriebsbeschreibung des Aldehydbetriebes

1) Aldehyd-Synthese HÜ 441

In der Aldehyd-Synthese wird das von der Tiefkühlanlage 462 gelieferte Acetylen

($97\ \%\ C_2H_2$, $1\ \%\ CO_2$, $2\ \%\ N_2$ und K.W.)

mittels Hg-Kontakt (pro Liter: 0,5 g Hg, 40 g Gesamteisen, 4 g Fe (III), 200 g SO_4 (II), 20 cm³ NO, d_{15} 1,160) zu Acetaldehyd umgesetzt. Der Umsatz beträgt ca. 55 %, die Ausbeute 93 – 95 %. An Nebenprodukten entstehen Essigsäure (ca. 2 %, bezogen auf Aldehyd, läuft in den Kanal) Aceton (0,5 – 0,7 %), Diacetyl und Crotonaldehyd.

In 7 Synthese-Systemen wird Frischgas aus HÜ 462 und Kreislaufacetylen aus den Generatoren (je etwa 50 %) mittels Elmopumpen auf einen Druck von etwa 1,2 atü gebracht. Das Elmowasser wird im Kreislauf geführt. Nach Durchströmen von als Wasserabscheider dienenden Windkesseln wird das Gas mit Dampf auf 90° aufgewärmt und unten in die Generatoren (gummierte Türme) eingeleitet. In den Generatoren, die mit der Kontaktlösung gefüllt sind, bildet sich bei 97° Acetaldehyd.

Aus den Generatoren geht oben unter einem Druck von 100 – 200 mm Quecksilber (der Druckverlust von 1,2 atü auf diesen Druck entspricht der Flüssigkeitssäule im Generator) ein Gemisch von Wasserdampf, Acetaldehyd und unverändertem Acetylen ab. In diesem Gemisch wird in V4A – berohrten Kondensatoren bei 85° der größte Teil des Wasserdampfes kondensiert. Das Kondensat läuft den Generatoren wieder zu.

Nach den Kondensatoren geht das Gas über Zyklone, um mitgerissenes Quecksilber abzuscheiden. Das abgeschiedene Quecksilber läuft mit zugesetztem frischem Quecksilber den Generatoren zu.

Nach den Zyklonen wird das Gas in V2A – berohrten Gaskühlern auf 60° abgekühlt. Der sich kondensierende wässrige Aldehyd läuft ins Tanklager HÜ 449, abgeschiedenes Quecksilber in die Generatoren zurück.

Nach den Gaskühlern wird das Gas in den sogenannten Essigsäurewaschern (gummierte Raschig-Ring-Türme) zur weiteren Quecksilberabscheidung mit in den nachfolgenden Aldehydwaschern gebildetem wässrigem Aldehyd berieselt. Dieser wässrige Aldehyd läuft in das Tanklager HÜ 449, das abgeschiedene Quecksilber in die Generatoren zurück.

Hinter den Essigsäurewaschern wird das Gas in den Aldehydwaschern (eiserne Tunnelbödenkolonnen) mit aus der Acetylenrückgewinnung gelieferten acetylenhaltigem Trinkwasser berieselt. Der dampfförmige Acetaldehyd wird aus dem Gase ausgewaschen, während gleichzeitig das Acetylen aus dem Wasser ausgegast wird. Der wässrige Aldehyd läuft gemeinsam mit dem in den Gaskühlern und Essigsäurewaschern gebildeten über 6 Koksfilter, die zur weiteren Quecksilberabscheidung dienen, und von denen jeweils 2 oder 3 in Betrieb sind, in das Tanklager HÜ 449. Das aus den Aldehydwaschern abziehende Gas geht als Kreislaufacetylen auf die Kimos zurück, nachdem vorher ein Teil als Restgas entnommen worden ist, welches in die Acetylenrückgewinnung HÜ 455 geht.

Die verbrauchte Säure wird aus den Generatoren über Spitzbehälter abgezogen. Sie läuft in einer Sammelleitung über einen Abscheider den sogenannten Druckminderer (gummierter Spitzkessel) auf 2 Säureausgaser (gummierte Raschig-Ring-Türme), von denen jeweils einer in Betrieb ist, in denen mit Dampf bei ca. 100° der in der Säure gelöste Acetaldehyd ausgetrieben wird. Die ablaufende Säure geht nach HÜ 346, das abgehende Dampf- und Acetaldehydgemisch wird kondensiert und dem wässrigen Aldehyd zugegeben.

2) Aldehyd-Destillation HÜ 441

In der Destillation wird der in der Synthese erzeugte wässrige Aldehyd (ca. 7%ig) in 4 Kolonnen auf Reinaldehyd verarbeitet. Daneben werden die im wässrigen Aldehyd vorhandenen Nebenprodukte (Aceton, Diacetyl, Crotonaldehyd) als sogenannter Seitenstrom gewonnen und in entsprechenden Kolonnen aufgearbeitet.

Der vom Tanklager HÜ 449 heraufgepumpte wässrige Aldehyd durchläuft zunächst V2A - Wärmeaustauscher, wo er mit dem Sumpfablauf der Kolonnen auf ungefähr 100° aufgewärmt wird. Mit dieser Temperatur tritt er etwa in der Mitte der Hauptkolonnen ein.

Die Hauptkolonnen bestehen aus dem Sumpf aus V2A, 4 V2A Schüssen zu je 4 Böden, 2 Raschig-Ring Schüssen mit je 1 Meter Raschigringen (dazwischen ist der Einlauf angeordnet), nochmal 1 V2A Schuß zu 4 Böden und einem eisernen Schuß zu 8 Böden. Die Beheizung der Kolonnen erfolgt durch Niederdruckdampf, der unten direkt eingeblasen wird. Die Destillation erfolgt unter 2 atü, die Sicherheitsventile sind auf 2,2 atü eingestellt.

Der aus den Kolonnen oben abziehende dampfförmige Aldehyd wird in eisernen Kondensatoren kondensiert. Der flüssige Aldehyd läuft in eiserne Verteilergefäße, in denen mittels Loch-Scheiben die Verteilung auf Rücklauf und Produktion (Rücklaufverhältnis 1 : 3) erfolgt. Die Produktion geht über Quecksilber-Abscheider zu den Acetylenausgasern (Eiserne Glockenbödenkolonnen mit 18 Böden). in denen der Aldehyd mittels Umlaufverdampfern zum Sieden erhitzt wird, wobei das im Aldehyd gelöste Acetylen entweicht. Ein Teil des Aldehyds verdampft und geht zurück auf die Kondensatoren. Aus den Acetylenausgasern läuft der Aldehyd über sogenannte Produktkühler (eiserne Nadelkühler) in Vorlagen mit Schwimmreglern, die den Aldehyd ins Tanklager HÜ 449 entspannen.

Aus den Verteilergefäßen wird das Restgas entnommen, welches zur Kondensation des darin vorhandenen Aldehyddampfes eiserne Kühler durchläuft (das Kondensat geht vor den Acetylenausgasern zur Produktion). Es wird dann mittels eines automatischen Druckhalteventils entspannt und in dem sogenannten Hilfswascher (eiserner Raschig-Ring-Turm) durch Berieseln mit Wasser von dem Rest seines Aldehydgehaltes befreit. Das Rieselwasser läuft in die Sammelleitung für wässrigen Aldehyd. Das Restgas des Hilfswaschers geht gemeinsam mit dem Restgas der Synthesen in die Acetylenrückgewinnung HÜ 455.

Vom ersten Boden über dem Einlauf der Hauptkolonnen wird der sogenannte Seitenstrom (bestehend aus Acetaldehyd, Aceton, Diacetyl, Crotonaldehyd und Wasser) abgezogen. Der Acetaldehyd wird daraus in den Aldehydausgasern (Raschig-Ring-Kolonnen aus V2A) durch Einblasen direkten Dampfes ausgetrieben und geht über die Hilfswascher in die Tanks für wässrigen Aldehyd. Die übrigen Bestandteile laufen in den Seitenstrom-Tank I (Eisen). Vom Seitenstrom wird vorerst nur das Aceton aufgearbeitet. In der Aceton-Vorkolonne werden Acetaldehyd und Aceton abdestilliert, während mit dem Sumpfablauf Diacetyl und Crotonaldehyd vorläufig noch in den Kanal gehen. Acetaldehyd und Aceton werden in der Aldehydabtreibkolonne getrennt. Der Acetaldehyd läuft über den Hilfswascher zu den Tanks für wässrigen Aldehyd. Das wässrige Aceton dagegen geht in die Reinaceton-Kolonne, wo es nochmals destilliert wird.

3) Acetylenrückgewinnung HÜ 455

In der Acetylenrückgewinnung HÜ 455 wird das Restgas der Synthese und Destillation (etwa 70 % Acetylengehalt) auf Reinacetylen aufgearbeitet.

Das Restgas geht drucklos durch einen Abscheider und dann über einen Elmo (1,2 atü) zu 3 Dreistufenkompressoren, in denen es in der ersten Stufe auf 4 atü komprimiert wird. Nach Passieren eines Abscheiders durchläuft das Gas den Vorwascher (eiserner Turm mit Raschig-Ringen), in dem es zur Befreiung von Spuren Acetaldehyd mit wenig Wasser berieselt wird. Das Rieselwasser läuft auf einen Aldehydwascher der Synthese. Vom Vorwascher geht das Gas auf den Hauptwascher (eiserner Turm mit Siebböden, 4 atü), wo es mit 0,5 %-iger Natronlauge von Kohlensäure befreit wird. Die Umlauflauge wird vom Hauptwascher auf den Entspannungsturm (aus Eisen) gedrückt, wo das in der Lauge gelöste Acetylen entspannt wird.

Dieses Rückacetylen (96 % C_2H_2, ferner N_2 und K.W.) geht über die Frischgas-Sammelleitung vor die Synthese. Nach einem Abscheider gelangt das im Hauptwascher nicht gelöste Gas (ca. 25 % Acetylen) auf die zweite Stufe des gleichen Kompressors (7 atü) und von da über einen weiteren Abscheider auf den Nachwascher (eiserner Raschig-Ring-Turm), wo durch Berieseln mit viel Wasser das Acetylen aus dem Gas ausgewaschen wird. Das acetylenhaltige Wasser geht auf die Aldehydwascher der Synthese.

Das vom Acetylen zum größten Teil befreite Gas (3 % C_2H_2, ferner N_2 und K.W.) wird mittels eines automatischen Druckhalteventils in die Fackelleitung zum Kraftwerk entspannt.

4) Kontaktsäureregeneration HÜ 346

In der Kontaktsäureregeneration HÜ 346 wird die ablaufende Säure aus der Synthese filtriert und mit Salpetersäure oxydiert und die dabei anfallenden NO-haltigen Gase auf Salpetersäure aufgearbeitet. In einem oppanierten Ansatzbehälter (HÜ 445) wird Kondenswasser, Ferrosulfat und 96 %ige Schwefelsäure (aus 2 eisernen Tanks) gemischt. Die fertige Kontaktsäure wird in den Behälter für filtrierte Säure (gummiert) gepumpt.

Die aus der Synthese kommenae verbrauchte Säure läuft in den sogenannten Schlammsäurebehälter (gummiert) und wird von da mittels Pesi-Pumpen auf die beiden Filterpressen (gummiert) gefördert. Nach den Filterpressen läuft die Säure in den Behälter für filtrierte Säure.

Aus dem Behälter für filtrierte Säure drücken Pesi-Pumpen die Säure über einen Vorwärmer (aus V4A) in 3 Überlaufgefäße (gummiert), von denen sie über Dosierscheiben in die Voroxydierer ein-läuft. Der Rest der Säure fließt aus den Überlaufgefäßen zurück in den Behälter für filtrierte Säure.

In den 6 Voroxydierern (ausgemauerte Behälter) wird das Ferrosulfat der Kontaktsäure mittels 30 %iger Salpetersäure zu Ferrisulfat oxydiert. Je 2 Voroxydierer laufen auf einen Nachoxydierer (ausgemauerte Behälter) ab, in denen die Oxydation zu Ende geführt wird. Zur Vernichtung der letzten Spuren Salpetersäure wird hier noch eine kleine Menge nichtoxydierter Säure zugegeben. Aus den Nachoxydierern läuft die Säure in die Behälter für oxydierte Säure (gummiert), aus denen sie durch Pumpen der Synthese zugeführt wird.

Das gesamte System der Voroxydierer, Nachoxydierer und Säurebehälter wird durch einen Ventilator (aus V2A) bei geringem Unterdruck gehalten. Die abgesaugten stickoxydhaltigen Gase strömen nach Zugabe von Luft durch 5 Absorptionstürme (Raschig-Ring-Türme aus V2A). In diesen Türmen, von denen jeweils 3 hintereinander geschaltet in Betrieb sind, wird mittels Pumpen aus V2A Wasser umgepumpt, das sich langsam mit Salpetersäure anreichert.

Die so erhaltene verdünnte Salpetersäure wird mit 60 %iger Salpetersäure (aus dem Lagerbehälter aus V2A) zu 30 %iger Salpetersäure gemischt. Sie läuft dann in den Lagerbehälter für verdünnte Salpetersäure (aus V2A). Von hier aus wird sie mittels Pumpen über ein Überlaufgefäß (aus V2A) zu den Voroxydierern gefördert. Das Abgas der Absorptionstürme geht über Dach.

Der quecksilberhaltige Schlamm, der in den Filterpressen anfällt, wird mit Natronlauge und Kalk neutralisiert und in einem gemauerten Ofen (Hü 348) mittels Heizgas auf helle Rotglut erhitzt. Ein Ventilator saugt die Verbrennungsgase durch einen indirekten und 2 direkte Kühler ab. Unter den 3 Kühlern befinden sich Abscheider, in denen das destillierte und kondensierte Quecksilber aufgefangen wird.

5) Tanklager Hü 449

Das Tanklager Hü 449 dient zur Aufnahme des in der Synthese erzeugten wässrigen Aldehyds, des Reinaldehyds und der Nebenprodukte der Aldehyddestillation. Für den Reinaldehyd stehen die eisernen Tanks 1 - 8 mit je 70 m³, für den wässrigen Aldehyd die eisernen Tanks 9 und 10 mit je 300 m³ und für die Nebenprodukte die eisernen Tanks 11 - 14 mit je 30 m³ zur Verfügung.

6) Hafentanklager 9001

Im Hafentanklager 9001 stehen 14 Reinaldehydtanks zu je 70 m³ zur Aufnahme von auf dem Lippekanal ankommendem und abgehendem Aldehyd und als allgemeine Lagerreserve zur Verfügung.

Betriebsbeschreibung des Aldehydbetriebes
([illegible])

A) Korrosionen und Gegenmaßnahmen:

1) Kontaktsäure enthält 1 % Schwefelsäure.

Gegenmaßnahmen: Gummierte Eisenrohre und Apparate; ferner V4A-Rohre und Apparate; dann gemauerte Eisenbehälter.

2) 30- und 53%ige Salpetersäure.

Gegenmaßnahmen: Apparate aus V2A, Rohrleitungen aus V17P extra.

3) Wässeriger Acetaldehyd enthält etwa 0,1 % Essigsäure und geringe Mengen Crotonaldehyd.

Gegenmaßnahmen: Destillationshaupt- und Seitenstromkolonnen aus V2A.

B) Sicherheitsmaßnahmen:

Außer den üblichen Sicherheitstauchungen und ~~Ventilen [illegible]~~ schnellverschlüsse am Reinacetylengasometer und in der 700er und 500er Acetylenleitung vor Bau 441.

C) Luftschutzmaßnahmen:

1) Aufstellung von Trockenmauern zwischen den Apparaten und Betonhauben über den Motoren.

2) Gegebenenfalls Abriegelung des Betriebes.

3) Aufstellung von 2-Mann-Splitterschutzbunkern für die Notbelegschaft in den Bedienungsständen und im Betriebsgelände.

D) Engpaß des Betriebes ist die Acetylenrückgewinnung. Die max. Flüssigkeitsbelastung für den Nachwäscher beträgt etwa 120 cbm/h Wasser, das entspricht ca. 3 Synthesesystemen.

E) Lebenswichtige Apparate:

Reinacetylengasometer und Säurepumpen (Fesi).

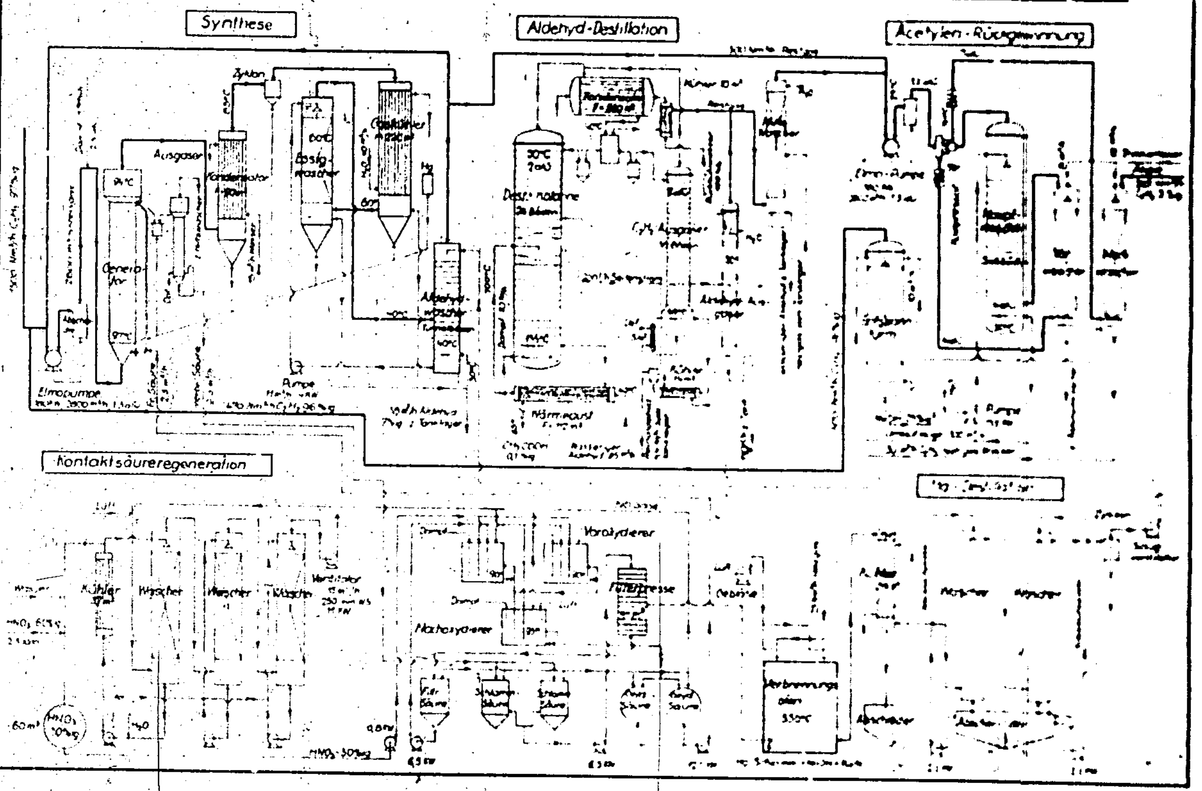
F) In der Acetylenrückgewinnung können Elmo und Hauptwäscher umgangen werden. Das bedeutet allerdings einen Ausbeuteverlust von etwa 10 %.

G) Reserveteile:

3 Generatoren, Apparateteile für 2 Synthesesysteme, 10 % Reserve an gummierten Eisenrohren, Fesi-Rohren, V2A- und V4A-Rohren; ferner etwa 10 % an Säurepumpen.

32

Verfahren zur Erzeugung von Acetaldehyd aus Acetylen mittels Quecksilber-Kontakt als Katalysator



Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Krs. Recklinghausen

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Aktenvermerk!

Betr.: Arbeitsweise der Gleichrichtersteuerung bei den Lichtbogengruppen.

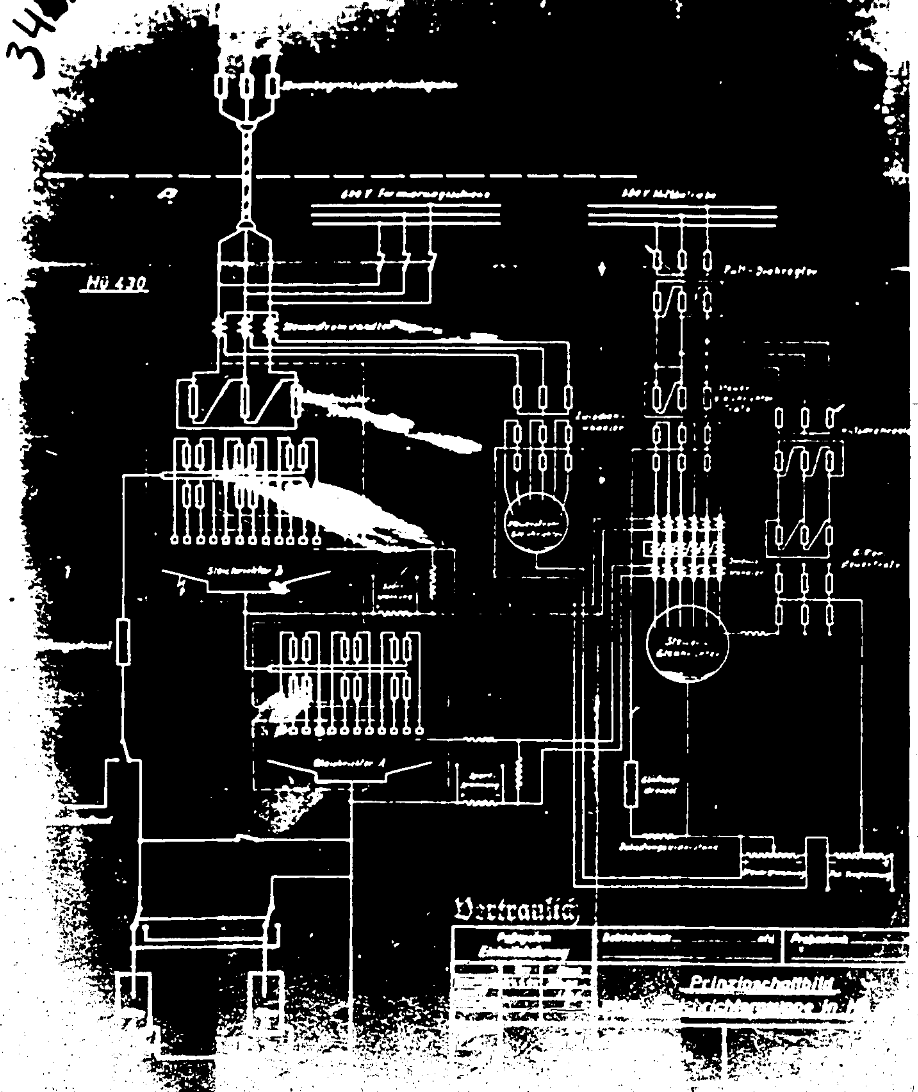
Bei den Lichtbogengleichrichtern muß die Gleichspannung in eine solche Abhängigkeit vom Ofenstrom in dem durch die Widerstandsänderung des Lichtbogens verursachten Bereich der Stromschwankungen gebracht werden, daß ein stabiles brennen des Ofenlichtbogens ermöglicht wird. Diese Aufgabe obliegt der Steuerung, deren Arbeitsweise anhand des Prinzipschaltbildes ES 70130 zusammenfassend dargelegt werden soll.

Steuerung der BBC-Gleichrichtergruppen.

Der dem Gleichstrom proportionale Drehstrom der Gruppe wird über Steuerstromwandler und Zwischenwandler in einen Steuerstrom übersetzt, der nach seiner Gleichrichtung im Steuerstromgleichrichter einen dem Ofenstrom proportionalen Spannungsabfall am Steuerwiderstand erzeugt. Diese Steuerspannung liegt in Reihe mit einer bezüglich der Polarität entgegengeschalteten, konstanten positiven Vorspannung und sechs um je 60° verschobenen sinusförmigen wechselspannungen, die vom Gittersteuertransformator geliefert werden, an den Gittern des Steuergleichrichters. Im Betriebspunkt (850 Amp. Gleichstrom) sind Steuerspannung und positive Vorspannung annähernd gleich, sodaß die Gitter des Steuergleichrichters nur mit den wechselspannungen des Gittersteuertransformators beaufschlagt werden. Beim Übergang dieser Wechselspannungen in die positive Halbwelle zünden die betreffenden Anoden des Steuergleichrichters mit einem steilen Stromstoß, da der Steuergleichrichter nur schwach ausgesteuert ist (Zündwinkel etwa 50°). Diese Stromstöße werden über die Impulswandler durch die Ableitwiderstände geführt, an denen die Impulse für die Steuergitter der Hauptgleichrichter abgegriffen sind. Bei sinkendem Ofenstrom überwiegt die positive Vorspannung gegenüber der Steuerspannung und hebt dadurch die an den Gittern des Steuergleichrichters liegenden Wechselspannungen um einen dementsprechenden Betrag gegenüber der Kathode. Die hierdurch bewirkte Verlegung des Zündzeitpunktes in voreilendem Sinne verschiebt die Zündstromstöße der Anoden des Steuergleichrichters und damit auch die Gitterimpulse der Hauptgleichrichter im Sinne einer Vergrösserung der Gleichspannung.

gez. Grunert.

34



Vertraulich

Prinzipschaltbild

2.) Steuerung der SSW-Gleichrichtergruppe

Der wiederum über Steuerstromwandler und Zwischenwandler übersetzte Steuerstrom erzeugt nach seiner Gleichrichtung im Steuerwiderstand einen Gleichspannungsabfall, der einer mit dem Betriebsregler abgegriffenen konstanten Gleichspannung entgegengeschaltet ist. Beide Spannungen sind so gewählt, dass am Gitter der Eingangsröhre des Verstärkers eine positive Spannung liegt, die in einem genügend grossen Bereich der Betriebsstromstärke eine Vergrößerung des Verstärker-Ausgangsstromes bei sinkendem Betriebsstrom bewirkt.

Der Ausgangsstrom des Verstärkers durchfliesst eine Wicklung des Gittertransformators und bewirkt dadurch eine Gleichstromvormagnetisierung. Dem hochgesättigten Gittertransformator wird durch Vorschaltung schwachgesättigter Vordresselspulen ein Dreieckförmiger Magnetisierungsstrom aufgezwungen, der bei seinen Nulldurchgängen spitze Spannungsimpulse in die Wicklungen induziert, deren Sekundärseite an die Gitter des Steuergleichrichters geführt ist. Mit der Grösse des Verstarker-Ausgangsstromes ändert sich die Vormagnetisierung, sodass die Spannungsimpulse dementsprechend zeitlich varschoben werden. Über eine weitere Wicklung ist der Gittertransformator durch einen konstanten Gleichstrom so vormagnetisiart, dass in dem möglichen Änderungsbereich des Verstärkerstromes die Spannungsimpulse um 100° zeitlich verschiebbar sind.

Zwischen den Anoden und der Kathode des Steuergleichrichters liegen Kondensatoren, die, uber Trockengleichrichter aufgeladen, im Zündaugenblick Entladestromstösse über den Steuergleichrichter zum Fliessen bringen, die über die Stromstosswandler wiederum an die Gitter der Hauptgefasse galangen. Bei sinkendem Ofenstrom bewirkt somit die Vergrosserung des Verstarker-Ausgangsstromes eine Verschiebung der Gitterimpulse für den Steuergleichrichter in dem Sinne, dass die Entladestromstösse zeitlich früher erfolgen, und damit die Hauptgefasse höher ausgesteuert werden.

Die Anwendung der Kondensatoren zur Erzeugung von Stromstössen hat den Vorzug, dass diese Stromstösse über einen grossen Bereich bei gleichbleibender Höhe verschoben werden können, sodass die Ofenspannung mit der Steuerung bis Null kontinuierlich geregelt werden kann.

Beim Anfahren des Lichtbogenofens wird über den Anfahrregler eine Gleichspannung zusätzlich in den Gitterkreis der Eingangsröhre gebracht, die , da im Steuerwiderstand zunächst noch kein Strom fliesst, im gleichen Sinne wie die Steuerspannung wirkend dem Verstärker einen hohen Gleichstrom vortäuscht, wodurch der Ausgangsstrom des Verstärkers soweit verkleinert wird, dass die Gleichspannung der Gruppe auf Null heruntergesteuert wird.

Nach dem Kurzschliessen des Ofens durch die Zündstange wird der Anfahrregler wieder herausgedreht, sodass mit zunehmender Aussteuerung der Ofenkurzschlussstrom von Null aus bis zu einem Stromwert ansteigt, der mit dem Betriebsregler eingestellt werden kann und unabhängig von dem weiteren Herausdrehen des Anfahrreglers von der nunmehr verhandenen Steuerspannung gehaltern wird. Bei Erreichen der Endstellung des Anfahrreglers wird die Zundstange selbsttätig geöffnet, woraufhin die Steuerung den Ofenstrom in den mittels Betriebsregler einstellbaren Betriebswert regelt.

Ein weiterer Einfluss im Gitterkreis der Verstärker-Eingangsröhre von der Ofenspannung her kann noch wirksam gemacht werden; er ist jedoch nicht unbedingt erforderlich und bei dem derzeitigem Betrieb ausgeschaltet.

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FIAT FINAL REPORT 925

FORMAMIDE PRODUCTION
AT THE I.G. FARBENINDUSTRIE PLANT
AT OPPAU

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OFFICE OF MILITARY GOVERNMENT FOR GERMANY (US)

FIAT FINAL REPORT NO. 925   30 August 1946

FORMAMIDE PRODUCTION
AT THE I.G. FARBENINDUSTRIE PLANT
AT OPPAU

BY

WILLIAM L.E. DEWLING

TECHNICAL INDUSTRIAL INTELLIGENCE DIVISION

U.S. DEPARTMENT OF COMMERCE

THIS REPORT IS ISSUED WITH THE WARNING THAT IF THE SUBJECT MATTER SHOULD BE PROTECTED BY U.S. PATENTS OR PATENT APPLICATION, THIS PUBLICATION CANNOT BE HELD TO GIVE ANY PROTECTION AGAINST ACTION FOR INFRINGEMENT.

FIELD INFORMATION AGENCY, TECHNICAL

ABSTRACT

Reviews formamide manufacture at the Oppau Plant of I.G. Farbenindustrie and gives information on process and equipment.

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Reviews formamide manufacture at the Oppau Plant of I.G. Farbenindustrie and gives information on process and equipment.

TABLE OF CONTENTS

INTRODUCTION

Objective:

The purpose of the investigation was to obtain information on formamide manufacture at the Oppau Plant of the I.G. Farbenindustrie.

Evaluation:

The Oppau formamide plant is said to be the only plant in Germany for producing formamide from the interaction of methanol, carbon monoxide and ammonia. Process and design features incorporated in the Oppau formamide plant are considered of particular interest. Formamide produced at Oppau is normally sent to Ludwigshafen to be dehydrated to yield hydrocyanic acid. The normal capacity of the plant is 30 metric tons per day.

Guide to the Reader:

The reader is referred to Appendix 2 for a list of previous reports on this subject.

GENERAL

Formamide is produced at Oppau, in a batchwise manner, by the interaction of methanol, carbon monoxide and ammonia as follows:

a. Methanol and carbon monoxide are reacted, in the presence of a catalyst, to form methyl formate according to the following reaction:

$$CH_3OH + CO \longrightarrow HCO_2CH_3$$

b. Methyl formate is reacted with liquid or gaseous ammonia to produce formamide as follows:

$$HCO_2CH_3 + NH_3 \longrightarrow HCONH_2 + CH_3OH$$

The methanol and unreacted ammonia are recovered and returned to the system.

PROCESS DESCRIPTION

Reference is made to Figure 1 which presents a process diagram for the formamide plant at Oppau. For purposes of description the process is divided into the following steps:

Methyl formate synthesis
Methyl formate recovery
Conversion to formamide
Product recovery and finishing
Methanol and ammonia separation
Recovery of residual methanol, ammonia and methyl formate

Methyl Formate Synthesis:

For preparing the sodium methylate catalyst used for methyl formate synthesis, metallic sodium in small pieces is added to methanol in catalyst mixing tank 1, the sodium reacting with methanol to form sodium methylate. One percent by weight of metallic sodium is added to the methanol which must not contain over 0.01% water. Hydrogen liberated in the mixing tank is cooled and sent to the Oppau ammonia plant. The use of metallic sodium is considered necessary in order to remove the last traces of water from the methanol.

The methyl formate autoclave 4 is charged from catalyst mixing tank 1 by means of methanol pump 2, and the charge is heated to 60°C. Carbon monoxide gas is then injected at the base of the autoclave and the pressure in the autoclave is gradually increased. Conversion to methyl formate begins at about 30 atm. and 60°C, and cooling is then required to maintain the temperature of the batch between 90 - 110°C. At higher temperatures decomposition takes place and at lower temperatures the conversion efficiency is low. As the reaction proceeds, the pressure in the autoclave is gradually increased until a pressure of 200 atm. is reached. The completion of the reaction is indicated by a drop in temperature of the reactants.

When the reaction is completed, the batch is cooled to 30°C. and the pressure in the autoclave is then reduced to 70 atm. The finished batch is discharged into formate solution storage tank 5 which operates at a pressure of 0.5 atm.(ga). Gases released from the batch due to the reduction in pressure are vented to the atmosphere from the formate solution storage tank. Approximately 4 hours are required for the completion of one batch, including filling and emptying operations. About 82% of the methanol is converted to methyl formate.

The carbon monoxide gas used is received from the CO removal system of the Oppau ammonia plant. This gas, containing 97 - 98% CO, is scrubbed with ammoniacal liquor in order to reduce the CO_2 contents to less than 0.005%. When the carbon monoxide gas contains CO_2 in excess of this value, or the methanol contains water in excess of 0.01%, the yield of methyl formate is greatly decreased. From 75 - 80% of the CO injected into the methyl formate autoclave is utilized. Unreacted gas vented from the autoclave is sent to the CO conversion system of the Oppau ammonia plant. This gas contains 1 - 2 gms. per cu.m. of methyl formate plus methanol.

Methyl Formate Recovery

Solution from methyl formate storage tank 5, containing about 90% methyl formate, is fed to one of the formate boilers 8, there being 4 of these boilers provided. The methyl formate vaporized from this unit, containing from 5-10% methanol, passes through the formate column 9, is condensed in formate condenser 10 and is then sent to the formate storage tank 11. Normally, a reflux is not required on the formate column as the presence of a small amount of methanol is desirable in the final reaction to formamide. If, however, pure methyl formate is desired for other processes it can be obtained by refluxing the formate column. The formate boiler operates at 50 - 60°C., and 0.5 atm.(ga).

When one of the formate boilers becomes 2/3 filled with methanol, the feed to it is stopped and it is disconnected from the formate column. Methanol is then evaporated from the disconnected boiler, first at atmospheric pressure, and then under a vacuum of 400 - 500 mm. of Hg. in order to recover the methanol from the precipitated sodium methylate catalyst. The recovered methanol is liquefied in methanol condenser 7 and returned to catalyst mixing tank 1. The sodium methylate catalyst removed from the boiler is normally wasted as it has very poor catalytic properties when re-used.

Conversion to Formamide

Methyl formate solution is charged to formamide autoclave 13 which operates in a batchwise manner. The charge is first heated to a temperature of 40°C., and then ammonia is added to the charge at a pressure of 10 - 15 atm. The reaction is exothermic and cooling is necessary to maintain a temperature of 60 - 70°C. Approximately 3% excess of ammonia over the stoichiometric amount required is injected into the autoclave in order to increase the percentage of methyl formate converted to formamide A conversion of methyl formate to formamide of 98% is claimed.

The total time required for each batch is one hour, which includes the time required for charging and discharging the autoclave. The reaction time was said to be only 20 minutes. When the reaction is completed, the batch is cooled to 20°C., and is discharged under the pressure existing in the autoclave to formamide solution storage 14.

Product Recovery and Finishing

Crude formamide solution containing 47% formamide, 50% methanol, 3% ammonia and traces of methyl formate is continuously fed from formamide solution storage 14 to formamide fractionator boiler 15, which is provided with formamide fractionator column 16 and a partial condenser for refluxing. The major portion of the methanol and ammonia are remov from the formamide solution in this equipment. The boiler operates at 130 - 135°C. and the temperature at the top of the column is 60 - 70°C. The normal operating pressure is 0.5 atm. (ga). Ammonia and methanol removed from the formamide in this step are recovered as described later in this report.

Bottoms from the formamide fractionator boiler, containing about 1% methanol and traces of ammonia and methyl formate are fed to a fractionating unit consisting of, recovery boiler 28, recovery column 29, and recovery reflux condenser 30. In this equipment the major portion of the residual methanol, ammonia and methyl formate are removed. This separation is accomplished under a vacuum of 600 – 700 mm. of Hg. The recovery boiler operates at 115°C., and the temperature at the top of the recovery column is 35°C. The recovery of the ammonia, methanol and methyl formate removed are described later in this report.

Formamide from the recovery boiler 28 is cooled and pumped through filter 36 in which oil is removed. The final product has the following approximate composition, 99.7% formamide, 0.1% methanol and 0.2% ammonium formate, the later impurity resulting from traces of water entering the system with the ammonia feed.

Methanol and Ammonia Separation

Methanol and ammonia vapors leaving the partial condenser 17 are condensed in final condenser 18 and are fed to a fractionating unit consisting of ammonia fractionator boiler 19, ammonia fractionator column 20, and ammonia partial condenser 21. The boiler operates at 55 – 60°C. and 0.5 atm. (ga), the temperature at the top of the column is 30 – 40°C. Ammonia vapor leaving the partial condenser 21 is cooled in ammonia gas coolers 22 and 23, compressed in ammonia compressors 24 to 15 atm, and returned to the formamide autoclave 13.

Methanol from the bottom of the ammonia fractionator boiler 19, containing traces of ammonia and 1 – 2% formamide, is fed to methanol boiler 25 which operates at a pressure of 0.5 atm. (ga) and a temperature of about 65°C. Vaporized methanol from methanol boiler 25 is condensed in methanol condenser 27 and is returned to the catalyst mixing tank 1. This methanol is said to be free of water and contains 0.01% – 0.02% ammonia. Bottoms from the methanol boiler, containing traces of formamide in methanol, are sent to recovery return storage 31.

Recovery of Residual Methanol, Ammonia and Methyl Formate

Methanol and formamide condensed in recovery reflux condenser 30 is sent to recovery return storage 31. Recovered solution is returned from this storage to formate solution storage 14. Uncondensed gases from recovery reflux condenser 30 are boosted in pressure by recovery vacuum pump 32, to about atmospheric pressure. These gases are then scrubbed with formamide in absorber 33 to recover the last traces of methanol, ammonia, and methyl formate. The formamide used for scrubbing must be replaced with pure formamide when its sp. gr. has dropped to 1.0 (the sp. gr. of pure formamide being 1.14). Spent solution is sent to the recovery

return storage 31.

Description of Equipment

According to Dr. M. Günther, a large portion of the equipment now used at Oppau for formamide production was originally designed for other uses. Drawings for this equipment are not available at Oppau.

The following description for the important pieces of equipment is based on information given from memory by Dr. Günther. The capacity of the plant, containing this equipment, is 30 metric tons per day.

Item 3. 3 CO Compressers – 4 stage, reciprocating-type, provided with interstage coolers. Each compresser has a capacity of 1100 cu.m. per hour measured at suction conditions. The maximum discharge pressure is 200 atm.

Item 4. 3 Methyl formate autoclaves – Vertical, high pressure vessels, 800 mm. I.D. x 6 m. high. These units are provided with loose liners of stainless steel, type V2A (18% Cr, 8–9% Ni, 0.1%C., and trace of Ti), and jackets for heating steam or cooling water. Each autoclave has sufficient capacity, per batch, for 1 ton of final product. Maximum working pressure, 200 atm.

Item 4. 1 Methyl formate autoclave – Same as above with the exception that this unit has a height of 12 m. and twice the capacity.

Item 8. 4 Formate boilers – Kettle type boilers, provided with steam heating cools. The capacity of each boiler is 10 cu.m. Steel construction.

Item 9. 1 Formate column – Vertical packed tower, 1 m. I.D. x 10 m. high. This unit is constructed of steel and is packed with 50 mm. x 50 mm. porcelain Raschig rings.

Item 11 3 Formate storage tanks – Horizontal vessels of steel, each having a capacity of 17 cu.m. Steel construction.

Item 13. 2 Formamide autoclaves – Vertical, high pressure vessels, 800 mm. I.D. x 6 m. high. These vessels are constructed of stainless steel-clad plate. The stainless steel is type V2A (18% Cr, 8–9% Ni, 0.1C., and trace of Ti). Jackets are provided for heating steam or cooling water. The normal maximum working pressure is 15 atm.

Item 14. 1 Formamide solution storage – Horizontal tank lined with rubber. Capacity 10 cu.m.

Item 15. 1 Formamide fractionator boiler - Vertical, tray-type fractionating boiler. This unit contains 5 shallow trays each tray 1.2 m. in diameter x 0.2 m. deep. The trays are constructed of silumin (12 - 13% Si and 87 to 88% Al) and each is provided with a silver heating coil.

Item 16. Formamide fractionator column - Vertical, packed tower, 1.1 m. I.D. x 10 m. high. This unit is constructed of aluminum and is packed with 30 mm. x 30 mm. aluminum Raschig rings.

Item 20. 1 Ammonia Fractionator column - Vertical, packed tower, 1 m. I.D. x 12 m. high. This unit is constructed of steel and is packed with 50 mm. x 50 mm. steel Raschig rings.

Item 24. 2 Ammonia compressors - 2 stage, reciprocating type. Capacity each 100 cu. m. per hour measured at suction conditions. Normal discharge pressure 12 to 15 atm.

Item 28. 1 Recovery boiler - same as the formamide fractionator boiler except that only 3 trays are provided.

Item 29. 1 Recovery column - Vertical, packed tower, 900 mm. I.D. x 8 m. high. This unit is constructed of aluminum and is packed with 30 mm. x 30 mm. aluminum Raschig rings.

Item 30. 1 Recovery reflux condenser - Double-pipe condenser containing approximately 5 sq.m. of cooling surface. The inside pipes, carrying the condensing vapors are constructed of V2A steel.

Item 33. 1 Absorber - Vertical, packed tower, 500 mm. I.D. x 8 m. high. This unit is constructed of steel and is packed with 30 mm. x 30 mm. steel Raschig rings.

Item 36. 1 Filter press - Seitz wine filter with asbestos mats. This unit is constructed of steel. Inside surfaces are tinned.

The following equipment, not described above, is constructed of aluminum. Items 17, 31, 35 and 37.

YIELDS AND UTILITIES

It is claimed that 82% of the methanol fed to the methyl formate autoclave is converted to methyl formate, and that about 98% of the methyl formate fed to the formamide autoclaves is converted to formamide. Losses of methanol and ammonia from the system were said to be very small. From 75 to 80% of the CO fed to the methyl formate autoclave is reacted.

For a production of 30 tons of formamide per day, the following

utilities are required per metric ton of formamide:

Electricity	260-270 KWH
High pressure steam (12 atm. ga.)	1.7 tons
Low pressure steam (1.5 atm. ga.)	2.5 tons
Cooling water	400 cu.m.
Operating labor	8 man hours

APPENDIX 1

LIST OF GERMAN PERSONNEL INTERVIEWED

Name	Position	Location
Dr. M. Günther	Formamide Plant Superintendent	Oppau

APPENDIX 2

BIBLIOGRAPHY

Copies of the reports listed below were transmitted to Washington, D.C. Inquiries should be addressed to:

Office of Publications Board,
U.S. Department of Commerce,
Washington 25, D.C.

1. Related Reports Published by Allied Intelligence Agencies:

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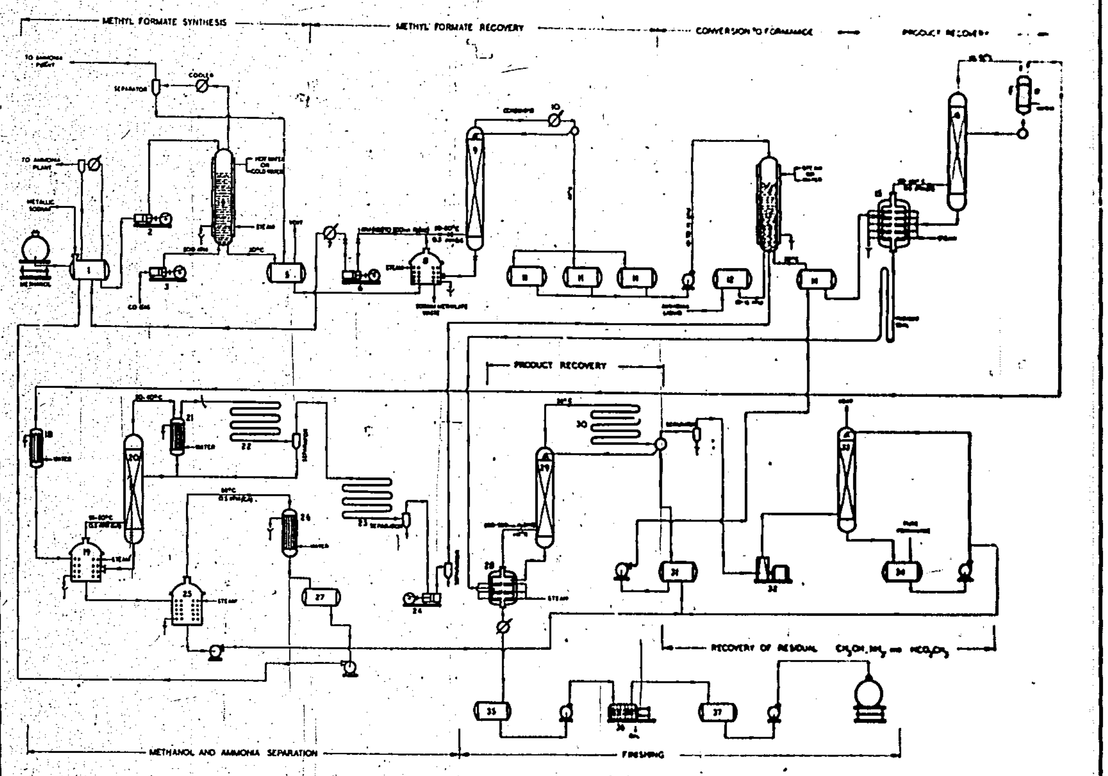
CIOS Report, Item No. 22, File No. XXXII-64: "Manufacture of Hydrocanic Acid, I.G. Farbenindustrie, Oppau", located at FIAT Records Branch, File TP 200/P632.



1	CATALYST MIXING TANK	14	FORMAMIDE SOLUTION STORAGE	27	METHANOL STORAGE TANK
2	METHANOL PUMP	15	FORMAMIDE FRACTIONATOR BOILER	28	RECOVERY BOILER
3	CO COMPRESSOR	16	FORMAMIDE FRACTIONATOR COLUMN	29	RECOVERY COLUMN
4	METHYL FORMATE AUTOCLAVE	17	PARTIAL CONDENSER	30	RECOVERY REFLUX CONDENSER
5	FORMATE SOLUTION STORAGE TANK	18	FINAL CONDENSER	31	RECOVERY RETURN STORAGE
6	METHANOL VACUUM PUMP	19	AMMONIA FRACTIONATOR BOILER	32	RECOVERY VACUUM PUMP
7	METHANOL CONDENSER	20	AMMONIA FRACTIONATOR COLUMN	33	ABSORBER
8	FORMATE BOILER	21	AMMONIA PARTIAL CONDENSER	34	ABSORBER STORAGE TANK
9	FORMATE COLUMN	22	AMMONIA GAS COOLER	35	RAW PRODUCT STORAGE
10	FORMATE CONDENSER	23	AMMONIA GAS COOLER	36	FILTER
11	FORMATE STORAGE TANKS	24	AMMONIA COMPRESSOR	37	FINAL PRODUCT STORAGE
12	AMMONIA STORAGE TANK	25	METHANOL BOILER		
13	FORMAMIDE AUTOCLAVE	26	METHANOL CONDENSER		

FORMAMIDE MANUFACTURE

FIAT FINAL REPORT NO [illegible]

PROCESS DIAGRAM

DATE AUG 23 1946

FIG. 1

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COAL PREPARATION PRACTICE IN WESTERN GERMANY

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FIAT FINAL REPORT NO. 634 7 March 1946

COAL PREPARATION PRACTICE IN WESTERN GERMANY

BY

Thomas FRASER
M.G. DRIESSEN

Joint Intelligence Objectives Agency

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ABSTRACT

Reviews German practice in coal preparation. Describes the differences in equipment for de-dusting, flash drying, conveyor drying, resonance screening, froth breaking and anti-breakage loading. Also describes coal purifying by multiple-stage froth flotation, heavy suspension separation, electrostatic separation, acid extraction of ash minerals, and tar oil extraction of coal matter.

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I. Introduction

II. Summary

III. General Appraisal of the Coal Preparation Industry

IV. Organizations of Coal Preparation Service

Subject **Page No.**

V. **Economic Factors**

VI. **Field Data on Plants**

VII. **Devices and Techniques**

Unless otherwise specified all measurements in this report are expressed in metric units. Sieve sizes are in meshes per square centimeter.

Metric ton	2204.6 lbs.	cubic meter	35.3144 cu. ft.
Kilo	2.2046 lbs.	cubic meter	264.17 gallons
Meter	3.2407 ft.	liter	1.0567 qt.
Centimeter	.3937 in.	400 mesh	50 per lineal in.
Kilometer	.62137 mile	10000 mesh	250 per " " .

COAL PREPARATION PRACTICE IN WESTERN GERMANY

I - INTRODUCTION

1. Objectives

A study of coal preparation practices in western Germany was undertaken, first, to discover any technological or organizational developments that might be of value to the American coal industry; second, to discover any special devices or techniques that may have been developed in Germany to prepare special grades of coal to meet emergency war needs; and third, to ascertain the grades of fuel and methods of preparation thereof that were particularly adapted to the synthetic liquid fuels industry with a view to profiting by German experience in the selection and preparation of American coals for conversion to liquid fuels.

2. Method and Scope of Investigation

To facilitate rapid and thorough coverage of technological developments in the field, a preliminary survey contacted the equipment manufacturers and plant builders who service the coal industry. This is a very small group of highly specialized firms that dominates the coal preparation plant development and construction field. Through the engineering staffs of these firms and the specialists of the Bergbauverein now in the employ of the North German Coal Control Agency at Heisingen, Essen, contact was established with operating plants embodying new developments of promise, so that further field work could be concentrated on these plants. These plants were studied usually in company with technical representatives of the designers and builders.

II - SUMMARY

3. General Practice

The coal preparation industry in Germany is organized very much as in America, in that the erection of new plants and extensive alterations are generally handled on contract by specialized equipment manufacturers who also control very largely the exploitation of new technologic developments.

The dominant washing process is the jig which handles 87 to 88% of all the coal washed in the Ruhr area. General plant engineering and design does not appear to be as well developed as in American and British practice. Compared to our conditions at least, the plants are very large and over-staffed in relation to the through-put. However, there are some items of individual devices and techniques that may be adaptable to

V. Economic Factors

VI. Field Data on Plants

VII. Devices and Techniques

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American practice. Fine coal jigs, both with and without feldspar, are used almost to the exclusion of other methods for coal under 8 mm. or 10 mm. except that dust under 0.75 mm. is commonly handled by froth flotation.

In the preferred plant design, the 10 mm. x 0 is dedusted before washing. This appears to have great advantages in the subsequent dewatering of the coal and clarification of the water.

4. Special Techniques and Devices

Mechanical developments that may be in part new include dedusting devices, flash dryers, conveyor dryers, the resonance screens, froth breakers, and anti-breakage loading devices.

In the field of special preparation for exacting war uses, the Germans devised several intensive coal purifying methods embracing (1) multiple stage froth flotation, (2) controlled heavy suspension separation, (3) electrostatic separation, (4) acid extraction of the ash minerals, and (5) tar oil extraction of the coal matter.

These methods were applied with widely varying measures of success to produce very low ash coal to serve as a substitute for other carbon materials not available to the Germans. Some of these processes might conceivably be of use to us ultimately for production of low ash coal for such use as electrodes for the aluminum industry, for carbide and carborundum industries, for sugar refinery carbon, and activated carbon.

III - GENERAL APPRAISAL OF THE COAL PREPARATION INDUSTRY

5. General Preparation Practice

The jig washery is the predominant plant for the preparation of general industrial coal. Both air actuated and mechanically actuated jigs are in wide use in current practice. The piston jig box of Coppee type is much more popular in current design than in America, where the mechanical jig has generally been considered obsolete, except in the anthracite region, since the introduction of the Baum type in the early thirties.

Probably the most striking feature of German practice (and continental practice generally), contrasted with current American practice, is the wide use of jig wash boxes for fine coal. It is standard practice to wash the fine coal of 10 mm. x 0 or 8 mm. x 0 separately on multiple compartment piston jig boxes, in some instances with feldspar on the screens of the primary washers but generally with a natural slate bed on the rewash boxes. The practice of rewashing the middlings from the fine coal jigs on rewash jigs is general. In many instances the middlings from the nut coal jigs is crushed and rewashed with the fine coal middlings or added raw to the fine washed coal. Where that is used for colliery power plant or other local use, the crushed coarse middlings are diverted to that use.

In current design, the leading plant builders favor thorough dedusting of the fine coal at approximately 1/2 mm. before washing. The development of aspirator dedusters has greatly facilitated this practice and led to a revival of interest in the bin drainage of fine coal as well as the dedusting practice itself. Complete removal of the dust not only improves the performance of the washers but also greatly increases the rate of drainage of the washed product. There is now a trend from centrifugal drying back to bunker drainage. Both Klockner-Humboldt Deutz and Westphalia-Dinnendahl-Gröppel, A.G. have introduced effective aspirator type dry dedusting systems, and the leading plant builders are introducing specially equipped draining bunkers into their designs.

Most effective dedusting is obtained by following the aspirator dedusters with rinsing sieves that remove the last of the dust from the 8 mm. x 0.5 mm. raw coal before it goes to the jigs. High speed horizontal or near horizontal short stroke shaking sieves are used for this service. With half millimeter wedge wire cloth, these sieves will handle 8 to 10 mm. top size pre-dedusted coal at the rate of about 8 metric tons per hour per square meter of cloth and deliver a feed practically free of dust under 0.35 mm. and less than 1.0% through half millimeter.

This practice simplifies the clarification of the wash water and hence new designs incorporate smaller settling cones and thickeners.

The half millimeter dust is shipped raw in admixture with the washed fines or cleaned by froth flotation, as the condition may dictate. At the representative plants visited, the dry dust was used in the raw state and the slurry from the rinsing screens only was sent to the flotation plant.

The technique of jigging fine coal is well developed in Germany and the adaptability of this system to American practice might well be profitable, particularly for use in conjunction with tables or launder washers that operate on a stratification principle which is complementary to the fundamental jig stratification.

The German and continental coal cleaning industry has not made use of concentrating tables or rising current classifiers. The Rheolaver in modified form has been introduced in a few isolated instances but is not popular.

An estimate of the breakdown of tonnage between the different types of washing plants recently operative in the Ruhr was furnished by Dr. Meyer of the North German Coal Control. This is presented in Table 1.

TABLE 1 - RECENT COAL WASHING PRACTICE IN THE RUHR

Type of Washer	Percentage of Total Tonnage
Jig Boxes	87 to 88
Heavy Medium Washers	8
Dry Cleaners	2 to 3

These data do not include the fines of flotation size. Of this material about one-third is cleaned by froth flotation and two-thirds receives no special treatment.

The dry cleaners are of the pneumatic shaking table type and are reported to be used only for the treatment of fine anthracite coal for briquetting. Small dry cleaning plants are operated by seven anthracite producers and four briquetting plants.

6. Jig Wash Boxes

In the coarse coal category, (sizes larger than 8 mm. or 10 mm) washing practice at commercial washeries is much more uniform than in the American industry. Lump coal larger than 80 mm. is generally screened out in the head house and handpicked dry on rubber belt picking bands.

The 80 mm. x 8 mm. size is commonly separated into two grades for washing, either at 30 mm. or at 25 mm., and these two sizes are washed separately whether the washing is done in jig boxes or heavy media separators.

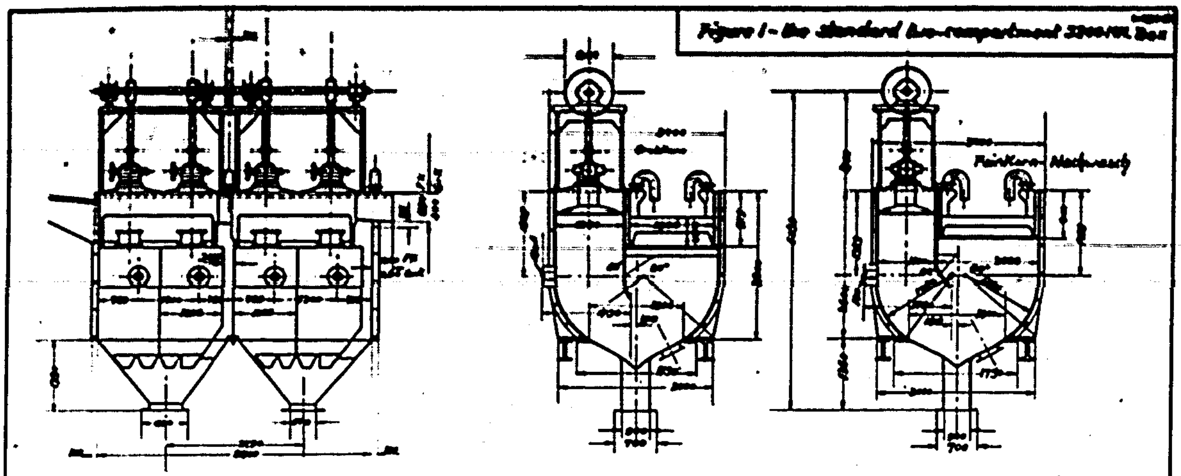
Wash box sizes and general construction features have tentatively been standardized by agreement among the manufacturers. Figure 1 shows the standard dimensions of the 3200 mm. two compartment box.

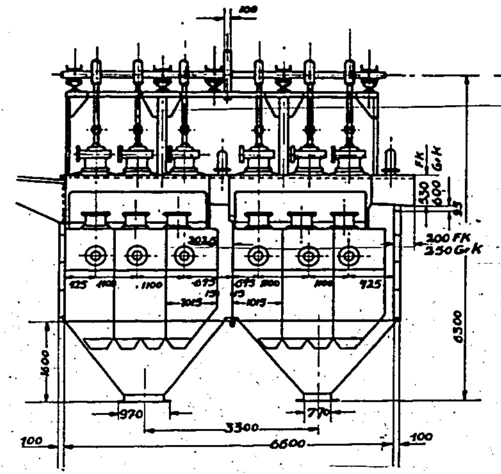
The specifications set up in the table of Figure 1 are transcribed with the corresponding English captions in Table 2.

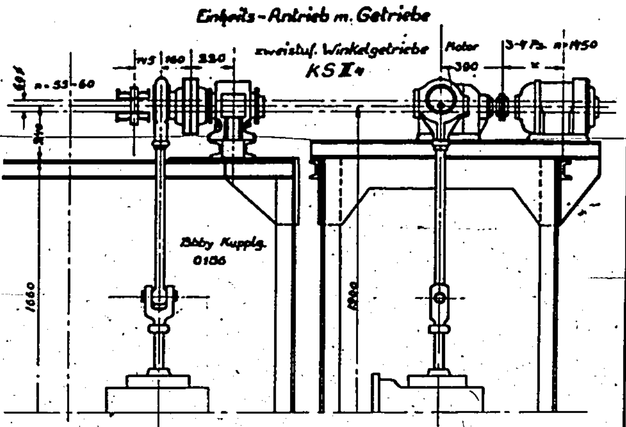
TABLE 2 - SPECIFICATIONS OF JIG BOXES OF FIGURE 1
Width 3200 mm.

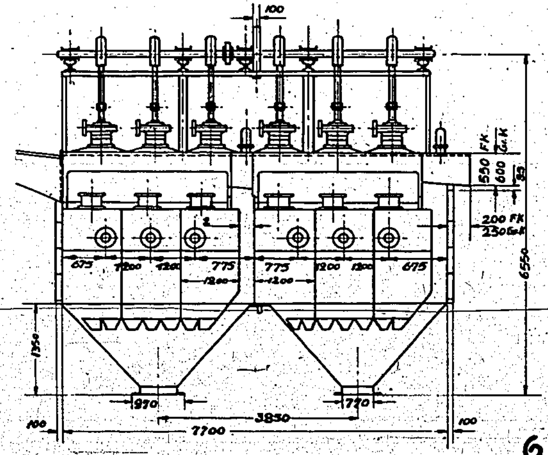
	Length	For Coarse Coal	For Fine Coal and Rewash
Effective Area in Square Meters	5500	9.1	10.0
	6600	11.0	12.3
	7700	13.2	14.6
Air Consumed in Cubic Meters per Minute	5500	41	15 (25)
	6600	49.5	18.5 (31)
	7700	59.5	22.5 (36.5)
Total Weight in Kilograms Cast Iron Design	5500	27150	26900
	6600	32200	32000
	7700	35500	35300
Total Weight in Kilograms Steel Design	5500	23150	23000
	6600	27600	27400
	7700	30500	30000
Weight of Hutches in Kilograms	5500	3200	3200
	6600	4000	4000
	7700	4800	4800
Water Content of Box in Cubic Meters	5500	42.000	42.000
	6600	50.000	50.000
	7700	57.000	57.000

Figure I - the standard two-compartment 3200 MM. Box









Maschinenbrt. = 3200

	Länge	Grob Korn	FeinKorn Nachwasch
Setzfläche m²	5500	9,1	10,0
	6600	11,0	12,3
	7700	13,2	14,6
Luftverbrauch m³/min	5500	41	15-(25)
	6600	49,5	18,5(31)
	7700	59,5	22,5(36,5)
Gewicht Kg Ausf. Ge.-Fass 1)	5500	27150	26900
	6600	32200	32000
	7700	35500	35300
Gewicht Kg Ausf. Stahl-Fass 1)	5500	23150	23000
	6600	27600	27400
	7700	30500	30000
Auslauftrichter Kg	5500	3200	3200
	6600	4000	4000
	7700	4800	4800
Wasserinhalt m³ 2)	5500	42,000	42,000
	6600	50,000	50,000
	7700	57,000	57,000

1) siehe nebensteh
2)

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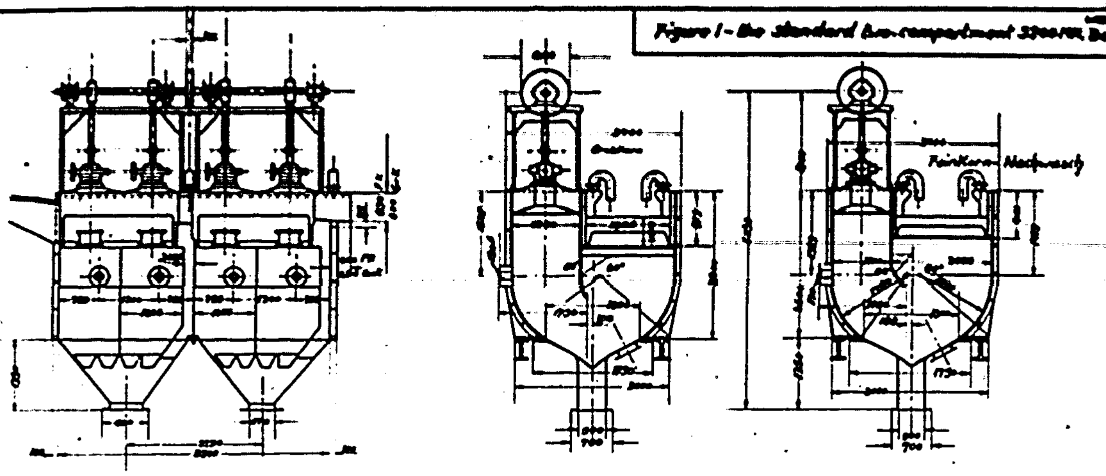
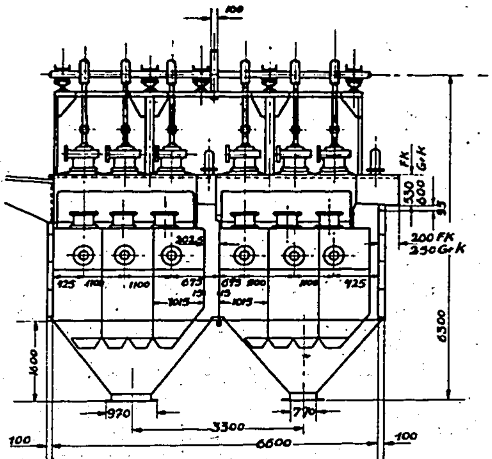
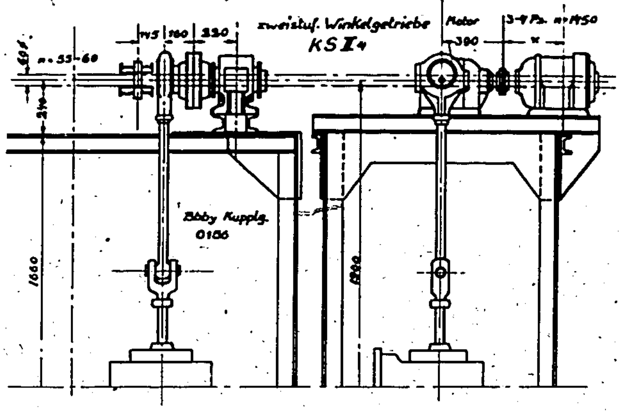
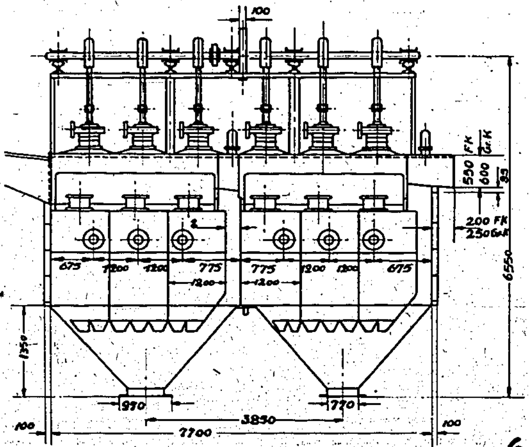




Einheits-Antrieb m. Getriebe





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	6600	50,000	50,000
	7700	57,000	57,000

1) siehe nebenstehd
2)

Wash boxes generally are of about the size and general structural design of the Baum jig as of the period when the Simon-Carves and Norton Baum jig designs were introduced into the United States. Development of very large capacity heavy wash boxes that characterized the Americanization of the Baum jig was not paralleled by such a development on the continent. Many of the present plants are equipped with cast iron boxes, but the modern practice is to fabricate the bodies of steel plate by welding.

German jigs are generally equipped with automatic controls of the float type connected to the slate gate operating mechanism. A modification of this device, developed by Schüchtermann and Kremer-Baum, articulates the float movement also with the air valves so that an excessive accumulation of slate brings about a corrective increase in the air impulse on the pulsion stroke.

The same company is now studying the relationship of stroke period and phase characteristics to performance with a view to developing more effective use of the jig stroke, such as has been featured by the research at Battelle Institute on the phase characteristics of the air actuated jig stroke.

Typical data on the throughput and performance of a German Baum jig washery are presented in a table assembled from data provided by Dr. Ernst Dupierry, director of Schüchtermann and Kremer-Baum, A.G.. These data are presented in Table 3.

TABLE 3 - SOME DETAILS OF SPECIFICATIONS AND PERFORMANCE DATA OF A JIG WASHERY RATED TO HANDLE 300 TONS OF RAW COAL PER HOUR WITH 10% OVERLOAD OR 330 TONS MAXIMUM HOURLY CAPACITY OF 80 mm. x 0 RAW COAL (ALL TONS ARE METRIC.)

		Tons Per Hour	Cubic Meters Per Hour
I. Prescreening			
Coarse 80 mm. x 25 mm.	25%	82.5	
Middle 25 mm. x 10 mm.	18%	59.5	
Fine 10 mm. x 0	57%	188.0	
Total Raw Coal 80 mm. x 0	100%	330.0	

TABLE 3 (Continued)

		Tons Per Hour	Cubic Meters Per Hour
II. Preparation of Nut Coal			
Wash box for 80 mm. x 25 mm 11 square meters			
Feed:			
Raw coal		82.5	
Transport Water			165
Underwater			296
Total Feed Coal and Water		82.5	461
Products:			
Coal and Slurry	65.5%	54	459.0
Middlings	7.0%	5.8	0.5
Refuse	27.5%	27.7	1.5
Total Products	100.0%	82.5	461.0
Crushing Middlings 80mm x 25 mm.:			
Crushed Middlings 25 mm x 10 mm		3.8	
Crushed Middlings 10 mm x 0		2.0	
Total Crushed Middlings		5.8	
Wash box for 25 mm. x 10 mm. - 11 square meters			
Feed:			
Raw Coal		59.5	
Crushed Middling 25 mm x 10 mm from above		3.8	0.4
Transport Water			120.0
Underwater			264.0
Total Feed Coal and Water		63.3	384.4

TABLE 3 (Continued)

		Tons Per Hour	Cubic Meters Per Hour
Products:			
Coal and Slurry	78%	49.4	382.4
Middlings	8%	5.0	1.0
Refuse	14%	8.9	1.0
Total Products	100%	63.3	384.4

The middlings are crushed in hammer mill to 10 mm., deslimed on the middling screen, and fed to the rewash-jig.

	Tons Per Hour	Cubic Meters Per Hour
Sizing and Loading of Nut Coal		
Total feed to nut-sizing screens from both jigs:		
From coarse coal jig	54	459
From middle coal jig	49.4	382.4
Total Coal and Water	103.4	841.4
From the above, undersize degradation and slurry to slurry tank	7	
Feed to bins for nuts and to loading screens plus spray water (clear)	96.4	5.0 48.0
Total	96.4	53.0
The above is divided as follows:		
To be loaded	94.0	6.0
To tank for degradation from nut coal	2.4	47.0
Drain-water from fine coal bins (VII)		25.7
Drain-water from middle coal bins (VIII)		1.8
Total to tank for degradation from nuts	2.4	74.5
Total feed to dewatering screen for nut undersize:		
From degradation screen	2.4	74.5
From slurry tank	7.0	836.4
Drain-water from refuse bins (VIII)		1.6
Total to slurry tank	9.4	912.5

TABLE 3 (Continued)

	Tons Per Hour	Cubic Meters Per Hour
Total to slurry tank	9.4	912.5
Removed by elevator	6.0	2.0
Water and solids to clarification from coarse coal jigs	3.4	910.5

III. Preparation of Fine Coal

Dedusting

Estimated on the basis of 21% of 0.5 mm. x 0 dust in the 10 mm. x 0 raw coal, a dedusting efficiency of 80% (at 4% moisture) and 16% oversize in the dust. The dust tonnage is:

$$\text{Dust Product} = \frac{188 \times .21 \times .80}{.84} = 37.5$$

Remaining dedusted coal	150.5
Total Feed	188.0

Desliming before washing

	Tons Per Hour	Cubic Meters Per Hour
Feed to screen from the deduster:	150.5	
Transport Water		226.0
Spray Water (Reclaimed)		40.0
Spray Water (Fresh)		35.0
Total to pre-desliming screens	150.5	301.0

Fine coal cleaning

Wash box area - 2 x 12.3 = 24.6 square meters

	Tons Per Hour	Cubic Meters Per Hour
Raw coal 10 mm x 0.5 mm	134.5	45.0
Returned from nut coal degradation	6.0	2.0
Transport water		180.0
Underwater		442.0
Total to fine coal jig	140.5	669.0

TABLE 3 (Continued)

Products:		Tons Per Hour	Cubic Meters Per Hour
Coal and slurry	86.2%	121.0	663.5
Middlings	6.0%	8.5	3.0
Refuse	7.8%	11.0	2.5
Total	100.0%	140.5	669.0

IV. Retreatment of Fine Coal

Screening:

	Tons Per Hour	Cubic Meters Per Hour
Crushed middlings from coarse coal jig - reference II - 1	5.8	0.5
Transport water		25.0
Crushed middlings from middle coal jig - reference II -2	5.0	1.0
Transport water		25.0
Spray water (reclaimed)		15
Spray water (fresh)		12
Total feed to middlings screen	10.8	78.5

Sized Products:

	Tons Per Hour	Cubic Meters Per Hour
Size 25 mm. x 10 mm (to middling-jig - II,2)	3.8	0.4
Size 10 mm. x 0.7 mm (to rewash - jig - IV,2)	5.0	12.0
Size 0.7 mm. x 0 mm (to pre-desliming cones IX,2)	2.0	66.7
	10.8	78.5

Rewash of Middlings:

Wash box for 10 mm. x 0.7 mm. - 9 square meters

	Tons Per Hour	Cubic Meters Per Hour
Middlings from fine coal jig (II,3)	8.5	3.0
Transport water		15.0
10 mm. x 0.7 mm from middlings (IV,1)	5.0	12.0
Transport water		5.0
Underwater		178.0
Total to rewash - jig	13.5	213.0

TABLE 3 (Continued)

Products:		Tons Per Hour	Cubic Meters Per Hour
Coal and slurry	10%	2.2	209.4
Middlings	64%	8.6	3.0
Refuse	20%	2.7	0.6
	100%	13.5	213.0

V - Dewatering Screens for Clean Fine Coal

	Tons Per Hour	Cubic Meters Per Hour
Feed to dewatering screens:		
From fine coal-jig (III,3)	121.0	663.5
From rewash-jig (IV,2)	2.2	209.4
Spray water (white)		35.0
Total to dewatering screens	123.2	907.9
Products:		
Oversize - dewatered fine coal	110.0	35.0
Undersize (to water clarification, VI)	13.2	872.9
	123.2	907.9

VI - Water Clarification and Slurry Screens

	Tons Per Hour	Cubic Meters Per Hour
Feed to clarification cones:		
Overflow from slurry tank (II,3)	3.4	910.5
Undersize from fine coal-dewatering screens (V)	13.2	872.9
Total	16.6	1783.4
Feed to slurry screens:		
Overflow of pre-clarification cones (VI,1)	11.0	22.0
Spray water (white)		20.0
Total to slurry screens	11.0	42.0
Products:		
Oversize — coarse slurry	5.0	2.0
Undersize — fine slurry	6.0	40.0
	11.0	42.0

TABLE 3 (Continued)

VII - Drainage of Fine Coal in Drainage Bins

	Tons Per Hour	Cubic Meters Per Hour
Feed:		
Fine coal from dewatering screens (V)	110.0	35.0
Coarse slurry from slurry screens (VI,2)	5.0	2.0
Total to drainage bins	115.0	37.0
Products:		
Coal as shipped (9% H_2O)	115.0	11.3
Drippings from fine coal bins (II,3)		25.7
	115.0	37.0

VIII - Dewatering of Middlings and Refuse in Drainage Bins

	Tons Per Hour	Cubic Meters Per Hour
Dewatering of middlings:		
Middlings from rewash-jig (IV,2)	8.6	3.0
Dewatered middlings (12% H_2O)	8.6	1.2
Drippings from middle coal bin (II,3)		1.8
	8.6	3.0
Dewatering of refuse:		
Refuse from coarse coal jig (II,1)	22.7	1.5
Refuse from middle coal jig (II,2)	8.9	1.0
Refuse from fine coal jig (III,3)	11.0	2.5
Refuse from rewash coal jig (IV,2)	2.7	0.6
Total to refuse drainage bins	45.3	5.6
Dewatered refuse (8% moisture)	45.3	4.0
Drippings from refuse drainage bins		1.6
	45.3	5.6

IX - Slurry Treatment with Dewatering of Clean Fine Coal in Drainage Bins

Main Clarification

Feed:

TABLE 3 (Continued)

	Tons Per Hour	Cubic Meters Per Hour
Overflow from pre-clarification (VI)	5.6	1761.4
Fine slurry from slurry screens (VI)	6.0	40.0
Total to main pump tank	11.6	1801.4
Products:		
Overflow to wash-water-tank		1770.0
Settled slurry (to flotation)	11.6	31.4
	11.6	1801.4
Distribution of wash water:		
Transport water to coarse coal jig		165
Underwater to coarse coal jig (II)		296
Transport water to middle coal jig (II)		120
Underwater to middle coal jig (II)		264
Spray water to pre-desliming screens (III)		40
Transport water to fine coal jigs (III)		180
Underwater to fine coal jigs (III)		442
Transport water for coarse middlings to middle coal screen (IV)		25
Transport water for middle middlings to middle coal screen (IV)		28
Spray water to middle coal screen (IV)		15
Transport water for fine middlings to rewash-jig (IV)		15
Transport water for middlings 10 mm x 0.7 mm. to rewash jig (IV)		5
Underwater to rewash-jig (IV)		178
		1770
Clarification of Desliming Screen Underflow		
Feed to clarifier:		
Underflow of desliming screens on fine coal (III)	16.0	256.0
Underflow of middle coal screen (IV)	2.0	66.1
Total to clarifier	18.0	322.1
Outside thickener		
Feed:		
Overflow from clarifier cones (IX)	4.0	40.1

TABLE 3 (Continued)

		Tons Per Hour	Cubic Meters Per Hour
Products:			
Thickened slurry		4.0	6.0
Overflow — clarified water			34.1
		4.0	40.1
Flotation:			
Feed:			
Slurry from main clarification (IX)		11.6	31.4
Slurry from desliming screens cones (IX)		14.0	56.0
Slurry from outside thickener (IX)		4.0	6.0
Dust from deduster		10.4	
Filtrate (IX)			50.0
Fresh water			16.0
Total to flotation		40.0	160.0
Products:			
Concentrate	78.0%	31.2	58.0
Refuse	22.0%	8.8	102.0
	100.0%	40.0	160.0
Filters:			
Feed:			
Concentrate from flotation (IX)		31.2	58.0
Filtercake (20% water)		31.2	8
Filtrate (effluent water)			50
		31.2	58

X - <u>Summary of Product</u>

Washed nut coal	94.0
Washed fine coal	115.0
Filtercake	31.2
Dust from deduster (dry unwashed)	27.1
Middlings	8.6
Refuse	45.3
Flotation tailings	8.8
	330.0

XI - Fresh Water Distribution

Fresh Water Leaving with Products

Products	Cubic Meters Per Hour
Washed Nut Coal	6.0
Clean Fine Coal	11.3
Filter Cake	8.0
Middlings	1.2
Refuse	4.0
Sailings from Flotation	103.0
Overflow from Outside Thickener	34.1
	166.6

Fresh Water Entering System

Admission Point	Cubic Meters Per Hour
Loading of Nut Coal	48.0
Desliming Fine Raw Coal	35.0
Middling Screen	12.0
Fine Washed Coal Dewatering	35.0
Slurry Screen	20.0
Flotation	16.6
	166.6

XII - Slurry Treatment with Dewatering of Clean Fine Coal in Centrifuges

	Tons Per Hour	Cubic Meters Per Hour
The Centrifuges		
Feed:		
Predewatered fine coal and coarse slurry (VII)	115.0	37.0
Products:		
Coal centrifuged (7% water)	111.0	8.3
Slurry	4.0	28.7
	115.0	37.0
Pre-clarification Cones		
Feed: (VI)	16.6	1783.4
- Drain water from drainage bins		-25.7
+ Slurry from centrifuge	4.0	28.7
Total to pre-clarifier	20.6	1786.4

ABLE 3 (Continued)

		Tons Per Hour	Cubic Meters Per Hour
Products:			
Slurry from cones		11.0	22.0
Overflow to main pump tank		9.6	1764.4
		20.6	1786.4
Main clarification:			
Feed:			
Overflow from pre-clarification (XII)		9.6	1764.4
Fine slurry from slurry screens (VI)		6.0	40.4
Total to main clarifier		15.6	1804.4
Products:			
Overflow to wash-water-tank (IX)			1770.0
Settled slurry		15.6	34.4
		15.6	1804.4
Flotation:			
Feed:			
Slurry from main clarifier (XII)		15.6	34.4
Slurry from pre-deslimer cones (IX)		14.0	56.0
Slurry from outside thickener (IX)		4.0	6.0
Dust from deduster		6.4	
Filtrate (XII)			52.0
Fresh water			11.6
Total to Flotation		40.0	160.0
Products:			
Concentrate	80.0%	32.0	60.0
Tailings	20.0%	8.0	100.0
	100.0%	40.0	160.0
Filters:			
Feed:			
Concentrate from flotation		32.0	60.0

TABLE 3 (Continued)

Products:	Tons Per Hour	Cubic Meters Per Hour
Filter cake (20% H_2O)	32.0	8.0
Filtrate, effluent water		52.0
	32.0	60.0

XIII - Summary of Products (from centrifuging of washed fine coal)

Clean Nuts	94.0
Centrifuged Fine Coal	111.0
Filter Cake	32.0
Dust	31.1
Middlings	8:6
Refuse	45.3
Flotation Tailings	8.0
	330.0

XIV - Fresh Water Distribution (in centrifuging of washed fine coal)

Fresh Water Leaving With Products

Products	Cubic Meters Per Hour
Nuts	6.0
Centrifuged Fine Coal	8.3
Filter Cake	8.0
Middlings	1.2
Refuse	4.0
Flotation Tailings	100.0
Overflow from Outside Thickener	34.1
	161.1

Fresh Water Entering System

Admission Place	Cubic Met Per Hou
Loading of Nuts	48.0
Pre-desliming	35.0
Middling Screen	12.0
Fine Coal Pre-dewatering	35.0
Slurry Screen	20.0
Flotation	11.6
	161.1

With regard to cleaning performance, it is impossible to make a irect appraisal of German and continental jigging practice in relation to merican standards, because in Europe the primary pressure is on the yield ide whereas in America it is more on the product side. This condition ill be evident from a study of the examples of plant performance guarantees presented in the Appendix. Tested at normal washing gravities, the efuse will be relatively freer from float coal and the washed product ill carry more sink material than corresponding operations in America. omparison is further complicated by the practice, much more common in urope than in America, producing a middling product.

It is the general opinion of the investigators that jigging operaions in Germany are not significantly different in cleaning efficiency from American practice although specific test data were not assembled with sufficient coverage to substantiate this opinion by factual proof. Leaders of the equipment industry expressed the view that, in the past decade, there has been an improvement in jig practice represented by a reduction of the tolerance of ash in the cleaned coal from 1½% above the float coal ash at the same yield to a tolerance of around 0.5 to 0.8% above the float coal ash at the same yield; and that this improvement has been brought about primarily by better plant engineering and by improvements in operational and control techniques.

However, examination of representative plants did not bring to light any important developments in the field of control, such as automatic sampling, product testing, or control rountine. In fact the plants visited, which were represented to be modern, well-designed plants representing the best practice, are not as well-equipped as the best American plants in this respect.

Any appraisal of this specification of the washed product must take into consideration the circumstance noted above that in European practice the economic emphasis is on yield. In coarse coal jig plants producing commercial coal, the slate end will probably be guaranteed to contain lees than 1% float material.

7. The Heavy Medium Processes

The practice of heavy media separation in Germany has generally followed the trends developed in the Dutch mining industry of South Limburg. The Barvoys process has been introduced into the German field by Westphalia-Dinnendahl-Gröppel, A.G.. Here it is called the Sophia Jacoba process. This company has built several heavy media plants in Germany and some plants using the Tromp process have been installed by Klöckner-Humboldt-Dentz and Schüchtermann and Kremer-Baum. Both of these firms build Tromp plants.

These processes have been well described and explained in the technical literature.

In German plants, heavy media separation boxes have, as a rule, been introduced into conventional jig plant designs without any substanti revision of general plant arrangement. The coarse coal adapted to heavy media separation is divided into two sized grades just as in jig practice (80 mm. x 25 mm. and 25 mm. x 8 mm.), and these two sizes are washed separately then rinsed, sized, and loaded through bunkers to transportatio facilities.

The Chance Sand Flotation process is not used in Germany, and there is no evidence of an acknowledged influence of the long-standing American experience in heavy media separations in German or continental practice.

Developments in this field have been concentrated on the use of so-called "stable" suspensions, such as the barytes-clay mixture of the Sophia Jacoba process in which the medium settles only very slowly and on processes using unstable suspensions but adapted to compensate for a predetermined and presumably uniform settling rate by control of the characteristic of the return suspension material.

In these processes, pulverized magnetite has been used but curiously the potential advantages of magnetic recovery and reconditioning of the medium have been ignored up to the time of the experimental installation in the special Reinstkohle plant at Koenigen Elizabeth Colliery. This may have been due to a lack of effective magnetic separator equipment At Koenigen Elizabeth, a machine somewhat like the Dings was installed by Krupp-Gruson, A.G.. This is referred to later in the description of the Koenigen Elizabeth plant.

At the works of Humboldt, a research project is now being carrie on to develop a magnetite suspension washer of the overflow discharge type which appears to follow more nearly than any other European development, the trend of American practice in the use of heavy suspensions. The general idea of this apparatus is shown in Figure 2, which is self-explanatory.

The Germans apparently have contributed little to perfecting heavy suspension concentrating processes. This general method of gravity separation pioneered in America by Chance, Conklin, and Wuensch, with the early development and reduction to practice of the Chance Sand Flotation process, was then given a revolutionary turn to the use of substantially stable suspensions by the Dutch with the development of the Barvoys process, in Staatsmijnen Loess process, and the Tromp process; and these processes have probably reached their best development from the standpoint of plant design and control in British practice.

8. Summary of General Practice

To summarize generally the comparison of contemporary German and American coal preparation practices, there is (1) the greater preponderanc of conventional jig washers in the German field (although the Baum jig is

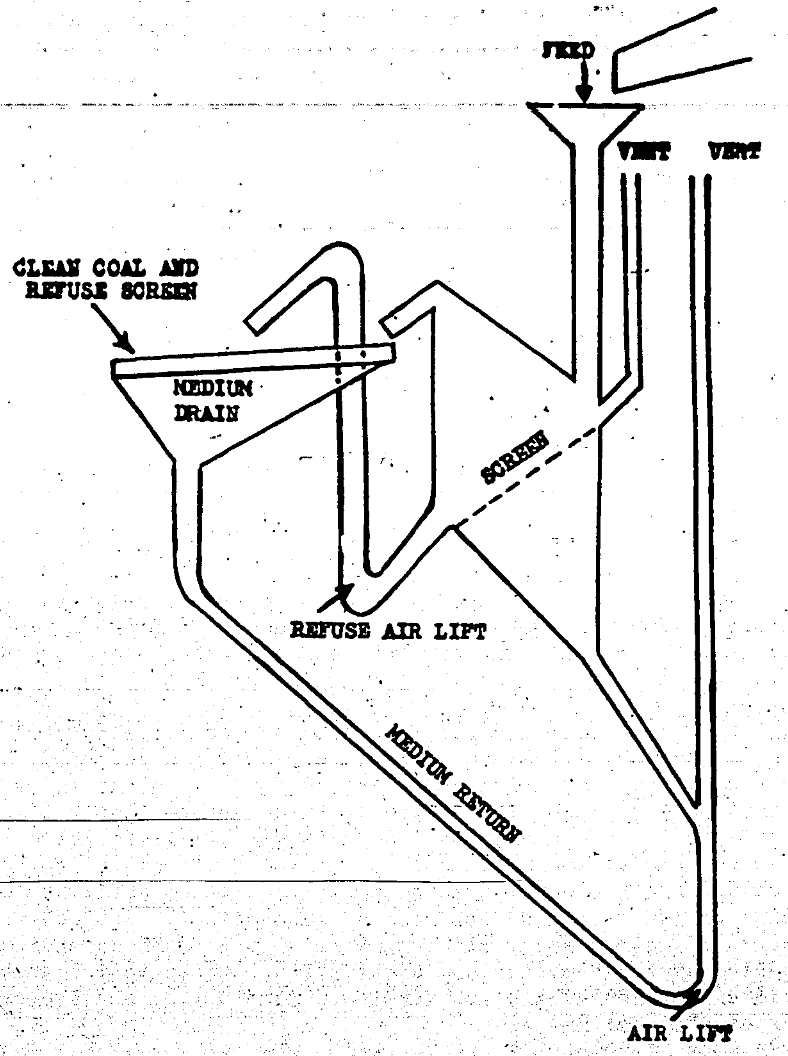

FIGURE 2 - SCHEMATIC DRAWING OF EXPERIMENTAL MAGNETITE HEAVY MEDIUM SEPARATOR

also the most popular washer in America), (2) presizing of coal before j ging as compared to almost universal practice of jigging unsized coal in America, (3) treatment of fine coal on jigs in Germany contrasted to use of tables, launders, and classifiers in America, (4) extensive use of froth flotation in Germany, (5) wide use of the Chance Sand Flotation pr in America with no direct counterpart in Germany but with a more definit trend to the use of substantially stable suspensions, (6) loading of the product through bins in Germany compared with the practice of direct continuous loading of prepared grades from preparation units to transport facilities in American practice. These differences in practice apply in large measure to continental practice generally.

There is little material evidence of interchange of ideas and methods between the coal preparation industries of continental Europe and America in the past.

9. Special Coal Preparation Projects

Aside from the preparation of coal for general industrial and commercial use, there are several special coal preparation projects of interest that have been undertaken primarily as the result of emergency war conditions.

Those projects center around the preparation of super-cleaned coal products for very special purposes, such as the production of carbo electrodes for the aluminum industry, generator fuel for automotive vehicles, and coal adapted to be converted into liquid fuel.

The most exacting specification is for the electrode coal which must be of substantially less than 1.0 per cent ash content. To produce coal of this grade, the first step in every instance was the selection of seams that would respond most effectively to super-cleaning. Schemes for ultra refining of the coal, most of which were still in the development stage at the end of the war, included (1) multiple stage froth flotation in which the froth product was repeatedly reflected until the ultimate end point of ash reduction was reached substantially without regard to the yield, (2) attempts at ultra-refinement of heavy suspension processe represented by the Laminarstrom washer, (3) electrostatic separation, (4) acid extraction of the ash forming minerals, (5) and use of solvents to extract the coal matter.

Such ultra-cleaning schemes were actually incorporated into only two commercial plants; both of these were bombed and are not now in condition to operate. Such data as could be obtained regarding the technological functioning and the performance of these plants is given in some detail in the fifth section of this report designated Field Data

IV - ORGANIZATIONS OF COAL PREPARATION SERVICE

10. Development and Construction

The development of coal preparation technology and the construction of plants is very largely in the hands of a few contracting firms that service the industry in much the same way as the American equipment manufacturers service the coal industry of America.

The principal builders of coal preparation plants are:

(1) Schüchtermann and Kremer-Baum, A.G., with factories in Dortmund and Herne, Ernst Dupierry, managing director. This firm manufactures a complete line of screens, wash boxes, dedusters, and conveyors for coal handling, and builds complete coal handling and preparation plants on contract. This firm has the German license for the Tromp Process but shares the field with Klöckner-Humboldt-Deutz, A.G., of Cologne.

(2) Westphalia-Dinnendahl-Gröppel, A.G., with works in Bochum and Dinnendahl, Dr. Möllenberg and Carl Gröppel, directors. This firm likewise manufactures a complete line of coal handling, screening, and washing appliances with patented dedusters and auxiliary devices. It is the German Licensee of the Barvoys process designated the Sophia Jacoba process in the German trade.

(3) Klöckner-Humboldt-Deutz, A.G., Cologne Kalk, Dr. Ernst Maruhn, director. This firm manufactures ore dressing as well as coal preparation appliances, the briquetting plants and general coal handling equipment. It holds a license to sell Tromp magnetite washing plants, and it is one of three firms associated in a syndicate to manufacture the Schieferstein Resonance screens. The other two firms are Krupp-Gruson, A.G., Magdeburg, and Carlshütte Maschinen and Stahlbau Gesellshaft m.b.H., Waldenburg-Altwasser. The latter two firms in the Russian Zone of occupation were not visited, but some engineers formerly on their staffs were interviewed at their Essen Office.

In the field of coal handling, J. Pohlig, A.G., Cologne and Demag, Duisburg are leaders in the installation of cranes and bridges for handling coal over Redewater piers.

Büttner-Werke, A. G., Uerdingen, is especially active in the development of dryers adapted to use in the coal preparation industry. These dryers are incorporated into the designs of the coal preparation and handling plants.

In Germany it is the most common practice to handle the erection of new preparation plants and extensive alterations of existing plants by also the most popular washer in America), (2) presizing of coal before j ging as compared to almost universal practice of jigging unsized coal in America, (3) treatment of fine coal on jigs in Germany contrasted to use of tables, launders, and classifiers in America, (4) extensive use of froth flotation in Germany, (5) wide use of the Chance Sand Flotation pr in America with no direct counterpart in Germany but with a more definit trend to the use of substantially stable suspensions, (6) loading of the product through bins in Germany compared with the practice of direct continuous loading of prepared grades from preparation units to transport facilities in American practice. These differences in practice apply in large measure to continental practice generally.

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In Germany it is the most common practice to handle the erection of new preparation plants and extensive alterations of existing plants by contract with one of these recognized firms specializing in that line who customarily tender firm bids on a competitive basis.

The contractor undertakes manufacture and erection of equipment, buildings, and supplementary facilities usually on a fixed lump sum basis with performance guarantees. He places the plant in operation and turns it over to the customer in operation after an acceptance test is completed. In this regard, contracting practice is virtually identical with American practice.

The manufacturers are active also in the development of new equip ment and techniques. Developments initiated at producers' plants likewise fine their way into general use through the agency of the appliance manufacturer who furnish the only practical means by which new devices can be reduced to practice and introduced to the general trade.

11. Standardization of Equipment

As a step to minimize confusion and misrepresentation in the solicitation of contracts and the formulation of guarantees, the principal contracting firms have tentatively agreed upon standard designs and specifications for certain items of preparation plant equipment. Copies of tentative dimension sheets of such items as have presently been standardiz are reproduced in Figures 1, 3 and 4 (pages 6a, 21 and 25 respectively) fr sample sheets obtained from Schüchtermann and Kremer-Baum. These include standard designs of wash boxes, screens and draining bin stand pipes.

12. Standardization of Guarantees

Similarly, through the agency of Bergbauverein, a comprehensive scheme for the standardization of the plant performance guarantees has been proposed. In this plan, which has been published in a schedule entitled Richtlinien für Abnahme und Ueberwachung von Steinkohlen Aufbereitungsanlagen issued by the Bergbau, forms are suggested for the guarantees of the performance of washeries, screens, and filters.

Tolerances are not prescribed, but examples of generally accept able performance with respect to typical operating conditions are present A transcript of the salient parts of this document are reproduced in Appendix 1.

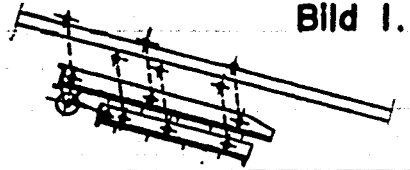
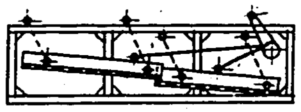
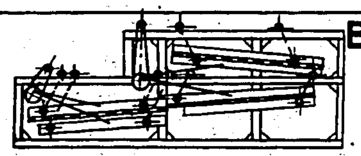
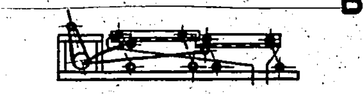
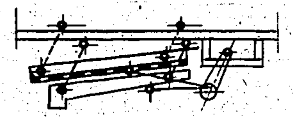
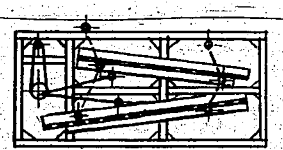
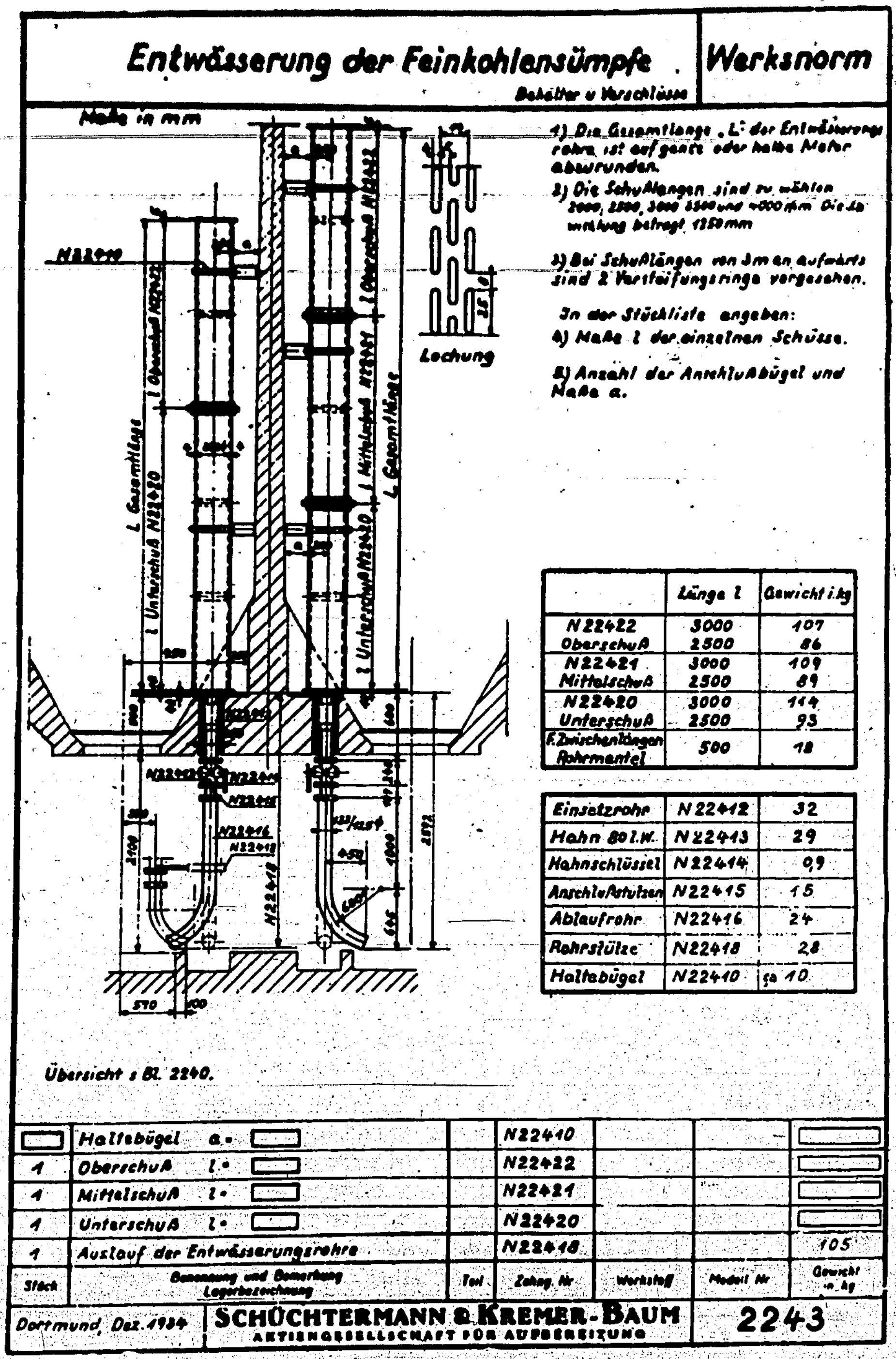
Schnellschwingsiebe **Bauformen.**	
Förderkohlensieb	 **Bild 1.**
Vorklassierung 1600 × 2 · 4000 1800 × 2 · 4000 2000 × 2 · 4000	 **Bild 2.**
Nußklassierung, kleine Leistung 1400 × 7000 1600 × 7000 1800 × 7000 **Sieb unter dem Wipper** 1600 × 2 · 2500	 **Bild 3.**
Nußklassierung 1400 × 9000 1600 × 9000 1800 × 9000	 **Bild 4.**
Feinkohlen-u. Vorentschlämmsiebe, 1200 × 2 · 2500 1400 × 2 · 2500 1600 × 2 · 2500 Schlammsiebe 1000 × 2 · 2500 1200 × 2 · 2500 1400 × 2 · 2500	 **Bild 5.**
Nußverladesieb 1400 × 3000 1600 × 3000	 **Bild 6.**
Kokssiebe 1000 × 4500 1400 × 4500 1600 × 5000	 **Bild 7.**
Dortmund.	**SCHÜCHTERMANN & KREMER-BAUM** ACTIENGESELLSCHAFT FÜR AUFBEREITUNG. DORTMUND.

FIGURE 3 - SCREEN STANDARD



Entwässerung der Feinkohlensümpfe

Behälter u Verschlüsse

Werksnorm

Maße in mm

1) Die Gesamtlänge „L" der Entwässerungsrohre ist auf ganze oder halbe Meter abzurunden.

2) Die Schußlängen sind zu wählen 2000, 2500, 3000 3500 und 4000 mm. Die Abwicklung beträgt 1350 mm

3) Bei Schußlängen von 3m an aufwärts sind 2 Versteifungsringe vorgesehen.

In der Stückliste angeben:

4) Maße l der einzelnen Schüsse.

5) Anzahl der Anschlußbügel und Maße a.

	Länge l	Gewicht i.kg
N 22422 Oberschuß	3000 2500	107 86
N 22421 Mittelschuß	3000 2500	109 89
N 22420 Unterschuß	3000 2500	114 93
F. Zwischenlängen Rohrmantel	500	18

Einsatzrohr	N 22412	32
Hahn 80 l.W.	N 22413	29
Hahnschlüssel	N 22414	0,9
Anschlußstutzen	N 22415	15
Ablaufrohr	N22416	24
Rohrstütze	N22418	28
Haltebügel	N22410	ca 10

Übersicht s Bl. 2240.

Stück	Benennung und Bemerkung Lagerbezeichnung	Teil	Zchng. Nr	Werkstoff	Modell Nr	Gewicht in kg
☐	Haltebügel a = ☐		N22410			☐
1	Oberschuß l = ☐		N22422			☐
1	Mittelschuß l = ☐		N22421			☐
1	Unterschuß l = ☐		N22420			☐
1	Auslauf der Entwässerungsrohre		N22418			105

Dortmund, Dez. 1934

SCHÜCHTERMANN & KREMER-BAUM
AKTIENGESELLSCHAFT FÜR AUFBEREITUNG

2243

FIGURE 4 - WASHED COAL DRAINAGE BIN STAND PIPES

13. **Price Relationships**

This investigation was strictly a technological project. The time and facilities were not available to make a study of the economic relationships affecting the German coal preparation industry. Yet it is difficult to make an intelligent appraisal of technologic practices, particularly as the features which differ radically from our own practice, without some knowledge of a few simple and fairly ascertainable factors affecting the practical operation of the industry.

Such factors as the prevailing price of coal, the wage rate, relative prices of other basic commodities, availability of competitive fuels, and the wartime demand for special and strategic forms of the product, may readily rationalize otherwise unexplainable peculiarities of technology.

In wartime Germany, the coal industry supplied three grades of coal refined beyond the normal industrial grade. A schedule showing the technical names, ash specifications, uses, and prices current as of January, 1945, of these grades and the ordinary industrial grade is presented in Table 4.

TABLE 4 - GRADES OF COAL PRODUCED IN THE RUHR IN JANUARY, 1945

Designation of Grade	Ash Specification Per Cent	Use	Price per Metric Ton F.O.B. Mine Marks**
Reinstkohle	0.5	Electrodes	107
Reinstkohle	0.8	Electrodes	95
Edelkohle	2.0	Electrodes*	55
Generatorkohle	3.0	Gas Producers	34
Hydrierkohle	4.0	Hydrogenation	24
Kokskohle	6.0 to 8.0	Coking and Industrial	16

* Used in Production of the Cheaper Grade of Aluminum

** Marks converted to dollars at rate of 38 cents per mark - See Table 6 on page 30.

The first three grades were entirely strategic products and presumably the prices may represent value that cannot be maintained in peacetime economies; although the aluminum industry, the sugar refining industry, carbide carborundum and other chemical industries may be expected to continue to supply a market for these grades of coal. It is conceivable that this market might even expand in the future. It is reported that the cleanest coal is also adapted to production of activated carbon.

In the matter of competitive fuels, petroleum and natural gas were obviously not in the picture during the war, but on the contrary the coal industry was probably under extreme pressure to furnish raw material for production of the largest possible supplies of synthetic oils. This condition also prevailed, to some degree at least, the nearby pre-war period.

The chief source of energy fuel in Germany, other than the bituminous coal fields of Western Germany and Silesia was the lignite or braunkohle which was produced in roughly the same volume (for Germany as a whole) as the steinkohle. For example, the 1938 production of hard coal was 186.2 million tons and brown coal was 195.0 million tons.*

Under normal conditions, Germany is better supplied with fuel than other European countries and has an excess available for export, but production costs are substantially higher than in the United States and the available reserves are very much smaller. The expenditure of a substantially greater effort in preparation of the product and prevention of waste is justified. A rough evaluation of the economic condition of the coal industry is presented by the ratio of the unit price of the product to the prevailing wage. The f.o.b. price of industrial coal in the late war years was 16 marks per metric ton and the basic miner's wage was 8.40 marks (subject to deduction of approximately 18%). The price of coal in labor units was thus 16 man hours per ton in Germany compared to about 2 man hours per ton in the United States. Approximately the same relationship prevailed in the pre-war years as there was little variation in basic commodity prices and wage rates under the rigid price control system prevailing in Germany during the war.

Very little attention was given to monetary cost data in this investigation as the wide differences in commodity price patterns in Germany and in the United States under the influence of war conditiona and artificially controlled prices would make it impossible to evaluete such data expressed in German price units.

However, some miscellaneous cost figures wero obtained as shown in Table 5 for what they might be worth to the reader. The tentative dollar equivalent was arrived at by using a value of 38 cents for the mark. This factor was obtained by a comparison of estimates of prevailing American prices and German prices of a list of 20 commodities shown in Table 6 as prepared by Dr. Joseph Becht, an interpreter in the FIAT office and checked by Dr. J. Nüsslein, formerly a director of I.G. Farben, both residents of Frankfurt.

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* See the Economic Survey of Germany, Section F, Ministry of Economic Warfare, 1944.

TABLE 5. COSTS (Incl. capital costs but no office overhead).

Cost per ton at Bergbauverein (typical of Ruhr)		
Washing	RM 0.25 to 0.42	$0.095 to 0.16
Froth flotation and filtering	" 1.00 to 2.00	$0.38 to 0.76
Cost per liter of aero-benzin at Gelsenberg Benzin a.g.		
Hydrogenation	RM 0.20 to 0.22	$0.076 to 0.084

TABLE 6. RELATION OF RM TO $.

	$	RM		$	RM
Bread per kilo	.15	.35	Shirt (cotton)	2.25	11.00
Eggs per doz.	.60	3.00	Haircut	.75	.80
Sugar (kilo)	.10	.80	Doctor (call)	4.00	10.00
Meat (kilo)	.80	1.80	Picture show	.35	1.50
Potatoes (kilo)	.045	.10	Carfare (city)	.08	.20
Cabbage (kilo)	.03	.15	RR fare (100 km)	1.34	4.00
Beer (glass)	.10	.30	Labor (hr)	.65	.91
Butter (kilo)	1.00	3.50	Domestic (mo)	60.00	50.00
Cigarettes (pkg)	.14	1.20	Rent (cheap)	30.00	80.00
Shoes (pr)	6.00	25.00	Coal (ton)	11.00	110.00

TABLE 7. FOB PRICES OF RUHR COAL BY SIZES (In RM per ton in August 1938. No change during war period)

Kind	MM	Fett Coal 18-29 vol.	Ess Coal 12-18 vol.	Anthracite #1 7-11 vol.	
Raw resultant	80 x 0	12.90	12.00	-	
Run-of-mine		14.00	13.00	-	
Lump over	80	18.25	18.25	19.00	
No. 1 Nut	80 x 50	17.00	19.00	27.00	
No. 2 Nut	50 x 30	17.00	19.00	31.00	
No. 3 Nut	30 x 18, 20 x 15	17.00	19.00	25.00	
No. 4 Nut	20 x 10, 18 x 12	16.85	17.00	18.50	18.00
No. 5 nut	10 x 6 , 8 x 5	16.00	16.00	15.00	
Washed Fines	5 x 0	12.50	12.25	11.25	
Raw Fines	5 x 0	12.00	12.25	11.25	
Coking Slack		15.00	-	-	

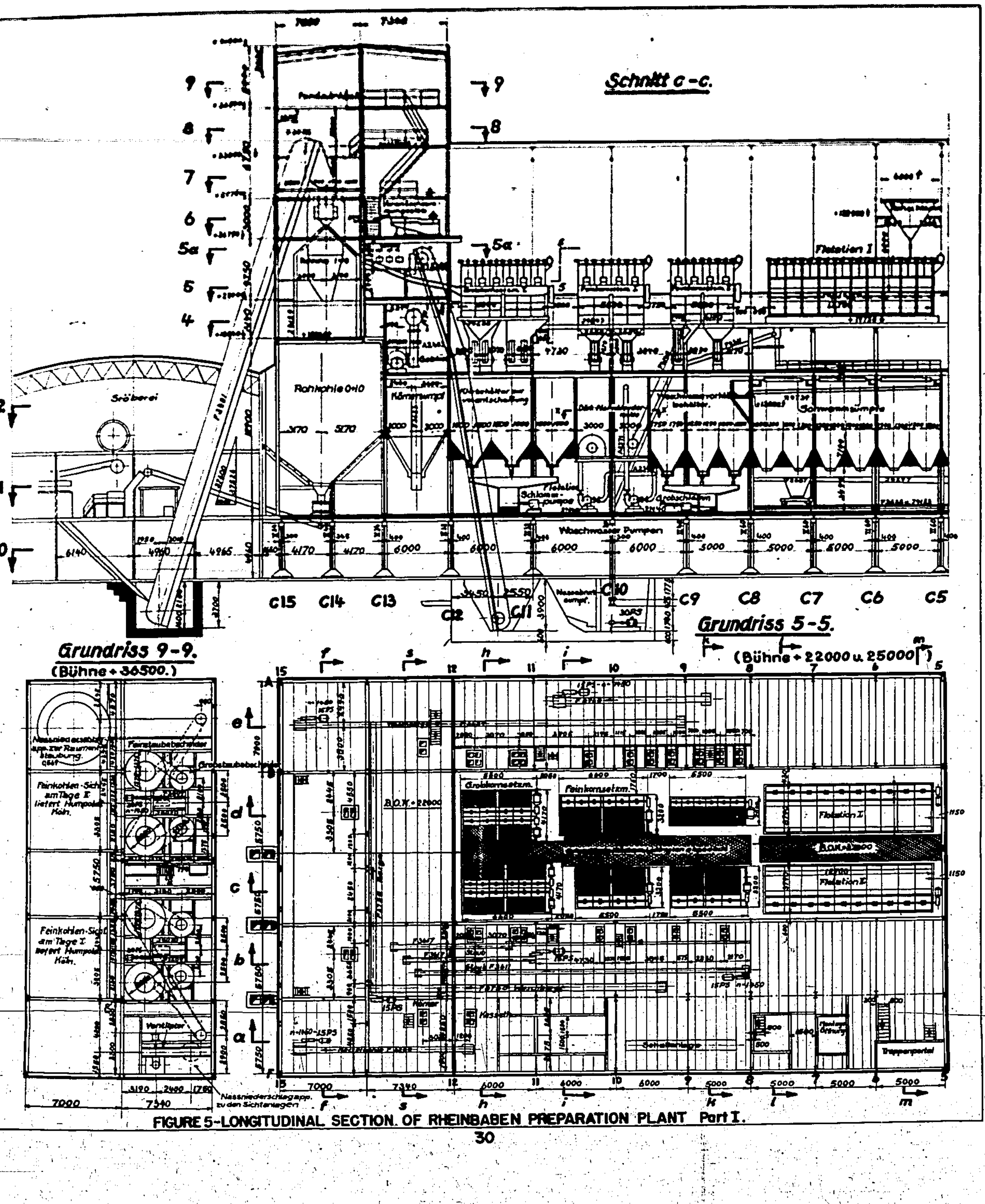
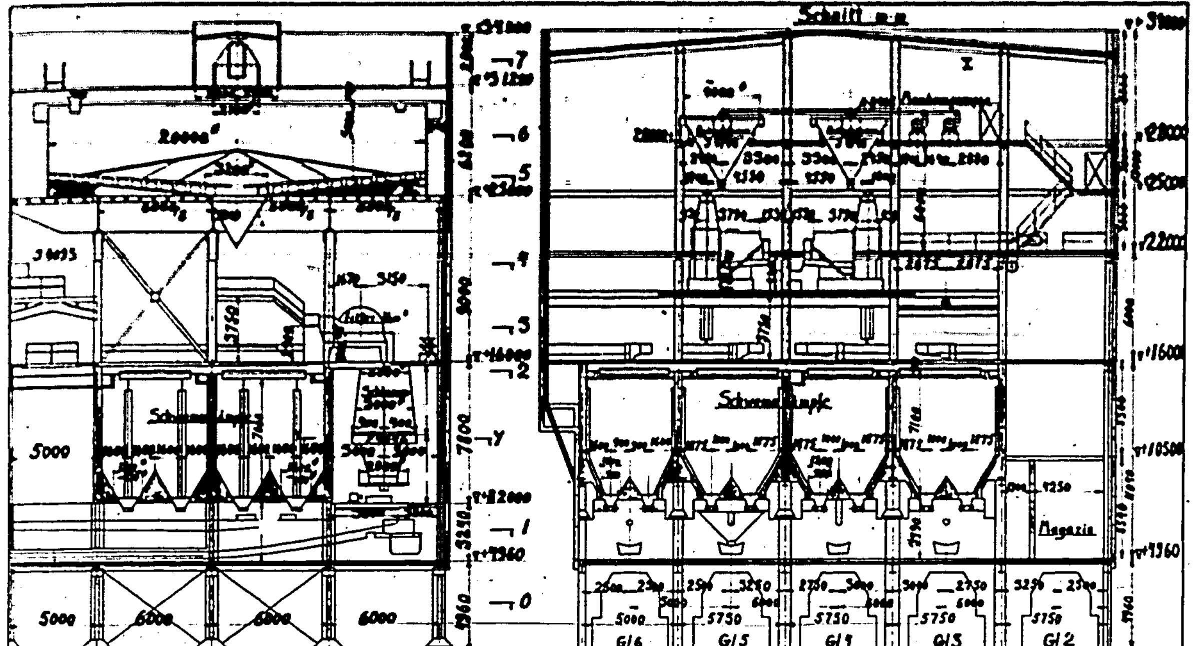


FIGURE 5-LONGITUDINAL SECTION. OF RHEINBABEN PREPARATION PLANT Part I.

FIGURE 5 - LONGITUDINAL SECTION OF RHEINBABEN PREPARATION PLANT PART II



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VI - FIELD DATA ON PLANTS

14. Rheinbaben Colliery

This is one of several operations of Hibernia A. G., the state owned colliery company. The Rheinbaben pit, near Bottrop, has a rated capacity of 5,000 tons of coal per day (16 hours). The product is a medium volatile bituminous coal (27% volatile) divided into two use classes i.e., "Gaskohle" and "Fettkohle", which are handled separately in preparation but by identical methods.

The preparation plant is a design of Schüchtermann and Kremer-Baum A.G., erected in 1939. It consists essentially of a sizing plant and a washing and loading plant in which the two grades of coal are washed by parallel Baum jig washing plants of 200 tons per hour rated capacity for the "Gaskohle" and 150 tons per hour for the "Fettkohle". The preparation plant is housed in steel and masonry buildings of permanent construction. Colliery and preparations plants are undamaged and were in full scale operation at time of inspection.

The operating force in the washing plant is 35 men on each shift

The Washery

A typical section of the preparation plant is shown in Figure 5. This is Sheet No. 1 of a series of six assembly drawings covering the entire plant. The complete set is available in the files of the Solid Fuels Sub-committee, J.I.O.A., Washington.

Inasmuch as the two washing units for "Gaskohle" and "Fettkohle" are identical, a description of the "Gaskohle" unit will suffice for the entire plant.

The run-of-mine coal is screened at 80 mm.. The 80 mm. lump goes to picking belts and the 80 mm. resultant only goes to the washery. In the washery the 80 mm. x 0 coal is screened into three sizes: 80 mm. x 25 mm., 25 mm. x 10 mm., and 10 mm. x 0 with the tonnages 43, 38 and 119 tons per hour respectively.

The two coarse sizes go to separate Baum jig boxes. All boxes are provided with automatic slate gate controls (SKB Austragregler) and all automatic devices at the time of inspection were working. Some of them are provided with additional air regulating devices (Luftzusatzregulierung) which are connected to the air inlet valves to regulate the amount of air admitted to the jigs for pulsation.

An important feature of the S.K.B. washery design is the method of handling the fine coal 10 mm. x 0. This coal is elevated to the top of the washery building where it is first dedusted pneumatically using the Humboldt rapid sichter dedusting equipment. This system, devised by Klöckner-Humboldt-Deutz A.G., Cologne, is described and illustrated in the equipment section of this report.

After dedusting, the fine coal is further deslimed over four S.K.B. desliming screens of 0.5 mm. wedge wire. Each screen has a surface of three square meters and a capacity of about 8 tons per square meter per hour. The thickness of the coal layer on the screens is about 4 cm.. These screens are sprayed intensively with water to wash the fines through the screen.

Dr. Kuhn, chief engineer of S.K.B., estimated that such screens running at 550 strokes a minute and operating on 10 mm. x 0 coal not previously dedusted would have a capacity of 5 to 6 tons per square meter per hour over bronze wire cloth screens with openings 1.0 mm. x 0.8 mm.. The deslimed coal would have no particles under 0.25 mm.. The feed may be as fine as 6 mm. x 0, but normally they would treat a feed of 10 mm. x 0. The spray water amounts to 1 ton of water to 0.8 to 1.0 ton of coal.

At Rheinbaben the dedusted and deslimed fine coal contains no more than 1 per cent under 0.7 mm.; whereas without preliminary dedusting, the feed to the jigs after prescreening would carry approximately 10 per cent of such fines. Due to the fact that the fine coal is so well deslimed, the wash water for the jigs has a solid content of no more than 18 grams per liter.

Dewatering of Fine Coal

The washed fine coal is transported from the jigs directly to large draining bins (Schwemm-Sümpfe). No dewatering screens are used ahead of these bins. Each bin has a capacity of 100 tons. After filling the bin, the valves of the four vertical dewatering pipes within the bin are opened, so that the water can be drained off. Small pipes for air admission, not shown on the drawing 2243, are provided on top of the drain pipes.

Because of the dedusting and desliming of the fine coal before washing, draining in the drainage bins is very rapid and effective: after four hours draining the moisture content of the fine coal in the bins is 2%, and after eight hours, it comes down to 10%, including about 1½ to 2% vein moisture. Standard specifications have been set up for these draining bin accessory pipe. See Figure 4.

The wash water from the desliming screens (under 0.7 mm.) goes to a cone of 4 meters diameter, and the settled material is treated by froth flotation. This plant consists of 20 cells and has a capacity of 80 tons per hour including the "Gaskohle" and "Fettkohle" together. S.K.B. mechanical frothers, similar to the Mineral Separation cells, are used.

The water and tailings from the flotation plant, 150 cubic meters per hour, goes to settling ponds.

The overflow water from the fine coal drainage bins goes to a cone, where the coarser material settles (Körnersumpf). The overflow of this cone goes to a 22 meter diameter Dorr thickener (Rundeindicker) which reduces the solids content from 20 grams per liter to 18 grams per liter. The settled material goes to the flotation plant.

The froth from the flotation plant is treated in an S.K.B. froth breaker before filtering. This treatment is said to increase the rate of filtering by a hundred per cent. There are two rotary drum filters each having a surface area of 16 square meters. An interesting feature of the filters is, that the feed is admitted to one side and not in the trough under the filters. The moisture content of the filter cake is approximately 18 per cent. See figure 6.

Storage of Filter Cake

The filter cake is conveyed to small conical loading hoppers with rotating plate dischargers at the bottom, which are said to be very effectiv for handling stubborn fines hard to handle in bins and chutes.

15. Augusta Victoria Colliery

This preparation plant is at shaft No. 111 near Recklinghausen. It is a conventional jig washery of 200 tons per hour capacity of typical Westphalia-Dinnendahl-Gröppel (Wedag) design erected in 1936.

The flow sheet of this plant is shown in Figure 7 and general arra ment drawings in Figure 8. The plant is housed in substantial steel and co crete buildings of a size indicating, together with a general appraisal of the total equipment, a capital investment very much larger than would generally be considered in American practice to be appropriate to a throughput of 200 tons of coal per hour.

In the presizing plant, which is housed in a separate building along with the lump coal picking tables, the run-of-mine coal is screened at 80 mm. and the lump coal goes to picking belts.

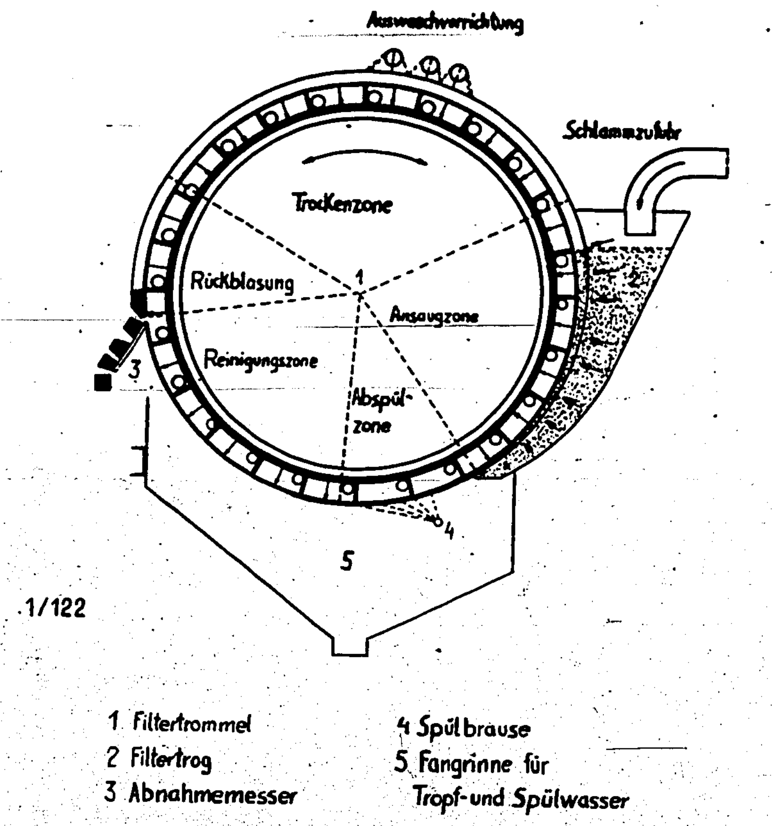

FIGURE 6 - THE S.K.B. VACUUM FILTER FOR COAL SLUDGE

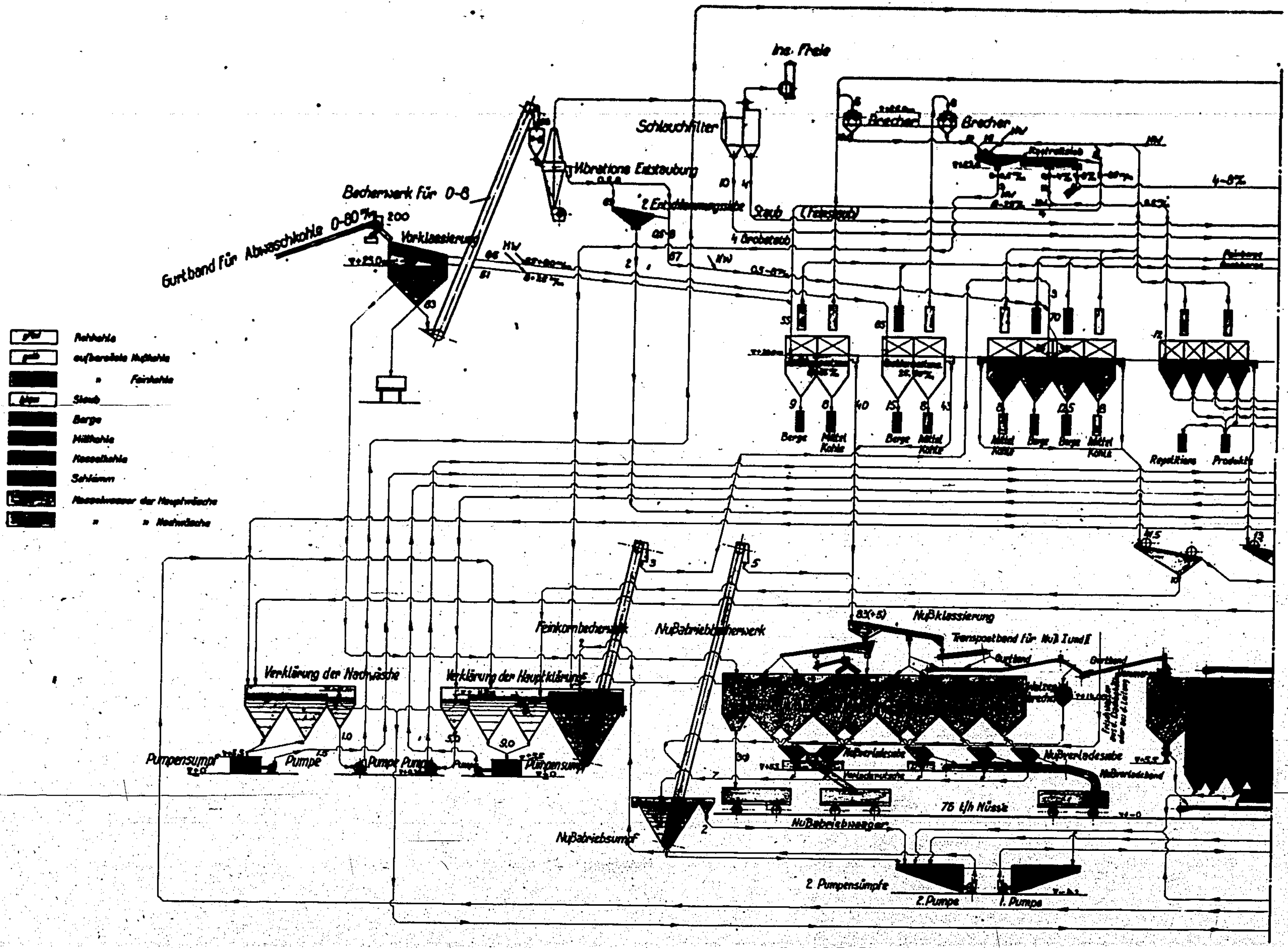

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FIGURE 7- FLOW SHEET OF AUGUSTA VICTORIA WASHING PLANT
PART I

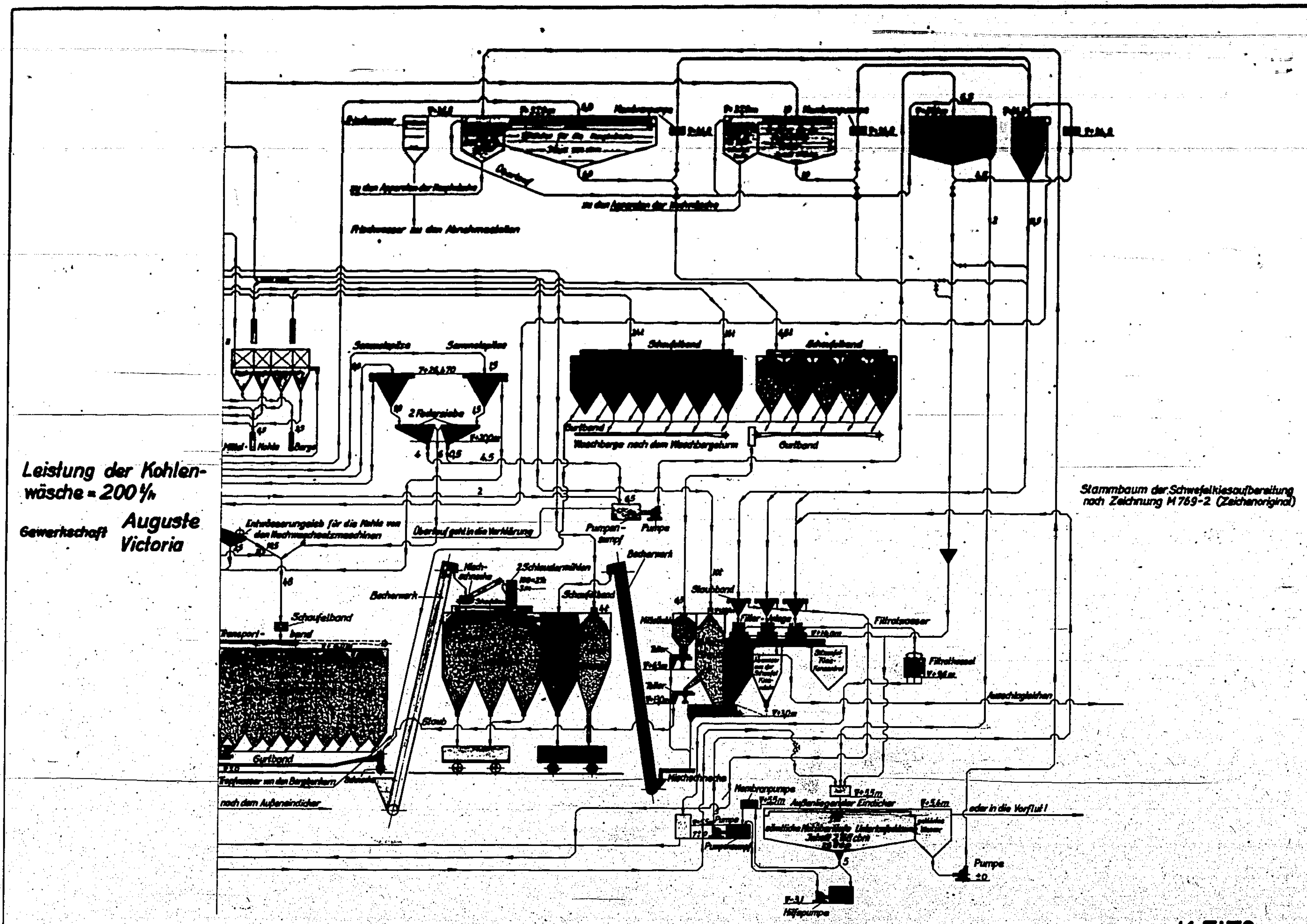

FIGURE 7 – FLOW SHEET OF AUGUSTA VICTORIA WASHING PLANT
PART II

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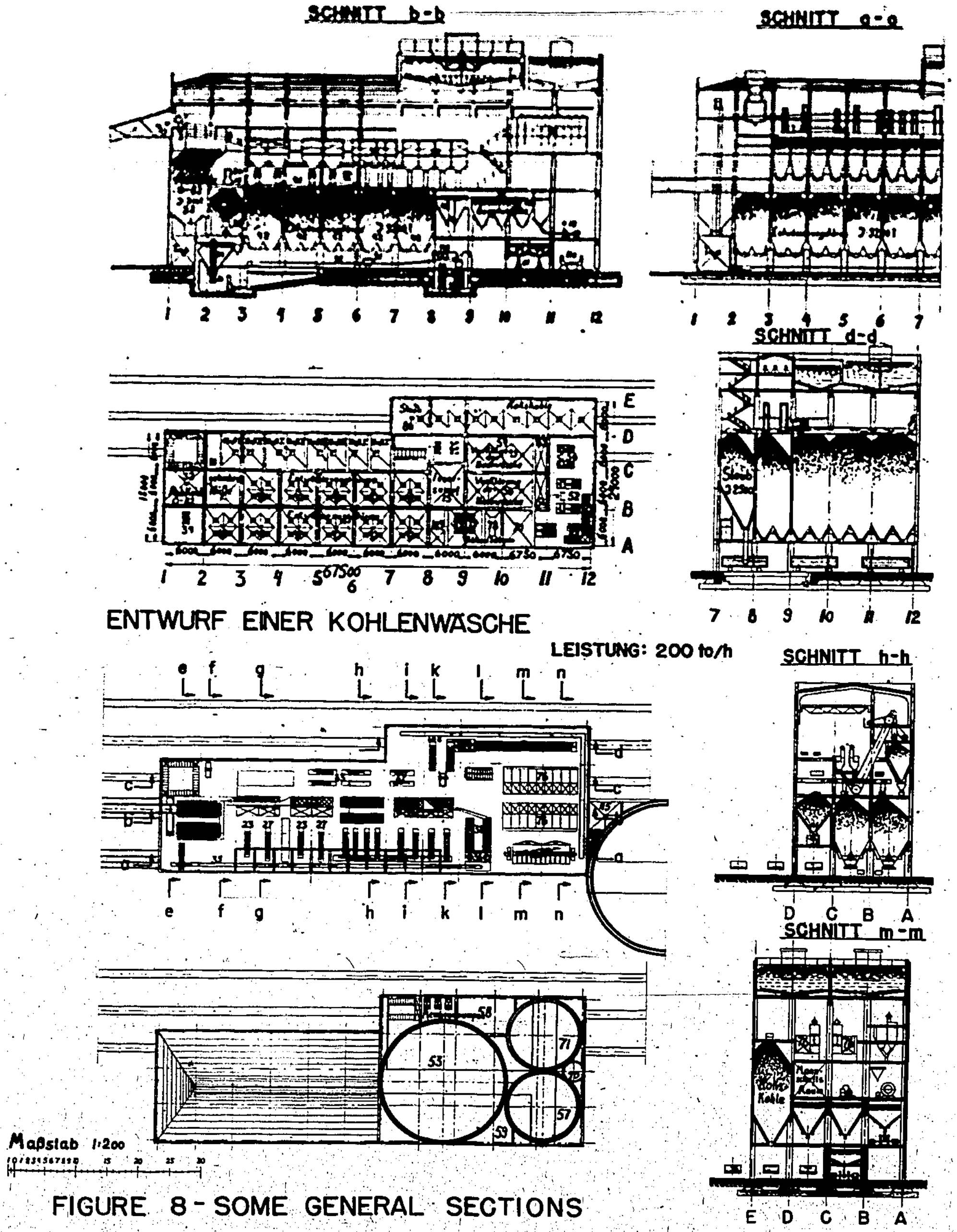

FIGURE 8 - SOME GENERAL SECTIONS OF ASSEMBLY DRAWING OF AUGUSTA VICTORIA WASHERY

Part I

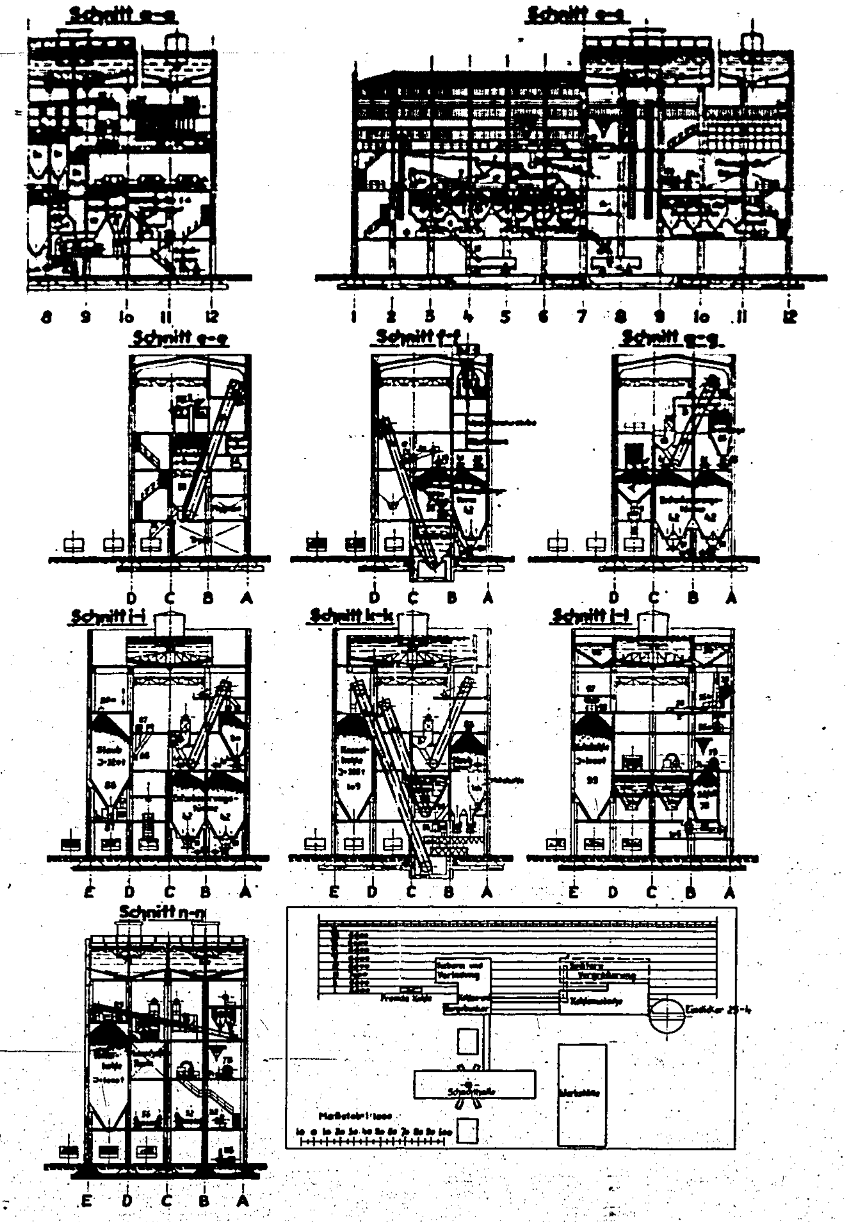

FIGURE 8- SOME GENERAL SECTIONS OF ASSEMBLY DRAWING OF AUGUSTA VICTORIA WASHERY

PART II

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The 80 mm. resultant coal is transported to the washery building by an inclined rubber belt conveyor of 1200 mm. width on 3-pulley carriers.

In the washery the coal is screened on large Schieferstein screens, which are housed in an enclosure so that the dust can be drawn away to dust collectors. These multideck screens divide the coal into the following sizes: 80 mm. x 25 mm., 25 mm. x 8 mm., and 8 mm. x 0.

The 80 mm. x 25 mm. size and the 25 mm. x 8 mm. size are washed in separate piston driven jig boxes 1.8 meters wide and 6.6 meters long. The 8 mm. x 0 size is washed in two jig boxes, each 2 meters wide and 5 meters long. The primary fine coal wash boxes have feldspar on the screens.

At this plant all the jigs are equipped with automatic slate gate controls, but most of them were set in a fixed position, so that they could not work. They indicate the height of the slate bed and the opening of the gates. The control is effected by floats connected to oil pressure cylinders to operate the slate gates.

The washed coal is screened into five different nut sizes and delivered to bunkers by spiral chutes.

The fine coal is dedusted before delivery to the wash boxes. This is done in the Wedag-Vibrator-Sichter, a kind of deduster in which the fine coal is fed over a vibrating screen through a high velocity air current. After dedusting, the coal is deslimed on 0.5 wedge wire screens before going to the wash boxes.

The washed fine coal is dewatered on 0.5 or 0.3 mm. wedge wire screens to a moisture content of about 30 per cent. Then it goes to draining bunkers with gratings around the discharge gates, where it is reduced to a moisture content of 10 percent.

Middlings of the coarse coal 80 mm. x 25 mm. and 25 mm. x 8 mm. go to crushers, and then to vibrating screens (Schnellschwingsiebe) to remove the 0.5 mm. slurry. The crushed middlings sizing screens make 25 mm. x 8 mm., 8 mm. x 4 mm., and 4 mm. x 0.5 mm. sizes. The 0.5 mm x 0 sli goes to a sump. The 4 mm. x 0.5 mm. and 8 mm. x 4 mm. gradings are rewashed.

The clean coal from the rewash jigs is dewatered by screens and bins in the same way as the primary clean coal. Even the slate is drained in bins after part of the water has been removed in the slate elevators.

Slurry Treatment

The water and slurry under 0.5 mm. go to cones (Vorklassierungs-Spitzen). The settled material (about 0.5 mm. x 0.1 mm.) from these cones

is pumped to other cones (Spitzen) and the final settlings thence to 0.2 mm. screens. The product of these screens is added to the washed fine coal. The effluent (0.1 mm. x 0) of the cones flows to a large Dorr thickener (on the roof of the washery) having a diameter of 20 meters.

The clarified overflow of the Dorr thickener is used again as wash water in the jigs. The settled slime goes to filters.

The water from the raw coal slurry screens is kept separate and settled in smaller Dorr thickeners of 10 meters diameter.

It is proposed in the future for the thickened slurry to go to a froth flotation plant, but at present it is settled in cones (Spitzen) and the settled slurry from these cones is brought to filters.

The general washery effluent goes to a large 25 meters diameter Dorr thickener in order to obtain a final effluent of no more than 0.5 grams per liter solids in the water.

16. Nordstern Colliery

Data on the product of this mine, which was used in the hydrogenation plant at Gelsenberg, were obtained from Dr. Jacob of Gelsenberg Benzin A.G., and from the builder, Westphalia-Dinnendahl-Gröppel. The colliery company and the consuming company are associated as subsidiaries of Gelsenkirchen Bergwerk A.G., which belongs to Vereinigte Stahlwerke. The coal is subject to inter-company movement only. For this reason, the specifications as to grade of coal are not too rigidly enforced.

The coal of selected seams of high volatile content is prepared in a "Sophia Jacoba" washery (Sophia Jacoba is the German name of the Barvoys process licensed to Westphalia-Dinnendahl-Gröppel) which delivers a washed coal of 4.0 per cent ash content by separation at approximately 1.45 specific gravity.

A typical petrographic report on the coal selected for hydrogenation is: Fusain 2.2 per cent; Vitrain 45 per cent, Clarain-durain 52.8 per cent. They have used at Gelsenberg coals of as much as 5.0 per cent fusain and 1.5 per cent of semi-fusain.

Fusain is regarded as an inert diluent constituent which is not convertible; but it is not difficult to handle in the plant. The ash is not only inert but very troublesome as an obstruction in mechanical functioning of the converters.

Four per cent ash was reported to be the most economical grade of hydrogenation fuel to have at Gelsenberg in view of the overall economy of mine and conversion plant although the operators of Gelsenberg Benzin would prefer a lower ash.

The washed coal is shipped from Nordstern to Gelsenberg in the nut and slack size. At the works of Gelsenberg Benzin, it is dried to 1.0 per cent moisture and pulverized in Ball mills to pass a screen of 400 mesh to the square centimeter, approximately 40 per cent will pass a screen of 10,000 mesh per square centimeter (equals 0.06 mm.).

The statistical price of coal transferred from Nordstern to Gelsenberg Benzin A.G., was 24 marks per metric ton.

Coal hydrogenation plants using high volatile bituminous coals from Western Germany and Silesia were (1) Gelsenberg, considered above, (2) the Scholven Plant near Buer, (3) the Poelitz plant near Stettin, and (4) Blechhamer in Upper Silesia.

Other plants using Braun coal were operated at (1) Leuna, (2) Wesseling, (3) Braunkohle Benzin A. G., and Brux. Ruhröl, G.m.b.H. near Welheim Bottrop operated on pitch.

The Heavy Medium Washery

The preparation plant at Nordstern is a typical heavy media plant of conventional Gröppel design. Drawings of this plant were not available, but general arrangement schemes were available for a similar plant presently being designed for Duhamel, a colliery at Hirschbach near Saarbrücken.

This plant will be designed to handle a total raw coal load of 270 to 315 tons per hour of which 65 per cent over 10 mm. in size is to be handled in heavy media separators, Sophia Jacoba process, and 35 per cent under 10 mm. in size will be handled in fine coal jigs. The coarse coal will be pre-screened into two sizes 80 mm. x 30 mm. and 30 mm. x 10 mm to be washed in separate heavy media boxes.

The coarse coal 80 mm. x 30 mm. is dedusted on dry screens before going to the wash boxes.

The slack 30 mm. x 10 mm. is deslimed on wet screens before entering the wash boxes. Then the specific gravity of separation is 1.40, the make-up suspension must be about 1.55 in order to take care of the dilution of the medium by the wet coal entering the box.

If a separation at 1.6 specific gravity is required, the 30 mm. x 10 mm. coal should be deslimed first by water and afterwards sprayed with a suspension of 1.6 specific gravity. The suspension coming from the screens and having a specific gravity of about 1.45 would then be thickened by a "star" centrifuge and returned to the system.

At present, the "star" centrifuge is in operation only at the Sophia Jacoba mine in Hückelhofen, but will be introduced in other washeries of "Wedag".

The dirt from the heavy medium boxes is rewashed in an upward current to take the middlings out.

The width of the baths, each for 75 to 100 tons per hour, is two meters.

In order to thicken the barytes, a 30 meter diameter thickener will be used, but 20 meters diameter would do also according to the makers. Another 30 meter diameter thickener takes care of the washery and spray water from the washed nuts and the slack.

The slurry from the washed fine coal is thickened and put to vacuum filters.

The plant has a closed circuit on the water, but floculation is not used.

17. Zeche Carl Alexander

This operation at Baeswyler near Alsdorf is especially directed to the production of Reinstkohle of 0.5 to 0.7 per cent ash adapted to the very special purpose of preparing carbon electrodes for the aluminum industry. It is a privately owned mining property, but the entire product was delivered to the Rheinisch Westfälisches Kohlensyndikat for sale.

The coal is a bituminous coking variety of 20 to 21 per cent volatile. Mining and preparation operations were concentrated on the working of certain selected seams that respond best to super-cleaning.

The washery is divided into a normal part for making coking coal and a special part for making electrode coal. The numbers in the description below refer to the flow sheet, Figure 9.

The raw coal is screened at 80 mm. and the resultant coal passing through the screen is sized on screen (2) into 80 mm. x 0.75 mm. and 0.75 mm. x 0 grades. The 80 mm. x 0.75 mm. size again is screened into 80 mm. x 10 mm. nuts and 10 mm. x 0.75 mm. fine coal, which are washed on two Baum jig wash-boxes (3). The 0.75 mm. x 0 from the pre-screen (2) goes to a preflotation plant (9), which consists of flotation boxes, similar to the Mineral Separation type but manufactured by Schüchtermann and Kremer-Baum.

The froth from these machines can either go to drum filters (11) or to the main flotation plant for further refining by selective flotation.

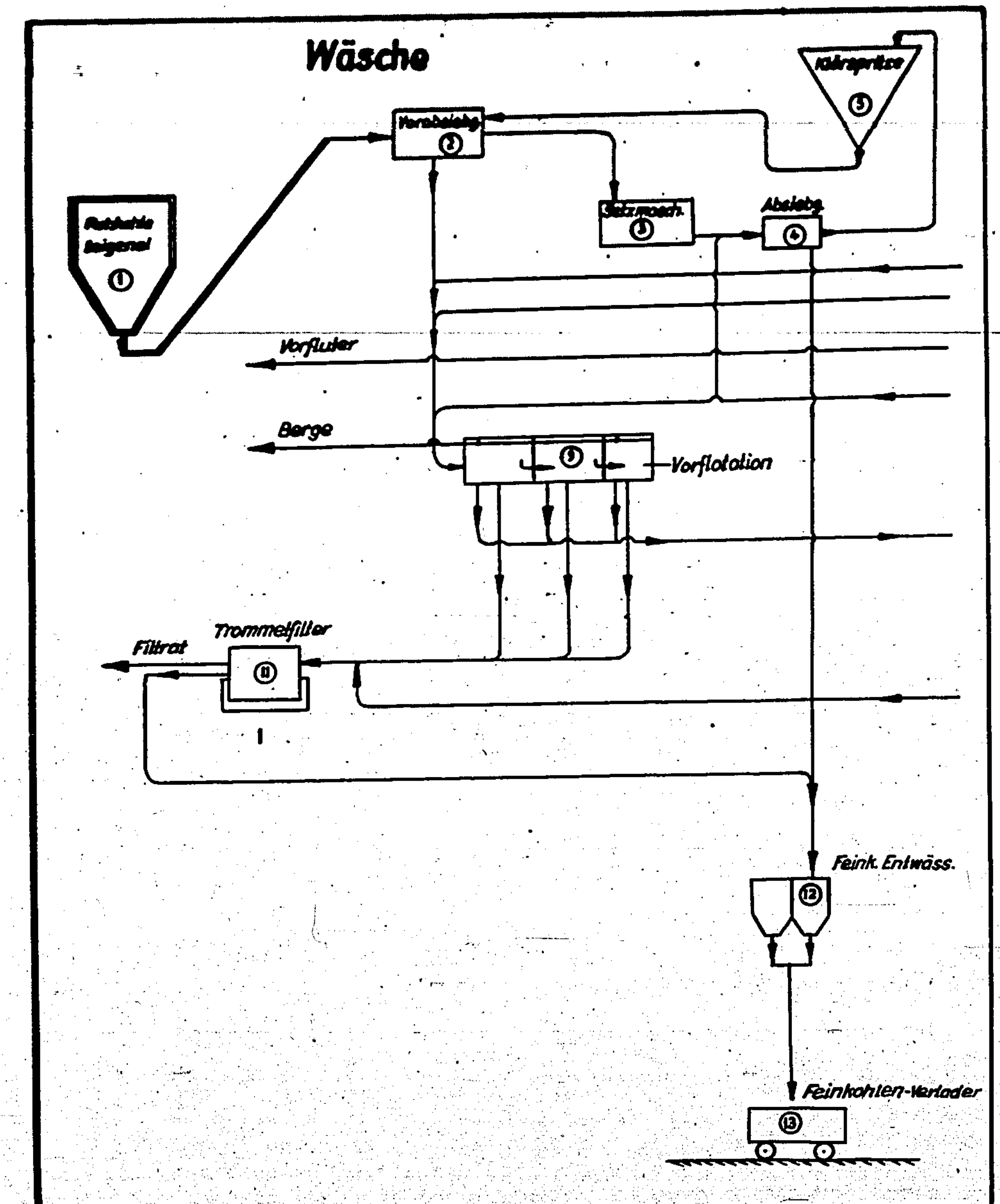

FIGURE 9 - FLOWSHEET OF REINSTEINKOHLE PLANT AT CARL ALEXANDER COLLIERY
Part II

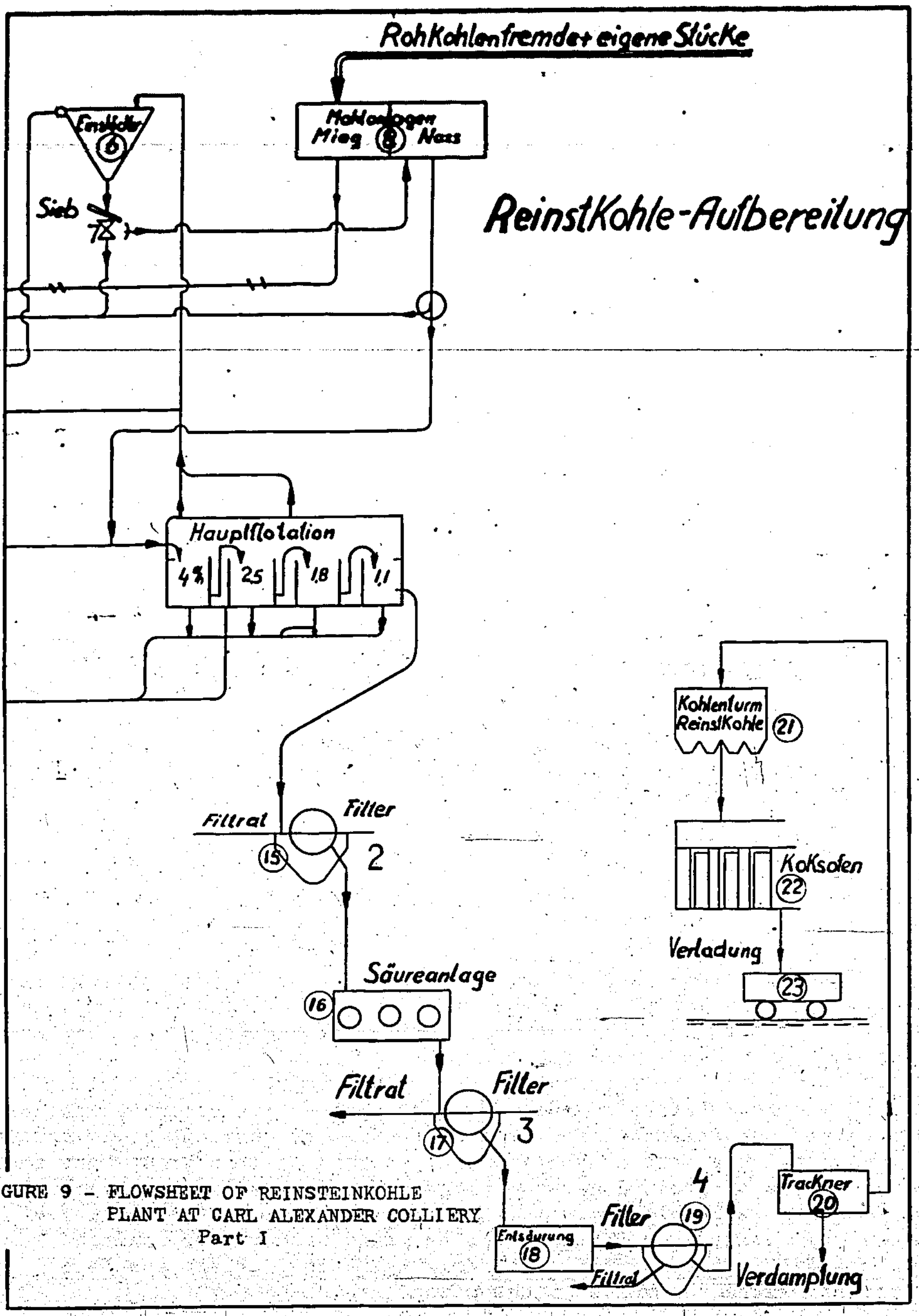

FIGURE 9 - FLOWSHEET OF REINSTEINKOHLE
PLANT AT CARL ALEXANDER COLLIERY
Part I