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INDEX - MICROFILM REEL 239
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AUTHORS

BARDGETT - PRENTICE (C.I.O.S.)
BARDGETT-POSTLEWAITE(B.I.O.S.)

	C.I.O.S. File No.	Item No.
BARDGETT, H. Coke-oven installation of Reichwerke A. G., Hermann Goring Werke at Watenstedt near Brunswick. (1945) 62 p., incl., tables, (part fold.), diagrs., (part fold.) (PB 46962) Investigators: H. Bardgett(Br.); F. H. Reed, L. D. Schmidt (U.S.)	XXXIII-37	30
BAYER, O. Polyurethanes, (by) Dr. O. Bayer (I. G. Farbenindustrie, Leverkusen). Translated from C.I.O.S. Report No. XXIX-12 (PB 12635, Roberts, F. H.) by L. V. Chilton. (1946) 22 p., illus., diagrs. (PB 46961) Lecture given at T. A. Meeting, Frankfurt/Main, Sept. 24, 1941.	XXIX-12 (Appendix)	22
CHAFFEE, C. C.; ATWELL, H. V. I. G. Farbenindustrie, Mainkur - Hoechst. 1945. 33 p. diagrs. Pages 12 - 33 in German. (PB 2443)	XXV-26	30, 8, 22, 24
CHAFFEE, C. C.; OZOL, R. J. Compilation of German fuels and lubricants specifications (1945) 29 p. tables. (PB 23020)	XXXI-58	30
HIRSCHKIND, W.; LOWDERMILK, F. R. Fischer-Tropsch and allied processes, 26 June 1945. 24 p., incl. diagrs. (PB 474)	XXVII-82	22, 30
NEUMANN, R.; BATES, H. H. Nordstern coke-oven plant, Gelsenkirchner Bergwerke A. G. (1946) 7 p. fold. diagr. (PB 46963) Investigators: R. Neumann, H. H. Bates (Br.); R. Powell (U.S.).	XXXIII-40	30

	C.I.O.S. File No.	Item No.
PRENTICE, T. M. Styroflex, a plastic produced by Norddeutsche Seekabelwerke. (1945?) 6 p. (PB 1847)	XXX-63	22

	B.I.O.S. Final Report Number	Item No.
BARDGETT, H. Drying, briquetting and low-temperature carbonisation of brown coal in Lurgi-Spülgas retorts at Braunschweigische Kohlen Bergwerke Schwelwerk Offleben near Helmstedt. (1946) 19 p., incl. diagrs. (B.I.O.S. Trip No. 2153)	626	30
BARDGETT, H. Gesellschaft für Teerverwertung Varziner-Strasse, Duisberg-Meiderich, Ruhr. (1946?) 16 p., diagr. Investigators: H. Bardgett,(Br.); F. H. Reed, L. D. Schmidt,(U.S.). Date of visit: July 31, 1945.	596	30
(BELL, W. C.; COOKE, V. R.; CHAMBERLAIN, H. L., and others.) Top stripping equipment used in open caste coal workings, reported by members of M.O.S. (1946?) Tube Mission Team. 6 p. diagrs. (PB 48433) (B.I.O.S. Trip No. 993, Sept. 1945.) Investigation made at Knapsack and Hermulheim.	617	31
BOOTH, J. C. H.; REAY, J. L. German methods of manufacture of copper tuyeres for blast furnaces. (1946) 18 p., diagrs. (B.I.O.S. Trip No. 2033 — Mar. 1946) (PB 42745) Date of investigation: April, 1946.	670	21
BROOKBANK, T.; GREEN, J. A.; WEMYSS, W. D. Band oil filter, oil refinery at Porta (nr. Minden). (1946?) 9 p., incl. diagrs. (PB 23813)	324	30
BROWN, W. The microanalytical methods employed in the analytical laboratories of I. G. Farben, Elberfeld-Wuppertal, Germany. (1946) 7 p. (B.I.O.S. Trip No. 1552.) (PB 45245)	715	22
CAMBRON, A. Catalytic hydrogenation of acetylene to ethylene, Dr. Alexander Wacker Ges. fur Elektrochemische Industrie, Burghausen, Sept. 1945. 7 p., incl. diagrs. (B.I.O.S. Trip 1155) (PB 44954)	171	22

	B.I.O.S. Final Report Number	Item No.
CLARK, G. C. Manufacture of cyclohexanol, cyclohexanone, cycloketone resins, at I. G. Ludwigshafen. (1946) 12 p. diagrs. (B.I.O.S. Trip 1124.) (PB 47719)	743	22
(EVANS, E. C.; FROST, R.; ELLIOT, G. D.; RIDGION, J.M.) Iron ore preparation in Germany. Reported by members of B.I.O.S. Trip 1234, Nov., 1945. (1946) 22 p. tables, diagrs. (PB 40293)	592	21
GREGG, R. J.; BRINSLEY, F.; EWART, J. H. German domestic gas appliance industry. (1946) 46 p. (B.I.O.S. Trip 1829.) (PB 34731)	588	30
HOUSEMAN, C. R. Production of nitrous-oxide GMI. (1946) 12 p. pl., diagrs. (B.I.O.S. Trip 2207)	672	22
HOWLETT, J. R. B. German steel drum industry. (1945) 103 p., incl. 22 diagrs. (B.I.O.S. Trip 1546.) (PB 34734)	531	21, 28
HUNTER, W. C. F. Boehringer und Soehne, Mannheim-Waldhof. Commercial organic solvent production. (1946) 9 p., incl. diagr. (B.I.O.S. Trip 1124.) (PB 47724)	740	22
HUNTER, W. Hydrocyanic acid, I. G. Oppau and Ludwigshafen. (1946) 7 p. diagr. (B.I.O.S. Trip 1124.) (PB 47717)	754	22
HUNTER, W. The production of tetranitromethane and nitroform at I. G. Farbenindustrie, Hochst-am-Main. July, 1946. 15 p., incl., diagrs. (B.I.O.S. Trip 1919) (PB 47730)	709	22
HUNTER, W. Manufacture of phthalic anhydride and phthalates at I. G. Ludwigshafen. (1946) 20 p., incl. diagrs. (B.I.O.S. Trip 1124) (PB 47718)	753	22
JONES, F. A.; GORDON, M.; BROWN, F.; COOPER, W. Interview with Professor Otto Bayer, formerly member of the Directorate and head of Scientific Laboratories of the I. G. Farbenindustrie, Leverkusen. (1946) 8 p. (PB 45246)	719	22

	B.I.O.S. Final Report Number	Item No.
LANNING, H. J. Manufacture of synthetic resins by I. G. Farbenindustrie, Ludwigshafen. (1946?) 5 p. (B.I.O.S. Trip 1124.) (PB 47731)	663	22
LANNING, H. J.; CLARK, G. C. Manufacture of fatty acids by oxidation of paraffins, hydrogenation of the fatty acids, at I. G. Ludwigshafen-Oppau. (1946) 22 p., incl. diagrs. (B.I.O.S. Trip 1124) (PB 47720)	748	22
LOVELESS, A. H. Distillation of phenols at I. G. Farben Fabrik, Wolfen. (1946) 7 p. (B.I.O.S. Trip 1901) (PB 34743)	560	22, 30
MATTHEWS, M. A. Ethylene oxide by direct oxidation of ethylene, at Zweckel, I. G. Farbenindustrie. (1946?) 4 p., incl. diagr. Date of visit: Oct. 2, 1945.	686	22
MATTHEWS, M. A. Manufacture of ethylene cyanhydrin at I. G. Ludwigshafen. (1946) 3 p. diagr. (B.I.O.S. Trip 1124.) (PB 47716) Interrogation of Dr. Roser, Sept. 26, 1945.	757	22
MATTHEWS, M. A. Pilot plant for manufacture of acrylonitrile at I. G. Oppau. (1946?) 4 p. diagr. (B.I.O.S. Trip 1124.) (PB 47715) Interrogation of Drs. Bauer and Keller, Sept. 28 & Oct. 1, 1945.	759	22
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PHILIP, T. B. Notes on items of chemical plant at works of I. G. Farbenindustrie, Knapscak; Dr. Alexander Wacker, Burghausen; Anorgana, Gendorf; I. G. Farbenindustrie, Hoechst. (1946) 17 p. (PB 45244)	713	22, 31
PLUMMER, G. A. LaMont boilers in Germany for naval and mercantile marine use. (1945) 261 p., incl. illus., tables, diagrs., (part fold.) (PB 31236)	382	29

	B.I.O.S. Final Report Number	Item Number
POSTLETHWAITE, J. P.; BONDY, H. F. The continuous tar distillation still at Gesellschaft für Teerverwertung. (1946) 15 p. (B.I.O.S. Trip 2153) (PB 42744) (Vacuum still designed for fractionation of dehydrated high temperature tar.)	615	30

ITEM No. 30

FILE No. XXXIII—37

Copy 1

COKE-OVEN INSTALLATION OF REICH-WERKE A.G., HERMANN GÖRING WERKE AT WATENSTEDT near BRUNSWICK

Bardgett, H.

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SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

Coke-Oven Installation of Reichwerke A.G., Hermann Göring Werke at Watenstedt near Brunswick

Reported by

H. Bardgett (Br.)

on behalf of

Fuel Research Station, D.S.I.R.

and

U.S. Technical Industrial Intelligence Committee.

C.I.O.S. Black List Item 30
CIOS Target Number: C30/409

Fuels & Lubricants

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Table of Contents

Subject	Page No.
Location of target	1
Condition of target	1
Personnel interrogated	1
Date of investigation	1
Introduction	1
Coal Supply	2
Coal handling, preparation and blending	3
Control of blends	4
Coke Ovens	5
Heating of the ovens	6
Coke	7
By-product recovery	8
Proposed high-pressure benzol recovery	9
Gas purification and sulphur recovery	9
Benzol fractions	10
Liquor	11
Tar	11
Gas	11
Labour	12
Costs	12
Table 1 - Analyses and Properties of Coking Coals	13 & 14
Table 2 - Typical Flue Temperatures	15
Appendix I - List of Documents	(i)
" II - Guarantee Test on Still Ovens	(ii)
" III - Guarantee Tests on Four types of Ovens	(xiii)
" IV - Description of High-Pressure Benzol Recovery Process	(xxi)
" V - Description of Sulphur Recovery Process	(xxiii)
Fig. 1 - Temperatures in Gas Space above Charge	
" 2 - Typical Flue Temperatures - No. 4 Battery	
" 3 - Flow Sheet - No. 1 Coke Oven Plant	
" 4 - Temperatures of Cooling Water and Gas - Pressure gas - Contents of NH_3 in Liquor and H_2S in Gas.	
" 5 - Process of Operation for Benzol Purification - Jan. - Nov. 1944	
" 6 - Flow Sheet for Production of Coumarone-Resin	
" 7 - Flow Sheet of an Acid Regeneration Plant - Menzen Bochum System	

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Coke-Oven Installation of Reichwerke A.G., Hermann Göring Werke at Watenstedt, near Brunswick

Location of target: The plant is situated at Watenstedt near Salzgitter, about 10 miles South of Brunswick.

Condition of target: Approximately 10-15 per cent. damaged, including bomb damage to 7 ovens in one battery, and destruction of one benzole washer, ammonia still and one gas purification and sulphur recovery unit.

Personnel interrogated:

Dir. Strickrodt - Acting General Manager
Eduard Schiegries - Coke Oven and Blast furnace Superintendent
Heinrich Joswig - Coke Oven engineer

Most of the information obtained was from Herr Schiegreis, who was very helpful in supplying data and in preparing copies of reports, diagrams etc. which were desired.

Date of investigation: 16th - 23rd July, 1945.

Introduction

The installation of ovens which comprises in all eight batteries of four different types, the first of which commenced operation in 1939, is one of the most modern in Germany with a rated capacity of 10,000 tonnes of coal (moisture 8%) per day. There are three batteries each of Otto and Koppers ovens and one battery each of Becker and Still ovens.

The coking plant had been planned to be the largest in the world, the layout being so arranged that the plant could be eventually extended to comprise 16 batteries in line covering a distance of 4 kilometres. At the time of the visit six batteries were completed and had been in operation, the seventh was completed but had not worked and the eighth was under construction. The ovens are arranged in two sets of four batteries, each with its separate by-product plant.

The main purpose of the ovens is to provide coke for the 16 blast furnaces of the adjacent iron and steel plant. Eight of these modern furnaces are of 6.8 metres hearth diameter and are rated at 350 tonnes of iron per day, and eight are of diameter 7.2 metres and are rated at 650 tonnes per day. They operate on oolitic ore (Fe_2O_3), concentrated to about 40% Fe, from a local deposit. The coke from four batteries of ovens supplies eight blast furnaces.

Additional features of interest at the works are :-

1. A combined Velox boiler and gas turbine power unit. The boiler is of capacity 40 tons of steam per hour and is heated by blast-furnace gas, the waste combustion gases being used to drive the gas turbine. The turbine was designed by Schattschneider of the firm of Brown Bouverie.

2. A gas turbine in course of erection, by the same designer, intended to operate on the products of combustion, cooled to 500°C., of blast furnace gas. The turbine, which was 2-stage, was said to develop 4000 H.P. and would have an efficiency of 30%. It was intended for driving compressors to supply air to the blast furnaces.

The coke ovens were selected as a target for investigation mainly because it was understood that weakly caking coals from Upper Silesia were utilized in the production of blast furnace coke and also because the installation comprised different types of ovens operating on similar coal. It was hoped that the results obtained on the plant might throw light on the relative performance of the different ovens.

Coal Supply

The ovens were intended originally to operate entirely on coking coals from the Ruhr. During the war, however, when the supplies obtained from the Ruhr were insufficient it had been necessary to augment the supplies by obtaining coal from other areas and it had been necessary to utilize a proportion of weakly caking coal from Upper Silesia. The management would prefer, in order to make good coke, to use only Ruhr coals but they found it possible to make satisfactory coke from blends containing up to 30% of Upper Silesian coal.

Table 1 shows the types and sources of the various coals used with proximate analyses, sulphur content, swelling pressure and coking indices as far as this information was available. Swelling pressure was determined by the method of Damm* and coking indices by the method of R. Kattwinkel.

Coal was received at the plant as washed slack of size 10 mm. - 0 except that from Upper Silesia which was generally unwashed. The Upper Silesian coals were fairly high in ash content and the ash was said to be higher in silica and lower in alumina than that of the Ruhr coals.

* Koppers and Jenkner, Gluckauf 1931, 67, 358.

A typical blend of Rhenish-Westphalian and Upper Silesian coals supplied to the ovens was as follows :-

Coal	Per cent. in blend	Vol. matter %	Ash %
Bergmannsglück	20	19 - 20	7-8
Blumenthal	10	20 - 21	7-8
Julia	40	24 - 25	7-8
Ewald and Heinrich Robert	10	28 - 29	7-8
Upper Silesian	20	32 - 34	10

Coal handling, preparation and blending

The works are equipped to handle coal received either by rail or by barge via the Mitteland Canal, a branch of which adjoins the plant. Coals from the Ruhr are received normally by barge, those from Upper Silesia generally by rail but sometimes by barge. The coal is unloaded by means of grabs, operated from overhead travelling bridges, either directly on to a belt conveyor or into concrete-lined pits located along the side of the canal basin. The unloading bridges comprise three of Lauchhammer type capable of dealing with 250 tonnes per hour and two of Pohlig type to deal with 500 tonnes per hour. There are sixteen storage pits each about 6 metres deep and of capacity 30,000 tonnes. They can be flooded with water if necessary to minimise oxidation of the coal by storage under water, or in the event of spontaneous heating.

Coal from the main conveyor belt is transferred to a transverse belt leading to the coal preparation plant, of which there is a separate and similar system for each set of four batteries of ovens. This system comprises two magnetic separators for the removal of tramp iron, three hammer mills, eight bunkers for storage of the separate crushed coals and two coal mixers. The hammer mills, of Humbold-Köln type, each has a rated capacity of 250 tonnes of coal per hour. Two only are normally in use and produce 5,000 tonnes of crushed coal per day, grinding the coal from size 10 mm. -0 to 90 per cent. below 3 mm. The mills, which are fed at constant rate by a drum and gate arrangement, contain hammers of manganese steel. After about four months use when one side of the tips of the hammers have become worn the hammers are turned and can then be used for about a further one month, after which time new tips are welded on to the old shanks. The crushed coal discharges from the mills through punched plate screens having slots 90 x 15 mm. spaced at 45 mm. centres.

The eight bunkers for storage of the crushed coals, each having a capacity of 500 tonnes are located in a blending tower. Coals from these separate bunkers, as required for the blend, are drawn off by volume on to steel pan conveyors feeding on to a belt conveying the mixed coals. The volume of coal discharged from any particular bunker is determined by the setting of a gate. The gate settings

are periodically checked by discharging the bunkers separately and weighing the coal on a length of the belt.

The measured quantities of the coals are next conveyed to two helical paddle mixers in series. The mixers are cylindrical shells about 12 ft. long each containing two horizontal shafts with 3 ft. diameter paddles rotating in opposite directions. The management considered that these mixers are essential to mix the coals sufficiently well to ensure the production of coke of uniform quality.

Coal from the mixers is conveyed by belt to five bunkers from which the ovens are charged.

Control of Blends

Constituent coals in the blends are normally proportioned in amounts to give a resultant blend containing 24-25 per cent. of volatile matter. Uniformity of blending is controlled by determinations of size grading, moisture, ash, volatile matter, caking index and swelling pressure. Coals are tested for swelling pressure by the method of Damm. Those showing a pressure of less than 1 kilo. per sq.cm. are considered not dangerous, those between 1.0 and 1.1 can be used up to 10% in a blend, while if the pressure exceeds 1.1 kg. per sq.cm. the coal is classed as dangerous and in normal conditions would not be employed. Coal from the Ewald mine which has the particularly low swelling pressure of 0.175 kg. per sq.cm. has been used in blends for controlling the swelling pressure within safe values.

Care is taken in the preparation of the blends to maintain the moisture content uniform at about 10 per cent. Sprays are fitted over the belt conveying the blend so that the necessary amount of water can be added. This is controlled by taking special samples each hour from the blend on the conveyor belt and also at the ovens. Moisture contents are reported within 30 minutes of sampling and if necessary the valve controlling the water spray is adjusted.

Facilities are also available for spraying tar on to the blended coals being conveyed from the mixing station. Experience has shown that where the blend contains Upper Silesian coal the coke produced is of inferior strength. The micum drum abrasion value of coke from blends containing 30 per cent. of Upper Silesian coal is only about 70 as compared with about 82 for good quality coke from Ruhr coal. The addition to such blends of up to 6-7% of raw coal tar, heated to 40-50°C. and sprayed on to the blend, increases the micum index up to 75-78. Brown coal tar was said to be more effective than bituminous coal tar in improving the resistance to abrasion of the coke.

Coke ovens

The oven installation consists of seven completed batteries each of 55 ovens and one battery of 55 ovens partly constructed. The types of ovens are as follows :-

Cokery No.	Battery No.	Type of Oven	Date of Completion
1	1	Otto Regenerative	started 1939 - 1940
	2	Becker Regenerative	
	3	Koppers Recirculation (Kreisstrom)	
	4	Still Regenerative	
2	5	Otto Regenerative	started 1944
	6	Otto Regenerative	" "
	7	Koppers Recirculation (Kreisstrom)	Finished but not started.
	8	Koppers Recirculation (Kreisstrom)	Under construction.

Drawings of sections of each type of oven were obtained. These are listed as items II, IV, V, and VI in the list of documents - Appendix I.

The principal dimensions of the ovens in each battery are similar and are shown below :-

Length of oven between doors	12,270 mm. (Koppers 12,440)
Height to crown	4,100 "
Mean width	445 "
Horizontal taper	75 "
Distance between centres	1,160 "
Thickness of roof	1,120 "
Depth of charge	3,800 "
Bulk density of charge (basis dry coal)	735 kg. per M^3
Useful capacity of oven (dry coal)	15.2 tonnes
Normal coking period	17.5 hours
Minimum coking period	16 hours

The ovens have self-sealing doors on the Wolff system and the management considered that the sealing was very good, the percentage of inerts in the gas being normally 9-10. Only minor repairs to the doors have been necessary up to the time when the plant ceased operating on 10th April, 1945.

Analytical data relating to the bricks used in the Koppers and Otto batteries were obtained. These show that the oven bricks is both types contained about 94.5% SiO_2 and 2.0 Al_2O_3. In the Otto batteries the bricks in the regenerators contained 75% SiO_2 and 20% Al_2O_3 and those on the doors 70% SiO_2 and 25% Al_2O_3.

One feature of difference between the different types of oven which was considered of importance by the management is the distance between the top of the heating flues and the oven top. With the Still ovens, where this dimension is least, considerable trouble was said to have been experienced due to excessive cracking of the gas above the charge, with consequent carbon deposition. Considerable amounts of scurf of thickness over 1 inch which it was said had been removed from the Still ovens were observed during the visit. Determinations had been made of the temperature in the gas space above the charge in each type of oven when operating at similar flue temperatures. These determinations were made by inserting thermo-couples through an end charging hole, so as to bring the junction to the centre of the height of the gas space. Determinations made 11 hours from the start of coking in a total coking period of 17.6 hours gave the following results.

Temperatures in Gas Space above Charge

Battery No.	1	2	3	4
Type of Oven	Otto	Becker	Koppers	Still
Dimensions from Top of Flues to Top of Oven - mm.	470	600	700	370
Temperatures in Gas Space after 11 hours - deg.C.	800	780	750	820

Other determinations showing the rise of temperature in the gas space above the charge in each type of oven are shown in Fig. 1. The temperatures above the charge are normally maintained as far as possible between 740 and 780°C. This range is considered the optimum for maximum benzole production and for the avoidance of troubles due to excessive cracking and deposition of carbon in the ovens and of naphthalene deposits in the condensers.

Details were obtained of a guarantee test made on the Still ovens in 1942 after they had been in use for a period of two years. The test was over a period of six days during which the ovens were heated with coke oven gas. The test included determinations of (1) coal throughput (2) fuel consumption (3) flue temperatures (4) temperature and composition of the waste gases. The report on this test forms Appendix II of this report.

Heating of the ovens

All batteries are designed for heating either by coke oven

gas or by blast furnace gas from the adjoining iron works. Table 2 gives typical average flue temperatures on both machine side and coke side for each of the ovens in each of the batteries 1 to 4, when heated by blast furnace gas. These values were taken from records for the same day, 8th April, 1944. Typical cross flue temperatures for four ovens in the No.4 battery of Still ovens are shown in Fig.2.

Appendix III shows details of the results of guarantee tests carried out in July - August, 1942, on each of the four types of ovens, heated both by coke oven and blast furnace gases. The values for heat consumption vary between about 530 and 580 k.cal. per kg. of coal as charged containing approx. 10% moisture.

Coke

The oven installation was designed for a minimum coking period of 16 hours. Experience, however, has shown that a period of 17 - 17.5 hours is necessary to ensure satisfactory pushing of the coke. The coke after discharge is quenched automatically for 120 seconds using 1 M^3 of water. After quenching the coke is screened twice over 25 mm. shaker screens. All coke over 25 m.m., said to be about 90% of the total, is used in the blast furnaces. Considerable breakage of the large coke apparently occurs in handling between the ovens and blast furnaces as the maximum size of pieces received at the furnaces was said to be about 50 m.m. Coke of size 0-25 m.m. is used for ore sintering in the Dwight-Lloyd process.

Information was not available relating to yields, screen analyses, or physical properties of cokes from the different ovens, but it was stated that the size of coke from each type of oven was similar. Data were not available relating to uniformity of temperature of coke throughout the oven, but measurements by optical pyrometer of the coke at discharge gave usually about 1050°C.

Tests normally applied to the coke are proximate analysis and micum trommel test. The trommel index varies usually from 75 to 80 from blends containing a proportion of Upper Silesian coal. Good quality Ruhr coking coal gives coke of index 85. Some preliminary tests had been made on determinations of coke friability in the Hoesch A.G. Iron and Steelworks test*, (the Wolff test), in which a definite volume of coke is compressed against an outlet flap valve and the coke then screened on a 30 m.m. sieve to determine the amount of undersize. This test, in which cokes are subjected to a pressure of 1-2½ kg. per sq.cm., is said to differentiate more clearly than does the trommel test between cokes of different abradability. Details of the results of these tests were not available.

* See Grundlagen der Koks-Chemie - Simmersbach - Schneider, 1930, p.326.
and W. Wolff : Stahl u. Eisen 1928, p.33.

The coke oven and blast furnace superintendent, Schiegries, who had had much practical experience of operating blast furnaces, considered that none of the tests normally applied to blast furnace cokes was of much value in evaluating cokes for this use. He preferred to judge the coke by appearance and placed much value on the appearance of the coke near the tuyeres as seen through inspection holes. A good quality coke, in his experience, remained of uniform size at this level in the furnace and had very little small coke between the larger pieces.

By-product recovery

The layout of the plant is such as to provide a complete recovery system for each set of four batteries. Fig.3 shows diagrammatically the flow sheet of the gas. The system consists of the following :-

1. Five primary coolers. These are indirect tube coolers each of 3,000 sq.metres cooling surface and each containing 1,200 tubes of outside diameter 89 m.m. through which the cooling water flows. The gas is cooled from about 90 to 25°C.

2. Gas exhauster of standard turbine type of capacity 110,000 M³ per hour, operated at about 75% of capacity. The gas temperature rises to 35°C. at the exhauster outlet.

3. Two secondary coolers of indirect type and each of 2,000 sq.metres cooling surface. These cool the gas again to 25°C. Crude gas containing tar is admitted to the last coolers to act as a solvent for naphthalene.

4. Four Lurgi-type electrostatic precipitators operated at 60,000 volts.

5. Four ammonia washers each 5 metres diameter and 45 metres high containing wood hurdles. Pre-softened water is used as unsoftened water had been found to cause troubles due to ammonium carbonate. In order to avoid destruction of the wooden hurdles the naphthalene deposited is not steamed out but is periodically removed by washing through with tar oil.

6. Ten Bamag type purifier towers each containing 600 tonnes of iron oxide for the removal of H_2S from the gas. The gas temperature rises from 25 to 45°C. during passage through the towers.

7. Two indirect coolers, similar to those in item 3, in which the gas temperature is reduced to 25°C.

8. Benzol washer consisting of a cylindrical shell 20 metres diameter divided into five segments containing wooden hurdles. The wash-oil is a tar fraction 200-300°C. The benzol content of the gas is reduced from 25-30 to 1.5 gm. per M³. The oil is enriched to 2.5% benzol before distillation.

9. Gas holder. This is a 250,000 M³ Klonne waterless type holder which for reasons of safety was not in use during the war.

10. Gas compressors. The gas is compressed to 19 atmospheres in six compressors each of 24,000 M³ capacity and is then supplied to the grid feeding Berlin, Magdeburg, Hanover, Brunswick, Hildersheim, etc.

Fig.4 shows temperatures of cooling water and gas, and pressures of gas throughout the system together with contents of NH_3 in liquor and H_2S in gas.

Proposed high-pressure benzol recovery

Arrangements had been made to install plant designed by Koppers for gas purification and benzol stripping at 19 atmospheres pressure to follow the existing plant, but the process had not been applied. It was proposed to cool the gas from 150° to 25°C. in four coolers, the remaining H_2S, 2 gm. per 100 M³, being reduced at 19 atmospheres by iron oxide to 0.25 gm. per M³. The gas was then to be scrubbed with tar oil to remove all but traces of the residual 1.5 gm. of benzol per M³. Any residual naphthalene would also be removed at this stage. The scrubber was to contain a series of 7 perforated trays.

Drawings showing the flow-sheet and arrangement of the plant for this proposed process were obtained and are quoted as items XII and XIII in the list of documents - Appendix I.

A description of the process, prepared by the contractors H. Koppers G.m.b.H. is given in Appendix IV.

Gas purification and sulphur recovery

Drawings were obtained, listed in Appendix I as items XIV and XV, of the gas purification plant showing the layout and details of the tower. Each tower contains 16 baskets containing iron ore in two layers 30 cm. deep with a 10 cm. space between the layers.

Gas enters the tower at the bottom, is distributed by a spiral baffle in the bottom compartment and then enters a central manifold from which it passes through the baskets in parallel. Air, equivalent to one per cent. oxygen, is added to the gas entering the purifiers and the relative humidity is maintained at about 90% by means of water sprays at the bottom of the towers. Removal of H_2S is from 8 gm. per M^3 in the gas entering the first tower to 2 gm. per 100 M^3. The gas temperature rises from 25 to 45°C. during passage through the towers.

The oxide is used until the content of sulphur rises to about 45% at which stage it is removed from the tower and the sulphur recovered by extraction with CS_2. A description of the process of sulphur recovery is given in Appendix V. Drawings of the plant were obtained and are listed as items XVI, XVII and XVIII in the list of documents.

The iron oxide used in the purifiers could, it was stated, be used and extracted two or three times, after which it was sold to paint manufacturers.

The cost of oxide purification was said to be 25 pg. per M^3 of gas.

The cost of recovery of sulphur by CS_2 extraction was quoted as 60 RM per tonne of raw sulphur (98%) or 70 RM per tonne of refined sulphur (99.5%). The selling prices were given as 100 and 115-120 RM per tonne of raw and refined sulphur respectively. Spent oxide if sold prior to recovery of sulphur would only realise, it was stated, 10 RM per tonne of sulphur present. Refining of the raw sulphur is effected with oleum with 20-25% SO_3.

Benzol fractions

The total yield of crude benzols represents 0.95 - 1.0 per cent. by weight of the dry coal.

Fig. 5 shows a flow diagram, with yields, of the system of benzol purification. According to requirements the crude benzol is distilled into the following products :-

Motor benzole : Initial b.p. 80°C. : 95% distills up to 155°C.
Pure 90's benzole : 90% distills up to 100°C.
Pure benzol : Initial b.p. 80°C. : 95% distills up to 85°C.
Benzene : 90% distills within 0.6°C. of the initial b.p.
Pure toluol : Initial b.p. not less than 100°C. : at least 90% distills up to 120°C.
Toluene : 90% distills within 0.6°C. of the initial b.p.
Pure xylenol : Initial b.p. not below 120°C. : at least 90% distills up to 145°C.

Xylenes : 90% distills within 3.6°C. of the initial b.p. : 90% within 4.5°C.
Pure solvent naphtha I : Initial b.p. not below 120°C. : at least 90% up to 160°C.
Pure solvent naphtha II : Initial b.p. not below 135°C. : at least 90% up to 180°C.

Coumarone resin, used for lining food containers and in aeroplane lacquers is recovered from the light oil by the scheme shown in Fig. 6. The production cost and selling price were quoted as 250 and 400 RM per tonne respectively.

Liquor

Ammonia is recovered by the indirect system. The sulphuric acid used in the production of ammonium sulphate is regenerated from the waste acid from benzol purification. The scheme of acid regeneration is shown in Fig. 7.

Phenol is recovered from the liquor as sodium phenate by the Pott-Hilgenstock process in which the phenol is washed out with benzol and then treated with caustic soda.

Tar

The yield of crude tar of specific gravity 1.12 - 1.15 and containing 3-5% water represents 2.7 - 3 per cent. by weight of the dry coal.

The crude tar is not distilled but is delivered to Gesellschaft für Teerverwertung in Castrop-Rauxel.

Gas

The yield of gas calculated to a basis of 4,000 k.cal. per M^3 at 0°C. and 760 m.m. is normally 330 - 340 M^3 per tonne of dry coal.

Typical analyses of the coke oven gas before and after benzol scrubbing (taken on different days) and of blast furnace gas used for heating are as follows :-

	Coke oven gas		Blast furnace gas
	Unscrubbed	Scrubbed	
CO_2	3.2	2.4	7 - 8
C_2H_4	2.8	2.0	-
C_6H_6	0.7	-	-
O_2	0.5	0.8	nil
CO	9.3	7.1	27 - 30
H_2	48.8	53.0	1.5 - 2.5
CH_4	27.3	22.6	-
N_2	6.4	12.1	up to 60
Gross C.V. k.cal/M^3	5030	4422	950

As previously stated, the gas is compressed and supplied to the grid. Surplus gas is used for power station and steel foundry purposes and peak quantities for heating the ovens.

Labour

The total personnel employed on the coke oven plant, working eight hour shifts, was about 520 men. These were for the following operations :- Handling and storing coal, crushing plant, blending tower, coal tower, operation of ovens, coke screening and loading, condensers, phenol plant, engine room, ammonia washer, ammonia works, gas purification, sulphur extraction and refining, recovery of crude benzol, benzol purification, compressor station, workshop and works control and supervisory staff.

Costs

The following table shows costs data supplied by the management. The costs are based on the use of coking coal of volatile matter content about 23%.

	Per ton of coal as coked		Per ton of blast furnace coke. Yield 70%
I Including capital costs	R.M.		R.M.
Coking coal	17.80		25.43
Operating costs	6.30		9.00
Capital outlay	1.23		1.75
Administration	0.32		0.45
	25.65		36.63
Credits for coke breeze, tar, ammonia, benzol and gas	12.43		17.75
Coke for despatch (0.7 t)	13.22	(1.0 t)	18.88
2 Excluding capital and Administrative costs			
Coking coal	17.80		25.43
Operating costs	6.30		9.00
	24.10		34.43
Credits	12.43		17.75
Coke for despatch (0.7 t)	11.67	(1.0 t)	16.68

Table 1.

Analyses and Properties of Coking Coals

Source of Coal (Mine)	Volatile Matter %	Moisture %	Ash %	Sulphur %	Swelling Pressure Kg/Cm²	Coking Index kg.
Group I. 24-28% V.M.						
Ewald unwashed	28.79	3.70	12.62	1.38	0.175	6.16
washed	24.12	8.70	6.34	1.20	1.000	5.25
Friedrich Heinrich	24.47	9.10	5.26	1.07	0.800	6.04
Gneisenau	25.85	8.90	6.28	1.54	0.500	5.86
Grimberg	26.87	7.70	6.05	1.21	0.900	5.59
Gustav	26.27	11.10	7.96	1.55	-	7.22
Heinrich Robert	29.25	10.20	7.38	1.41	0.620	5.43
Julia	24.50	10.21	6.16	1.24	0.875	8.35
Minister Stein	25.82	11.40	6.48	1.32	-	7.79
Monopol Shaft Grimberg	24.46	9.60	5.92	1.15	1.000	6.28
Monopol Shaft Grillo	24.46	9.60	5.92	1.15	0.900	6.38
Rheinbaben	24.69	10.40	4.79	-	-	-
Rheinpreussen	26.97	7.90	5.81	1.12	-	5.25
Sachsen	25.45	10.00	7.34	1.17	1,000	6.80
Viktoria	26.81	11.30	7.67	1.19	1.050	5.71
Viktor	24.08	10.40	9.42	1.64	1.060	6.31
Nordstern	28.79	-	-	-	-	-
Group II. 20-24% V.M.						
Carolus Magnus	22.00	-	-	-	0.700	12.31
Constantin der Grosse	19.29	-	-	-	1.000	6.30
Erin	23.38	-	-	-	0.750	4.83
General Blumenthal	21.27	11.20	7.54	1.23	0.500	5.18
Hannibal	22.09	11.12	8.19	1.00	0.550	5.40
Holland	22.74	-	-	-	1.020	5.77
König Ludwig	22.93	9.36	10.21	1.57	0.850	6.28
Lothringen	20.74	-	-	-	-	-
Neumühl	21.57	9.80	9.26	1.17	0.625	7.19
Präsident	20.95	9.90	5.53	1.17	0.775	5.52
Prosper III	23.22	5.60	10.25	1.32	0.375	5.72
Robert Müser	22.49	9.30	6.38	1.10	0.600	6.48
Schwerin	21.43	9.50	8.43	1.09	0.975	13.64
Waltrop	20.40	6.50	7.94	1.26	0.975	3.72
Shamrock	22.25	-	-	-	-	-
Westerholt	20.43	-	-	-	0.750	5.78
Zollern I	21.23	9.70	7.92	1.32	0.925	7.10
Zollern II	23.68	-	-	-	0.550	5.67
Group III						
Dorstfeld	24.72	11.60	6.86	1.15	1.100	12.16

Table 1 - continued.

Source of Coal (Mine)	Volatile Matter %	Moisture %	Ash %	Sulphur %	Swelling Pressure Kg./Cm²	Coking Index kg.
Group III						
Emscher Lippe	20.40	8.70	7.71	1.32	1.100	7.37
Konigsborn 1/2	21.09	10.30	7.41	1.28	1.100	6.16
Westfalen 1/2	23.87	9.20	5.44	1.17	1.100	4.84
Group IV. - Lager Coal						
Bergmannsglück	19.91	8.60	6.81	1.42	1.100	6.31
Centrum I/III/VII	18.53	9.90	6.22	1.00	1.025	5.60
Konigsborn 3/4	19.59	7.50	6.66	1.30	1.200	4.35
Hansfeld	19.61	11.30	7.47	1.02	1.050	5.30
Neu Iserlohn	19.57	9.90	6.56	1.12	1.700	7.21
Ospel	19.68	9.80	6.93	1.06	0.850	2.98
Prinz Regent	15.44	13.00	7.84	1.09	0.660	1.72
Wolfsbank	18.02	11.60	10.11	1.04	0.675	5.84
Sälzer-Amalie	17.90	-	-	-	-	-
Group V - Gasflamm Coal						
Bismark	32.66	-	-	-	-	-
Schlägel und Eisen	28.94	-	-	-	-	-
Upper Silesian Coal						
Annagrube	33.02	9.40	6.35	0.72	-	16.00
Charlottegrube	33.62	7.00	10.72	0.84	-	13.00
Emmagrube	32.45	10.10	10.81	1.11	-	-
Ferdinandgrube	30.48	15.20	9.50	0.97	-	0
Hoymgrube	35.16	10.10	6.81	0.73	-	13-14
Knurowgrube	32.48	9.80	7.64	1.01	-	-
Königsgrube K.	29.47	6.90	13.90	1.11	-	14.00
" E.	33.77	8.50	13.4	1.18	-	-
Myslowitzgrube	37.12	11.20	3.92	0.52	-	0
Oehringen E.	29.71	4.80	16.33	1.14	-	-
" W.	28.75	3.80	12.90	1.04	-	16.00
Olsa	26.99	2.60	19.75	0.59	-	17.00
Paxgrube	29.66	3.60	16.46	0.87	-	0
Richterschächte	31.71	11.40	9.20	1.38	-	0
Rönergrube	33.88	3.80	5.77	0.75	-	15.00
Sashitza	30.58	4.20	12.58	1.07	-	16.00
Königsgrube W.	34.59	7.40	8.65	1.41	-	15.00
Lower Silesian Coal						
Gluckhilf-Hermsdorf	20.52	10.00	7.91	1.07	-	-
Bahnschacht	22.79	9.80	7.93	1.16	-	-

Table 2 - Typical Flue Temperatures

Average temperatures of all flues on both machine side and coke side

Data relates to 8th April, 1944.

WALL No.	BATTERY No. 1 OTTO M.S.	BATTERY No. 1 OTTO C.S.	WALL No.	BATTERY No. 2 BECKER M.S.	BATTERY No. 2 BECKER C.S.	WALL No.	BATTERY No. 3 KOPPERS M.S.	BATTERY No. 3 KOPPERS C.S.	WALL No.	BATTERY No. 4 STILL M.S.	BATTERY No. 4 STILL C.S.
1	1330	1360	29	1230	1360	111	1280	1320	139	1210	1290	166	1260	1290	194	1260	1240
2	1270	10	30	30	50	112	1190	1270	140	60	1320	167	30	90	195	70	70
3	80	1250	31	30	1290	113	70	50	141	40	20	168	40	80	196	70	70
4	20	90	32	30	86	114	60	50	142	30	10	169	20	80	197	80	70
5	50	80	33	30	87	115	1200	80	143	10	1300	170	50	60	198	80	70
6	50	1300	34	30	88	116	1190	60	144	20	1270	171	30	40	199	50	70
7	50	1290	35	30	89	117	80	60	145	20	1310	172	1190	1300	200	80	60
8	10	90	36	50	90	118	70	40	146	50	10	173	1200	1280	201	80	70
9	10	90	37	40	91	119	80	40	147	10	10	174	10	80	202	80	70
10	30	90	38	60	92	120	1230	70	148	10	1290	175	40	1300	203	80	70
11	80	1350	39	60	93	121	30	80	149	30	90	176	20	1280	204	60	60
12	40	40	40	50	94	122	20	70	150	1200	1320	177	20	80	205	70	70
13	1170	00	41	70	95	123	10	70	151	30	10	178	40	90	206	70	70
14	1230	00	42	60	96	124	10	70	152	10	1290	179	40	70	207	70	80
15	20	1280	43	30	97	125	50	90	153	30	90	180	40	90	208	50	80
16	40	1330	44	40	98	126	70	1300	154	10	70	181	70	90	209	30	80
17	40	00	45	60	99	127	20	1280	155	50	10	182	70	90	210	30	60
18	30	20	46	70	100	128	10	80	156	50	00	183	60	70	211	30	70
19	50	1270	47	80	101	129	1180	60	157	30	70	184	60	90	212	30	80
20	20	80	48	80	102	130	1240	90	158	10	90	185	80	70	213	40	60
21	50	60	49	60	103	131	50	1300	159	10	80	186	80	90	214	1190	30
22	70	50	50	60	104	132	40	1280	160	40	1300	187	80	80	215	90	30
23	60	90	51	40	105	133	60	1300	161	10	1270	188	60	80	216	1200	50
24	90	60	52	50	106	134	40	1280	162	00	60	189	40	60	217	10	40
25	10	1310	53	50	107	135	40	1310	163	00	80	190	50	80	218	1180	30
26	30	1280	54	60	108	136	40	10	164	10	60	191	70	60	219	80	30
27	30	60	55	60	109	137	40	1300	165	10	1330	192	80	60	220	1210	50
28	1200	60		70	110	138	20	1300		20		193	60	40			

Consumption of Blast Furnace Gas : Cu. metres per hour.

Battery No.	M³/hr.
1	38,000
2	37,000
3	35,000
4	37,000

Appendix I - List of Documents - Bag No. 4501.

I	Untersuchungsvorschriften für die Kokeri - Laboratorien.
II	Regenerativ - Verbund - Koksofen, Bauart, "Still" D.R.P.
III	Ofenquerschnitte der Ofengruppe I bis IV. - Drawing No.613.
IV	Schnittdurch der Kreisstrom - Verb. - Ofen (Koppers).
V	Schnittdurch der OTTO Verbund ofen.
VI	Schnittdurch der Becker-ofen (transparency).
VII	Einfluss der unteren Füllochweite auf die Kohlenfüllhöhe im Koksofen bei einem Schüttwinkel von 55° - Drawing No.612.
VIII	Lageplan der Kokerei mit Nebengewinnung Ausbaustufe I - Drawing No.12002/14.
IX	Lageplan der Kokerei - Ausbau II - Drawing No. 22002/6B.
X	Elevation showing Plant lay-out
XI	Schema der Kokerei I
XII	Anordnung der Hochdruck-Benzol-wäsche u. Gaskühlung
XIII	Schaltungsschema der Hochdruckbenzolwaschung, Gastrocknung u. Entnaphthalinung.
XIV	Einbau einer Kühl-und Befeuchtungseinrichtung in die Gaskammern der Reinigertürme - Drawing No. 3919
XV	Übersicht zur Turmreiniger-Anlage und Lasse-Aufbereitung- Drawing No. He 62729 a.
XVI	Schwefelgewinnung - Schnitt AA, B-B
XVII	Schwefelgewinnung - Schnitt EE, K-K.
XVIII	Schwefelgewinnung - Schnitt D-D.
XIX	Schema einer Säureregenerieranlage.
XX	Schema der Cumaronharzherstellung.
XXI	Verbarbeitungsgang der Benzolreinigung nach den Betriebsergebnissen - Jan. - Nov. 1944 (Transparency)
XXII	Betriebsablauf der Gaskühl - und Waschanlage.
XXIII	Garantie-nachweis.- Tests on heat requirements of Ovens (8 sheets).
XXIV	Flue temperatures across Still ovens after 8 hours(Transparency)
XXV	Kokskohlen - Analyses. - 3 pages.
XXVI	Analyses of Silica bricks etc. - 12 pages.

GUARANTEE TEST ON STILL OVENS

Report on the Performance of the Coke Oven Setting (No. 4 Battery) Erected at the Hermann Goering Works in Watenstedt by the firm of Carl Still, Recklinghausen.

I. Heating with Coke Oven Gas

The coke oven setting consisting of 55 ovens was started up on the 16th July, 1940. The chief measurements are as follows -

Length of oven between fixing posts	12982 mm.
Length of oven between doors	12270 mm.
Total height of ovens to crown	4100 mm.
Mean width of ovens	445 mm.
Taper	75 mm.
Distance between centres of ovens	1160 mm.
Thickness of roof	1120 mm.
Height of filling	3800 mm.

Confirmation of the guaranteed performance with heating by rich gas took place from Friday, 3rd July, to Thursday, 9th July, that is after two years. This included as arranged a determination of (1) the throughput of the coal, (2) fuel consumption, (3) heating flue temperature, waste gas temperature and composition of waste gas, (4) the temperature of the make-gas in the descension pipe after the collecting main and the content of inerts in the make-gas. During this time the number of ovens to be discharged in 24 hours was fixed at 77; this corresponds to a mean period of operation of 17.2 hours per oven.

The course of the investigation involves the following details -

(1) The throughput is determined under the conditions that the coal supplied for coking is a well-washed, well mixed, homogeneous fine coal without inclusions of slurry and is non-swelling with at the most 12 per cent of moisture with a bulk density of 750 kg/m^3 of dry coal with at least 80 per cent below 3 mm. and not more than 25 per cent of volatile matter. The moisture content of the coal which must amount to 12 per cent at the most may only vary over 2 per cent. If it varies by more than 1 per cent this circumstance must be taken into consideration in an appropriate manner. The coal supplied must have not been stored for more than three months. The

throughput with a carbonising time of 17½ hours, a bulk density of 750 kg/m³ of dry coal and a moisture content of 10 per cent amounts to 1170 metric tons daily of dry coal. The bulk density of the coal was determined as 741 kg. of dry coal per cu.m. with a moisture content of 9.2 per cent. This corresponds to a throughput of 1156 metric tons of dry coal or of 1285 metric tons of coal with 10 per cent of moisture. The moisture content of the coal was measured in an average sample taken every shift. This sample was taken by sampling 5 charging cars every shift. The weights of coal in every charging car were taken.

(2) The measurement of the heating gas was carried out by means of an orifice plate. The differential pressure was recorded by means of a ring balance that had previously been calibrated. At the same time, the temperature of the gas was registered and the gas pressure was read before every changeover. The calorific value of the heating gas was determined in a Junkers hand calorimeter on a 24 hour average sample and the density in the same sample was measured in a Bunsen-Schilling apparatus.

(3) The heating flue temperatures were measured daily at 8.0 a.m. on both sides of the oven in No. 4 heating tube with a Pyropto (partial radiation pyrometer), and the waste heat temperature was registered. The waste gases were examined daily in individual ovens in the bend of the waste heat flue for their composition.

(4) Places for measuring the make-gas temperature are arranged at equal heights in the ascension pipes in all the batteries; further, the gas temperature was measured in the gas collecting space and through the lid of the ascension pipe at the height of the roof of the oven. The gas temperature immediately behind the collecting mains was read on two thermometers. Two average samples of the make-gas were taken daily. When the collecting mains were being cleaned secondary air was able to get into these samples and this gives a false picture of the composition of the gas. Therefore, the oxygen content was determined in separate samples. It amounted to 0.2 per cent. The make-gas analyses were recalculated to the basis of this oxygen content.

B. Experimental Results

The oven setting was changed over to heating by rich gas from a throughput of 71 ovens in 24 hours with lean gas heating on Thursday, 2nd July, at 9.0 a.m. The

experiment commenced on Friday, 3rd July, at 6.0 a.m. At the request of the works management the changeover was effected in such a way as not to interfere with running arrangements so that, from the start a well carbonised coke was discharged. It goes without saying that the oven setting was not brought to a standstill at the beginning of the experiment. No. 4 battery was charged as a unit together with No. 3 battery. In this way it was arranged that the individual ovens retained their prescribed periods of carbonisation, but the load on the oven setting at different times of the day was consequently different. The consequence of this was that fluctuations in the weight of the charge and in the water content of the coal must have considerable effect. The carbonising periods conditioned by the operation of the ovens varied between 16 and 18 hours. Disturbances in operation occurred in the night of the 2nd-3rd July, owing to air-raid warnings, owing to delays in the discharging of the neighbouring setting on the 4th July, and owing to damage to the discharging car on the 6th July. These disturbances had their effect on the subsequent carbonising times.

1. Throughput of Coal

As a result of an observed bulk density of 741 kg/m³ the mean throughput of the coal during the 6 days of experiment amounted to 1291 metric tons of wet coal with a moisture content of 10.1 per cent. This corresponds to 1159 metric tons of dry coal. The individual weights of coal are recorded with a moisture content in Table 3.

2. As can be seen from Table 5 the fuel consumption with a moisture content of the coal of 10 per cent, amounts to about 576 calories per kg. The limits of tolerance had to be claimed on the following grounds -

(a) The oven battery had not reached the requisite temperature owing to the short changeover period at the beginning of the experiment. Only on the fourth day of the experiment was equilibrium reached; until this time the heating flue and waste gas temperatures rose. Heat had still to be stored up in the oven space and in the regenerator during the experiment, and this was at the expense of the consumption of heat. Thus the mean heating flue temperature at the beginning of the experiment amounted to 1295° and at the end of the experiment to 1322°. The waste gas temperature rose from 245 to 277°.

(b) Owing to the great fluctuations in the moisture

content of the individual coal charges, combined with great fluctuations in the weight of coal charged to the individual ovens, the ovens had to be operated at a higher temperature, in order to obtain properly carbonised coke under these limiting conditions, than would have been necessary under constant operating conditions.

(c) The same holds for the individual carbonising time of the ovens themselves.

3. The composition of the flue gases can be seen from Table 5, also the mean heating flue and waste gas temperatures. The composition of the flue gas was fairly uniform during the six days of the experiment. The excess air amounted to about 20 per cent, and under these conditions a waste gas temperature of 267° was reached. The heat utilised accordingly during regeneration was good.

4. The make-gas temperature was determined in the ascension pipe at the places where it was measured, which were arranged in all batteries at the same height, to be about 590°; in the gas collecting space, 790°; in the ascension pipe at the height of the oven roof, 760°. A temperature of 850° was guaranteed. The inert constituents of the make-gas amounted on the average to 6.6 per cent, compared with 9 per cent in the guarantee.

Summarising, it may be said that the guaranteed conditions were complied with in all respects for heating the setting with rich gas.

Watenstedt, 10th July, 1942.

Carl Still Reichswerke Hermann Goering.

Table 1 Coking Periods (Gross) and Charging Times of Ovens.

Day of Test	1.					2.					
Oven No.	Coking Period hrs.min.	Time Charged hr. min.	Oven No.	Coking Period hrs.min.	Time charged hr.min.	Oven No.	Coking Period hr.min.	Time charged hr.min.	Oven No.	Coking period hr.min.	Time charged hr.min.
198		6. 10	216		19. 15	192	17. 20	6. 10	220	16.00	17. 25
193		6. 20	211		19. 23	187	17. 25	6. 20	215	17. 27	19. 03
188		6. 30	206		19. 30	182	17. 25	6. 30	210	17. 27	19. 10
183		6. 40	201		19. 36	177	17. 20	6. 40	205	17. 23	19.16
178		6. 48	196		19. 45	172	17. 20	6. 50	200	17. 25	19. 23
173		6. 55	191		19. 53	167	17. 30	7. 05	195	17. 26	19. 30
168		7. 02	186		20. 00	219	17. 10	9. 20	190	17. 23	19. 36
215		8. 35	181		20. 10	214	17. 14	9. 30	185	17. 15	19. 40
210		8. 45	176		20. 20	209	17. 10	9. 35	180	17. 11	19. 46
205		8. 55	171		20. 33	204	17. 10	9. 45	175	17. 10	19. 53
200		9. 00	166		20. 40	199	17. 07	9. 52	170	17. 10	20. 00
195		9. 10	220		20. 25	194	17. 05	10. 00			
190		9. 15				189	17. 10	10.10	217	17. 35	22. 10
185		9. 25	218		22. 15	184	17. 02	10. 15	212	17. 27	22. 18
180		9. 30	213		22. 23	179	17. 03	10. 25	207	17. 20	22. 25
175		9. 40	208		22. 23	174	17. 09	10. 35	202	17. 15	22. 33
170		10. 10	203		22. 39	169	17. 07	10. 40	197	17. 14	22. 39
217		12. 05	198	16. 35	22. 45	216	17. 35	12. 50	192	17. 36	22. 46
212		12. 15	193	16. 34	22. 54	211	17. 32	12. 35	187	17. 33	22. 53
207		12. 25	188	16. 38	23. 08	206	17. 30	13. 00	182	16. 28	22. 58
202		12. 38	183	16. 53	23. 33	201	17. 34	13. 10	177	16. 25	23. 05
197		12. 45	178	16. 57	23. 45	196	17. 30	13. 15	172	16. 23	23. 13
192		12. 50	173	17. 04	23. 59	191	17. 27	13. 20	167	16. 16	23. 21
187		12. 55	168	17. 06	0. 08	186	17. 28	13. 28	219	15. 36	0. 56
182		13. 05	215	17. 01	1. 36	181	17. 25	13. 35	214	15. 33	1. 03
177		13. 20	210	16. 58	1. 43	176	17. 20	14. 40	209	15. 38	1. 13
172		13. 30	205	16. 58	1. 53	171	17. 15	13. 48	204	15. 39	1. 24
167		13. 35	200	16. 58	1. 58	166	17. 12	13. 52	199	16. 08	2. 00
			195	16. 54	2. 04				194	15. 07	2. 07
219		16. 10	190	16. 58	2. 13	218	17. 41	15. 46	189	16. 06	2. 15
214		16. 16	185	17. 00	2. 35	208	17. 20	15. 55	184	16. 01	2. 26
209		16. 25	180	17. 05	2. 50	203	17. 22	16. 00	179	16. 11	2. 36
204		16. 35	175	17. 03	2. 43	203	17. 31	16. 10	174	16. 08	2. 43
199		16. 45	170	16. 40	2. 50	198	17. 35	16. 20	169	16. 13	2. 53
194		16. 55	217	16. 30	4. 35	193	17. 36	16. 30	216	15. 55	4. 45
189		17. 00	212	16. 36	4. 51	188	17. 44	16. 36	211	16. 00	4. 55
184		17. 13	207	16. 40	5. 05	183	17. 22	16. 55	206	16. 05	5. 05
179		17. 20	202	16. 40	5. 18	178	17. 28	17. 03	201	16. 10	5. 20
174		17. 26	197	16. 40	5. 25	173	17. 11	17. 10	196	16. 10	5. 25
169		17. 33				168	17. 08	17. 16			

Table 1 (continued)

Day of Test	3					4					
Oven No.	Coking period hrs. min.	Time charged hr. min.	Oven No.	Coking period hr. min.	Time charged hr. min.	Oven No.	Coking period hr.min.	Time charged hr.min.	Oven No.	Coking period hr.min.	Time charged hr.min.
191	16.50	6.10	219	17.39	18.35	195	17.47	6.11	218	17.56	19.12
186	16.52	6.20	214	17.42	18.45	190	17.50	6.20	213	17.57	19.20
181	16.55	6.30	209	17.42	18.55	185	17.50	6.30	208	17.55	19.25
176	16.55	6.35	204	17.36	19.00	180	18.05	6.50	203	17.52	19.32
171	16.57	6.45	199	17.10	19.10	175	18.10	7.05	198	17.48	19.38
166	17.03	6.55	194	17.08	19.15	170	18.12	7.12	193	17.52	19.48
218	16.37	8.20	189	17.07	19.22	217	17.18	8.53	188	17.47	19.52
213	16.37	8.30	184	17.04	19.30	212	17.20	9.00	183	17.47	20.00
208	16.35	8.35	179	16.59	19.35	207	17.20	9.10	178	17.48	20.08
203	16.30	8.40	174	17.02	19.45	202	17.21	9.16	173	17.36	20.12
198	16.30	8.50	169	16.57	19.50	197	17.20	9.25	168	17.35	20.18
193	16.30	9.00				192	17.22	9.35			
188	16.34	9.10	216	17.25	22.10	187	17.23	9.43	220	17.50	22.10
183	16.25	9.20	211	17.21	22.16	182	17.25	9.50	215	17.50	22.20
178	16.22	9.25	206	17.18	22.23	177	17.21	9.56	210	17.50	22.30
173	16.20	9.30	201	17.10	22.30	172	17.18	10.03	205	17.50	22.40
168	16.24	9.40	196	17.11	22.36	167	17.20	10.10	200	17.55	22.50
220	17.30	10.55	191	16.33	22.43	219	17.25	12.00	195	16.49	23.00
215	16.55	11.58	186	16.30	22.50	214	17.23	12.08	190	16.50	23.10
210	16.52	12.02	181	16.30	23.00	209	17.20	12.15	185	16.50	23.20
205	16.52	12.08	176	16.35	23.10	204	17.25	12.25	180	16.40	23.30
200	16.52	12.15	171	16.35	23.20	199	17.25	12.35	175	16.35	23.40
195	16.55	12.25	166	16.35	23.30	194	17.30	12.45	170	16.34	23.46
190	16.54	12.30	218	16.56	1.16	189	17.33	12.55	217	16.32	1.25
185	17.00	12.40	213	16.53	1.23	184	17.35	13.05	212	16.30	1.46
180	16.54	12.45	208	16.55	1.30	179	17.40	13.15	207	16.30	1.40
175	17.02	12.55	203	17.00	1.40	174	17.40	13.25	202	16.30	1.46
170	17.00	13.00	198	17.00	1.50	169	17.40	13.30	197	16.28	1.53
			193	16.56	1.56				192	16.25	2.00
217	17.25	15.35	188	16.56	2.05	216	18.15	16.25	187	16.27	2.10
212	17.25	15.40	183	16.53	2.13	211	18.56	17.12	182	16.30	2.20
207	17.25	15.51	178	16.55	2.20	206	18.55	17.18	177	16.34	2.30
202	17.22	15.55	173	17.06	2.36	201	18.52	17.22	172	16.37	2.40
197	17.26	16.05	168	17.03	2.43	196	19.02	17.28	167	16.35	2.45
192	17.27	16.13	220	18.15	4.20	191	18.49	17.32	219	16.55	4.55
187	17.27	16.20	215	16.32	4.30	186	18.48	17.38	214	16.57	5.05
182	17.27	16.25	210	16.38	4.40	181	18.50	17.50	209	17.00	5.15
177	17.30	16.35	205	16.42	4.50	176	18.48	17.58	204	17.00	5.25
172	17.32	16.45	200	16.40	4.55	171	18.42	18.02	199	16.55	5.30
167	17.29	16.50				166	18.40	18.10			

Table 1 (continued)

Day of Test	5					6					
Oven No.	Coking period hr.min.	Time charged hr.min.	Oven No.	Coking period hr.min.	Time charged hr.min.	Oven No.	Coking period hr.min.	Time charged hr.min.	Oven No.	Coking period hr.min.	Time charged hr.min.
194	17.37	6.12	217	17.13	18.38	193	17.25	6.09	216	17.23	18.48
189	17.25	6.20	212	17.09	18.42	188	17.28	6.20	211	17.20	18.55
184	17.23	6.28	207	17.10	18.50	183	17.27	6.27	206	17.25	19.09
179	17.20	6.35	202	17.14	19.00	178	17.28	6.38	201	17.29	19.15
174	17.15	6.40	197	17.17	19.10	173	17.29	6.48	196	17.30	19.20
169	17.20	6.50	192	17.20	19.20	168	16.32	6.57	191	17.20	19.25
216	16.31	8.56	187	17.15	19.25	220	17.32	9.12	186	17.22	19.35
211	15.52	9.04	182	17.15	19.35	215	17.15	9.15	181	17.22	19.42
206	15.54	9.12	177	17.10	19.40	210	17.30	9.35	176	17.24	19.50
201	15.58	9.20	172	17.10	19.50	205	17.37	9.47	171	17.30	20.00
196	16.04	9.32	167	17.20	20.05	200	17.43	9.58	166	17.35	20.10
191	16.08	9.40				195	17.40	10.05			
186	16.10	9.48	219	17.15	22.10	190	17.42	10.12	218	17.25	22.10
181	16.06	9.56	214	17.11	22.16	185	17.36	10.16	213	17.02	22.20
176	16.05	10.03	209	17.08	22.23	180	17.32	10.22	208	17.11	22.26
171	16.10	10.12	204	17.05	22.30	175	17.32	10.27	203	17.08	22.33
166	16.10	10.20	199	17.10	22.40	170	17.30	10.35	198	17.10	22.40
218	16.52	12.04	194	16.38	22.50	217	17.32	12.10	193	16.41	22.50
213	16.52	12.12	189	16.40	23.00	212	17.36	12.18	188	16.40	23.00
208	16.55	12.20	184	16.48	23.16	207	17.35	12.25	183	16.43	23.10
203	16.56	12.28	179	16.50+	23.25	202	17.33	12.33	178	16.42	23.20
198	16.58	12.36	174	16.55	23.35	197	17.36	12.46	173	16.42	23.30
193	16.56	12.44	169	16.53	23.43	192	17.34	12.54	168	16.43	23.40
188	17.00	12.52	216	16.29	1.25	187	17.36	13.01	215	17.05	2.20
173	17.08	13.20	211	16.29	1.33	182	17.33	13.08	210	16.51	2.26
178	17.02	13.10	206	16.28	1.40	177	17.40	13.20	205	16.46	2.33
173	17.08	13.20	201	16.26	1.46	172	17.39	13.29	200	16.42	2.40
168	17.07	13.25	196	16.20	1.50	167	17.30	13.35	195	16.41	2.46
			191	16.33	2.05				190	16.43	2.55
220	17.40	15.50	186	16.33	2.13	219	17.40	15.50	185	16.49	3.05
215	17.40	16.00	181	16.32	2.20	214	17.42	15.58	180	16.41	3.13
210	17.35	16.05	176	16.36	2.26	209	17.39	16.02	175	16.43	3.20
205	17.30	16.10	171	16.27	2.30	204	17.40	16.10	170	16.51	3.26
200	17.25	16.15	166	16.23	2.35	199	17.40	16.20	217	17.05	5.15
195	17.25	16.25	218	16.25	4.55	194	17.40	16.30	212	17.07	5.25
190	17.20	16.30	213	16.56	5.00	189	17.35	16.35	207	17.10	5.35
185	17.20	16.40	208	17.05	5.15	184	17.29	16.45	202	17.12	5.45
180	17.20	16.50	203	17.05	5.25	179	17.30	16.55	197	17.04	5.50
175	17.15	16.55	198	17.02	5.30	174	17.30	17.05	192	17.04	5.58
170	17.25	17.05				169	17.32	17.15			

Table 2 Coal Throughput and Moisture content

Day of test	1		2		3		4		5		6	
		H_2O		H_2O		H_2O		H_2O		H_2O		H_2O
Morning Shift												
No. of ovens discharged	28	11.4	28	-	28	9.3	28	8.9	28	9.2	28	9.7
Coal throughput t.	479.5	-	471.6	-	456.5	-	465.1	-	471.6	-	468.5	-
Oven No. & moisture %	183	10.9			171	9.0	175	9.5	184	10.5	190	10.0
	195	10.5			198	9.3	212	7.9	211	10.2	180	10.0
	175	10.9			173	10.1	219	9.5	181	11.5	217	10.4
	197	10.8			195	10.0	194	9.7	218	10.2	212	10.0
	172	10.4			175	9.9	179	9.8	193	10.8		
Day Shift												
No. of ovens discharged	23	12.4	22	-	22	9.1	22	9.5	22	9.2	22	10.3
Coal throughput t.	394.7	-	372	-	368.7	-	359.7	-	369.9	-	367.8	-
Oven No. and Moisture %	219	9.8	213	9.2	212	8.3	196	8.6	200	9.5	199	10.6
	184	10.5	183	8.8	187	9.8	171	9.4	175	10.7	174	10.6
	211	11.7	215	10.4	167	8.5	198	9.5	197	9.6	196	9.2
	191	11.9	195	10.5	194	8.3	173	9.7	172	9.1	171	9.6
	171	10.8	170	10.8	169	9.4						
Night Shift												
No. of ovens discharged	26	11.6	27	-	27	8.6	27	12.0	27	10.5	27	10.5
Coal throughput t.	439.1	-	455.5	-	448.9	-	456.4	-	449.1	-	451.6	-
Oven No. and moisture %	198	11.5	212	10.7	211	9.9	105	10.0	199	9.3	203	10.3
	173	11.4	197	11.2	186	8.0	180	10.0	174	10.2	178	10.7
	183	11.3	177	11.1	166	8.0	202	11.8	201	10.0	205	10.9
	215	11.3	199	9.5	198	10.0	177	11.0	208	10.7	180	11.1
	185	12.3	174	9.1	168	8.6	209	10.3	176	10.5	202	10.0

Table 3 Flue-gas Analyses

Day of Test	1			2			3			4			5			6		
	Oven	CO_2	O_2	Oven	CO_2	O_2	Oven	CO_2	O_2	Oven	CO_2	O_2	Oven	CO_2	O_2	Oven	CO_2	O_2
Coke Side	215	8.0	1.2	119	8.0	3.2	169	7.0	1.5	209	7.0	3.1	171	8.8	1.7	169	8.1	2.6
	201	8.6	1.6	212	8.9	1.5	197	8.3	1.8	191	7.9	3.1	191	7.8	2.4	184	7.2	3.4
	181	8.8	0.4	202	8.8	1.4	215	8.0	2.8	176	7.8	2.6	208	7.6	3.2	199	8.4	1.8
	169	5.9	1.0	194	8.9	1.2	-	-	-	-	-	-	-	-	-	215	8.2	2.4
Machine Side	171	9.0	0.6	216	8.0	2.2	169	9.0	1.1	170	8.0	2.6	214	7.6	2.8	215	7.2	3.6
	183	8.3	0.9	204	7.8	2.3	197	8.4	1.6	184	8.0	2.0	196	8.0	1.8	195	8.6	0.6
	195	9.0	0.8	192	9.0	1.0	215	7.8	2.0	201	7.6	2.8	196	8.0	1.8	183	8.0	1.8
	217	8.8	1.2	180	9.0	0.7	-	-	-	-	-	-	-	-	-	174	8.9	1.3

Table 4 Make-gas Analyses

Day of Test	1	2	3	4	5	6
CO_2	0.7	1.6	1.0	0.8	0.6	0.8
Unsats.	2.3	-	2.3	-	2.2	2.4
O_2	0.2	0.2	0.2	0.3	0.2	0.2
CO	5.3	-	5.5	-	5.3	5.4
H_2	62.0	-	61.8	-	59.7	61.6
CH_4	24.1	-	24.2	-	24.3	24.0
N_2	5.4	4.6	5.0	5.2	7.7	5.6
Inerts	6.3	6.4	6.2	6.3	8.5	6.6

APPENDIX III

Guarantee Tests on Four Types of Ovens

PLANT: Battery I
55 OTTO COKE OVENS

Guarantee Certificate
Kind of gas: RICH Gas

from 24 July
to 29 July 1942

Length of oven between the doors 12270 m.m.
Height to which filled 3800 m.m.
Mean breadth. 445 m.m.

Measuring pipe 510 m.m.
Orifice 389.5 m.m.

Date	No. of Ovens Discharged	Coking Time	Oven charge		Coal Throughput		H2O	V.M.	Ash	Coal Size in m.m.								Gas Quantity	Calorific Value		Heat Consumption		Gas in Foul Main Inerts			Waste Gases			Gas Temps.		
										>6	6-3	3-2	2-1	1-0.5	0.5-0.2	<0.2	<3		gross	nett			CO_2	N_2	CO_2+N_2	CO_2	O_2	CO	Upriser	Above charge	Stack
		Nett	gross	dry	wet	dry	wet															Kcal/ Kg coal (wet)									
July		hr.	hr	tonnes	tonnes	tonnes	tonnes	%	%	%	%	%	%	%	%	%	%	%	$Nm^3/24hr.$	Kcal/Nm^3		Kcal/24hr.		%	%	%	%	%	%	°C.	°C.	°C.
24/25	77	16.62	17.14	15.245	17.091	1173.88	1316.01	10.8	23.64	7.62	2.5	7.5	8.0	17.0	27.5	19.5	18.0	90.0	184,757	4392	3859	712,977,000	541.8	0.8	6.2	7.0	8.2	2.3	0.0	590	750	235
25/26	77	16.83	17.14	15.274	17.084	1176.09	1315.54	10.6	23.27	7.11	3.0	7.0	8.5	17.5	27.0	19.0	18.0	90.0	182,681	4373	3853	703,870,000	535.0	1.0	6.3	7.3	8.1	2.9	0.0	595	745	240
26/27	77	17.16*	17.14	15.033	16.778	1157.56	1291.92	10.4	23.73	7.30	2.0	8.0	10.0	19.0	28.0	18.0	15.0	90.0	180,398	4381	3869	697,960,000	540.2	-	-	-	7.9	2.8	0.0	580	740	245
27/28	77	17.18	17.14	15.067	16.648	1160.15	1281.93	9.5	23.53	8.57	2.0	6.5	8.5	20.0	26.5	21.0	15.5	91.5	182,312	4396	3897	710,470,000	554.2	1.2	6.4	7.6	7.4	3.3	0.0	585	740	245
28/29	77	16.97	17.14	15.335	16.945	1180.82	1304.77	9.5	21.87	8.15	2.5	7.5	8.0	17.5	29.5	20.0	15.0	90.0	173,388	4331	3831	664,249,000	509.1	-	-	-	7.7	3.0	0.0	590	750	245
Mean.	77			15.191	16.910	1169.70	1302.03	10.3	23.21	7.55	2.4	7.3	8.6	18.2	27.5	19.5	16.5	90.3	180,707	4375	3862	697,905,000	536.0	1.0	6.3	7.3	7.66	3.46	0.0	588	745	242
Guarantee Given								10.0													4000 ~~1950~~		520.0						850		

Attained 332.7 (17.5 hr. period, gross)
= +12.7
= + 2.45%

Observations: * Change-over time = 47 sec./change-over; time during which cocks were open = 23.06 hr./24 hr.
1st day: Shortest coking time (nett) of one oven 15.12 hr.; longest coking time (nett) of one oven 18.08 hr.
2nd " " " " " " " 15.94 " " " " " " " 17.50 "
3rd " " " " " " " 16.27 " " " " " " " 18.47 " (danger of air attack).
4th " " " " " " " 15.87 " " " " " " " 18.33 "
5th " " " " " " " 16.53 " " " " " " " 17.33 "
For whole test " " " " " " " 15.12 " " " " " " " 18.47 "
Owing to the variations in the bulk density of the coal, the throughput fluctuated greatly. Also the quality of the gas for underfiring varied from day to day. Rise in mean flue temperature from 1320°C. to 1329°C. = +9°C.
Heat consumption at 8% H_2O = 509 Kcal/Kg.

* Danger of air attack: Oven stood over.

PLANT: Battery I
55 OTTO COKE OVENS

Guarantee
Certificate
Kind of gas:
BLAST FURNACE GAS

from 17 July
to 22 July 1942

Length of oven between the doors 12270 m.m.
Height to which filled 3800 m.m.
Mean breadth 445 m.m.

Measuring pipe 1008 m.m.
Orifice 840.7 m.m.

Date	No. of Ovens Discharged	Coking Time Nett	Coking Time gross	Oven charge dry	Oven charge wet	Coal Throughput dry	Coal Throughput wet	H_2O	V.M.	Ash	Coal Size in m.m. >6	6-3	3-2	2-1	1-0.5	0.5-0.2	<0.2	<3	Gas Quantity	Calorific Value gross	Calorific Value nett	Heat Consumption	Heat Consumption	Gas in Foul Main Inerts CO_2	Inerts N_2	Inerts CO_2+N_2	Waste Gases CO_2	Waste Gases O_2	Waste Gases CO	Gas Temps. Uprisers	Above charge	Stack
July		hr.	hr.	tonnes	tonnes	tonnes	tonnes	%	%	%	%	%	%	%	%	%	%	%	Nm^3/24hr.	Kcal/Nm^3		Kcal/24hr.	Kcal/Kg coal(wet)	%	%	%	%	%	%	°C.	°C.	°C.
17/18	77	16.90	17.14	15.234	16.927	1173.09	1303.43	10.0	22.68	7.11	2.0	9.5	8.5	19.5	26.5	12.0	22.0	88.5	776,450	973	963	747,647,000	573.6	1.0	5.8	6.8	22.6	0.4	0.0	580	740	245
18/19	77	16.95	17.14	15.328	17.116	1180.24	1317.97	10.5	23.08	7.05	2.5	10.0	9.0	20.5	26.0	13.0	19.0	87.5	783,030	961	951	744,662,000	565.0	1.2	7.2	8.4	22.3	0.6	0.0	570	750	235
19/20	77	17.05	17.14	15.488	17.123	1192.54	1318.45	9.6	23.41	7.29	3.0	9.0	9.5	22.5	27.0	14.0	15.0	88.0	776,761	952	942	731,709,000	555.0	0.8	6.2	7.0	21.3	1.5	0.0	575	745	235
20/21	77	16.88	17.14	15.443	17.159	1189.12	1321.24	10.0	23.08	7.42	2.0	9.0	9.0	21.5	27.0	19.0	12.5	89.0	780,140	937	927	723,190,000	547.4	1.0	6.2	7.2	22.1	1.7	0.0	580	740	235
21/22	77	15.98	17.14	15.446	17.089	1190.89	1315.90	9.5	23.71	7.43	2.5	11.5	11.5	19.0	25.0	17.0	13.5	86.0	789,587	955	944	745,370,000	566.4	2.0	6.4	8.4	21.9	1.5	0.0	590	750	235
Mean.	77	16.95	17.14	15.392	17.083	1185.18	1315.39	9.9	23.20	7.25	2.4	9.7	9.5	20.5	26.3	15.0	16.3	87.6	781,193	955	944	738,530,000	561.4	1.2	6.35	7.56	22.0	1.1	0.0	580	745	237
Guarantee Given	75		17.30	15.520		1170.00		10.0												1050			560.0							850		360

at bulk density
750 Kg./m^3 dry

Attained 562.7 (17.5 hr. period, gross)
= + 2.7
= + 0.48%

Observations: Change-over time = 89 secs; time during which cocks were open = 22.23 hr./24 hr.
The gross and nett coking time of each separate oven is different because of the pause imposed between the individual series. Heat consumption at 8% H_2O = 538.7 Kcal/Kg.
1st day Shortest coking time (nett) of one oven 16.57 hr.; longest coking time (nett) of one oven 17.58 hr.
2nd " " " " " " " " 16.49 " " " " " " " " 17.87 "
3rd " " " " " " " " 16.73 " " " " " " " " 17.70 "
4th " " " " " " " " 16.52 " " " " " " " " 17.40 "
5th " " " " " " " " 16.62 " " " " " " " " 17.53 "
For whole test " " " " " " " " 16.49 " " " " " " " " 17.87 "
Increase of mean temperature of heating flues from 17th to 22nd July 1324 to 1343°C. = + 19°C.

PLANT: Battery II
55 BECKER COKE OVENS

Guarantee Certificate
Kind of gas:
RICH GAS

from 17 July
to 21 July 1942

Length of oven between the doors 12270 m.m.
Height to which filled 3800 m.m.
Mean breadth 445 m.m.

Measuring pipe 510 m.m.
Orifice 369.5 m.m.

Date	No. of Ovens Discharged	Coking Time		Oven charge		Coal Throughput		H_2O	V.M.	Ash	Coal Size in m.m.								Gas Quantity	Calorific Value		Heat Consumption		Gas in Foul Main Inerts			Waste Gases			Gas Temps.		
		Nett	gross	dry	wet	dry	wet				>6	3-6	2-3	1-2	0.5-1	0.2-0.5	<0.2	<3		gross	nett			CO_2	N_2	CO_2+N_2	CO_2	O_2	CO	Upriser	above charge	Stack
July		hr.	hr	tonnes	tonnes	tonnes	tonnes	%	%	%	%	%	%	%	%	%	%	%	%	$Nm^3/24$ hr.	Kcal/Nm^3		Kcal/24hr.	Kcal/Kg coal(wet)	%	%	%	%	%	%	°C.	°C.	°C.
17	77	16.55	17.08	15.120	16.772	1163.640	1291.500	10.70	22.68	7.11	2.0	9.5	8.5	19.5	26.5	12.0	22.0	88.5	174,057	4235	3747	652,403,866	505	1.0	6.9	7.9	7.3	2.8	0.0	590	790	268
18	77	16.55	17.08	15.219	16.952	1171.910	1305.310	10.22	23.08	7.05	2.5	10.0	9.0	20.5	26.0	13.0	19.0	87.5	181,195	4280	3740	678,938,414	520	1.2	7.2	8.4	7.6	2.4	-	580	-	270
19	77	16.55	17.08	15.293	16.922	1177.580	1303.070	9.63	23.41	7.29	3.0	9.0	9.5	22.5	27.0	14.0	15.0	88.0	185,265	4265	3746	694,004,930	532.5	0.8	6.8	7.6	7.9	1.7	-	-	810	270
20	77	16.55	17.08	15.381	16.997	1184.306	1308.770	9.51	23.08	7.42	2.0	9.0	9.0	21.5	27.0	19.0	12.5	89.0	185,900	4302	3800	706,420,000	539.8	-	-	-	8.0	1.6	-	585	-	275
21	77	16.55	17.08	15.253	16.929	1174.463	1303.510	9.90	23.71	7.43	2.5	11.5	11.5	19.0	25.0	17.0	13.5	86.0	185,904	4311	3784	703,646,640	539.8	-	-	-	7.8	1.8	0.0	595	790	278
Mean.	77	16.55	17.08	15.252	16.914	1174.380	1302390	9.99	23.19	7.26	2.0	9.8	9.5	20.6	26.3	15.0	16.4	87.8	182,466	4279	3763	687,082,770	527.4	1.0	6.96	7.96	7.72	2.06	0.0	590	793	272
Guarantee Given	-	-	-	-	-	1140.000	-	8.00	-	-	-	-	-	-	-	-	-	-	-	-	4000	-	525.0	-	-	8.00	-	-	-	850	-	330

Attained 512.7
= - 42.3
= - 7.65%

Observations: Heat consumption, coal at 8% H_2O = 512.7 Kcal/Kg.

PLANT: Battery II
55 KOPPERS COKE OVENS

Guarantee
Certificate
Kind of gas:
BLAST FURNACE GAS

from 24 July
to 28 July 1942

Length of oven between the doors 12270 m.m.
Height to which filled 3800 m.m.
Mean breadth 445 m.m.

Measuring pipe 1208 m.m.
Orifice 894.0 m.m.

Date July	No. of Ovens Discharged	Coking Time Nett hr.	Coking Time gross hr.	Oven charge dry tonnes	Oven charge wet tonnes	Coal Throughput dry tonnes	Coal Throughput wet tonnes	H_2O %	V.M. %	Ash %	Coal size in m.m. >6 %	3-6 %	2-3 %	1-2 %	0.5-1 %	0.2-0.5 %	0.2 %	<.3 %	Gas Quantity Nm^3/24hr.	Calorific Value gross Kcal/Nm^3	Calorific Value nett Kcal/Nm^3	Heat Consumption Kcal/24hr.	Heat Consumption Kcal/Kg coal(wet)	Gas in Foul Main Inerts CO_2 %	Inerts N_2 %	Inerts CO_2+N_2 %	Waste Gases CO_2 %	Waste Gases O_2 %	Waste Gases CO %	Gas Temps Upriser °C.	Gas Temps Above charge °C.	Gas Temps Stack °C.
24	77	16.55	17.08	15.170	16.912	1168.100	1302.230	10.30	23.64	7.62	2.5	7.5	8.0	17.0	27.5	19.5	18.0	90.0	774,096	967	957	741,583,968	569.4	1.0	6.3	7.3	21.6	1.1	0.0	-	760	258
25	77	16.55	17.08	15.086	16.894	1161.620	1300.810	10.70	23.27	7.11	3.0	7.0	8.5	17.5	27.0	19.0	18.0	90.0	732,864	949	939	688,892,160	529.5	1.0	6.6	7.6	21.4	0.4	0.0	-	765	250
26	77	16.55	17.08	14.903	16.596	1147.554	1277.900	10.20	23.73	7.30	2.0	8.0	10.0	19.0	28.0	18.0	15.0	90.0	737,342	964	953	702,687,307	549.9	-	-	-	20.6	1.5	0.0	-	760	248
27	77	16.55	17.08	14.936	16.464	1150.048	1267.690	9.28	23.53	8.57	2.0	6.5	8.5	20.0	26.5	21.0	15.5	91.5	736,660	971	967	712,350,993	561.9	-	-	-	21.3	1.5	0.0	620	755	250
28	77	16.55	17.08	15.068	16.793	1160.272	1293.070	10.27	21.89	8.30	2.5	7.5	8.0	17.5	29.5	20.0	15.0	90.0	737,990	955	944	696,662,560	538.7	1.0	5.8	6.8	21.7	1.3	0.0	610	760	250
Mean	77	16.55	17.08	15.033	16.732	1157.518	1288.340	10.15	23.21	7.78	2.4	7.30	8.60	18.20	27.70	19.50	16.30	90.30	743,784	961.2	952	708,435,398	549.9	1.0	6.23	7.23	21.32	1.16	0.0	615	760	251
Guarantee Given						1140.000		8.00													1050		555.0			9.00				850		360

Attained $\frac{534.4}{-20.6}$
$= -3.71\%$

Observations: Heat consumption, coal at 8% H_2O = 534.4 Kcal/Kg.

PLANT: Battery III
55 Koppers-Circulation-Compound-COKE OVENS

Guarantee Certificate
Kind of gas: RICH GAS

from 4 August to 6 August 1942

Length of oven between the doors 12440 m.m.
Height to which filled 3800 m.m.
Mean breadth 445 m.m.

Measuring pipe 510 m.m.
Orifice 369.5 m.m.

Date	No. of Ovens Discharged	Coking Time Nett	Coking Time gross	Oven charge dry	Oven charge wet	Coal Throughput dry	Coal Throughput wet	H_2O	V.M.	Ash	Coal size in m.m. >6	3-6	2-3	1-2	0.5-1	0.2-0.5	0.2	<3	Gas Quantity	Calorific Value gross	Calorific Value nett	Heat Consumption	Heat Consumption Kcal/Kg coal (wet)	Gas in Foul Main Inerts CO_2	Gas in Foul Main Inerts N_2	Gas in Foul Main Inerts CO_2+N_2	Waste Gases CO_2	Waste Gases O_2	Waste Gases CO	Gas Temps. Upriser	Gas Temps. Above charge	Gas Temps. Stack
		hr.	hr.	tonnes	tonnes	tonnes	tonnes	%	%	%	%	%	%	%	%	%	%	%	$Nm^3/24hr.$	Kcal/Nm^3		Kcal/24 hr.		%	%	%	%	%	%	°C.	°C.	°C.
4 Aug.	77	16.55	17.08	15.226	16.874	1172.376	1299.320	9.77	22.75	8.19	2.0	11.0	10.0	22.0	26.0	16.5	12.5	87.0	189,180	4400	3901	737,991,180	567.9	-	-	-	8.0	1.6	0.0	590	740	290
5 Aug.	77	16.55	17.08	15.142	16.746	1165.929	1289460	9.58	22.18	7.23	2.0	8.0	9.5	21.0	27.5	20.0	12.0	90.0	180,562	4299	3796	685,414,490	531.5	1.0	6.6	7.6	7.8	2.0	0.0	590	750	295
6 Aug.	77	16.55	17.08	15.157	16.712	1167.154	1286830	9.30	22.67	7.26	2.5	7.5	9.0	19.5	26.5	21.0	14.0	90.0	185,184	4408	3901	722,587,968	561.5	1.2	6.2	7.4	7.9	2.2	0.0	590	745	300
Mean.	77	16.55	17.08	15.157	16.777	1168.486	1291.870	9.55	22.53	7.56	2.17	8.83	9.50	20.83	26.66	19.77	12.84	89.0	184,975	4369	3866	715,331,213	553.6	1.1	6.4	7.5	7.9	1.93	0.0	590	745	295
Guarantee Given						1140.000		8.00													4000		555.0			9.0				850		330

Attained 542.6
= - 12.4
= - 2.23%

Observations: Heat consumption, coal at 8% H_2O = 542.6 Kcal/Kg.

With 77 ovens/day the cross section of the heating gas channels and heating flues is fully loaded.

PLANT: Battery III
55 Koppers-Circulation-Compound-COKE OVENS

Guarantee Certificate
Kind of gas: Blast furnace gas

from 1 July
to 8 July 1942

Length of oven between the doors 12440 m.m.
Height to which filled 3860 m.m.
Mean breadth 445 m.m.

Measuring pipe 1008.0 m.m
Orifice 840.7 m.m

Date	No. of Ovens Discharged	Coking Time Nett	Coking Time gross	Oven charge dry	Oven charge wet	Coal Throughput dry	Coal Throughput wet	H_2O	V.M.	Ash	Coal size in m.m. 6	6-3	3-2	2-1	1-0.5	0.5-0.2	0.2-0.5	<0.5	Gas Quantity	Calorific Value gross	Calorific Value nett	Heat Consumption	Heat Consumption	Gas in Foul Main Inerts CO_2	Inerts N_2	Inerts CO_2+N_2	Waste Gases CO_2	Waste Gases O_2	Waste Gases CO	Gas Temps. Upriser	Above charge	Stack
July		hr.	hr.	tonnes	tonnes	tonnes	tonnes	%	%	%	%	%	%	%	%	%	%	%	$Nm^3/24$ hr.	Kcal/Nm^3		Kcal/24hr.	Kcal/Kg coal(wet)	%	%	%	%	%	%	°C.	°C.	°C.
1	77	16.55	17.08	15.134	16.796	1165.299	1293.340	9.90	24.40	7.53	3.0	10.0	8.5	18.5	25.5	13.5	21.0	87.0	753,168	973	957	721,534,944	557.8				22.7	1.0	0.0		675	250
2	77	16.55	17.08	15.149	16.888	1166.484	1300.428	10.30	24.20	7.53	2.5	9.0	8.0	19.5	26.0	17.0	18.0	88.5	722,263	976	964	696,983,988	535.9	1.4	6.4	7.8	22.1	1.4	0.0	570		252
3	77	16.55	17.08	15.523	17.580	1195.280	1353.660	11.40	24.30	7.32	2.5	8.0	8.5	20.5	28.5	15.5	17.0	89.5	740,009	973	963	713,451,772	527.0				21.5	1.2	0.0		690	254
4	77	16.55	17.08	15.598	17.340	1200.330	1335.180	10.10	24.10	7.67	3.5	10.5	9.5	19.5	25.0	4.0	18.0	86.0	728,709	963	951	693,731,539	519.6	1.0	5.0	6.0	23.2	0.7	0.0	560		253
5	77	16.55	17.08	15.509	17.080	1194.170	1315.160	9.20	24.50	6.89	2.0	10.0	8.5	20.5	26.0	16.5	16.5	88.0	732,214	955	947	694,138,493	528.0	1.2	5.4	6.6	21.8	0.7	0.0		670	255
6	77	16.55	17.08	15.325	17.009	1180.030	1309.690	9.90	24.00	7.74	2.5	8.0	7.5	17.5	24.0	7.5	33.0	89.5	737,095	961	951	700,977,535	535.2				22.4	0.9	0.0	570		260
7	76	17.11	17.22	15.470	17.150	1175.190	1303.430	9.80	23.30	7.35	3.0	9.0	7.5	18.5	27.0	17.0	18.0	88.0	746,020	958	947	707,227,718	542.6	0.9	6.2	7.1	21.8	1.2	0.0		690	253
8	77	16.55	17.08	15.436	17.163	1188.590	1321.540	10.06	23.00	7.58	2.0	7.0	9.0	20.0	28.0	16.0	18.0	91.0	748,610	979	968	724,655,207	548.3				22.6	0.9	0.0	560		255
Mean.	76.88	16.57	17.10	15.392	17.126	1183.234	1316.554	10.08	23.97	7.42	2.62	8.93	8.37	19.31	26.23	14.62	19.92	88.44	738,511	967.2	956.0	706,587,649	536.8+	1.16	6.0	7.16	22.26	1.0	0.0	565	681	253
Guarantee given						140.000		8.00															555.0			9.0				850		360

Attained 521.6
= - 33.4
= - 6.02%

Observations: + Heat consumption, coal of 8% H_2O specified in guarantee = 521.6 Kcal/Kg.

PLANT: Battery IV
55 Still Coke Ovens

Guarantee Certificate
Kind of gas: RICH GAS

from 3 July to 8 July 1942

Length of oven between the doors 12270 m.m.
Height to which filled 3800 m.m.
Mean breadth 445 m.m.

Measuring pipe 510 m.m.
Orifice 369.5 m.m.

Date	No. of Ovens Discharged	Coking Time Nett	Coking Time gross	Oven charge dry	Oven charge wet	Coal Throughput dry	Coal Throughput wet	H_2O	V.M.	Ash	Coal Size in m.m. >6	>3	>2	>1	>0.5	>0.2	<0.2	<3	Gas Quantity	Calorific Value gross	Calorific Value nett	Heat Consumption	Heat Consumption	Gas in Foul Main Inerts CO_2	Gas in Foul Main Inerts N_2	Gas in Foul Main Inerts CO_2+N_2O	Waste Gases CO_2	Waste Gases O_2	Waste Gases CO	Gas Temps. Upriser	Gas Temps. Above charge	Gas Temps. Stack
July		hr.	hr.	tonnes	tonnes	tonnes	tonnes	%	%	%	%	%	%	%	%	%	%	%	Nm^3/24 hr.	Kcal/Nm^3		Kcal/24hr.	Kcal/Kg coal(wet)	%	%	%	%	%	%	°C.	°C.	°C.
3	77	16.7	17.2	15.07	17.05	1161	1313.2	11.6	24.3	7.3	2.5	8.0	8.5	20.5	28.5	15.5	17.0	89.5	194,600	4406	3870	753,100,000	560	0.7	5.4	6.3	8.7	1.0	0.0	590	790	245
4	77	16.7	17.2	15.07	16.87	1168	1299.1	10.1	24.1	7.7	3.5	10.5	9.5	19.5	25.0	14.0	18.0	86.0	192,200	4406	3870	743,810,000	572	1.6	4.6	6.4	8.6	1.7	0.0	595	780	258
5	77	16.7	17.2	15.02	16.54	1156	1274.0	9.2	24.5	6.9	2.0	10.0	8.5	20.5	26.0	16.5	16.5	88.0	198,100	4467	3914	775,360,000	613	1.0	5.0	6.2	8.1	2.0	0.0	585	790	273
7	77	16.7	17.2	15.07	16.77	1161	1290.6	10.1	23.3	7.4	3.0	9.0	7.5	18.5	27.0	17.0	18.0	88.0	187,700	4370	3838	720,390,000	564	0.6	7.7	8.3	8.2	2.1	0.0	590	795	275
8	77	16.7	17.2	15.02	16.73	1157	1287.9	10.2	23.0	7.6	2.0	7.0	9.0	20.0	28.0	16.0	18.0	91.0	198,800	4450	3926	780,490,000	601	0.8	5.6	6.6	8.1	2.2	0.0	590	790	277
Mean.	77	16.7	17.2	15.04	16.77	1159	1291	10.2	24.0	7.4	2.6	8.8	8.4	19.6	26.4	16.4	20.1	88.7	192,770	4406	3871	746,340,000	576	0.9	5.6	6.6	8.3	2.0	0.0	590	790	267
Guarantee Given	75.4		17.5			1156		10.0	25.0									80.0			4000		550			9.0				850		330

Attained 573.4
= + 23.4
= 4.27%

Observations: Gas temperature in ascension pipe at the measuring point = 590°C.
" " " " " at height of oven roof = 760°C.

Heat consumption, coal at 8% H_2O = 560 Kcal/Kg. The heating flue temperature at the beginning of the test was 1295°C., at the end of the test 1322°C. On account of the short change-over period the oven was not in equilibrium at the beginning of the test, this can be seen from the heating flue temperature and the waste gas temperature. Further the H_2O content of the coal and the weight of the coal charge fluctuated. Therefore the heat consumption was higher than would have been necessary with constant operating conditions.

PLANT: Battery IV
55 Still Coke Ovens

Guarantee Certificate
Kind of gas:
Blast Furnace Gas

from 29 July
to 2 August 1942

Length of oven between the doors 12270 m.m.
Height to which filled 3800 m.m.
Mean breadth 445 m.m.

Measuring pipe 1008 m.m.
Orifice 820 m.m.

Date	No. of Ovens Discharged	Coking Time Nett	Coking Time gross	Oven charge dry	Oven charge wet	Coal Throughput dry	Coal Throughput wet	H_2O	V.M.	Ash	Coal Size in m.m. >6	>3	>2	>1	>0.5	>0.2	<0.2	<3	Gas Quantity	Calorific Value gross	Calorific Value nett	Heat Consumption	Heat Consumption	Gas in Foul Main Inerts CO_2	Inerts N_2	Inerts CO_2+N_2O	Waste Gases CO_2	Waste Gases O_2	Waste Gases CO	Gas Temps. Upriser	Above charge	Stack
		hr.	hr.	tonnes	tonnes	tonnes	tonnes	%	%	%	%	%	%	%	%	%	%	%	Nm^3/ 24 hr.	Kcal/Nm^3		Kcal/24 hr.	Kcal/ Kg coal (wet)	%	%	%	%	%	%	°C.	°C.	°C.
29 July	77	16.7	17.2	14.58	16.31	1123	1255.7	10.6	21.9	7.8	3.5	8.5	8.0	19.0	30.5	17.5	13.0	88.0	743,500	1009	995	739,800,000	584	1.4	6.8	8.6	22.3	0.8	0.0	620	770	240
30 July	77	16.7	17.2	14.64	16.35	1127	1254.1	10.5	23.8	7.1	2.0	7.0	9.0	19.0	28.0	20.0	15.0	91.7	743,200	979	966	717,900,000	566	1.0	5.8	7.0	22.5	0.7	0.0	615	780	240
31 July	77	16.7	17.2	14.85	16.34	1144	1258.1	9.1	24.0		1.0	9.0	10.0	22.0	24.0	19.0	15.0	90.0	748,000	979	967	723,400,000	582	0.9	6.1	7.3	22.7	0.8	0.0	610	775	240
1 Aug.	77	16.7	17.2	14.66	16.22	1128	1248.1	9.6	24.8	7.2	1.0	9.0	9.5	21.0	26.0	19.5	14.0	90.0	737,500	976	965	711,700,000	574	1.0	6.4	7.7	23.0	0.5	0.0	620	780	245
2 Aug.	77	16.7	17.2	14.82	16.36	1142	1260.1	9.4	23.5		1.5	9.0	9.0	21.0	26.0	19.5	14.0	89.5	729,200	985	973	709,500,000	568	1.6	6.6	8.5	22.9	1.2	0.0	610	790	245
Mean.	77	16.7	17.2	14.71	16.32	1131	1256.2	9.9	23.6		1.8	8.5	9.1	20.4	26.9	19.1	14.2	89.8	740,300	986	973	720,500,000	575	1.2	6.3	7.9	22.7	0.8	0.0	615	780	245
Guarantee Given	75.4		17.5			1128		10.0	25.0									80.0			1050		580			9.0				850		360

Attained $= \sqrt{-\frac{576}{4}}$
$= - 0.68\%$

Observations:
1. Wide fluctuations in H_2O content in individual ovens (7.1 to 12.3%)
2. Bulk density of coal essentially lower than in rich gas experiment (Fine grinding)
3. From the throughput of dry coal and the water content, and from the bulk density determined in the rich gas test, a guaranteed coal throughput of 1128 tons of dry coal/day is obtained.
4. Gas temperature in the foul main 93°C. Heating flue temperature at start of test 1324°C., at the end of the test 1334°C.
Heat consumption, coal at 8% H_2O, 559 Kcal/Kg. coal.

Both in the rich gas test of 3-8 July and in the lean gas test of 29th July - 2 August the guarantees were fulfilled.

(xx)

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Table 5 Summary of Experimental Results.

Day of Test	1	2	3	4	5	6	mean	Guarantee
No. of ovens discharged	77	77	77	77	77	77	77	75.4
Mean coking period hr.	17.2	17.2	17.2	17.2	17.2	17.2	17.2	17.5
Coal throughput (wet) tonne	1313.2	1299.1	1274.0	1281.0	1290.6	1287.9	1291	-
" " (dry) "	1161	1168	1156	1152	1161	1157	1159	1156
Mean charge (wet) "	17.05	16.87	16.54	16.64	16.77	16.73	16.77	-
" " (dry) "	15.07	15.07	15.02	14.96	15.07	15.02	15.04	-
Moisture in coal %	11.6	10.1	9.2	10.1	10.1	10.2	10.2	10.0
Volatile matter %	24.3	24.1	24.5	24.0	23.3	23.0	24.0	25
Coal grading below 3 mm. %	89.5	86.0	88.0	89.5	88.0	91.0	88.6	80
Heating gas $1000Nm^3$	194.6	192.2	198.1	185.2	187.7	198.8	192.77	-
Calorific value (nett) Kal/Nm^3	3870	3870	3914	3806	3838	3926	3871	4000
Fuel consumption (raw coal) Kal/Kg	573	573	607	550	565	603	578	-
" " (10% H_2O) Kal/kg	560	572	613	549	564	604	576	550
" " (8% H_2O) Kal/kg	544	556	597	533	548	585	560	555
* Flue temperature (mean) °C	1313	1307	1319	1322	1317	1323	1322	1295
Waste gas temperature (mean) °C	245	258	273	275	275	277	267	330
CO_2 in waste gas %	8.7	8.6	8.1	7.8	8.2	8.1	8.3	-
O_2 in waste gas %	1.0	1.7	2.0	2.7	2.1	2.2	2.0	-
Temp. in Ascension pipe °C	590	595	-	-	766	760	-	850
Temp. after collecting main °C	87	91	90	93	91	91	91	120
Inerts in coal gas %	6.3	6.4	6.2	6.3	8.5	6.6	6.6	9

* Flue temperature at start of test 1295°C, at end of test 1322°C, mean during test 1318°C.
Temperature in gas space above charge 790°C

APPENDIX IV

DESCRIPTION OF HIGH-PRESSURE BENZOL RECOVERY PROCESS

Description of request for concession for erection of a high-pressure benzol washer at the Brunswick iron-works in Watenstedt.

The Deutche Bergwerks - u. Hüttenbau G.m.b.H. intend to erect a high pressure benzol washer in the grounds of the Brunswick iron-works in Watenstedt. The gas-drying plant and the naphthalene removal introduced into the scheme are already available. The sulphur purifier is added on the works side.

The coke oven gas compressed to 20 atm. by the compressors enters at a temperature of about 150°C. in four equal streams, passing into the coolers (1) in which it is cooled to a final temperature of about 20°. After the coolers the four streams are combined in a common main that leads the gas to the benzol-washers (3). In these benzol washers, through each of which an equal amount of the gas is passed, the gas, containing benzol, is scrubbed with benzol wash oil. The debenzolised gas issues from the benzol washers, in a split stream through the oil separators and passes into the sulphur purifiers (10), arranged in parallel; in these it is freed from hydrogen sulphide by a purifier-mass. From the sulphur purifiers it goes to the gas-drying plant, which is already available, and in which the greater part of the moisture still present in the gas is washed out by a calcium chloride solution in the drier (13). The dried gas then passes through the anthracene oil scrubber (14), and passes into the delivery main. The change-over tower (11) and the change-over framework (12) serve in the replacement of the exhausted purifier-mass. The baskets with the exhausted mass are drawn out of the sulphur purifier electrically and deposited on the change-over framework (12) and the new charge is lifted out of the change-over tower (11) into the purifier. The fresh and exhausted masses are moved to and fro by means of trucks.

The condensate thrown down in the coolers, consisting of light oil and water, flows into the separators (2) and is separated continuously from the gas stream by condensate displacement vessels into the separated condensate vessel (9). The condensate occurring in the two intermediary coolers of the first and second compression stages are likewise led into the separators (9). Light oil and water are separated from

one another because of the difference in their specific gravities and flow through separate pipe-lines to the benzol and ammonia factories. The removal of both liquids is effected by a positive pressure, which is kept constant from the gas main through a reducing valve in the separator vessels. The pressure amounts to 2.5 atm. gauge. The gases freed when the pressure of the condensate is let down as it enters the separator vessel are led back through an over-flow valve into the induced gas main leading to the compressors. The cooling water necessary for cooling the gas is taken partly from the returning cooling water system of the works and partly from the fresh water supply. The issuing warm water is led to a cooling tower, which is already present.

The wash oil necessary for scrubbing the benzol out of the gas is pumped from the existing benzol factory into the supply tank (5). From here the washer pumps (7), one of which acts as a reserve, transfer it to the pressure washer (3). The wash-oil passing counter-current to the gas through the washer is enriched in benzol and leaves the washer through a discharge vessel, from which it is continuously displaced into the container (6). The enriched oil is transferred from the vessel by the pumps (8) to the benzol factory. The gases freed when the pressure of the wash oil is let down at its entry into the oil container are led into the induced gas line to the compressors.

The wash oil, light oil and condensed water are transferred in closed pipe-lines, which, like the rest of the plant are kept tight so that leaks are impossible. The control of operations is carried out with suitable measuring and regulating instruments.

Further, all the necessary safety precautions are provided for, and the lives and safety of persons occupied in the process are guaranteed.

Essen, 21.3.1944.

The contractor:

Heinrich Koppers G.m.b.H.

The numbers in parenthesis in the above description refer to the drawing, Item XII in the List of Documents.

APPENDIX V

DESCRIPTION OF SULPHUR RECOVERY PROCESS

Description II of an Extraction and Refining Plant for a Sulphur-Containing Purifier Mass for 10,000 tons of Sulphur per annum.

The extraction and refining plant consists essentially of the individual items named, together with the appropriate pipelines and steel framed buildings. The plant as a whole is divided up into the extraction bay (40), a refining plant (41), distilling plant (42) and the sulphur store (43).

The modus operandi of the plant is as follows -

(A) Sulphur Extraction.

The sulphur containing mass for gas purification, taken from the tower purifier, is transported by means of a travelling crane in wrought-iron buckets (2), over the works rails and deposited on platform balances (18). The buckets have perforated bottoms and are screwed together to form a unit of six individuals. The material is then transferred by hand down to the travelling crane at (16) operated by compressed air in the extraction bay and by means of this crane is deposited in the extractors (caustic alkali vessels). The twelve extractors (1) are connected by means of pipes in such a way that the solvent can run through various extractors from the high level tank (11). Each extractor can occupy the first position by suitable operation of the valves and connections provided, and then the subsequent extractors are traversed by the solution in turn.

The extractor that has had its sulphur content most reduced occupies for the time being, the first place. The carbon di-sulphide required for the extraction is circulated in such a way that it is transferred by pressure of inert gas from the solvent tank (3) to the high level solvent tank (11) whence the carbon di-sulphide flows to the individual extractors (1). The control of the quantity of solvent flowing is effected by regulating valves built into the pipelines through which the carbon di-sulphide flows away.

From the extractors the carbon di-sulphide charged with sulphur flows under its own head as a crude

solution into the crude solution tank (19). If the crude solution is refined it can be led directly to the stills (7) where it is kept constantly at 75-85°C. by means of steam so that the sulphur remains behind in the still while the carbon di-sulphide is distilled off. The liquid sulphur is indirectly heated to 15° in the still by means of steam for the time during which the mass is freshly charged into the extractor. During this time no crude solution is admitted to the still so that the plant is not under pressure. The sulphur at the temperature named above is then let out into the liquid sulphur tank (14) whence it is transferred to the sulphur store by means of the sulphur pump (6). The sulphur having solidified to large blocks can easily be removed and can be transferred in skips and the bucket conveyor directly for loading into railway wagons. The carbon di-sulphide distilling continuously from the still (7) goes directly to the priming boilers (8) in which a certain quantity of carbon di-sulphide should always be condensing so that the sulphur which by priming has got out of the still (7) into the priming boiler (8) is re-dissolved. The carbon di-sulphide then passes from the priming boiler (8) into the condensers (5) of the distilling plant which is accommodated in the refinery building (41); here it is condensed and flows back in liquid form to the solvent tank (3). From this point the above described circulatory process begins again.

If the crude solution obtained, however, is to be refined, it is first collected in the crude solution tanks (19) and then transported by means of inert gas into a crude solution collecting tank. At the same time a definite quantity of oleum is pumped through the pumps (34) from the oleum tank (20) to the oleum measuring tank (21). A definite quantity of oleum and crude solution is then supplied to the stirring tank (23) from the measuring tanks (21) and (22); in the stirring tank it is intimately mixed. The tarry constituents and other impurities contained in the crude solution are destroyed by the oleum. After a definite time for settling the specifically lighter solution still mixed with oleum residues is drawn off into two intermediate tanks (25). The oleum consumed is brought into the oleum separating tank (35). From the intermediate tank (25) the pure solution still mixed with traces of oleum is neutralized in the soda washers (28). The soda solution is prepared in solution tank (27). On the way between the intermediate tank and the soda washer the pure solution flows through filters with granulated fullers earth (26) in order to keep back the remaining impurities.

From the soda washers (28) the pure solution passes into the pure solution tank (29) and from there is transported by means of inert gas into the intermediate high level tank (30), thence to the stills (7). The carbon di-sulphide in the pure solution is now distilled as has already been described and passes in liquid form into the solvent tank (3). To equalize the pressure of the containers and tanks filled with inert gas an inert unloaded gas container (38) is provided and it serves for the breathing of the containers and tanks in the extraction plant up to the crude solution tank. A smaller similarly unloaded inert gas container (36) is provided for the breathing of the tanks and apparatus containing oleum; in order to avoid corrosion this inert gas container is lead lined and filled with sulphuric acid. The pure solution tanks also possess their own unloaded breathing tank (35). The inert gas stored in the tanks and containers becomes saturated with carbon di-sulphide vapour. In order to avoid a loss of carbon di-sulphide due to the necessary rising and fall in the pressure of inert gas an active charcoal filter (9) and (33) is provided, consisting of several filters which can be switched into the inert gas main alternately. The filters are arranged before the inert gas compressors (13) and (32) and after being charged with carbon di-sulphide are steamed out into the steaming vessel and then cooled in a circulation of inert gas. The refinery possesses its own inert gas compressor (32) and its own device for confining the inert gas (31). If an extractor (1) is completely free from sulphur, and this can be determined by measuring the density of the crude solution issuing from it, it is put out of circuit and connected to a steam lead. The carbon di-sulphide remaining in extractor (1) is forced in liquid form by means of steam pressure of 0.5 to 1.2 atmospheres gauge pressure into the solvent tank (3). The residual carbon di-sulphide is forced in vapour form to the steaming tanks (4) where at first the condensation of water takes place. As these steaming tanks (4) are kept at a temperature of about 90°, no carbon di-sulphide can be precipitated in liquid form; it passes from there as vapour to condensers (5) of the extracting plant and thence flows back liquified into the solution tanks (3). When the extractor is free of sulphur the steam is turned off and the residual vapour in the extractor is let down. The extractor can then be emptied and refilled. The bucket with the extracted gas purifying mass is lifted out by the compressed air crane at (16) and after a pause on the platform scales (18) is transferred via the rails to the bunker bay of the tower purifier plant for emptying (see description of tower purifier.)

All the tanks containing carbon di-sulphide and solution are so arranged that they lie in trenches underground and only the smaller tanks and the condensers and coolers which carry a little carbon di-sulphide are above ground. All carbon di-sulphide tanks are fitted with a water spray device and internal heating coils by means of which both in winter and in summer the carbon di-sulphide or the crude or pure solution can be maintained at a definite temperature. Under each vessel containing carbon di-sulphide there is a sump always filled with water and covered with a wooden grating. Any carbon di-sulphide thus getting out through leaks is caught under water and prevented from forming an explosive mixture by mixing in vapour with the air. Further, the large tanks for carbon di-sulphide solution are so fixed that it is possible to bring them quickly and completely under water in case of danger. All sumps can be pumped out by rotary pumps.

In order to produce an inert gas for protective purposes, a small inert gas producer is provided which is about 20 meters from the extraction and refining plant, set up in the bunker bay of the tower purifier. This device consists of a horizontal boiler in which coke oven gas is burned and cooled to 80-100° by means of cooling water. The further cooling of the inert gas is effected in a cooler lying below and goes down to about 10° above cooling water temperature. An inert gas compressor draws the inert gas through the boiler and the coolers, compresses it to about 3 atmospheres gauge, and transfers it partly to the inert gas containers (35), (36) and (38). A membrane governor is provided in front of the burner for keeping the gas pressure constant. If the permissible pressure is exceeded the inert gas gets out into the air through a safety valve. If the inert gas plant is to be let down a vent into the air is used until the desired composition of the gas is reached. For testing the inert gas a self acting oxygen recorder is provided. As the solvent and crude and pure solution tanks should not be put under pressure the transfer of inert gas is arranged in such a way that the liquid to be transported flows temporarily from the liquid tank itself, which is never under pressure, to two pressure vessels which are under inert gas pressure controlled by the regulator (31) or (12) alternately. While non-return valves prevent the pressure in the pressure vessel from getting back into the liquid tank the contents of the pressure vessel can be moved to the high level tanks (11) or (30) or to another vessel. By arranging the two pressure vessels and automatic change-over devices (12) or (31) it can be achieved that the

transfer by inert gas of solvent or pure or crude solution can be done almost continuously and is automatically interrupted when the tank from which it is being transferred empties for one reason or another. The inert gas is drawn out of the inert gas container through the active charcoal container (9) to (33) and compressed to the necessary pressure of about 3 atmosphere gauge by piston compressors (13) and (32). The inert gas producer is always running and serves for blowing out the extractors after the mass has been introduced.

The spraying device for the solvent and crude gas tanks and the flooding of the trenches can be effected from a service platform which is conveniently arranged 10 to 25 meters from the plant.

All the solvent and crude and pure solution tanks together with their pipe-lines are earthed, in order to avoid charging by static electricity. Furthermore, an effective lightning conductor system is provided which together with fire fighting and quenching devices will be built by the constructors. Doors and gates in sufficient numbers and of sufficient dimensions must be present and must open outwards.

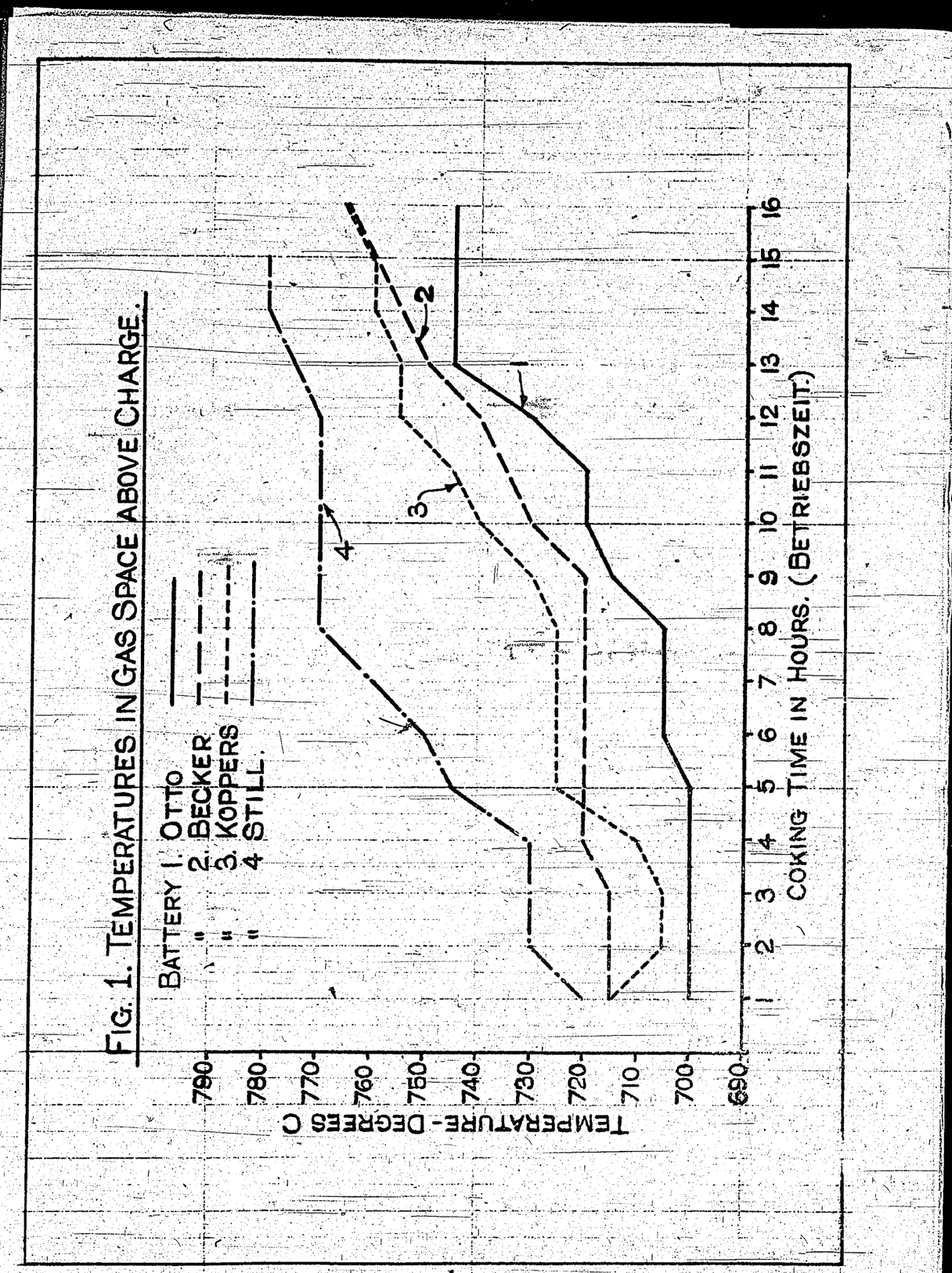
The staff room is accessible from outside and is separated from the extractor bay by a glass wall. Provision is made for the adequate ventilation of all operating spaces. Corrugated "ternite" is used for roofing. Provision is made for the sulphur extraction and refining plant to be at a sufficiently safe distance from the other operations. The electrical fittings of dynamos and motors comply with regulations for protection against fire-damp.

The travelling crane in the extractor bay is driven by compressed air on safety grounds.

Berlin, 15th December, 1938.

Bamag-Meguin.

The numbers in parenthesis in the above description refer to the drawings, Items XVI, XVII and XVIII in the List of Documents.



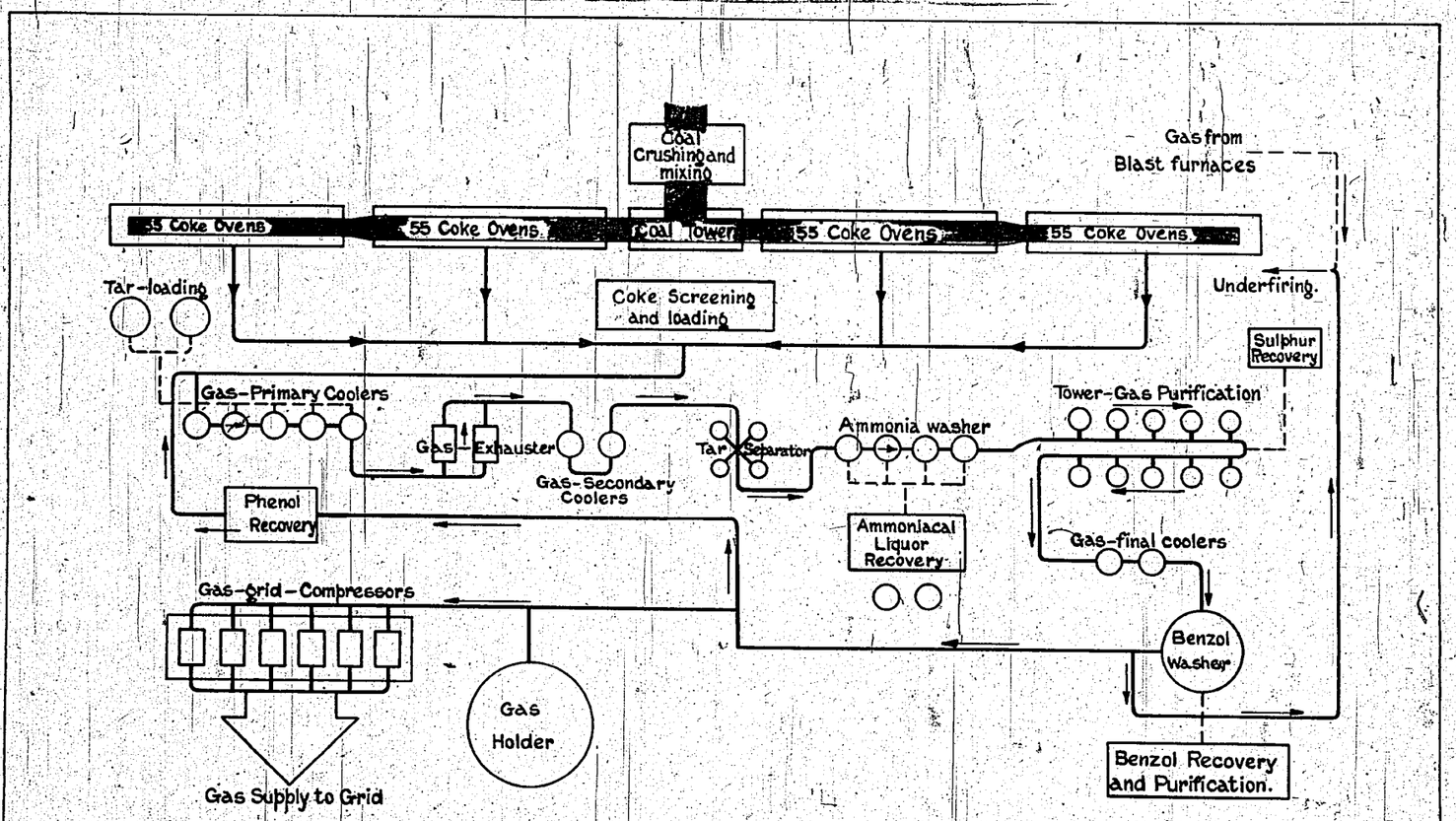
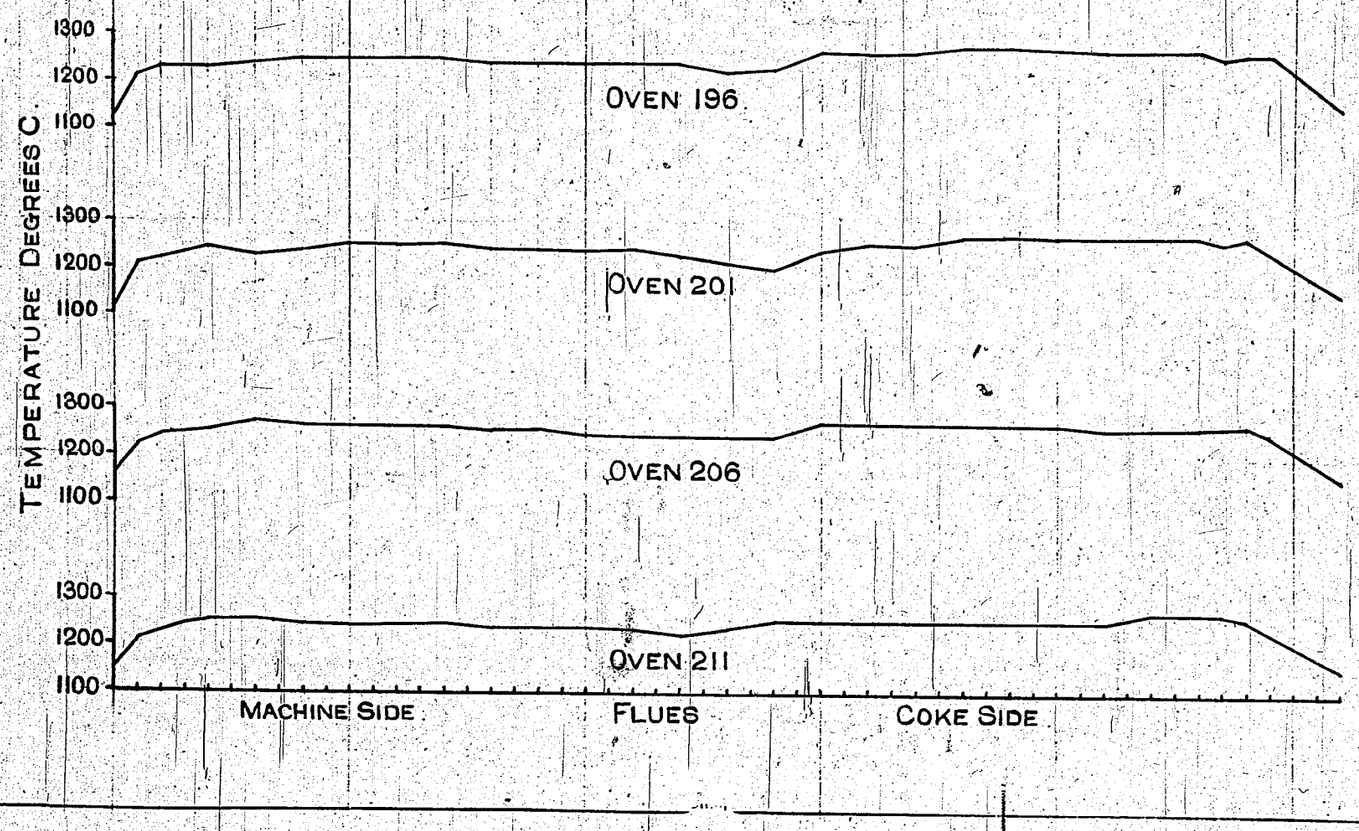


FIG. 3. FLOW SHEET - No. 1. COKE OVEN PLANT.

FIG. 2. TYPICAL FLUE TEMPERATURES.- No.4 BATTERY - STILL OVENS.
TAKEN 5 MINS. AFTER REVERSAL, AFTER 8 HOURS.- COKING PERIOD 17 HOURS.



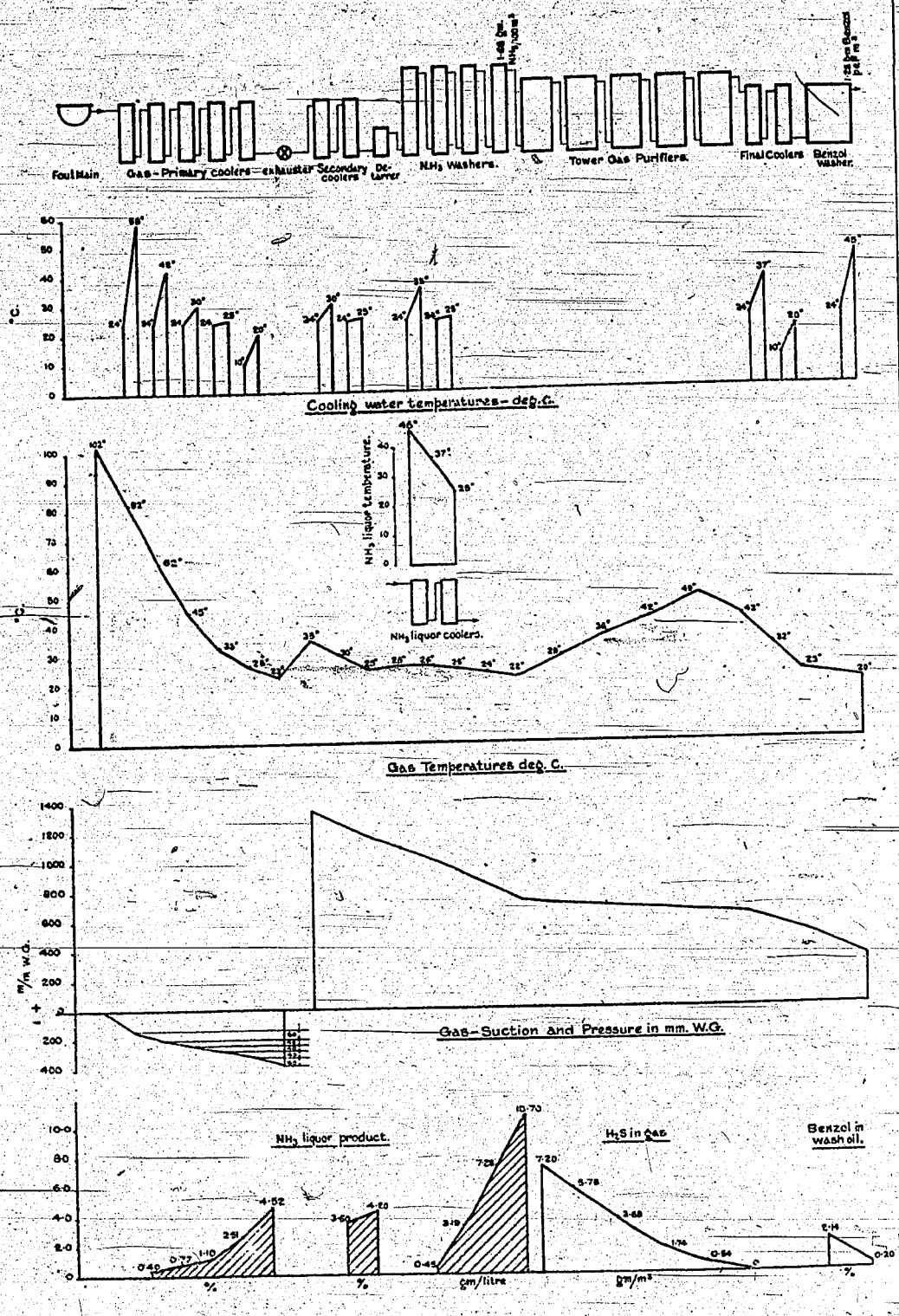


FIG. 4.

TEMPERATURES OF COOLING WATER AND GAS — PRESSURE GAS — CONTENTS OF NH_3 IN LIQUOR AND H_2S IN GAS.

Crude Benzol I 76·0

1

Washed with 4% wt. 65% H_2SO_4 *
Neutralise with 2% 18% NaOH.

* Mixture of waste acid and 77% H_2SO_4

Washed Crude Benzol. (98·5) 74·90

Loss. (1·5) 1·10

2

Motor Benzol. (96·7) 72·45 → 39 32

Residue. (2·2) 1·63

Loss. (1·1) 0·82

3

Crude Toluol. 15·5

5

Wash with 5% wt. H_2SO_4 – 66° Bé.
Neutralise with 3% 18% NaOH.

Washed Crude Toluol. (97·0) 15·04

Loss. (3·0) 0·46

6

Motor Benzol. (3·7) 0·55

Purified Benzol. (3·5) 0·53

Pure Toluol. (66·8) 10·05

Purified Toluol. (23·4) 3·52 → 7

Pure Xylol. (0·6) 0·09

Residue. (1·0) 0·15

Loss. (1·0) 0·15

Crude Benzol II 8·5

8

Wash with 1% wt. 77% H_2SO_4
Neutralise with 1% 18% Na.OH.

Washed Crude Benzol II. (98·0) 8·33

Loss (2·0) 0·17

9

Washed Distilled Crude Benzol II (94·0) 7·83 → 10

Residue (1·9) 0·16

Brown Resin (3·0) 0·25

Loss. (1·1) 0·09

FIG. 5. PROCESS OF OPERATION FOR BENZOL PURIFIC

72·45

1·63

0·82

0·55

0·53

10·05

3·52

0·09

0·15

0·15

7·83

0·16

0·25

0·09

39·48 Motor Benzol
32·97

3 — Wash with 5% wt. H_2SO_4 – 66° Bé. Neutralise with 2% 18% Na OH.

Washed Motor Benzol (97·2)

Loss. (2·8)

4

Motor Benzol (14·1) 4.52

Purified Benzol (84·0) 26·93

Residue (1·0) 0·32

Loss. (0·9) 0·28

7 — Purified Benzol (9·9) 0·35

Pure Toluol. (89·9) 3·16

Loss. (0·2) 0·01

10 — Wash with 1% 77% H_2SO_4 Polymerise with 1·5% H_2SO_4 – 66° Bé. Neutralise with 1·5% Soda Ash.

11 — Super Clear Coumarone Resin (11·7) 0·92

Solvent Naphtha (87·0) 6·81

Loss. (1·3) 0·10

12 — Wash with 5% wt. H_2SO_4 – 66° Bé Neutralise with 3% 18% Na OH.

Washed Solvent Naphtha (98·0) 6·67

Loss. (20) 0·14

Pure Xylol (46·2) 3·08

Motor Benzol (43·0) 2·87

Residue (9·7) 0·65

Loss. (1·1) 0·07

13

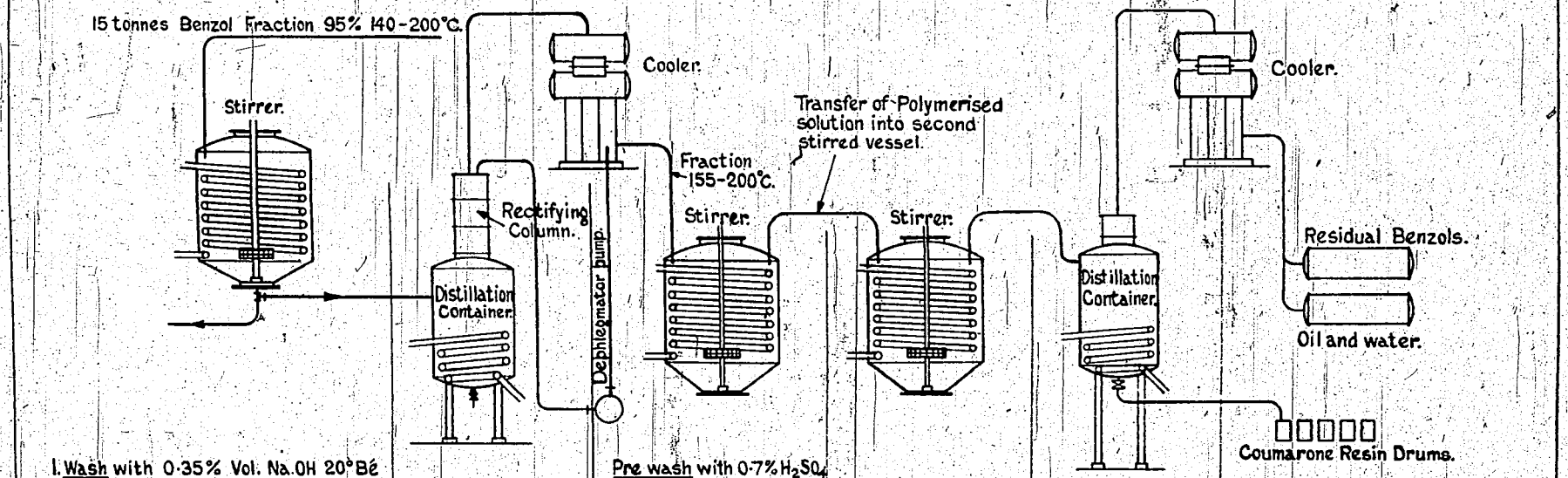
TOTAL PRODUCTS.

Motor Benzol	47·42
Purified Benzol	27·81
Pure Toluol	13·21
Pure Xylol	3·17
Super Clear Coumarone Resin	0·92
Brown Coumarone Resin	0·25
Residue	2·91
Loss.	4·31
Total	100·00

MATERIALS PER TON OF RAW MATERIAL.

	TONNE
H_2SO_4 – 60° Bé	0·014
H_2SO_4 – 66° Bé	0·033
Na OH – 45 %	0·027
Caustic Soda	0·001
Tar Oil	0·014
Steam at 18 atm. gauge for washed Benzol	0·64
for washed Toluol	2·06
Electric Power	21·6 K.W.H.

PURIFICATION BASED ON OPERATING RESULTS JAN.– NOV. 1944.



1. Wash with 0·35% Vol. Na.OH 20°Bé
Wash out Phenols and draw off alkaline liquid.

2. Wash with 0·35% Vol. H_2SO_4 60°Bé
Wash out bases and draw off acid.
Final wash with 100ℓ. water.
Draw off water.
Neutralise with 0·35% Vol. Na OH 20°Bé
Draw off alkaline liquor and lead off pre washed product into the container.

Distillation of 15 tonne of pre washed product for production of clear product.

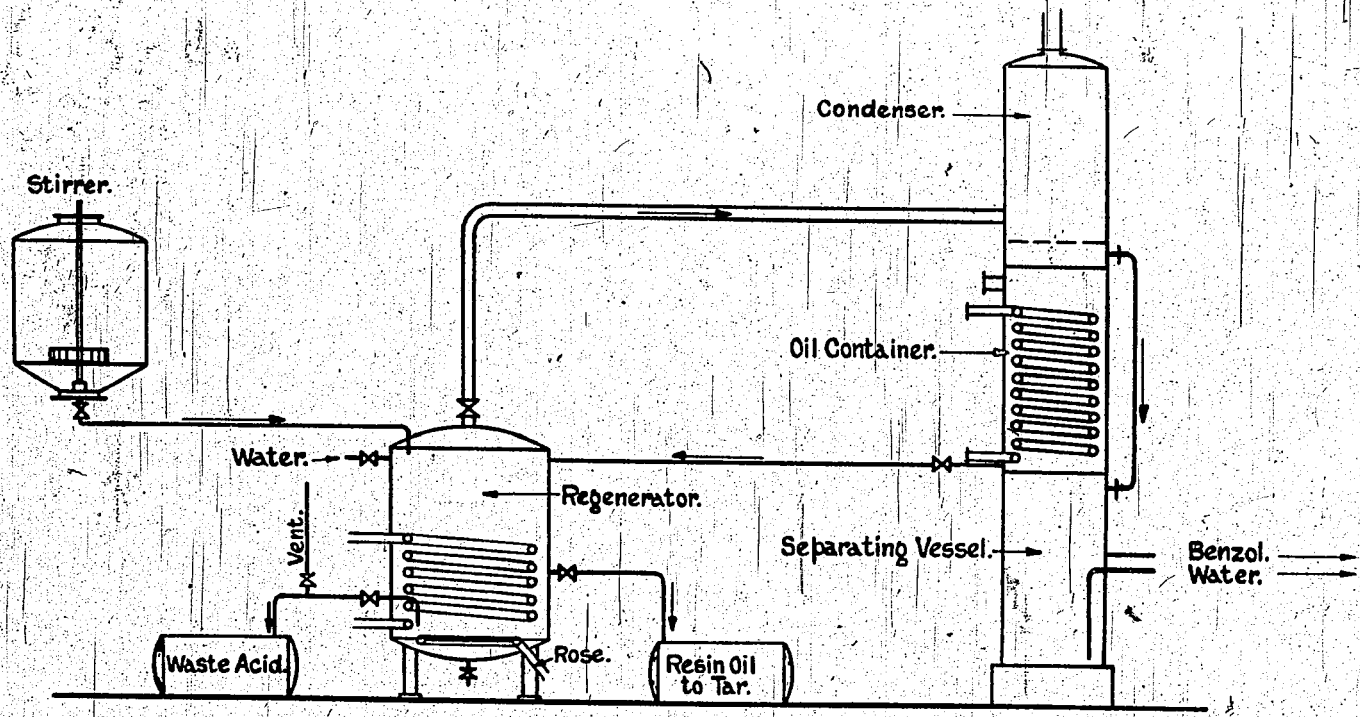
Pre wash with 0·7% H_2SO_4 60°Bé.
Polymerisation with 0·35-0·50% Vol. H_2SO_4 Bé.66°
Gradually let in with constant stirring. Temp. must not be allowed to exceed 50°C. Cooling must be carried out in good time.
Draw off acid.

Neutralise twice with Soda Solution Sp.Gr. 1·15 at about 85°C.
1. 200 kg. Soda
2. 200 kg. Soda
Leave to settle well and draw off.

Distillation with indirect steam until the correct softening point is reached.

Drawing off of Coumarone Resin with softening point of 55°C into drums.

FIG. 6. FLOW SHEET FOR PRODUCTION OF COUMARONE RESIN.



The waste acid with the resins is run down from the stirred vessel into the regenerator. Then about 1·5 tonnes of hot middle oil (100°C) is added from the oil container, the whole meanwhile being agitated with air. The oil must be hot to avoid resinification. While the oil is running in, about 150 litres of water are added simultaneously – the heating coil is put into action. The whole is then agitated with air for about 2 hours. The issuing vapours are condensed in the condenser and are lead away via the separating vessel. The acid–resin oil mixture is then allowed to stand for 2–3 hours, and the acid is drawn off through the dip pipe until traces of oil appear. Then steam is blown in through the rose again for 5–10 minutes in order to make the remaining acid settle. After ½ hour the resin oil is drawn off. Owing to the difference in the arrangement of the offtakes for oil and acid, one being a dip pipe with vent, there is always a certain quantity of acid and oil left in the regenerator, where by it is ensured that the acid is absolutely resin-free. The acid strength amounts to at least 45° Bé. which is 55% H_2SO_4.

FIG. 7. FLOW SHEET OF AN ACID REGENERATION PLANT ACCORDING TO THE MENZEN BOCHUM SYSTEM.

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ITEM No. 22
FILE No. XXIX - 12 (APPENDIX)

POLYURETHANES

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(I. G. Farbenindustrie, Leverkusen)

Bayer, O.

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P O L Y U R E T H A N E S

Dr. O. Bayer
(I.G. Farbenindustrie. Leverkusen)

Translated from C.I.OS. Report No. XXIX - 12

by

L.V. CHILTON, M.A.P.

CIOS Target No: 22/1(e).

Combined Intelligence Objectives Sub-Committee
32 Brysnston Square, London, W.1.

Translated from
CIOS Report No.XXIX-12.

POLYURETHANES

Dr. O. BAYER - LEVERKUSEN I.G. WERK.

(Lecture given at the T.A. meeting at Frankfurt/Main, 24th September, 1941.)

General Characteristics of Condensation and Polymerization Processes.

High-molecular compounds, as is well known, may be prepared by either of two fundamental methods, i.e., by condensation or by polymerization.

The characteristics of the condensation method are:

1. A great number of small molecules each having at least two functional groups react together, with the elimination of a low-molecular reaction product, such as water, HCl, NaCl, alcohol or the like.

2. For the realisation of the high-molecular state many vigorous reaction conditions, such as high temperature, condensation media, etc., are employed; and consequently the product consists in the great majority of cases of mixtures of the most varied condensation stages.

3. It is of importance that in condensation reactions there is the possibility of starting from a selected intermediate condensation product and permitting the reaction to go to completion under constant deformation, whether the material is a pressure moulding, a casting resin or an unmeltable lacquer-coating. Furthermore, mixed condensates may be prepared from the most varied partial condensation-products.

Typical condensation products of the class of two-dimensional linear high-polymers are for example the thiokols (from $ClCH_2.CH_2Cl + Na_2S_{4,5}$) and the superpolyamides (obtained with molecular weights from 10,000 - 20,000 by the condensation of adipic acid with hexamethylene diamine); and in addition, as examples of the more important members of the class of three-dimensional (i.e., fully "netted") synthetic materials, we may mention fully hardened Bakelite resins, urea-formaldehyde resins, and the Glyptals.

As contrasted with the condensation process, the essential points of polymerization reactions are:

1. The apparently smooth arranging together of many molecules of the monomer - up to 5,000 in a linear structure - to form a high-molecular polymer. The latter possesses the same percentage composition as the monomer. Polymerization

is an extraordinarily sensitive type of reaction, which results in all probability from the displacement of an activated hydrogen atom from molecule to molecule of the monomer. It takes place, by contrast with condensation, under extremely mild conditions. Viewed kinetically it is a pure chain reaction.

2. It is typical of polymerization reactions that only end-products are obtainable: it is not possible, as in condensation reactions, to start with reactive intermediate stages representing di-, tri- or tetramers and to polymerize these further either among themselves or with the monomer. On the contrary, these intermediate products act as polymerization poisons, as is the case, for example, of the polymerization of isobutylene to Oppanol.

3. A further important aspect of polymerization is that in practice it is possible only to proceed from reactive vinyl compounds (or their equivalents) and to obtain from these either linear or netted polymers in all of which there is a direct carbon to carbon linkage. It is true that mixed polymerization is possible between two or three polymerizable compounds, giving a range of possible variants (Igelite, Buna, etc.) but in practice high-molecular structures linked by hetero-atoms do not arise. In only a few exceptional cases are pure polymerization reactions known involving hetero-atoms: we may recall here the polymerization of highly stressed ring systems such as ethylene oxide, ethylene imine and caprolactam, and the vulcanization of rubber which must be regarded as a further polymerization by net-formation from the linear rubber, across sulphur molecules.

Although the complex totality of our synthetic materials is made by one or other of these fundamental processes, there has been lacking until recently a universal polymerization method which combines the possibilities and advantages of condensation and of classical polymerization methods - which will permit us, starting from selected bi- and polyfunctional low molecular compounds, whether partial condensates or partial polymerizates, to proceed by purely additive reactions to a desired endpoint, - to a synthetic material with the desired properties.

The Polyurethane Process: Complex-Formation by Addition.

Such a method was discovered in 1937 in the so-called polyurethane polymerization or di-isocyanate process, and has since then been extended (German Pat.Appl.J59,592 of 11th Nov.,1937).

The bases of the new method are as follows:

Wurtz, as long ago as 1848, found that

$C_2H_5N{=}C{=}O$ smoothly adds to C_2H_5OH to form $C_2H_5.NH.CO.OC_2H_5$,

and to $R\text{-}NH_1$ to form $C_2H_5.NH.CO.N\begin{matrix}R\\R_1\end{matrix}$,

the hydrogen atom of the hydroxyl or amino group being displaced to the nitrogen atom of the isocyanate group. A.W. Hoffmann established these reactions in the aromatic series, with phenyl isocyanate, in 1849. To utilize these reactions, which are quantitative and proceed with considerable heat of reaction, in the production of synthetic materials, it is necessary to

prepare diisocyanates and add these to glycols, diamines, polyhydroxy or polyamino compounds. You will recognise, from the following scheme of the simplest reaction between a diisocyanate and a glycol, that here we have a reaction running parallel to pure polymerization.

Mechanism of Polyurethane Formation

$$\text{HO.R.OH} + \text{OC:N.R.N:CO} + \text{HO.R.OH} + \text{OC:N.R.N:CO} + \;\downarrow$$

$$\text{- O.R.O.CO.NH.R.NH.CO.O.R.O.CO.NH.R.NH.CO -}$$

An essential difference between this and the customary polymerization reaction lies in that the molecular linkages are not through carbon alone but also through the hetero-atoms oxygen, carbon and nitrogen: and the enormous number of possibilities, considering the polyhydroxy compounds alone, implies a breadth of applications which could be claimed hitherto for neither condensation nor polymerization processes.

When Carothers' work on superpolyamides became known the I.G. was faced with the pressing problem of producing similar or better products not covered by the DuPont patents.

Typical Polyurethanes and their Properties.

We therefore studied first the addition of diisocyanates to diamines and to glycols, in order to prepare in this way high-molecular linear poly-ureas or polyurethanes. It was found that the use of long-chain aliphatic diamines and aliphatic diisocyanates led to the production of unmeltable and strongly hydrophilic polymers which could not be used in a melt-spinning process and were therefore useless as a practical contribution to the chemical technology of synthetic materials.

Polyurea

$$\text{- N:CO} + \text{NH}_2(\text{CH}_2)_6\text{NH}_2 + \text{OC:N}(\text{CH}_2)_6\text{N:CO} + \text{NH}_2(\text{CH}_2)_6\text{NH}_2 + \;\downarrow$$

$$\text{- NHCO.NH}(\text{CH}_2)_6\text{NH.CO.NH}(\text{CH}_2)_6\text{NH.CO.NH}(\text{CH}_2)_6\text{NH -}$$

A basic poly-urea made in this way nevertheless finds use as an animalizing medium for cuprammonium rayon.

Furthermore, linear polyurethanes proved to be useful in many directions and to have in some respects more valuable properties than the superpolyamides.

From the great number of linear polyurethanes so far prepared I can select only a few characteristic ones. The simplest in practice is the polyurethane from 1:6-hexane diisocyanate and 1:4-butylene glycol.

Polyurethane

from 1:6-hexane diisocyanate and 1:4-butylene glycol

$$- NCO + OH(CH_2)_4OH + OCN(CH_2)_6NCO + HO(CH_2)_4OH + OCN(CH_2)_6NCO + \downarrow$$

$$- NH.CO.O.(CH_2)_4.O.CO.NH(CH_2)_6NH.CO.O.(CH_2)_4.O.CO.NH(CH_2)_6NH.CO -$$

This is suitable for the production of artificial silk fabrics and above all for bristles. It is made in practice by the addition of 1 mol of the purest 1:6-hexamethylene diisocyanate to 1 mol of 1:4-butylene glycol in monochlorbenzene as solvent, whereupon the addition or polymerization proceeds with considerable evolution of heat (52 Cals per mol or 208 Cals./kg.polyurethane). The polyurethane separates after about 1 hour as a fine sandy powder, with a practically quantitative yield. According to the temperature and the period spent in solution (which can be adjusted by addition of dichlorbenzene, etc.), or by chain-breaking with mono-functional compounds, it is possible to synthesize polyurethanes having the most varied degree of polymerization.

The properties of such a polyurethane which is on the market as Igamid U for artificial fabrics (K-value of 60-70), and as Perlon U for silk and bristles (K-value 55-60), and its points of difference from Nylon and Perluran-materials, are as follows: Igamid U, owing to its mode of preparation by polymerization in a solvent and precipitation when a particular stage of complexity is reached, is more homogeneous than the superpolyamides, which are made by condensation. This is shown for example by the proximity of its melting point to its softening point.

Of all the technically important spinnable linear superpolymers at the moment, Perlon U possesses the lowest melting point - about that of acetate silk - and this is undesirable in a range of textile applications.

Table: Physical Properties of Igamids

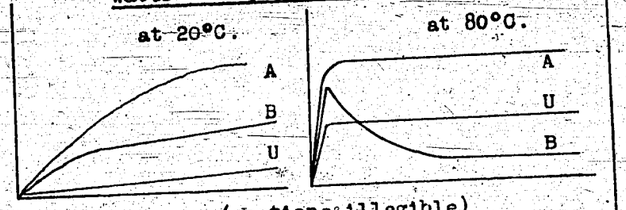
(mainly undecipherable, but see below)

	Igamid U	Igamid A	Igamid B
Density	1.21	1.14	1.15
Melting Point about	185°	255°	240°
.....	750	1000	600
Brinell Hardness	170°-180°	220°-225°	....
..... Temperature	100	100	....
Impact Strength about			
.....			
.....			300
Tearing Strength	...2.0	-	+ 15°
.....	+ 8°	-	
.....			
.....			

The strengths of silks and bristles made from polyurethanes are practically equal to those of superpolyamides. In the laboratory values of tensile strength up to 7.5 g./den. have been obtained for Perlon U (natural silk, 3.5 g./den.). It has been possible thus to prepare fibres of a strength more than 100% greater than that of the best natural silk.

Igamid U and the other polyurethanes are especially characterized by a low water-absorption.

Water-Absorption of Igamids.

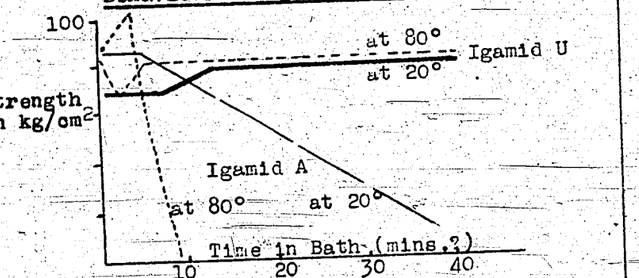


(captions illegible)

With increasing numbers of CH_2 groups in the polyurethane chain the water absorption rapidly decreases but, as the molecule becomes more paraffinoid, the strength and the melting point also decrease.

Polyurethanes are superior to polyamides, too, in their electrical properties and in their stability to weathering. They are also characterized by a superior resistance to acids.

Behaviour of Igamids in 52% HCl



(captions illegible or difficult to read with certainty

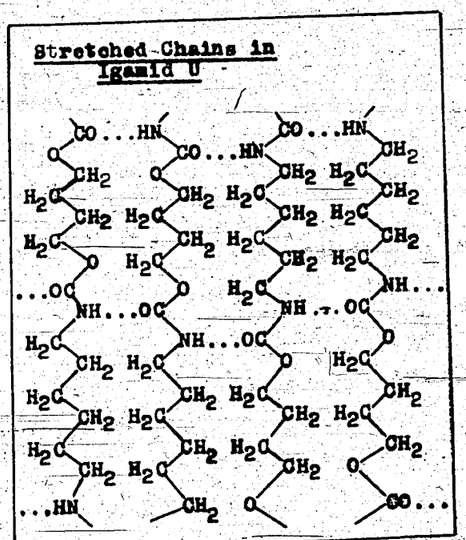
These special properties, taken in conjunction with the usual good properties of superpolyamides, make Igamid U a valuable high-melting thermoplastic for synthetic fabrics. Threads spun from it are unaffected by water or by atmospheric moisture and, above all, keep their dimensions. All these properties open up to Igamid U and to Perlon U a series of special technical

applications, for example, in the preparation of filter-cloths stable to boiling and to acid, clothing resistant to acids and to mustard gas, driving belts, ropes and cable-insulation. In the textile field the low water-absorption of polyurethanes is a disadvantage in many cases, e.g., for underclothes, but it acts as an advantage in the manufacture of ladies' hydrophobic stockings.

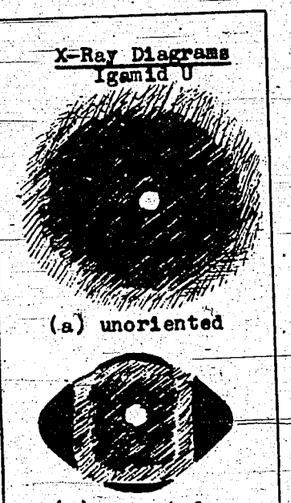
Perlon U, in consequence of its hardness, its quasi-metallic elasticity and its resistance to water at temperatures up to 80°, is a valuable material for the manufacture of bristles and horsehair. Perlon U differs from the superpolyamides in its behaviour with dyes: since it contains no basic groups it is not restricted like the latter to acid dyes, but can be dyed with acetate-silk and dispersed dyestuffs. Because of its freedom from basic groups, Igamid U in the melted condition is much less sensitive to atmospheric oxygen than are the superpolyamides, so that browning of the material does not occur during melting, spraying and spinning, even when air is not completely excluded.

In its usual physical behaviour and in its fabrication, e.g., the fact that it may be stretched to some four times its original length (the molecular chains being oriented thereby), and in its destructive deformation by boiling water when strongly stretched ("board setting"), it most closely resembles the superpolyamides.

Stretched Chains in Igamid U



X-Ray Diagrams Igamid U



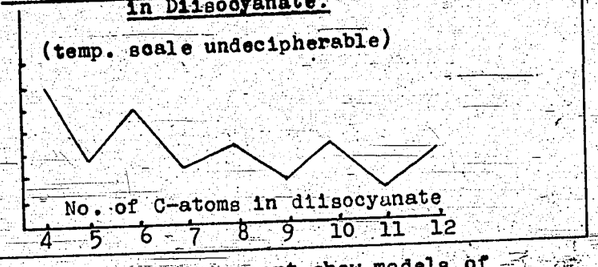
(a) unoriented

(b) oriented

Melting Points of some Polyurethanes

Nos. of C-atoms in Diisocyanate	Glycol	M.Pt.
4	4	490°
4	6	180°
4	10	170°
6	3	167°
6	5	159°
6	9	147°
12	12	128°

Melting Points of Polyurethanes from..... as function of increasing number of C-atoms in Diisocyanate.



(Plates at this stage of the report show models of polyurethane chains in the unoriented and oriented condition; and of parallel chains as in the diagram on p. 6 of this translation, with the note that spinnable polyurethane molecules have a chain-length some 25 times longer than that illustrated in the latter model, i.e., have a chain with some 50 molecules of the glycol and 50 of the diisocyanate.)

Apart from Igamid U and Perlon U the following types are of technical importance:

The polyurethane from 1:4-butane-diisocyanate and 1:4 butylene glycol. This has practically the same properties as Igamid U but melts about 12° higher (M.Pt.194°). Probably it will be a cheaper substitute for Igamid U, and will make us independent of phenol or benzene as starting material, 1:4-diaminobutane being made from acetylene and hydrogen cyanide.

The lower members of these unitary polyurethanes are completely insoluble, in the cold, in all solvents except a phenol-water mixture (90:10). On the other hand, mixed polyurethanes obtained from a mixture of glycols or diisocyanates have good solubility in chlorinated hydrocarbons even in the cold; and have, moreover, lower melting-points, a greater thermoplastic range, greater softness and better compatibility with plasticizers. They provide materials, therefore, for the production of films, foils and leather-substitutes.

An Igamid U in which only 10% of the butylene glycol is replaced by methyl-1:6-hexane diol is much more thermoplastic and therefore more easily sprayed than the unitary material. Of importance, too, is the fact that it can be made without the use of a solvent, by melting the components together. It has been shown to be broadly immaterial in practice what glycols are copolymerized with the cheap 1:4-butylene glycol. Igamid UL is a mixed polyurethane intended as a leather substitute, obtained by melting together 1 mol 1:6-hexane diisocyanate + ½ mol 1:4-butylene glycol + ½ mol methyl hexane diol. A mixed polymer with quite similar properties is made, for example, from ½ mol 1:4- + ½ mol 1:6-diisocyanate and 0.8 mol 1:4-butylene glycol + 0.2 mol methyl hexane diol.

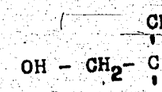
Igamid UL is a very soft material readily soluble in chloroform-alcohol mixtures. In many of its properties it surpasses sleeked leather.

Physical Properties of Igamids as Leather Substitutes

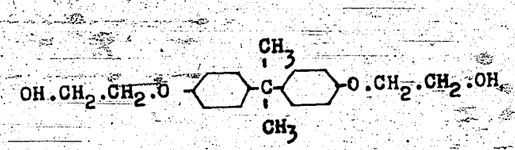
Igamid UL	.....	Sleeked leather
(undecipherable)		

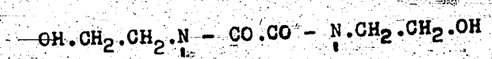
Of the Igamid varieties worth considering as leather substitutes, it has a somewhat higher L-strength and, above all, the advantage of being practically completely resistant to water.....(passage missing)..... as shown by heating in water.

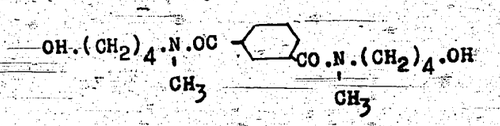
The polyurethane from 1 mol 1:6-diisocyanate + ½ mol 1:4-butylene glycol + ½ mol (2-dimethyl-)propylene glycol



because of its elastic properties, is especially suitable for the impregnation and lamination of textile fabrics. Other interesting types of polyurethanes, which are still in course of thorough test, are made in part from the cheaper of the aromatic diisocyanates + aliphatic glycols, or from aromatic glycols with hetero-atoms, e.g.,







or from aliphatic linear polyesters with free hydroxyl groups, etc. They will find application as electrical insulating foils, sausage-skins resistant to boiling, supports for photographic film, and in military requirements, etc.

In the course of this summary lecture I cannot unfortunately go fully into the numerous special fabrication processes and applications of polyurethanes. I might mention in passing that a whole series of new and difficult problems have arisen in the production and handling of polyurethanes, such as the production of polyurethanes of a particular K-value, the stabilization of the material against further polymerization and against decomposition in the melt, etc. New methods of appraisal, new plasticizers, new stabilizers, etc. have to be found.

Patent Position.

In the production of polyurethanes and in their use in the realms of foils and synthetic products we are completely free from the DuPont patents. In their application to silk and bristles we have succeeded, in conjunction with Dormagen and Wolfen, in devising working-methods clear of patents.

The patent position in U.S.A. is, however, still obscure. DuPont, whom we informed of our work in its very earliest stages, have applied for U.S.A. patents on polyurethanes Igamid U is practically no dearer than superpolyamides: the I.G. will thus save in future quite considerable expenditure on licences. Moreover, with the new material we shall be free from the territorial restrictions of the polyamide contract.

Polyurethane Lacquers.

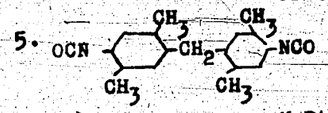
Linear polyurethanes are practically all thermoplastic and may be moulded or worked by heat or with solvents or plasticizers. If, however, diisocyanates are reacted with compounds having more than two reactive hydrogen atoms, e.g., with glycerol, cellulose acetate, polyesters with free hydroxyl groups, polyvinyl alcohol, etc., three-dimensional macromolecules are obtained which are insoluble in all organic solvents and are unmeltable. As such they are of only very restricted use in the manufacture of synthetic products (resins for casting, workable only with machine tools for shaping). If it is desired to make use of the great number of known polyhydroxy compounds to produce synthetic products, the addition of the diisocyanate must be made only during the finishing process. In the most important applications in the realm of lacquers and synthetic materials, it is thus necessary to allow the two components to react either on the object to be coated or by prolonged pressure moulding as in formaldehyde moulding compositions. (German Pat.Appl. J66,687).

An isocyanate lacquer is prepared for example by dissolving a polyalcohol and a diisocyanate, in an approximate ratio of OH- to NCO- groups of about 1:3, in a solvent not containing hydroxyl groups, and with pigment added if desired. Such a lacquer is then sprayed on and hardened by standing at room temperature or by heating up to 160°. There results a completely insoluble and unmeltable lacquer coat, the elasticity and hardness of which can be adjusted as desired by proper choice of the predominant aliphatic or aromatic components.

It is an obvious requirement of this mode of working that the reagents shall not immediately react at ordinary temperatures, or the lacquer solution will gel up. This requirement is fortunately met by some diisocyanates. According to conditions and choice of starting materials, the freshly prepared solutions may be stable for many hours or even days.

It has been discovered, moreover, that the stability may be raised by certain "stabilizers", especially when organic cellulose derivatives are used (as hydroxy-compounds? Trans.). These are, for example, azo dyes soluble in organic solvents and certain acid-reacting compounds like thiourea and its derivatives (German Pat. Appl, J67,162).

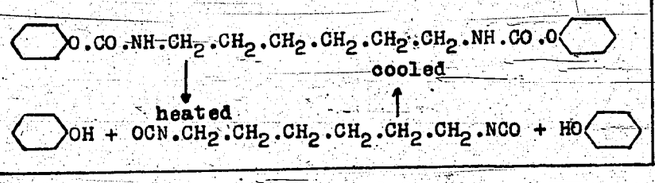
Of the technically available diisocyanates the following, which are arranged according to their reactivity, come up for consideration:-

1. 1-chlorphenylene-2:4-diisocyanate (Most reactive)
2. m-toluylene diisocyanate
3. 1:6-hexane diisocyanate
4. diisocyanate from dianisidine M.Pt.126° (+ ½ mol chlorbenzene of crystallization)
5.  M.Pt. 129°. (Least reactive)

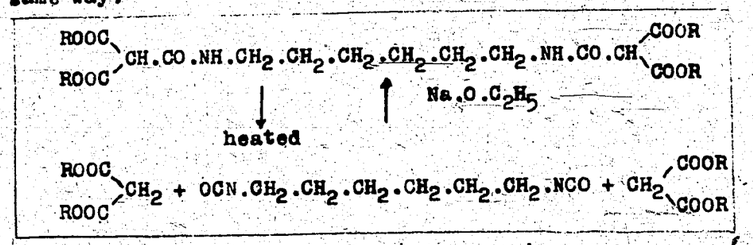
Polyurethane Lacquers using "Masked" Diisocyanates.

The problem of the stability of lacquer components can be tackled in a fundamentally different way. It is possible to use, instead of diisocyanates, compounds which when heated react like diisocyanates, i.e., which are split up with the liberation of diisocyanates. For example, one may take bisphenylurethane, which can be obtained either from a diisocyanate and phenol or from phenyl chloroformic ester and a diamine.

Mechanism of "Masked" Diisocyanates



Another class of compounds can be used for the same purpose, viz. the addition product of a diisocyanate and malonic ester or acetoacetic ester, which splits on heating, in the same way.



Other suitable addition products, which are easily split on heating, can be made from diisocyanates with phenyl methyl pyrazolone, diphenylamine or with C ≡ C-C(CH3)2-OH. A further splittable addition product may be prepared from hexane-1:6-diisocyanate + 1 mol ammonia.

These "masked diisocyanates" have the great advantage that lacquers made with them have unlimited stability. Indeed, it is even possible to use solvents containing hydroxyl groups with them since such solvents generally evaporate before the diisocyanate splits off. On the other hand, there are the disadvantage that a higher hardening temperature is needed and the inconvenient fact that undesirable fission products arise. Extensive experiments have shown, however, that the liberated phenol, for example, or the residual malonic ester and its decomposition products, evaporate together during the hardening process and cause no further trouble. By these means it is possible in any case to carry the complex-formation more readily to the desired degree than with free diisocyanates, since completely netted polyurethanes are not always by any means the best.

Of the vast number of cheaply producible polyhydroxy compounds the following have proved especially useful:-

1. A saponified copolymer of 80% vinyl chloride and 20% vinyl acetate;
2. A hydroxyl-containing polyester (prepared by esterification in vacuo) from about 3 mols adipic acid or thiodipropionic acid, phthalic acid, etc., and 4 mols trimethylolpropane, 1:2:4-trihydroxybutane, etc. By mixing the acid components it is possible to vary the elasticity between wide limits.
3. Luphenes ("Die Luphene");
4. Benzyl cellulose;
5. Condensation products from esterglycols (from 2 above) + oxygen-rich xylol-formaldehyde resins.

To summarize, the new lacquers have been found, as the result of thorough testing by the lacquer divisions of Le and Lu, to have the following advantages:-

1. As compared with alkyd lacquers they may be hardened at much lower temperatures - in some cases even at room temperature - and can therefore be applied to materials such as paper, textiles, leather, wood, rubber products, and synthetic materials.
2. They possess a range of variability of properties which is not often possible with other lacquers.
3. They are characterized by an unusual degree of adhesion to the support and by a very high gloss and are in the majority of cases very stable to cold. Wet-strength and pigment-carrying properties, and stability to water and to solvents are outstanding.

4. Since the diisocyanate groups react with all mobile hydrogen atoms, which are responsible for electrical conductivity, diisocyanate lacquers exhibit a high electrical resistance (10^{15} - 10^{16} ohm.cm.) and also a remarkably good leakage-current strength, which is of considerable significance for cable-lacquers.

The disadvantages of the new lacquers are:-

1. The low stability of the finished lacquer solutions. They must be used within a day.

2. The use of hydroxyl-free solvents and the complete absence of water.

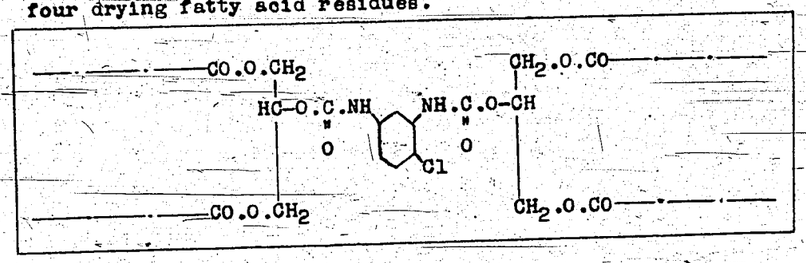
3. The occasionally pungent smell of the low-molecular diisocyanates.

The good adhesion, the high elasticity, and the stability to boiling water, of all isocyanate lacquers appear to make them especially suitable as coatings on deep-drawn sheets for canning. Since complex-formation occurs without fission products the lacquer coats have in any case a high gas-tightness. Diisocyanate lacquers are noteworthy, in addition, for their good resistance to mustard gas, which considerably exceeds even that of Oppanol. A paper impregnated with them (66 g./m^2.) with a lacquer coating weight of only 14 g./m^2. has an L* value of 13 hours (!) according to MR 30 and of 4 hours according to MR 50. The testing of these L-stable papers and textiles is being very thoroughly carried out at present.

Polyurethanes in the Refining of Linseed Oil.

Our observation that the introduction of urethane groups gave materials of high resistance to water and that, moreover, molecular coupling by means of diisocyanates was an exceptionally gentle method of construction, gave us the means of attacking from the new point of view the important and oft-tackled problem of the refining of linseed oil.

We esterified linseed oil to the diglyceride with the calculated quantity of glycerol or trimethylolethane and combined this with ½ mol chlorphenylenediisocyanate containing four drying fatty acid residues.



* L = presumably "Lost", i.e., mustard gas (Trans.)

In this way, it is possible to prepare oils with any desired viscosity, according to the degree of esterification, and these, unlike stand-oil (from linseed-oil) and certain Alkydals, have not undergone the vigorous thermal disturbance of heating to 280°.

The modified linseed-oil so obtained is much superior to linseed-oil stand-oil and, in the opinion of the Uerdinger Alkydal Division, possesses properties similar to those of the highest-valued Alkydal L Extra, combined with the "Spitzeneigenschaften" of all Alkydals. It has the advantages over Alkydal L Extra of quicker onset and completion of drying, higher film elasticity, more rapid attainment of water-resistance, greater compatibility and the absence of after-thickening with basic pigments. Its only disadvantage is its slightly lower stability to light.

Table of Comparative Properties - Modified Linseed Oils and Alkydals. (undecipherable)

As soon as larger quantities of linseed oil become available Uerdingen will take on the production of Urethane-Linseed Oil on a large scale and at a cost price about the same as that of Alkydal L Extra.

These diisocyanate linseed-oils offer a range of interesting applications in the fields of rust-protection, the preparation of oil-soaps, electrical insulating lacquers, smoothing finishes for aircraft construction, etc., and there is the further possibility of converting slow-drying oils such as fish oils into oils which dry quickly and completely. (German Pat.Appl. J.67,805 and J68,374).

Polyurethanes for Pressure Moulding.

In the preparation of moulding compositions by the new process, the diisocyanate is mixed with the polyhydroxy compound in about equimolecular proportions (based on reactive groups), a rather greater quantity of filler, e.g., wood-dust, is added, and the complex-formation carried out under heat and pressure as with Bakelite and urea moulding. In this case also, the limited stability of the mixture of diisocyanate and polyalcohol is a drawback. Careless handling results in complex-formation on the rolls of the mixer. One is advised in practice, therefore, to use only the slow-reacting and high-melting diisocyanates from dianisidine and from di-p-xylylidenemethane (see table on p. 10 of this translation), but from these, in conjunction with e.g. the hydroxyl-rich polyester from adipic acid and trimethylolethane (which finds similar application as a lacquer addition), quite remarkable results are obtained. Mouldings are obtained at temperatures of about 140° - 160° and times of 5-10 mins., which at higher temperatures are still somewhat more thermoplastic than completely hardened phenol plastics, to which, however, they are superior in important respects:-

1. A very high alkali-resistance.

2. Higher electrical resistance (ca.3-5 million megohms, as compared with 20,000 megohms for Bakelite).

3. High leakage-strength, even in the wet state.

4. Notably easier flow characteristics than in Bakelite masses, which, according to data from Troisdorf, will permit the production of pressure-mouldings up to three times the weight of those possible at present with phenol-formaldehyde.

Press-compounds from 1 part polyester + diisocyanate + 1 part wood-dust		Impact Bending Strength cm.kg./cm²	Bending strength kg./cm²	Brinell hardness in kg./cm² at 10°	Brinell hardness in kg./cm² at 60°	Martens Temp.
3 mols adipic acid 4.1 mols trimethylol propane	C_{11}-C_{12} diamine	16	260	517	490	25°C
	1:6-hexamethylene-diamine	15	342	587	552	25°C
	m-toluylene-diamine	12.5	911	1215	1175	65°C
2.5 mols adipic acid 0.5 mol phthalic acid 4.1 mols trimethylol propane	Dianisidine	13.5	1367	1590	1530	74°C
3 mols adipic acid 4.2 mols 1:2:4-trihydroxy butane	Dianisidine	14.3	1170	1380	1280	55°C
3 mols thiodipropionic acid 4 mols trimethylol propane	m.toluylene diamine	13.1	1110	1580	1520	50°C
Phenoplast Type S		6	700	1300	-	125°C
Sliced Bakelite Type Tz		12	600	1300	-	125°C

5. The most important property of the new materials is, however, their high elasticity as shown by their impact bending strength. It would appear that the problem of producing a so-called "elastic Bakelite" has thus been solved. As in the realm of lacquers there is here the possibility of producing soft and highly elastic materials by the use of predominantly aliphatic components and harder and more brittle pressings from predominantly aromatic components.

It even seems possible to effect a great simplification of the heat-and-pressure finishing process by a method of cold-forming the compound and then baking it out under pressure in an oven - a method similar to the firing of porcelain. Complex-formation then occurs quickly, avoiding deformation and changes of dimension. (German Pat.Appl. J69,444).

Such strongly netted polyurethanes are principally used at the present time for the preparation of load-bearing constructional parts of aircraft and for forming certain large special objects in which metals may not be employed and for which Bakelite is unsuitable owing to its brittleness and lower strength. New and interesting applications will probably arise also in the field of telephony. Since the new press-compounds possess also an outstandingly high degree of adhesion to the surrounding materials, they should be exceptionally suitable, according to Troisdorf tests among many others, for the preparation of high-duty polishing wheels - a large and important field of application, for which the Bakelite Company already has a monopoly for formaldehyde press-compounds containing added furfurol derivatives.

Of the very many cheaply available polyhydroxy compounds we have also extensively studied Cellit (cellulose acetate), which, with diisocyanates, gives pressure mouldings which are unmeltable and can be ejected hot from the press.

It may be remarked at this point that insoluble and unmeltable acetate-silks can be made with the help of diisocyanates. Since Cellit is spun from an alcohol-acetone solution, it is expedient to work (as in the case of lacquers) from a "masked" diisocyanate, which goes over to diisocyanate and completely nets up the long cellulose chains when the fibre is heated to about 130°. This procedure is being fully tested by Aceta Ges., with whom the trials were jointly carried out. (German Pat.Appl. J67,163).

Modified Polyurethanes for Solid-Foam Production.

Isocyanate groups react not only with the reactive hydrogen atoms of hydroxyl and amino groups but also, as Wurtz discovered as long ago as 1848, with carboxyl groups, thus:-

$$CH_3.COOH + OCN.C_2H_5 \longrightarrow CH_3.CO.NH.C_2H_5 + CO_2$$

In this case, the formation of an acid amide is accompanied by the elimination of carbon dioxide. This reaction may be used for the production of new "expanded" materials of high strength. For example, if a polyester from a dicarboxylic acid + a trihydroxy alcohol (containing therefore both free hydroxyl and carboxyl groups) is brought together with a diisocyanate, the honeylike mass undergoes reaction at 50° with the formation of urethane and carbonamide linkages and the liberation of carbon dioxide. The thick mass is thus blown up like a dough and becomes harder and harder as complex-formation proceeds. It is therefore feasible to produce expanded or foamed materials of very high strength and any desired degree of elasticity. For example, a material with a compressibility of 10 kg./cm3. and adequate thermal stability up to 100° may be made from toluylene diisocyanate + a polyester from oxalic acid, adipic acid and trimethylolethane. (Density less than 0.1). (see J69,394).

The Luftwaffe has the very greatest interest in such light materials at present for the production of unsinkable aircraft parts. Owing to their high strength such expanded materials can be used even in load-bearing components. They are greatly superior in this respect to the balsa-wood used by the Americans and even to Iporit. The new foamed material may also be used for the insulation of containers against cold.

Vulcanization of Rubber by Diisocyanates: Adhesion of Rubber to Metal.

When now we turned to the rubber field, and tried to replace the vulcanization of Buna with sulphur by the addition of diisocyanates to hydroxyl-containing Buna mixed-polymers, we made the interesting discovery that Buna S itself undergoes a very marked vulcanization with diisocyanates. Whether the low oxygen-content of Buna or reactive double bonds or CH_2- groups were responsible for this was not clear. (In any case, fumaric esters react with isocyanates!) We took this opportunity of making the further useful observation that such "vulcanizates" stuck extraordinarily fast to the metal parts of the vulcanizing press. The practical study of this phenomenon led us to the long-sought adhesive for applying Buna to various supports. The thoroughgoing work of our Rubber Laboratory showed that it was possible with the aid of diisocyanates to produce temperature-stable adhesion of normal Buna-sulphur mixtures to iron, light metals, porcelain, etc., with strengths up to 80 kg./cm^2. (i.e., the Buna itself tears, and not the joint!) Especially suitable for this purpose were the diisocyanates of m-toluylenediamine and of hexamethylene diamine, which are already on the market as Vulkollan T and N. Only with their help is it possible today to dispense with natural rubber, for example in the track-chain cushioning for tanks, and to use throughout high-performance adhesives for rubbery substances. At Leverkusen, at the present moment, there is intensive study of the problem of producing tyres of metal-cord and rubber, which has hitherto been impossible owing to the poor adhesion of rubber to metal. If this important problem is solved, it will be possible thereby to produce tyres of high temperature-stability and, above all, tyres which can be repeatedly re-covered, e.g., for the People's Car.

The good adhesion of Buna-diisocyanate mixtures permits the production of new material for the construction of aircraft, proposed by the Gotha Waggonfabrik, consisting of a thin sheet of vulcanized fibre welded to a light-metal plate. Laminates so prepared tear in the vulcanized fibre or in the metal before they tear at the joint. This material, consisting of metal outside and cellulose inside, permits the construction of a metal airplane by a woodworking method!! By its use, some two-thirds of the time now needed in the construction of a heavy bomber can be saved. At the same time the new material gives a higher bullet-protection than unsupported light-metal sheets. After the war this constructional material will find uses in carriage-construction. Vulcanized fibre with a Buna topcoat may also become an interesting material, which may find use in packing-cases for the tropics.

Buna-isocyanate adhesives also find important applications in the realm of the new "Bunafederkonstruktion"

Diisocyanates as Adhesives in other Fields: Mechanism of Adhesion.

This discovery in connection with the adhesion of Buna naturally prompted experiments on the general applicability of diisocyanates as adhesives. It was found that with their help solutions could be found to many problems hitherto deemed insoluble. For example, it was found possible to attach Oppanol sheets to iron or wood, in the cold, with the aid of a polyester as adhesive; and to weld together vulcanized fibre foils to form thick plates, which otherwise would involve many years of work.

The assembly of paper sheets with diisocyanates, alone or in conjunction with a polyalcohol, gave a laminated material identical in all its properties with vulcanized fibre.

Table of Properties of Paper Laminates

(undecipherable)

It is also possible to attach metal very firmly to metal, a fact of great significance in the line-production of aircraft.

Use of Diisocyanates as Adhesives for Metal to Metal.

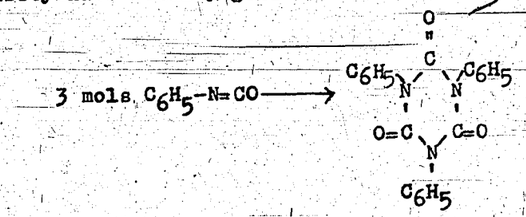
(mainly undecipherable)

We explain the chemical mechanism of this characteristic adhesion behaviour by supposing that diisocyanates:-

1. react even with hydrated oxide layers on the metal surface, so producing a clean surface wherein the urea groups of the adhesive most probably form chemical bonds, or at least subsidiary valency bonds, with the residual valencies of the metal lattice;

2. are soluble in practically every organic substance; and

3. under the action of compounds of metals (and particularly of iron), or of basic substances such as phenolates and pyridine, readily polymerize to high-molecular compounds in which many mutually linked and strongly netted triazine rings arise. Even the mono-functional phenyl isocyanate readily goes over to the corresponding trimer:-



Polyurethanes in the Animalizing of Cuprammonium Rayon.

We were also able with the aid of diisocyanates to solve satisfactorily the problem of developing an animalizing process for cuprammonium rayon. As the result of a thorough investigation we discovered that an animalizing medium, which shall make cellulose fibres as fast as wool to acid dyes, must have the following properties:-

1. very great molecular size, with outstanding absorptive power;
2. the basic nitrogen required for binding acid dyes should not amount to more than 3% of the molecule;
3. complete insolubility in all salts, and in boiling water;
4. complete freedom from colour, and from browning on illumination, and the light-fastness of the acid dye must not be affected;
5. the animalizing medium must be present in the finest possible state of division.

We have to this end worked out a pigment consisting of a basic polyurea, obtained by mixing chlorphenylene diisocyanate + γ:γ'-di(aminopropyl)-methylamine + suitable diamines.

Polyurethanes in Water-Repellent Finishes.

With our Persistol salt A (zirconium oxychloride) + Persistol foundation B (paraffin emulsion), or with their combination single-bath product Persistol N, there is obtained on textile materials a notably good water-repelling finish, fast to washing, but it has not been possible to obtain in this way a stable hydrophobic effect fast to benzine-washing.

Diisocyanates gave us the possibility of producing waxes which are hydrophobic but at the same time insoluble in benzine. A Persistol-foundation now in production, satisfactorily fast to washing and to benzine, and applied together with zirconium oxychloride and some Kaurit, contains a wax prepared from 1 mol 1:6-hexane diisocyanate + 2 mols of a mixture of higher fatty alcohols C_{12}-C_{18}. (German Pat.Appl. J68,271, J68,279 and J70,125).

Manufacture of Intermediates for Polyurethanes.

The starting materials necessary for the production of polyurethanes are diamines, phosgene, glycols and polyhydroxy compounds.

1:4-Butylene glycol and methyl-1:6-hexane diol are the principal glycols used for the production of linear polyurethanes. 1:4-Butanediol, made today by the Reppe (Roppe?) synthesis from formaldehyde and acetylene, is available very cheaply and on a technical scale. Methyl hexanediol is prepared by the hydrogenation of a mixture of esters of the raw methyl adipic acid obtained at Leuna by the oxidation of cyclohexanol-methylhexanol mixtures. Dimethyl 1:3-propanediol is now readily technically available

from isobutyraldehyde + formaldehyde with a combining hydrogenation. The introduction of further methyl groups or of longer side-chains leads to linear polymers with much lower mechanical properties and melting-points.

The starting materials for the diisocyanates are the corresponding aliphatic and aromatic diamines, which are converted to diisocyanates by the action of phosgene:-

$$COCl_2 + NH_2.R.NH_2 + COCl_2 \qquad O{=}C{=}N{-}R{-}N{=}C{=}O + 4HCl$$

1:6-Hexamethylene diamine is made by known methods from the aliphatic straight-chain diamines by hydrogenation of adipodinitrile. We tried, however, to start from 1:4-diaminobutane, which is more cheaply made from acetylene and HCN and is therefore independent of a phenol and benzene basis. We found that acrylonitrile may be very cheaply prepared directly from acetylene and HCN with the aid of a copper-chloride catalyst and that this, in the presence of an alkaline catalyst, smoothly takes on a further mol of HCN.

The succinodinitrile so obtained may be hydrogenated under very mild conditions.

Synthesis of 1:4-butanediisocyanate.

$$CH{\equiv}CH + HCN \longrightarrow CH_2{=}CH.CN \longrightarrow + HCN \longrightarrow$$

$$NC.CH_2.CH_2.CN \xrightarrow{+8H} NH_2.CH_2.CH_2.CH_2.CH_2.NH_2 \longrightarrow$$

$$+ 2\ COCl_2 \longrightarrow O{=}C{=}N{-}CH_2.CH_2.CH_2.CH_2{-}N{=}C{=}O + 4HCl$$

So far as the aromatic diamines are concerned, these are all dye-intermediates well known of old.

For the conversion of aliphatic diamines to diisocyanates there are available a number of variants in the mode of applying the phosgene: for example, it is possible by continuous or discontinuous methods to phosgenate the hydrochlorides of the bases, in suspension in dichlorbenzene, to the purest diisocyanates with a yield of 80% of the theoretical. In practice, we shall work by introducing the free base into the prepared dichlorbenzene-phosgene, probably obtaining thereby at first the hydrochloride of the aminocarbamic acid chloride $HCl.NH_2.(CH_2)_6.NH.CO.Cl$, which can be further phosgenated without difficulty. The purification of the diisocyanates, which are very sensitive compounds and readily polymerize, involved at first great difficulties.

The conversion of aromatic diamines to diisocyanates works very easily. The lower homologues are still distillable while diisocyanates with two benzene rings are generally not distillable, but in some cases the latter separate practically quantitatively from the chlorbenzene after phosgenation.

As I have already mentioned, linear polyurethanes cost about the same as polyamides; in the case of polyurethane leathers, which are made as a fused mass, we believe that the production cost may fall below that of competing polyamides. The cost of aromatic diisocyanates varies from RM1.80 - 2.80, so that for the production of high-performance lacquers and press-compounds, of which it is true only a part consists of diisocyanates, a productive cost-price will result.

Concluding Note.

I have given you a broad outline of the new and wide potential applications of diisocyanates. If today we are seeking approval of manufacturing plant to turn out initially 200 Moto* of aliphatic and 100 Moto of aromatic diisocyanates, this first development should be completely justified by their astonishingly many-sided fields of application; and it will be a real necessity after the war.

* tons per month? (Trans.)

Translator's Notes.

1. The diisocyanates were handled by I.G. under the names:

Desmodur H - Hexamethylene diisocyanate,

Desmodur R - Triphenylmethane-p-triisocyanate,

Desmodur T - Toluene diisocyanate,

Desmosit C - 1-chlorphenylene-2:4-diisocyanate,

Desmosit O - polyhexamethylene diisocyanate,

Desmosit T - 1:2:4-toluylene diisocyanate.

2. Among the polyhydroxy compounds produced by I.G. for use in polyurethane lacquers are the following:-

Desmophen 800S - polyester of trimethylol propane with adipic and phthalic acids,

Desmophen 1100 - polyester of 3 mols adipic acid, 2 mols 1:4-butylene glycol and 2 mols of trimethylol propane,

Desmophen 1200 - polyester of 3 mols adipic acid, 3 mols 1:4-butyleneglycol and 1 mol of trimethylol propane,

Desmophen 100 (cf. Desmophen 1100) - polyester of 3 mols adipic acid, 2 mols of 1:4-butylene glycol and 2 mols of hexanetriol or trimethylol propane.

Desmophen O-O - polyester of oxalic acid and hexanetriol.

(Data selected from CIOS Report No. XXVIII-62.

3. The cross-headings in the translation do not appear in the original.

28th February, 1946.

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ITEM No. 30, 8, 22, 24
FILE No. XXV-26

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Chaffee, E.B., and Atwell, W. V.

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C O N F I D E N T I A L

I.G. FARBENINDUSTRIE
MAINKUR-HOECHST

Reported By:

Captain C.C. CHAFFEE, U.S.
H.V. ATWELL, U.S.

On behalf of
British Ministry of Fuel and Power
and
U.S. Technical Industrial Intelligence Committee

CIOS Target Numbers 30/188, 8/59b, 22/1 G, 24/4
Fuels & Lubricants
Chemical Warfare
Miscellaneous Chemicals
Medical

June 15, 1945

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

C O N F I D E N T I A L

C O N F I D E N T I A L

TABLE OF CONTENTS

Subject	Page No.
Investigation Procedure.....	3
Description of Target.....	3
Crude Oil Demulsifying Agents.....	3
Miscellaneous Chemical Products.....	5
Samples.....	6
Recommendations.....	6
Table I..... Department Personnel.....	7
Table II.... Research Project Assignments.....	8
Manufacturing Instructions	
Humectol CX (Dismulgan V).....	10
Kaurit MKF.....	16
Trypaflavin.....	19
Thiodiphenylamin S.....	24
Dismulgan IV.....	27
Dismulgan VII.....	30
List of Captured Documents.....	33

Personnel of Team

Captain C.C. CHAFFEE, U.S. Army Ordnance
Captain G.A.A. MOIR, British General List
H.V. ATWELL, Petroleum Administration for War, U.S.A.

C O N F I D E N T I A L

1. Investigation Procedure

The I.G. Farbenindustrie plant at Mainkur was visited by CAFT assessors Capt. C.C. Chaffee and Capt. G.A.A. Moer on 5th April, 1945, at which time considerable information was obtained regarding organization, principal products, manufacturing processes and lines of research from the plant manager, Dr. Giesler. On 9th May, 1945, the plant was visited again by Mr. H.V. Atwell, who interrogated Messrs. Zerweck and Ritter specifically about their crude oil demulsifying agents known as "Dismulgans." The information obtained on both visits is consolidated in this report.

2. Description of Target

The Mainkur plant was normally engaged in the manufacture of dyestuffs and intermediates and employed 1,500 to 2,000 workers. During the war, this business declined and at the time of assessment only 1,000 to 1,200 workers were employed, and about 40% of the output was related to dyes. The distribution of technical personnel by Departments is shown by Table I, page 5, and the fields of research of most of the technical men in the scientific laboratory are shown by Table II, page 6. The principal current products are enumerated and discussed below

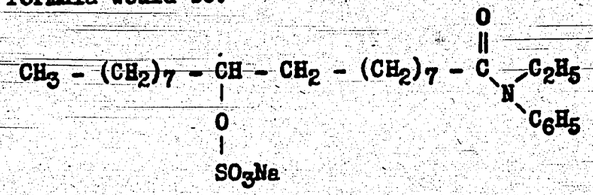
3. Crude Oil Demulsifying Agents

The Mainkur demulsifying agents are known as "Dismulgans" and are of four different kinds according to the crude oil emulsions which they are designed to treat, as follows:

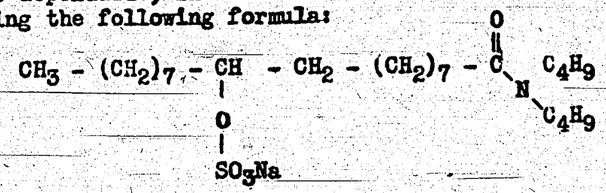
Dismulgan IV for Vienna
Dismulgan V for Elsass
Dismulgan VI for Vienna
Dismulgan VII for Hamburg

It was stated that crude oil emulsions usually require 0.3% of these agents, although sometimes less will suffice. At Erdoel Betrieb Reitbrook (Bergedorf) on 7th May, Mr. L.P. Evans of CAFT #3, 21st Army Group, was told that Dismulgan VII was used to treat Hamburg crude by adding 750 grams of Dismulgan (dissolved in about ten volumes of water) to each cubic meter of crude. The mixture was heated to 75°C. and settled for about 8 hours which resulted in a salt content of not more than 0.02% by weight. The same Dismulgan was not effective on Nienhagen crude. Mainkur production amounted to 30 to 40 tons per month. Written statements given to Captain Chaffee and oral statements to Mr. Atwell regarding the composition of these materials were somewhat contradictory but are summarized below.

Dismulgan IV: This was stated to be made by reacting oleylchloride with dibutylamine and then with sulfuric acid. However, the written instructions for preparation, reproduced in part on Page 25, indicate that N-ethylaniline rather than butylamine is used and that the final product is a sodium salt instead of the acid. Assuming the written record to be more accurate, and correcting typographical errors appearing therein, the formula would be:

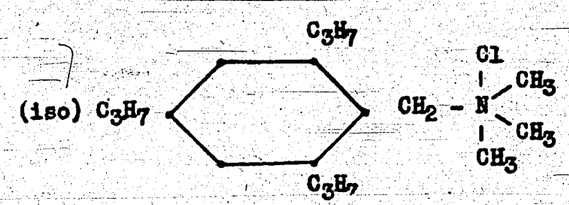


Dismulgan V: This was stated to be the sulfate of oleylethylanilide but the written record, which is presumably more dependable, indicates that it is the same as Humectol CX, having the following formula:



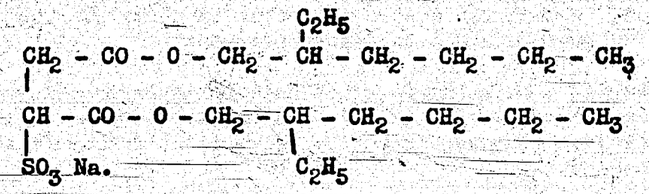
It is made like Dismulgan IV but using Diisobutylamine in place of N-ethyl aniline, as described in Page 8. Apparently in conversation the two compounds were inter-changed, and the final neutralization with caustic soda was not mentioned.

Dismulgan VI: This was stated to be a mixture, in proportions unknown to our informants, of Dismulgan V with the following:



No written instructions on Dismulgan VI were obtained. There is obviously some uncertainty about what was meant by the statement that Dismulgan V was used in this mixture.

Dismulgan VII: This material has the following structure.



Instructions for its preparation are reproduced on Page 28.

4. Miscellaneous Chemical Products

1. Dyes and Colors: Production amounted to 400-500 tons per month of sulfur dyes, vat dyes and azo dyes for dyeing and printing of military and civilian textiles.

2. Textile Aids: These included 60-80 tons per month of Humectol CX for printing vat colors and Katanole tannin substitute, and up to 30 tons per month of Kaurit MKF, a material for crease resistant fabrics from Melamine. Instructions for the preparation of Humectol CX and Kaurit MKF are reproduced in part on Pages 8 and 14 of the Appendix. Humectol CX was stated to be the same as Dismulgan V which is discussed on Page 2.

3. Detergents-Textile Aids from fatty Alcohol base: Of these the principal product was Ocenol-Sulfonate which was just going into production with an estimated ultimate capacity of 40 tons per month.

4. Intermediates for Synthetic Plastics: Melamine was made by a Swiss process to the extent of 40 tons per month, which was to be expanded to 120 tons per month.

5. Synthetic lacquer resins: Maprenal, a condensation product of malamine and formaldehyde was made to the extent of 100 tons per month. A second form of maprenal was made by condensing benzylguanidine with formaldehyde.

6. Other High Polymer Resins: Pollon I was an acronyl plastic used for waterproofing textiles.

7. Miscellaneous Medicinal Products: Trypaflavin, a prophylactic for colds, catarrh, etc., was made in amounts of about 2 tons per month. Directions obtained at Mainkur for preparing this material are reproduced in part on page 17..

8. Animal Medicinal Products: Phenothiazin, pure thiodiphenylamine, is used for destroying worms in animals. The Mainkur production amounted to 3 to 4 tons per month.

9. Insecticides: Thiamine S, crude thiodiphenylamine, is used for spraying swamps to kill malaria-bearing mosquitoes. Production amounted to 40 tons per month. Directions for manufacture are reproduced on Page 22.

Dr. Giesler stated that Mainkur had done no work on vaccines or sera, nor on any chemical warfare agents. He said they had no contact with, or knowledge of, Japanese activities. From conversation and observation at the plant and from inspection of captured documents there seemed to be no reason to doubt the truth of these statements.

5. Samples

Duplicate sets of the samples listed below were obtained:

Kaurit MKF
Ocenol Sulfonate (Laboratory Product)
Trypaflavin, pure
Thiodiphenylamine, pure
Thiodiphenylamine S
Dismulgan IV
Dismulgan V
Dismulgan VI
Dismulgan VII

One set of all samples was delivered to Col. P.R. Tarr of TIIC for examination and tests on behalf of the United States. The second set of Dismulgan samples was delivered to Mr. C.C. Chilvers of the Petroleum Board and the second set of pharmaceuticals, etc. was delivered to Mr. A.E. Childs for whatever tests the British agencies care to make. A third set of Dismulgan samples obtained by Mr. Atwell is being returned through TIIC to PAW for tests in the United States.

6. Recommendations

No further action on this target seems necessary until tests are obtained on captured samples. Then further details should be obtained regarding any product which appears important. In particular, if Dismulgans IV or V are found effective, the discrepancy between oral and written statements regarding their composition must be cleared up.

Capt. C.C. Chaffee
H.V. Atwell

TABLE I

DEPARTMENTAL PERSONNEL

Frankfurt a. Main, den 5.4. 1945

Liste der Chemiker

Werksleitung:

Direktor Dr. Giesler

Abteilung Schwefelfarben:

Dr. Schick
Dr. Frohneberg
Dr. Kohl
Dr. Murawski
Dr. Will

Abteilung Küpenfarben:

Dr. Ochwat
Dr. Pommer
Dr. Schulte

Abteilung Azofarben:

Dr. Ferreau
Dr. Wölfel
Dr. Kern

Analytisches Laboratorium:

Dr. Jassoy
Dr. Albrecht

Färberei:

Dr. Pässler
Dipl. Ing. Hübner

Physikalische Abteilung:

Dr. Rein — Dr. Farr
Dr. Heinrich — Dr. Gollnow
Dr. Menzer) Im
Dr. Ziegler) Heeresdienst

Wissenschaftliches Laboratorium:

Dr. Zerweck — Dr. Korten
Dr. Ritter — Dr. Müller
Dr. Riedmair — Dr. Fleischhauer
Dr. Schütz — Dr. Frister
Dr. Brunner — Dr. Sondag
Dr. Keller — Dr. Löwe im Heeresdienst
Dr. Salkowski
Dr. Schultis
Dr. Schwemberger
Dr. Kunze
Dr. Schubert
Dr. Honold
Dr. Hechtenberg
Dr. Gofferje
Dr. Tröskon
Dr. Kleinhans

TABLE II

RESEARCH PROJECT ASSIGNMENTS

1.) Dr. Brunner — Synthetische selbsttrockene Lackharze der aromatischen Reihe.

2.) Dr. Fleischhauer — Derivate der Cellulose, insbesondere der Amino-Cellulose

3.) Dr. Gofferié — a) Formaldehyd-Kondensationsprodukte von Kalkstockstoff, Cyanamid, Dicyandiamid etc.als Animalisierungsmittel und zur Verbesserung der Echtheiten substantiver Färbungen.
b) Prüfung von Stabilisierungsmittel.

4.) Dr. Hechtenberg — Schwefelfarbstoffe.

5.) Dr. Henold — a) Hochmolekulare Sulfonamide (entsprechend Superpolyamide)
b) Egalisierungsmittel für Küpen-und Schwefelfarbstoffe.

6.) Dr. Keller — Derivate des Melamins und ähnlicher Substanzen für Lackharze und Kunststoffe.

7.) Dr. Korten — a) Herstellung von Furfurol aus Haberschalen.
b) Herstellung von ungesättigten Verbindungen und Herstellung von Polysulfonen daraus mit SO^2.

8.) Dr. Kunze — Mischpolymerisate aus verschiedenen polumerisierbaren Verbindungen.

9.) Dr.Müller.Rud. — Herstellung von Stabilisierungsmittel für Poly-vinyl-chlorid und anderen chlorhaltigen Kunststoffen.

10.) Dr. Riedmair — a) Weichmacher für Kunststoffe (Sulfosäure-ester)
b) Derivate des Thio-diphenylamins als Schädlingsbekämpgungs und Wurmmittel.
c) Sulfonamide ("Prontosil").

11.) Dr. Ritte- — a) Ketone und Alkohole der aromatischen und hydroaromatischen Reihe als Weichmacher für Kunststoffe.
b) Tetrahydropyran aus Furfurol und Derivate desselben.

12.) Dr. Salkowski — Derivate des Melamins und ähnlicher Verbindungen für Lackharze und zur Verwendung in der Textillindustrie.

13.) Dr. Schubert — Kondensations-Produkte von heterocyclischen Aminen mit Formaldehyd.

14.) Dr. Schütz — Papierleimungs-und Hydrophorbierungs-Mittel (Uniformen).

15.) Dr. Schultis — Derivate des Kalkstockstoffes, die sich als Lösungsmittel eignen, insbesonders die Dialkyl-cyanamide.

16.) Dr. Schwemberger — Papierveredlungs-Mittel vor allen auf Grundlage Melamin.

17.) Dr. Trösken — Kapillaraktive Substanzen zur Verwendung in der Textil und Erdölindustrie.

Dr.Fr./Schi. Mainaur, den 4. Februar 1938.

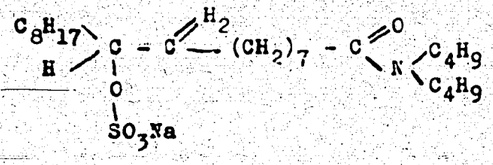
Verfahren zur Herstellung von

Humectol CX

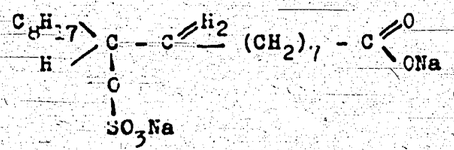
dient als Netz- und Egalisiermittel.

Chemischer Vorgang:

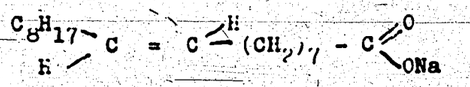
Ölsäure wird in Ölsäurechlorid übergeführt, dieses mit Diisobutylamin zu Ölsäure-di-isobutylamid kondensiert und mit Monohydrat in den Sulfoester übergeführt. Die vor der Sulfierung zugesetzte freie Ölsäure wird ebenfalls in den Sulfoester übergeführt, sodass das Humectol CX in der Hauptsache aus einem Gemisch des Sulfoesters des Ölsäure-di-isobutylamids und des freiem Sulfoesters der Ölsäure neben etwas Ölsaurem Natrium besteht.



Sulfoester des Ölsäure-di-isobutylamids Mol.518.



Sulfoester des ölsauren Na. Mol. 424.



Ölsaures Natrium Mol. 304.

37/32-z.

Ansatz:

500	kg	Ölsäure Mol. 282,
100	"	Phosphortrichlorid Mol. 137 (Theorie 81 kg),
344	"	Natronlauge 33° Bé = 93 kg 100%,
208	"	Diisobutylamin Mol. 129,
500	l	Schwefelsaure 50% = 485 kg 95%,
730	kg	Trichloraethylen,
180	"	Ölsäure,
748	"	Monohydrat (Theorie 236),
176	"	Trichloraethylen,
1600	"	Eis,
432	"	Natronlauge 33° Bé = 128 kg 100%,
50	"	Ölsäure.

A. Ölsäurechlorid:

In den sauer ausgesteinten Doppelkessel von 1000 l Inhalt (Pos. 1) laufen aus dem Ölsäuremassgefäss (Pos. 5a) 500 kg (= 536 l) Ölsäure heller Qualität der Hammonia Offenbach und werden auf 50° angewärmt. Unter die Oberfläche kommen dann aus

dem Massgefäss (Pos. 1a) 100 kg Phosphortrichlorid = 63,5 l, wobei die Temperatur auf 45° zurückgeht. Das Rührwerk lässt man dann 3 Umdrehungen machen, stellt wieder ab, erhitzt auf 55° und hält 3 Stunden bei dieser Temperatur. Nach dieser Zeit ist die Reaktion beendet. Die entstandene phosphorige Säure setzt sich als dickes Öl am Boden ab. Der Kesselinhalt wird dann in den 1000 l homogen verbleiten Scheider (Pos. 2) gedrückt, wo in 12 Stunden eine gute Trennung sich vollzieht. Im Winter wird schon nach 5-6 Stunden ein Teil der phosphorigen Säure abgezogen, da dieselbe sonst erstarrt. Die am Boden sitzende phosphorige Säure wird in den Kanal gelassen (ca. 45 kg). Im Scheider befindet sich dann das Ölsäurechlorid.

Ausbeute: 530 - 535 kg, Spez.Gew. 0,92 - 0,93.
Prüfung des Ölsäurechlorids siehe Probe Nr. 1).

B. Ölsäure-di-isobutylamid:

In dem eisernen Rührkessel von 2000 l Inhalt (Pos. 3) mit Mantel, eingerichtet, für Kältelauge und Dampfanschluss werden 265 l = 344 kg Natronlauge 33° Bé und 208 kg Diisobutylamin Mol. 129 vorgelegt und auf + 10° abgekühlt. Unter die Oberfläche läuft dann während 12 Stunden aus dem Ölsäurechloridscheider (Pos. 2) bei einer Temperatur von bis zu + 20° unter Kühlung mit Sole und fortwährendem Rühren das Ölsäurechlorid zu. Nachdem alles Ölsäurechlorid eingelaufen ist, wird die Suspension auf 50° angeheizt und eine Stunde gehalten. Nach dieser Zeit ist die Amidierung beendet und muss phenolphtalein-alkalisch reagieren. Die Suspension wird nun in den 3000 l verbleiten Scheider (Pos. 4) gedrückt und der Kondensationskessel (Pos. 3) mit 200 l Wasser nachgespült. Unter Rühren laufen dann aus dem Massgefäss (Pos. 4a) 125 l 50%ige Schwefelsäure in den Scheider (Pos. 4). Nachdem alle Schwefelsäure zugelaufen ist, wird das Rührwerk abgestellt, die Temperatur betrugt 45 - 50°. Das Diisobutylamid der Ölsäure hat ein spez. Gewicht von 0,87 - 0,88 und geht nach oben. Die sauren Salzbrühen werden nach 3 - 4 Stunden abgezogen. Sodann wird

das Amid nochmals mit 250 l 50%iger Schwefelsäure gewaschen, nach 3-stündigem Stehen die Salzbrühe wieder abgezogen, und die Reinigung nochmals mit 125 l 50%iger Schwefelsaure wiederholt. Im Scheider bleibt das Amid als klares Öl zurück.

Ausbeute: 835 - 840 l = 730 - 735 kg Ölsäurediisobutylamid.
Bestimmung des Gehaltes an freier Ölsäure siehe Probe Nr. 2.
Der Gehalt an freier Ölsäure beträgt 7-9 %.

C. Sulfierung:

Die Sulfierung erfolgt in dem 3400 l homogen verbleiten Doppelkessel (Pos. 5), der mit guter Kältelauge gekühlt werden muss.

Bei dem Ansatz ist darauf zu achten, dass auf je 130 kg Amid-Ölsäure 70 kg freie Ölsäure zugesetzt werden.

Beispiel: Nach Titration enthielten 735 kg Ölsäure-di-isobutylamid 8% freie Ölsäure d.h. aus 500 kg Ölsäure sind noch 58 kg freie Ölsäure vorhanden, also 500 - 58 = 442 kg Amid-Ölsäure. Um das Verhältnis Amid-Ölsäure zur freien Ölsäure zu erreichen, braucht man bei 442 kg Amid-Ölsäure 238 kg freie Ölsäure. Da im Amid aber bereits 58 kg freie Ölsäure enthalten sind, müssen noch 180 kg Ölsäure nachgesetzt werden. Es sind dann in der Sulfierung insgesamt 680 kg Ölsäure vorhanden.

Aus dem Scheider (Pos. 4) wird das Amid in den Sulfierkessel (Pos. 5) abgelassen, aus dem Massgefäss (Pos. 5a) 180 kg Ölsäure und aus dem Massgefäss (Pos. 4b) 730 kg Trichloraethylen zugefügt. Man kühlt nun auf - 3 bis - 5° ab und lässt unter Rühren und weiterem Kühlen noch aus dem Monohydratmassgefäss (Pos. 5b) während 12 - 14 Stunden 730 kg Monohydrat zufliessen, wobei die Temperatur nicht über 0° steigen darf. Temperatur der Sole - 15°.

Die Menge Monohydrat betrügt 10% mehr als die eingesetzte Gesamtölsäure bei 680 kg, also 748 kg Monohydrat. Die angegebene Sulfierzeit ist nur einzuhalten bei einer Kältesole von - 14 bis - 15°.

Nach beendigter Sulfierung wird noch ½ Stunde nachgerührt, zum besseren Drucke noch 1⅞ kg Trichloraethylen aus dem Massgefäss (Pos. 46) zugegeben, und dann die Sulfierung in den ausgesteinten Scheider (Pos. 6) von 7000 l, in dem 2800 l Wasser vorgelegt und auf + 5° abgekühlt waren, unter Rühren gedrückt. Durch Zugabe von Eis (ca. 1600 kg) sorgt man dafür, dass die Temperatur nicht über + 5° geht. Man lässt nun 4-6 Stunden stehen, wobei sich die Sulfierung gut unten absetzt.

D. Destillation:

Über eine ausgesteinte Vorlage (Pos. 7) kommt dann die ausgewaschene Sulfierung in den ausgesteinten Destillierkessel von 4000 l (Pos. 8). In dem Destillierkessel sind 300 l Wasser und 250 l = 325 kg Natronlauge 33° Bé (aus Pos.8a) vorgelegt; unter Rühren wird die Sulfierung zugedrückt, wobei die Reaktion deutlich phenolphtaleinalkalisch bleiben muss. Die Mischtemperatur beträgt 30-35°. Mit kaltem Wasser wird auf ein Volumen von 2600 l eingestellt. Zum Heizen dient eine Kupferschlange, die vollständig in das zu destillierende Gut eintaucht, um eine Überhitzung des wegzudestillierenden Trichloraethylens zu vermeiden. Gegen Ende des Zudrückens wird d. Phenolphtaleinreaktion schwächer, sodass nach und nach noch ca. 40 l = ca. 52 kg Natronlauge 33° Bé mit zugedrückt werden müssen. Unter stetem Rühren wird nun langsam hoch geheizt und dabei auf rotem Lackmuspapier die alkalische Reaktion dauernd verfolgt. Bis zum Ende der Destillation werden nun nach und nach 5 kg Natronlauge 33° Bé zugesetzt. Bei 78° fängt das Trichloraethylen an überzugehen, welches durch die beiden Schlangenkühler (Pos. 9 & 9a) in die Vorlage (Pos. 10) läuft. Bei 102° ist alles Trichloraethylen übergegangen. Es werden 816 kg Trichloraethylen wiedergewonnen, sodass mit einem Verlust von 10% zu rechnen ist. Nach 10 - 12 Stunden ist die Destillation beendet.

Um die Laugeaussalzbarkeit des Typs zu erreichen, wird nach Beendigung der Destillation dem Produkt noch 50 kg Ölsäure zugesetzt. Dann kommen noch ca. 50 kg Natronlauge 33° Bé hinzu. Siehe Probe Nr. 3. Nun wird auf 20° abgekühlt und mit kaltem Wasser auf ein Volumen von 2500 l eingestellt. Dann wird im Laboratorium der Netzwert bestimmt (Probe Nr. 4) und auf 2678 kg eingestellt, Spez.-Gew. 1,02 - 1,03. Ferner wird auf Verlangen eine Fettbestimmung ausgeführt, Probe Nr. 5, stets aber die Laugebeständigkeit geprüft. Probe Nr. 6.

Ausbeute: 2678 kg.

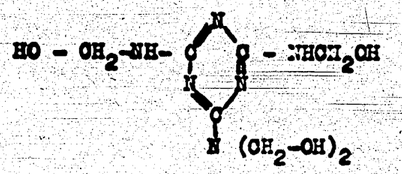
Für Exportzwecke wird auf Verlangen Humectol CX conc. hergestellt. Die Arbeitsweise ist dieselbe wie bei Humectol CX, nur wird vor der Destillation kein Wasser vorgelegt und statt Natronlauge 33° Bé, eine entsprechende Menge von 50° Bé, im Destillationskessel verwandt. Man erhält so ein Humectol CX conc. mit einer Stärke von 60-70:100.

Dr.Po./B. Mainkur, den 11. Januar 1944

Vorschrift zur Herstellung von K a u r i t MKF.

A. Chemischer Teil.

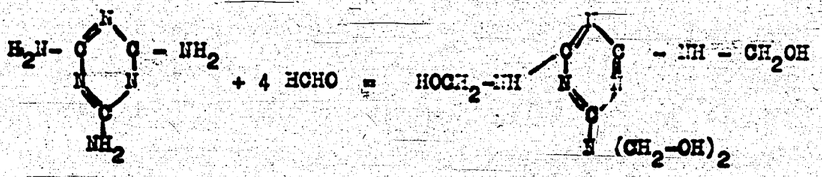
I. Konstitution:



$C_7H_{14}O_4N_6$

Mol.246,2

Chemischer Vorgang gemäss der Gleichung:



Melamin Mol.126 | Formaldehyd Mol.30 | Tetramethylolmelamin Mol.246,2

II. Einsatz:

283,5 kg Melamin (=Azamin rein), trocken in Form von 90-95%igem Filterpresskuchen = 2,25 Mol.
727,7 kg Formaldehyd 37 Gewichtsprozentig = 269 kg = 9 Mol.
55,9 kg Triaethanolamin
ca 1,2 kg Soda

III. Ausbeute:

1000 kg Kondensationsprodukt enthaltend ca 60% Trockensubstanz (ca 28% Azamin)

IV. Arbeitsweise:

In homogen verbleiten 1250 l Doppelkessel (1) werden 545,5 kg Formaldehyd (37 Gewichtsprozentig) vorgelegt, mit Sodalösung neutralisiert (Probe 1) und dazu 283,5 kg Azamin rein, trocken, in Form von Presskuchen, sowie 55,9 kg Triäthanolamin eingetragen. Unter Rühren wird mit Mantelddampf auf 70 - 75° Innentemperatur erwärmt, bis eine klare Lösung eingetreten ist. Man hält noch 15 Minuten bei 75°, lässt dann weitere 182,2 kg Formaldehyd

(37 Gewichtsprozentig), die vorher mit Sodalösung neutralisiert wurden (Probe 1) zulaufen, heizt wiederum auf 75° an und drückt schliesslich schnell den Kesselinhalt in eine gemauerte Kühlwanne (2) aus. Hier erstarrt die Lösung zu einer weissen Masse (E.P.47°), die nach längerem Stehen (im Winter 24 Std., im Sommer 48 - 54 Std.) so fest wird, dass sie mit Spachteln in faustgrossen Stücken ausgestochen werden kann. Die Stücke werden in einer Raspelmaschine (3) zu einer krümeligen Masse zerkleinert (Probe 2). Das Kaurit kommt in dieser Form, in Sperrholzfässern verpackt, zum Versand.

V. Proben:

a) Materialproben:

Azamin rein

126 g Azamin rein 100% sollen sich in 243 g Formaldehyd (37 Gewichtsprozentig mit Soda neutralisiert) bei 70 - 75° vollständig klar und farblos lösen. Der Gehalt an Azamin soll in der Paste mindestens 93 % betragen.

b) Betriebsproben:

1.) Formaldehyd wird mit ca 20%iger Sodalösung auf einen pH-Wert von 7,6 - 8 eingestellt.

2.) 170 g Kaurit MKF werden in 800 ccm heisses Kondenswasser eingestreut und auf 90° erwärmt. Nach 5 - 10 Minuten muss eine klare farblose Lösung eingetreten sein. Man kühlt auf 40° ab.

I. 200 ccm obiger Lösung werden mit 50 ccm Kondenswasser versetzt. Der pH-Wert soll ca 8,5 erreichen.

II. 200 ccm der Lösung werden mit 1,25 ccm Glykolsäure und 50 ccm Kondenswasser (= 5 ccm Säure je Liter) versetzt. Der pH-Wert soll ca 6,8 betragen, darf jedoch 6,5 nicht unterschreiten, da sonst die Haltbarkeit beeinträchtigt wird. Zur Feststellung des pH-Wertes wird Indikatorenpapier " Bayer " Nr. 8 benutzt.
Die Lösungen I und II sollen nach 20 - 24 stündigem Stehen noch klar sein.

B. Technischer Teil.

1.) Nr. 16237 Schmiedeeiserner, innen homogen verbleiter Doppelkessel mit Rührwerk n = 14 und n = 63, Inhalt 1250 l, Druckleitung aus Eisen nach 2)

2.) gemauerte Wanne, innen mit Kacheln ausgelegt, 3 x 2 x 0,3 m mit aufgesetzten Seitenwänden,und Verdachung aus Holz. Entlüftung durch Ventilator ins Freie. Zulauf von 1) mit eingebautem Siebtopf aus Eisen. Entleerung von Hand in verbleite Filterpressenwagen.

3.) Alexanderwerk-Haushaltsmaschine zum Raspeln. Beschickung von Hand, Entleerung in darunter stehende Fässer.

Mainkur, den 18.Juli 1938

Verfahren zur Herstellung von

T r y p a f l a v i n rein.

A.) Chemischer Teil.

I. Konstitution und Zusammensetzung:

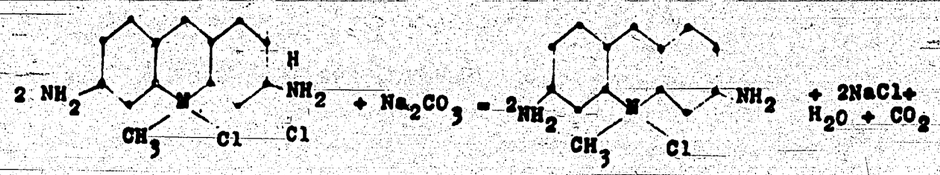
Trypaflavin rein ist eine Mischung von 3.6-Diamino-10-Methyl-Akridiniumchlorid und 3.6-Diamino-Akridinchlorhydrat; es enthält ausserdem 7 - 8 % Wasser und 2,5 - 3,5 % Kochsalz.

Normale Zusammensetzung:

63 - 67	%	Diaminomethylacridiniumchlorid	Mol.259,5
21,5-27,5	%	Diaminoacridinchlorhydrat	Mol.245,5
7 - 8	%	Wasser	
2,5 - 3,5	%	Steinsalz.	

II. Chemischer Vorgang:

Beim Neutralisieren der Lösung der Monochlorhydrate von 3.6-Diamino-10-Methylacridiniumchlorid und 3.6-Diamino-akridin (Trypaflavin sauer) wird mit Soda das 3.6-Diamino-10-Methylacridiniumchlorid frei gemacht, während das salzsaure 3.6-Diaminoakridin unverändert bleibt. Bei dem vorgeschriebenen Verdünnungsgrad bleibt das Gemisch in Lösung; es wird unter Zusatz von Carboraffin filtriert und im Filtrat das Trypaflavin mit Salzwasser abgeschieden.



III. Einsatz:

40-41 kg Trypaflavin sauer (Paste) = 1/3 Partie Trypaflavin sauer
7,5 kg Carboraffin, eisenarm
etwa 12 kg Soda calc.
etwa 380 l Salzwasser 24° Bé
etwa 2 l Salzsäure 1,11.

IV. Ausbeute:

Etwa 19 kg Trypaflavin.

V. Arbeitsweise:

Der Kessel 1), der ca.500 Ltr.Carboraffin-Waschwasser der vorangegangenen Partie enthält, wird bei einer Temperatur von 35-40° mit dem Trypaflavin sauer (Paste) beschickt und etwa 1 Stunde bei ca.40° gerührt. Alsdann gibt man unter weiterem Rühren 7,5 kg Carboraffin in den Kessel und soviel Soda (Vorsicht, starkes Schäumen), dass die Lösung ganz schwach (fast neutral) alkalisch reagiert. Rotes Lackmus - papier soll nach einigen Minuten nur einen sehr geringen Ueberschuss an Soda anzeigen. Nunmehr bringt man das Volumen mit Trinkwasser auf 580 Ltr.und rührt 3 Stunden bei 35-40°.

Nach Verlauf dieser Zeit drückt man den ganzen Kesselinhalt auf den Steinsauger 2) und saugt solange in den Kessel 1) zu rück, bis das Filtrat völlig klar abläuft (Schauglas am Stei filter). Dann wird umgestellt (vorheriges Evakuieren von Kessel 5!), sodass das Filtrat nunmehr in den Kessel 5) läuft Das in den Kessel 1) zu Beginn zurückgesaugte trübe Filtrat wird wieder auf den Sauger gedrückt. Der Carboraffinrückstand auf dem Sauger wird zunächst mit etwa 50 l Trinkwasser von 50° gewaschen; das Filtrat läuft in Kessel 5). Inzwischen hat man in Kessel 1) 500 l Trinkwasser auf 50° erwärmt und wäscht mit diesem den Carboraffinrückstand zum zweiten Mal unter Zusatz von soviel Salzsäure, dass das Waschwasser ganz schwach sauer reagiert (methylorange). Dieses Filtrat, das zum Ansatz der nächsten Partie dient, saugt man in Kessel 1) zurück. Die Carboraffinrückstände enthalten kein Trypaflavin mehr und werden fortgeworfen.

Damit der P.H.-Wert des Trypaflavins 6-7 beträgt, ist es nötig die schwach alkalische Trypaflavinlösung im Kessel 5) (etwa 600 l) vor dem Fällen mit Kochsalz vorsichtig mit soviel kons.Salzsäure zu versetzen (durchschnittl.150-200 ccm) bis eine Probe im Reagenzglas nach Zusatz von 3 Tropfen einer 0,4%igen alkoholischen Bromthylmolblaulösung sich nicht mehr verändert d.h.keine alkalische Reaktion mehr zeigt.

Inzwischen hat man im Kessel 7) Salzwasser von 24° Bé bereitet. Davon filtriert man etwa 380 l durch die Filterpresse 3) in den Kessel 6) und wärmt auf 30° an. Das filtrierte Salzwasser drückt man nun unter beständigem Rühren in 2-3 Minuten in den Kessel 5)

zu der neutralisierten Trypaflavinlösung. (Der Tonhahn der Leitung nach Kessel 5) ist ganz offen). Damit das fertige Produkt die richtige Kristallform erhält, die dem normalen Kochsalzgehalt von 2,5 - 3,5 % gewährleistet, ist es nötig, sowohl das Salzwasser, als auch die Trypaflavinlösung genau auf 32° Temperatur einzustellen. Ebenso muss auch das Wasserbad von Kessel 5) 32° warm sein, um ein zu schnelles Abkühlen der Fällung zu verhindern. Nur bei dieser Temperatur enthält das getrocknete Trypaflavin etwa 2,3% Kochsalz. Nach der Fällung rührt man noch solange (3-4 Minuten) bis das zuerst ölig ausgefallene Trypaflavin erstarrt ist.

Das Wasserbad von Kessel 5) darf während der ersten Stunden nicht gekühlt werden. Erst nachdem die Temperatur am nächsten Tag auf 23 - 24° gesunken ist, ist es angebracht, das Bad unter gleichzeitigem Rühren des Fällkessels durch Zugabe von Wasser auf eine niedrigere Temperatur zu bringen. Bei etwa 20° wird dann zentrifugiert.

Man schleudert bis keine Flüssigkeit mehr abläuft, was je nach der Form des Produktes 3-5 Stunden dauert.

Trocknen:

Je 3 Partien verteilt man partieweise, gleichmässig und gut zerkleinert auf je 9 Haveghorden und trocknet während der ersten 15-20 Stunden bei 35-40°, während der nächsten 12 Stunden bei 55-60°. Je nach der Form des Produktes geht die Trocknung langsam oder schnell von statten. Unmittelbar nach

dem Trocknen sollte der Wassergehalt 6,3% betragen (Probe 1). Da derselbe erfahrungsgemäss beim Mahlen und Umfüllen durchschnittlich um 1,3% zunimmt, enthält das versandfertige Produkt nun die gewünschten 7-8% Wasser.

Ausbeute je Partie: Etwa 19 kg trocken (Probe 2).

Mahlen:

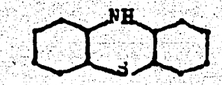
Je 3 Partien (ca.57 kg), die man nach den Analysenwerten zusammenstellt, werden etwa 24 Stunden in der Mahltrommel 9) gemahlen und dann baldmöglichst in 120 l Patentfässern versandt.

Mainkur, den 4. Oktober 1914.

Verfahren zur Herstellung von Thiodiphenylamin S.

A.) Chemischer Teil.

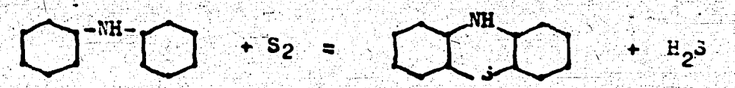
I. Konstitution:



Mol. 199

II. Chemischer Vorgang:

Diphenylamin und Schwefel werden unter Zusatz des Katalysators $AlCl_3$ bei ca. 170-180° zur Reaktion gebracht. Der freiwerdende H_2S wird abgeführt und auf Na-Sulfhydrat verarbeitet.



III. Einsatz:

1700 kg Diphenylamin
646 " Schwefel
20-25 " Aluminiumchlorid

IV. Ausbeute:

2000 kg Diphenylamin S

V. Arbeitsweise:

1700 kg Diphenylamin werden aus dem 20 cbm Lagerkessel über den Liegekessel in Kessel 1 eingedrückt. Dazu kommen 20 kg Aluminiumchlorid. Es wird auf 160-165° angeheizt. In Kessel 2 werden 646 kg Schwefel bei ca. 130° geschmolzen. Im Laufe von 2-3 Stunden wird nun der geschmolzene Schwefel in dünnem Strahl langsam in das Diphenylamin im Kessel 1 bei von 165-180° ansteigenden Temperaturen eingedrückt. Der dabei entstehende H_2S wird der Absorptionsanlage zur Darstellung von Na-Sulfhydrat zugeführt. Nach beendetem Eindrücken wird die Schmelze noch 2 Stunden auf 180-200° geheizt (Probe 1) und dann in 2 Wannen (3) ausgedrückt. Nach wenigen Stunden ist das Thiodiphenylamin in den Wannen erstarrt. Es wird von Hand herausgestossen und eingefasst. Das Produkt wird in der Zentralmühle gemahlen (Probe 2).

Ausbeute: 2000 kg Thiodiphenylamin S.

-2-

Laboratoriumsproben:

Probe 1: Eine Probe der fertigen Schmelze aus dem Schmelzkessel soll einen Schm.Pkt. von mindestens 173-175° haben. Liegt er tiefer, so hat das Aluminiumchlorid nicht ausgereicht. Die Schmelze muss dann unter 150° abkühlen und unter Zusatz von neuem $AlCl_3$ nochmals geheizt werden.

Probe 2: Das Endprodukt muss einen Schm.Pkt. von 173-175° haben.

Erfahrungen:

Beim Zusammenschmelzen von Diphenylamin und Schwefel in Gegenwart von $AlCl_3$ beginnt die Reaktion bei ca. 155° und steigert sich in wenigen Minuten zu grosser Heftigkeit, so dass der dabei freiwerdende H_2S – ca. 300 kg pro Partie – bewirkt ein starkes Aufschäumen der Schmelze, wobei Vorlagen und Rohrleitungen, die den H_2S abführen sollen, von dem übergeschäumten erstarrenden Produkt verstopft werden. Diese Schwierigkeiten sind zu umgehen, indem man die Umsetzung so vornimmt, dass man die eine Komponente vorgelegt und die andere bei Reaktionstemperatur langsam zulaufen lässt. Ausser der in diesem Verfahren beschriebenen Arbeitsweise kann man auch das Aluminiumchlorid vorlegen und das zusammengeschmolzene Gemisch von Diphenylamin und Schwefel bei 165-180° zulaufen lassen. Notwendig ist, stets dafür zu sorgen, dass der Einlauf durch das Schauglas gut überwacht wird und mindestens 2 Stunden dauert. Die Abgasleitung für den H_2S und die Vorlagen in dieser Leitung sind mit grösseren, leicht abnehmbaren Flanschen versehen, die die Reinigung der Gefässe wie auch der Leitungsstücke von mitgerissenem $AlCl_3$, Diphenylamin usw. leicht ermöglichen.

B. Technischer Teil.

Apparatur:

1.) Ein 2000 l gusseiserner Schmelzkessel mit hochgewölbtem Deckel, Rührer, Ölbad, Gasfeuerung. Der 400 mm weite Stutzen für die Abgasleitung des H_2S ist oberhalb des Kessels trichterförmig ausgeweitet. Die 200 mm Leitung führt durch 2 hintereinander geschaltete Vorlagen von je ca. 800 l Inhalt, von denen eine pro Partie mit einigen l verdünnter Natronlauge zum Zurückhalten von mitgerissenem sauren $AlCl_3$ gefüllt ist. Von der letzten Vorlage führt eine 150 mm Ø gusseiserne Leitung zur Absorptionsanlage für H_2S.

2.) Ein gusseiserner Doppelkessel mit Rührer, Dampfbad, isolierte Doppelrohrdruckleitung nach Kessel 1). Jn die Doppelrohrleitung ist eine Laterne zur Beobachtung des Schwefeleinlaufs und darüber ein Siebtopf mit düsenartigem Ablauf eingebaut.

3.) 2 schmiedeeiserne flache Wannen als Kühlwannen für die Schmelze.

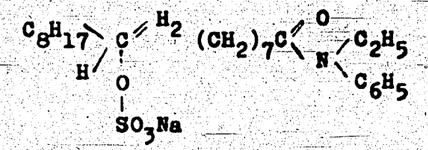
C. Produktionsmöglichkeit.

Täglich	2 Partien =	4000 kg
Monatlich	=	100 t.

Mainkur, den 24. April 1941.

Verfahren zur Herstellung von
Dismulgan IV

im wesentlichen Sulfoester des Olsäureaethylanilids Mol. 505.



Verfahren:

1.) Olsäurechlorid:

Jn den sauer ausgesteinten Doppelkessel von 1000 l Jnhalt (Pos. 1) laufen aus dem Olsäuremassgefäss (Pos. 5a) 500 kg = 536 l Olsäure und werden auf 50° angewärmt. Unter die Oberfläche laufen dann aus dem Massgefäss (Pos. 1a) 100 kg Phosphortrichlorid = 63,5 l, wobei die Temperatur auf 45° zurückgeht. Das Rührwerk lässt man dann 3 Umdrehungen machen, stellt wieder ab, erhitzt auf 55° und hält 3 Stunden bei dieser Temperatur. Nach dieser Zeit ist die Reaktion beendet. Die entstandene phosphorige Säure setzt sich als dickes Öl am Boden ab. Der Kesselinhalt wird dann in den 1000 l homogen verbleiten Scheider (Pos. 2) gedrückt, wo in 12 Stunden eine gute Trennung sich vollzieht. Jm Winter wird schon nach 5-6 Stunden ein Teil der phosphorigen Säure abgezogen, da dieselbe sonst erstarrt. Die am Boden sitzende phosphorige Säure wird in den Kanal gelassen (ca. 65 kg). Jm Scheider befindet sich das Olsäurechlorid.

Ausbeute: 530-535 kg spez.Gew. 0,92-0,93

Prüfung des Chlorids siehe Probe Nr. 1 bei Humextol CX.

2.) Olsäureaethylanilid:

Jn dem gusseisernen Doppelkessel von 2000 l (Pos. 3) werden 300 kg Lauge, 200 l Wasser und 214 kg Monoaethylanilin vorgelegt. Bei 20° läuft das Olsäurechlorid aus dem Scheider (Pos.2) unter Rühren während 8-10 Stunden zu, wobei dauernd mit Kältelauge gekühlt wird. Nachdem alles eingelaufen ist, wird auf 50° angeheizt, eine Stunde dabei gehalten, dann noch 1 Stunde bei 60° gehalten und schliesslich auf 70-80° angewärmt. Der Kesselinhalt wird dann in den 3000 l Spitzkessel (Pos. 4) gedrückt, in dem das Anilid mit 50%iger Schwefelsäure gewaschen wird. Aus dem

2000 l Mischkessel (Pos. 11) werden über das 500 l verbleite Massgefäss (Pos. 4a) = 125 l 50%ige Schwefelsäure in den 3000 l Spitzkessel (Pos. 4) gedrückt und bei 70-80° kurz gerührt, wobei dann 3 Stunden bei dieser Temperatur stehen gelassen, die untere saure Schicht in den Kanal abgezogen. Unter Rühren gibt man dann nochmal 250 l 50%ige Schwefelsäure zu, rührt kurz und lässt dann 6 Stunden stehen. Nachdem nun die untere Brühe wieder abgelassen ist, läuft das klare Aethylanilid in den Sulfierkessel (Pos.5), in dem 561 kg Trichloraethylen aus Pos. 4b zugegeben werden. Prüfung des Anilids s. Probe Nr. 2 bei Humextol CX.

Sulfierung:

Der Inhalt des Sulfierkessels wird unter 0° abgekühlt und während 10-12 Stunden bei 0° 250 kg Monohydrat aus dem Massgefäss (Pos. 5b) zugegeben. Die Temperatur darf nicht über 0° steigen, Sooletemperatur mindestens -15°.

Nach Beendigung der Sulfierung wird dieselbe sofort langsam in den homogen verbleiten Spitzkessel von 7000 l gedrückt (Pos.6), in dem 3000 l Wasser und ca. 300-400 kg Eis vorgelegt sind. Die Temperatur des vorgelegten Wassers muss 0° betragen, während des Eindrückens der Sulfierung wird noch mit Kältelauge gekühlt, dass die Temperatur nicht über + 5° steigt. Ist die ganze Sulfierung zugelaufen, wird das Rührwerk abgestellt und bis zum anderen Morgen stehen gelassen. Die unten sitzende Sulfoester des Ölsäureaethylanilids wird dann über die Vorlage Pos. 7 in den Destillationskessel (Pos. 8) gedrückt.

Neutralisation und Wiedergewinnung des Trichloraethylens:

In dem ausgesteinten Destillationskessel (Pos. 8) werden 200 l Natronlauge 33° Bé und soviel Wasser vorgelegt, dass das Rührwerk gerade noch greift. Während des Eindrückens der Sulfierung muss die Lösung schwach phenolphtaleinalkalisch reagieren. Dann wird langsam angeheizt und das Trichloraethylen abdestilliert. Bei 78° Innentemperatur beginnt das Gemisch von Wasser und Trichloraethylen überzugehen, bei 85° geht die Hauptmenge Trichloräthylen über und bei 98-100° ist die Destillation beendet. Während der Destillation werden Proben genommen, dienoch schwach alkalisch auf Lackmuspapier reagieren müssen. Destillationsdauer 10-12 Stunden. Bei eintretender Rötung des Lackmuspapiers ist ein Zusatz von Lauge nötig, jedoch ist zu grosser Überschuss von Lauge zu vermeiden, da das fertige Produkt keinesfalls laugealkalisch

(phenolphtaleinrot) sein soll. Zwischen 70-& 80° muss die Destillation besonders scharf beobachtet werden, da das Produkt ausserordentlich zum Schäumen neigt. Bei übermässigem Schäumen wird etwas Druckluft auf den Destillationskessel gestellt.

Bei der Destillation werden 540 kg Trichloraethylen wiedergewonnen, die zur Entfernung des Wassers von Pos. 19) nach dem Scheider Pos. 20) gedrückt werden.

Das im Destillationskessel befindliche Dismulgan IV wird mit Wasser auf 1800 kg eingestellt (spez.Gew. 1,05-1,06, Fettgehalt 35 %, siehe Probe 5 von Humextol CX). Die Farbe ist hellbraun. Netzwert 1,2 g im Liter = 110-120 Sek. Netzzeit. Laugebeständigkeit 2 g i.Ltr. = 12 ccm Natronlauge 40° Bé.

Ausbeute: 1800 kg.

Mainkur, den 25. Oktober 1944

Vorschrift zur Fabrikation von Dismulgan VII

(Holl.Nr.11742)

Konstitution:

C_2H_5

$CH_2.CO.O.CH_2.CH.CH_2.CH_2.CH_2.CH_3$

$CH.CO.O.CH_2.CH.CH_2.CH_2.CH_2.CH_3$

SO_3Na C_2H_5

Mol. 444

Chemischer Vorgang:

$CH.COOH$
$+ 2CH_3(CH_2)_3.CH.(C_2H_5).CH_2OH =$
$CH.COOH$

Mol.116 Mol.130

$CH.CO.CCH_2.CH(C_2H_5)(CH_2)_3.CH_3$
$+ 2H_2O$
$CH.CO.CCH_2.CH(C_2H_5)(CH_2)_3.CH_3$

Mol.340

$CH.CO.O.CH_2.CH(C_2H_5)(CH_2)_3.CH_3$
$+ NaHSO_3 =$
$CH.CO.O.CH_2.CH(C_2H_5)(CH_2)_3.CH$

$CH_2.CO.O.CH_2.CH(C_2H_5).CH_2.CH_2.CH_2.CH_3$
$CH.CO.O.CH_2.CH.(C_2H_5)CH_2.CH_2.CH_2.CH_3$
SO_3Na Mol.444

Maleinsäure wird mit Alpha-Äthylhexanol unter Zusatz von p-Toluolsulfosäure zum Maleinsäure-di-(Alpha-Äthylhexyl)ester umgesetzt. Durch Anlagerung von Natriumbisulfit wird der Ester in das Natriumsalz des Sulfobernsteinsäure-di-(Alpha-Äthylhexyl)-esters übergeführt (Dismulgan VII).

Einsatz:

4307 kg	Äthylhexanol 100%	
1830 "	Maleinsäure 100%	
75 "	p-Toluolsulfosäure 50%ige Paste	
4330 "	Spiritus 95%ig	
2194 "	Bisulfit Pulver 100% = 2250 kg effektiv	
190 "	Natronlauge 33° Bé	

Ausbeute:

Die Ausbeute beträgt 560 kg Dismulgan VII auf 100 kg Einsatz Maleinsäure.

Arbeitsweise:

In einem homogen verbleiten Doppelkessel von 8 cbm Inhalt (Nr.1) werden 4307 kg Äthylhexanol 100% (Probe Nr.1) eingesaugt und darauf unter Rühren 1830 kg Maleinsäure100% und 75 kg p-Toluolsulfosäure Paste (etwa 50%ige Ware) eingetragen. Es wird nun innerhalb einer Stunde auf 101°, weiter im Laufe von 7 Stunden langsam auf etwa 145° geheizt. Bei 101° beginnt das Abdestillieren des Wassers. Der Kessel ist dabei über einen Rückflusskühler (Nr.2) mit einem kleinen Scheider verbunden, der mitüberdestilliertes Äthylhexanol dem Veresterungsgemisch wieder zuführt. Nachdem die Hauptmenge des Wassers abdestilliert ist, wird der Kessel über Nacht weiter geheizt, wobei die Temperatur bis 151/152° steigt. Dabei geht nur noch wenig Wasser über. Zur Entfernung der letzten Reste Wasser wird der Rückflusskühler und der Scheider abgeschaltet und der Kessel über einen Schlangenkühler (Nr.3) auf eine kleine Vorlage von 100 Liter Inhalt geschaltet, die unter Vacuum gestellt wird. Das Vacuum wird dabei so schwach eingestellt, dass in den Schauglösern der Kühlschlangen das Destillat (Wasser und überschüssiges Äthylhexanol) fast tropfenweise erscheint. Nach etwa 5 stündigem Destillieren bei 150/152° ist die Destillation beendet. Der Kessel wird auf Raumtemperatur abgekühlt. Volumen etwa 6000 - 6100 Liter. Spez.Gewicht des Esters ca 0,940.

Sulfitierung des Esters:

In einen emaillierten Doppelkessel von 3,7 cbm Inhalt (Nr.4) werden 1100 Liter Ester übergedrückt (gemessen im Emaillekessel). Dazu laufen aus einem Messgefäss (Nr.5) 750 kg Spiritus 95%ig. Unter Rühren wird der Ester durch vorsichtige Zugabe von Lauge

schwach lackmusalkalisch gestellt. Es werden etwa 20 - 25 Liter Lauge verbraucht. Inzwischen werden in einem ausgemauerten Rührkessel von 3 cbm (Nr.6) 390 kg Natriumbisulfit wasserfrei = 380 kg 100% (durchschnittlicher Gehalt 97,5%) in 600 Liter Wasser gelöst. Nach Alkalischmacher des Esters wird die Bisulfitlösung zugedrückt. Gesamtvolumen 2900 Liter. Der Kessel wird geschlossen, langsam auf 98 - 100° geheizt und 12 Stunden bei dieser Temperatur gelassen. Der Druck beträgt etwa 1,6 Atü. Darauf wird auf 60° abgekühlt und nach Ablassen des restlichen Druckes wieder angeheizt zum Abdestillieren eines Teiles des Spiritus. Hierzu wird der Kessel über einen Kühler (Nr.7) mit einer Vorlage (Nr.8) verbunden. Bei 80° beginnt die Destillation. Die Temperatur wird langsam bis 91° gesteigert. Sobald 91° erreicht sind, wird die Destillation unterbrochen und der Kessel auf Raumtemperatur abgekühlt. Die Destillation dauert etwa 7 Stunden. Das fertige Produkt wird in Sperrholzdichtfässer von 200 Liter Inhalt ausgedrückt. Von jeder Partie wird sofort die Oberflächenspannung, der pH Wert und der Gehalt an flüchtigen Bestandteilen bestimmt (Probe 2). Der obige Sammelansatz Ester ergibt fünf Sulfitierungsansätze zu je 1100 Ltr., dazu noch einen sechsten Ansatz zu etwa 800 Ltr. (immer gemessen im Emaillekessel). Zum letzten Ansatz werden die Zusätze entsprechend der Literzahl des Esters berechnet. Nach Aufarbeitung der gesamten Estermenge wird der überdestillierte Spiritus alkalisch gestellt und in der Spiritus Kolonne destilliert. Wird bei der Sulfitierung ein schwächerer als 95%iger Spiritus eingesetzt, so muss die Wassermenge zum Lösen des Bisulfits entsprechend dem Wassergehalt des Spiritus verringert werden. Vom eingesetzten Spiritus werden etwa 70 - 82 % zurückgewonnen, die restlichen 18 - 30 % bleiben im Dismulgan.

Erfahrungen:

Bei der Esterbildung muss das Abdestillieren des Wassers sehr vorsichtig geschehen. Es hat sich herausgestellt, dass Partien, bei denen das Wasser schnell abdestilliert wurde, ein unbrauchbares Dismulgan lieferten.

Proben:

1.) Von jeder Sendung Äthylhexanol wird vom analyt. Laboratorium die Hydroxylzahl bestimmt. Die untere zulässige Grenze der Hydroxylzahl ist 415. Nach der Hydroxylzahl wird der Einsatz berechnet. Es wird ein Ueberschuss von 5% über Theorie genommen.

LIST OF CAPTURED DOCUMENTS

Instructions for Making and Testing Products

Process for preperation of Humectol CX	4 Feb. 1938
Kaurit MKF	Sep. 1943
Instructions for making Kaurit MKF	11 Jan. 1944
Process for making pure Trypaflevin	18 July 1938
Process for making Thiodiphenylamine S	4 Oct. 1944
Process for making Dismulgen IV	24 Apr. 1941
Instructions for making Dismulgan VII	25 Oct. 1944
List of personnel in different departments	5 Apr. 1945

List of projects supervised by different men.

Payroll records showing allocation of technical men during war.

25-84872-60

Classification Cancelled,
by authority of
The Joint Chiefs of Staff,
by Capt. Bosquet N. Wev, USN
BNW

ITEM NO. 30.
FILE NO. XXXI-58.

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COMPILATION OF GERMAN FUELS AND LUBRICANTS SPECIFICATIONS

Schaffer, B. Lo., Mt. Ogal, R. J.

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COMPILATION OF GERMAN FUELS AND LUBRICANTS SPECIFICATIONS

Reported by:

Captain C. C. Chaffee, U. S. Ord.
Lieutenant R. J. Ozol, U. S. Ord.

28 August 1945

CIOS ITEM 30
FUELS & LUBRICANTS

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

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TABLE OF CONTENTS

SUBJECT	PAGE NO.
Introduction	3
Discussion	3
Appendix A	5
Appendix B	7
Appendix C	18
Appendix D	25

PERSONNEL OF TEAM

Capt. C. C. Chaffee, U.S. Ord.
Lt. R. J. Ozol, U. S. Ord.

COMPILATION OF GERMAN FUELS AND LUBRICANTS SPECIFICATIONS

I. INTRODUCTION

Information has been obtained on German Armed Forces fuels and lubricants specifications from documents collected during CIOS field trips. The specifications have been translated and are briefly reviewed in this report. Information on the reason behind specification changes may be obtained by a detailed study of individual plant operations reported by CIOS investigators. It is intended that this report only summarize technical data on specifications.

II. DISCUSSION

The information on specification has been presented in four categories, that is, motor fuels, aviation fuels, lubricants and greases. The following general comments may be made at this time.

a. Motor Fuels. German Ground Forces fuel had to meet an Octane number of 72 and this was accomplished by using straight run, cracked and/or Fischer Tropsch gasoline and T.E.L., straight run, cracked and/or Fischer Tropsch gasoline T.E.L. and aromatics; and straight run crack and/or Fischer Tropsch gasoline and aromatics. Due to the need of aromatics in ammunition they were removed and T.E.L. substituted for them. Diesel fuel had to pass a minimum requirement of 45 Cetane number.

b. Aviation Fuels. Specifications have been obtained on fuels A-3 (80 ON), B-4 (89 ON) and C-3 (95 ON) and the components of these fuels made at Leuna, Scholven, Bohlen Gelsenberg and Brux. In general, the final product, was not altered greatly except that as the war progressed a larger amount of aromatics was allowable in each fuel, the maximum being 45% in C-3 gasoline. The only available specifications on aviation Diesel fuel reveal that a minimum octane value of 60 was allowed which was later reduced to 50.

c. Lubricants. Specifications have been obtained for both Air Force and Ground Force lubricants. They include lubricant S-3, V-2 and the synthetic lubricant components of these lubes and also summer and winter oil for Ground Force vehicles.

d. Greases. Specifications have been obtained for Rocker Arm Grease Protective Grease 40 G, Axle Grease, Refrigeration Grease, Aviation Instrument Grease, Aviation Grease-Blue, Protective Grease 40 (Tp); Protective Grease 40, Weapon Grease, Instrument Grease, Water Pump Grease, and Lubricating Grease.

The translated specifications and comments on test technique are appended. In general the quality of fuels and lubricants as set by specifications was fairly well maintained during the war.

APPENDIX A
MOTOR FUEL SPECIFICATIONS

OTTO ENGINE

	Motor Gasoline with Tetra Ethyl Lead	Motor Gasoline with Aromatics and Tetra-Ethyl Lead (Min of 20 wt % of Aromatics	Motor Gasoline with Aromatics (Min of 35 wt % of Aromatics)
General Characteristics:	The fuel must be clear free from undissolved water and solid impurities and should not attack copper.		
Color			
Density @ 15° C	0.720-0.780	0.740-0780	0.753-0.790
Octane No. (Motor Method)	72	72	72
Boiling Range			
up to 75°C	25 vol %	25 vol %	25 vol %
up to 100°C	30 vol %	38 vol %	45 vol %
up to 200°C	95 vol %	95 vol %	95 vol %
Reid Vapor Pressure @ 40° C	0.20-0.60 kg/cm^2	0.20-0.60 Kg/cm^2	0.20-0.60 kg/cm^2
Residue mg/ 100 c.c.	10 max.	10 max.	10 max.
Cloud Point °C	-20	-20	-20
Heat Value	7500 Kg cal/Liter	7650 Kg cal/Liter	7700 Kg cal/Liter
Sulfur Content (wt %)	0.2 max.	0.2 max.	0.2 max.
Tetra Ethyl Lead (vol %)	0.04 max.	0.04 max.	0

APPENDIX A
MOTOR FUEL SPECIFICATIONS

DIESEL ENGINE

	DIESEL FUEL	SPECIAL DIESEL FUEL
General Characteristics:	The fuel must be free from solid impurities	
Density @ 15° C	0.810-0.865	0.810-0.865
Viscosity °E @ 20°C	1.1-2.0	1.1-2.0
Pour Point	-30°C	-30°C
Cloud Point	-10°C	-10°C
Filtering Rate @		
Pour Point	200 cc/min. @-25°C	200 cc/min @-25°C
Cloud Point	200 cc/min. @- 5°C	200 cc/min @- 5°C
Flash Point	55°C min.	21°C min.
Acid Value (mgKOH/gm)	0.4 max.	0.4 max.
Zinc Corrosion	4.0 mg max.	4.0 mg. max.
Sulfur Content (wt %)	1.0 max.	1.0 max.
Minimum Heat Value	9900 Kg Cal/Kg	9900 Kg Cal/Kg
Centan Value	45 min.	45 min.
Water Content (wt%)	0.5 max.	0.5 max.
Ash (wt %)	0.05 max.	0.05 max.
Residue		
Hagemunn/Hammerich	2.0 wt %	2.0 wt %
Conradson	0.05 wt %	0.05 wt %
Boiling Range	80 vol % @ 360°C	80 vol % @ 360°C

Miscibility: All Diesel fuels must be miscible with one another

APPENDIX B
AVIATION FUEL SPECIFICATIONS--OTTO ENGINE

Specification Number	A-3 TL 147-257 1	A-3 TL 147-257 2	A-3 TL 147-257 3	A-3 TL 147-257 4
Date of Spec.		Jun. 1939	Oct. 1943	Nov. 1944
Appearance	Clear, free from undissolved water, acid and solids.			
Color	Dyed blue 2.0 mg/L Sudan blau G	Dyed blue 2.0 mg/L	Dyed blue 1.6 to 1.8 mg/ L Sudan blau GN	Dyed blue 1.6 to 2.6 mg/L Dye
Octane Rating	80 min.	80 min.	80 min.	80 min.
Density @15°C	0.710-0.755	0.715-0.750	0.710-0.760	0.700-0.770
Distillation				
10 vol. %	70°C	70°C	75°C	---
50 vol. %	100°C	100°C	105°C	100°C
90 vol. %	160°C	130°C	160°C	---
End Pt.	170°C max	150°C	170°C max	190°C max
Evap.	2 vol. %	2 vol. %	2 vol. %	2 vol. %
Reid Vapor Pres. (kg/cm^2)	0.5 max	0.5 max	0.5 max	0.5 max
Iodine No. (g./100 g.)	5.0 max	5.0 max	---	---
Melting Point	-60°C max		-60°C max	-50°C max
Corrosion Test	No discoloration or attack in the copper dish test.			
Residue (mg./100 c.c)	8 max	8 max	10 max	10 max
TEL(vol %) min	0.045-0.050	---	0.045-0.050	0.045
Aromatic Content (vol. %)	---	---	25 Max	35 max
Ethylene Dibromide (vol. %)	---	---	0.019-0.023	---
Inspection	After ½ yr storage, at 3 mos intervals(visual)		After ½ yr storage, at 3 mos intervals(visual)	Monthly intervals
Comments				Ethanol content of fuel =30 vol %

RESTRICTED

APPENDIX B

AVIATION FUEL SPECIFICATIONS--OTTO ENGINE

Specification Number	B-4 TL 147-304 1	B-4 TL 147-304 2	B-4 TL 147-304 3	B-4 TL 147-304 4
Date of Spec.		Jun. 1939	Oct. 1943	Nov. 1944
Appearance	Clear, free from undissolved water, acid and solids.			
Color	Dyed blue 3.6 mg/L Sudanblau G	Dyed blue 3.6 mg/L	Dyed blue 4.0-4.3 mg/L Sudanblau GN	Dyed blue 4.0-4.3 mg/L Dye
Octane Rating	89 min	89 min	89 min	89 min
Density @15°C	0.710-0.755	0.715-0.750	0.710-0.760	0.700-0.760
Distillation				
10 vol. %	70°C	70°C	75°C	75°C
50 vol. %	100°C	100°C	105°C	---
90 vol. %	160°C	130°C	160°C	---
End Pt.	170°C max	150°C	170°C max	190°C max
Evap.	2 vol %	2 vol %	2 vol %	---
Reid Vapor Pres. (kg/cm^2)	0.5 max	0.5 max	0.5 max	0.5 max
Iodine No. (g./100 g.)	5.0 max	5.0 max	5.0 max	---
Melting Point	-60°C max	-60°C	-60°C max	-50°C max
Corrosion Test	No discoloration or attack in the coppe dish test.			
Residue (mg/100 c.c)	8 max	8 max	10 max	10 max
TEL(vol. %)	0.115-0.120	0.115-0.120	0.115-0.120	0.115-0.120
Aromatic content (vol.%) max	---	---	25	35
Ethylene Dibromide (vol.%)	---	---	0.050-0.053	0.050-0.053
Inhibitor(wt %)	---	---	---	---
Inspection	After ⅓ yr storage visual inspec at 3 mos intervals		After ⅓ yr storage visinspec at 3 mos intervals	After ⅓ yr storage visual inspec at 3 mos intervals

APPENDIX B

AVIATION FUEL SPECIFICATIONS--OTTO ENGINE

Specification Number	C-3 TL 147-330 1	C-3 TL 147-330 2	C-3 TL 147-330 3	C-3 TL 147-330 4
Date of Spec.		Jun. 1939	Oct. 1943	Nov. 1944
Appearance	Clear, free from undissolved water, acid and solids.			
Color	Dyed green 0.5 mg Sudan blau GN & 0.5 mg Flurol per L		Dyed green 0.5 mg Sudan blau GN & 0.5 mg Flurol per L	Dyed green 0.5 mg Sudan
Octane Rating	95		95	95
Density @15°C	0.760-0.795		0.760-0.795	0.740-0.795
Distillation				
10 vol. %	---		80°C	80°C
50 vol. %	100°C		110°C	120°C
90 vol. %	---		100°C	180°C
End Pt.	180°C max		180°C max	190°C max
Evap.	2 vol. %		2 vol. %	---
Reid Vapor Pres. ($kg./cm^2$)	0.45 max		0.45 max	0.45 max
Iodine No. (g./100 g.)	4 max		---	---
Melting Point	-60°C max		-60°C max	-60°C max
Corrosion Test	No discoloration or attack in the copper dish test.			
Residue (mg/100 c.c)	10 max		10 max	10 max
TEL(vol.%)	0.115-0.120		0.115-0.120	0.115-0.120
Aromatic content (vol. %) max	---		45	45
Ethylene Dibromide(vol. %)	---		0.050-0.053	0.050-0.053
Inhibitor(wt %)	---		0.01	0.01
Inspection	After 3 mos storage, vis inspec at mo intervals		After 3 mos storage, vis inspec at mo intervals	After 3 mos storage, vis inspect at mo intervals

APPENDIX B

AVIATION FUEL SPECIFICATIONS--OTTO ENGINE

Specification Number	VT 702 TL 147-152 1	VT 702 TL 147-152 2	VT 702 TL 147-152 3
Date of Spec.	June 1939		
Appearance	Clear, free from undissolved water, acid and solids.		
Color	Water white		Water white
Octane Rating	70/--		70/89
Density @ 15°C	0.715-0.725		0.715-0.730
Distillation			
10 vol. %	60-70°C		70°C
50 vol. %	85-95°C		95°C
90 vol. %	120-130°C		130°C
End Pt.	135-145°C		145°C max
Evap.	2 vol. %		2 vol. %
Reid Vapor Pres. ($kg./cm^2$)	0.5 max		0.5 max
Iodine No. (g./100 g.)	3.0 max		3.0 max
Melting Point	-60°C max		-60°C max
Corrosion Test	No discoloration or attack in the copper dish test.		
Residue (mg./100 c.c)	5.0 max		5.0 max
TEL(vol. %)	0/		0/0.115
Aromatic Content (vol. %)	---		15
Ethylene Dibromide (vol. %)	---		---
Inhibitor (wt %)	---		---
Inspection	Yearly		Yearly
Comment:	This fuel is a hydro petrol made from German lignite by the high pressure hydrogenation process of the I.G. It must not contain straight run, cracked, or polymer gasoline, anti-knock or anti-corrosion dope or inhibitor. Maximum sulfur content--0.05 wt %.		

APPENDIX B

AVIATION FUEL SPECIFICATIONS--CTTO ENGINE

Specification Number	V.T. 705 TL 147-153 1	V.T. 705 TL 147-153 2
Date of Spec.	Sept. 1939	
Appearance	Clear, free from undissolved water, acid & solids.	
Color	Water white	Water white
Octane Rating	72/--	72/89
Density @15°C	0.725-0.740	0.730-0.745
Distillation		
10 vol. %	60-70°C	70°C
50 vol. %	85-95°C	100°C
90 vol. %	120-130°C	145°C
End Pt.	155°C	165°C max
Evap.	2 vol. %	2 vol. %
Reid Vapor Pres. ($kg./cm^2$)	0.45 max	0.45 max
Iodine No. (g./100 g.)	4.0 max	4.0 max
Melting Point	-60°C max	-60°C max
Corrosion Test	No discoloration or attack in the copper dish test.	
Residue (mg./100 c.c)	--	5 max
TEL (vol. %)	0/--	0/0.115
Aromatic Content (vol. %)	---	15 max
Ethylene Dibromide (vol. %)	---	---
Inhibitor (wt %)	---	---
Inspection	Yearly	Yearly
Comments	Maximum sulfur content--0.05 wt %.	

APPENDIX B

AVIATION FUEL SPECIFICATIONS--OTTO ENGINE

Specification Number	V.T. 810 TL 147-154 1	V.T. 810 TL 147-154 2
Date of Spec.	Sept. 1939	
Appearance	Clear, free from undissolved water, acid & solids.	
Color	Water white	Water white
Octane Rating	70/-	67/
Density @15°C	0.715-0.725	0.715-0.725
Distillation		
10 vol %	60-70°C	65°C
50 vol %	85-95°C	95°C
90 vol %	120-130°C	135°C
End Pt.	135-145°C	150°C max
Evap.	2 vol. %	2 vol. %
Reid Vapor Pres. ($kg./cm^2$)	0.5	0.5
Iodine No. (g./100 g.)	3.0 max	3.0 max
Melting Point	-60°C max	-60°C max
Corrosion Test	No discoloration or attack in the copper dish test.	
Residue (mg./100 c.c)	--	5.0 max
TEL (vol. %)	0/-	
Aromatic Content (vol. %)	--	11-15
Ethylene Dibromide (vol. %)	--	--
Inhibitor (wt %)	--	--
Inspection	Yearly	Yearly
Comments	Maximum sulfur content--0.05 wt %.	

APPENDIX B

AVIATION FUEL SPECIFICATIONS--OTTO ENGINE

Specification Number	VT 100 TL 147-100 1	VT 100 TL 147-100 2	VT 100 TL 147-100 3
Date of Spec.	October 1943		
Appearance	Clear, free from undissolved water, acid and solids.		
Color			Water white
Octane Rating			72/89
Density @15°C			0.710-0.755
Distillation			
10 vol. %			70°C
50 vol. %			100°C
90 vol. %			150°C
End Pt.			170°C
Evap.			2 vol. %
Reid Vapor Pres. ($kg./cm^2$)			0.5
Iodine No. (g./100 g.)			5 max
Melting Point			-60°C max
Corrosion Test	No discoloration or attack in the copper dish test.		
Residue (mg./100 c.c)			5 max
TEL (vol. %)			0/0.115 vol %
Aromatic Content (vol. %)			25 max
Ethylene Dibromide (vol. %)			---
Inhibitor (wt %)			---
Comments:			Straight run gasoline. Max sulfur content 0.05 wt %

APPENDIX B

AVIATION FUEL SPECIFICATIONS--OTTO ENGINE

Specification Number	VT 708 TL 147-158 1	VT 708 TL 147-158 2	VT 708 TL 147-158 3
Date of Spec	Oct. 1943		
Appearance	Clear, free from undissolved water, acid & solids.		
Color			Water white
Octane Rating			70 / 89
Density @15°C			0.710-0.740
Distillation			
10 Vol. %			70°C
50 Vol. %			100°C
90 Vol. %			150°C
End Pt.			170°C
Evap.			2 vol. %
Reid Vapor Pres. ($kg./cm^2$)			0.5
Iodine No. (g./100 g.)			4 max
Melting Point			-60°C max
Corrosion Test	No discoloration or attack in the copper dish test.		
Residue (mg./100 c.c)			5 max
TEL (vol. %)			0 /0.115 vol %
Aromatic Content (vol. %)			15 max
Ethylene Dibromide (vol. %)			---
Inhibitor (wt %)			---
Comments			Hydrogenated gasoline. Analine point of 55°C max. Maximum sulfur content 0.05 wt %.

APPENDIX B

AVIATION FUEL SPECIFICATIONS--OTTO ENGINE

Specification Number	VT 207 TL 147-157 1	VT 707 TL 147-157 2
Date of Spec.	October 1943	
Appearance	Clear, free from undissolved water, acid & solids.	
Color		Water white
Octane Rating		72 / 89
Density @ 15°C		0.720-0.740
Distillation		
Init.		40°C
10 Vol. %		70°C
50 Vol. %		100°C
90 Vol. %		145°C
End Pt.		165°C
Evap.		2 vol. %
Reid Vapor Pres. ($kg./cm^2$)		0.45 max
Iodine No. (g./100 g.)		3 max
Melting Point		-60°C max
Corrosion Test	No discoloration or attack in the copper dish test.	
Residue (mg./100 c.c)		5 max
TEL (vol. %)		0 / 0.115 vol %
Aromatic Content (vol. %)		15 max.
Ethylene Dibromide (vol. %)		---
Inhibitor (wt.%)		---
Comments		Hydrogenated gasoline. Analine point of 50°C. Maximum sulfur content-0.05 wt %

APPENDIX B
AVIATION FUEL SPECIFICATIONS--OTTO ENGINE

Specification Number	VT 302 TL 147-150 1	VT 811 TL 147-155 1	VT 812 TL 147-162 1
Appearance	Clear, free from undissolved water, acid & solids		
Color	Water white	Water white	Water white
Octane Rating	---	70 / 89	70 / 89
Density @ 15°C	0.875	0.725-0.750	0.720-0.740
Distillation			
Init.	80°C	40°C	40°C
10 vol. %	--	70°C	70°C
50 vol. %	--	105°C	110°C
75 vol. %	100°C	--	--
90 vol. %	--	145°C	150°C
95 vol. %	145°C		
End Pt.	--	165°C	165°C
Evap.	2 vol %	2 vol %	2 vol %
Reid Vapor Pres. ($kg./cm^2$)	0.25	0.50	0.50
Iodine No. (g./100 g.)	1.0 max	3.0 max	3.0 max
Melting Point	-10°C max	-60°C max	-60°C max
Corrosion Test	No discoloration or attack in the copper dish test.		
Residue (mg./100 c.c)	5 max	5 max	5 max
TEL (vol. %)	--	0 /0.115 vol %	0 /0.115 vol %
Aromatic Content (vol. %)	100.0	6-15.0	15.0 max
Ethylene Dibromide (vol. %)	--	--	--
Inhibitor (wt %)	--	--	--
Comments	Max. sulfur content 0.08 wt %	Hydrogenated gasoline. Aniline point of 52°C. Max Sulfur content 0.05 wt %	Hydrogenated gasoline. Aniline point of 44-54°C. Max sulfur content 0.05 wt %

APPENDIX B

AVIATION FUEL SPECIFICATIONS--DIESEL ENGINE

Specification Number	K-1 TL 147-351 1	K-1 TL 147-351 2
Date	April 1944	
Centane No.	60 min.	50 min
Density @ 20°C	0.810 min.	0.810 min.
Boiling Range	I.B.P. about 160°C.	I.B.P. about 160°C. 95 vol % off-350°C.
Setting Point °C	-45 max	-45 max
Viscosity	1.1°E min @20°C	1.1°E min @20°C
Flash Point °C	above 50	above 50
Acid Value mg KOH/gm	0.7 max	0.7 max
Sulfur Content wt %	1 max	1 max
Ash Content	Traces only	Traces only
Corrosion Test	Negative	4 mg max
Conradson Test Wt %	0.1 max	0.1 max
Water Content	Traces only	Traces only
Storage Stability	After 1 yr inspec at 6 mos intervals	After 1 yr inspec at 6 mos intervals

APPENDIX C

ACCEPTANCE CONDITIONS FOR THE AERO-ENGINE OILS.

S3 and V2

Military Name		S 3				V 2	
Filling Station Mark		VK,VB,NM,WD,WH,WN,WL, WR,WM,WY,WV,WF,WB,WG.		RG,HL,WD,WH,WN,WL,WR, WM,WG,WV,WF,WB,WG.		R G	
Technical Specific for Delivery (1)		TL 147-502 TL 147-503 TL 147-506		TL 147-(561-563) TL 147-(571-573) TL 147-(581-583)		TL 147-500	
Appearance		The lubricating oil must be clear, free from undissolved water and mineral acids and must contain no solid impurities.					
Refractive Index	Max.	1.4995		1.4990		1.5070	
Density @ 20°C. Kg/Ltr.	Max.	0.897		0.895		0.920	
Viscosity		cs.	°E	cs.	°E	cs.	°E
@ 50°C		125-137	(16.5-18.0)	125-143	(16.5-18.8)	133-144	(17.5-19.0)
@ 100°C	Min.	18.5	(2.70)	19.0	(2.75)	19.4	(2.80)
Pole Height	Max.	2.04		1.95		1.9	
Slope	Max.	3.45		3.35		3.4	
Viscosity Index	Min.	92		98		95	

ACCEPTANCE CONDITIONS FOR THE AERO-ENGINE OILS. (CONTD)
S3 and V2

Setting Point °C	Max.	-17(2)	-20	-25
Flash Point °C	Min.	260	225	235
Ignition Point °C	Min.	300	255	270
Acid Value mg.KOH/g.	Max.	0,06	0,06	0,1
Saponfication Value mg.KOH/g.	Max.	0,2	0,2	8,0
Evaporation Test (Dr.Noack) (3)				
250°C. % wt.	Max.	3,0(4)	11,0	5,0
Conradson % wt.	Max.	0.35	0,25	0,5
Ash Content % wt.	Max.	0,01	0,01	0,02
Hard Asphalt % wt.		0	0	0
Water Content % wt.		0	0	0

Notes:
1. Constructional specifications for BVM Aero-Engines "Test Regulation for Aero-Engine lub. oils for use in Otto engines". Can be obtained from the Scientific Reports section (ZWB) of the DVL, Berlin-Adlershof, Rudower Chaussee 16/25.
2. For S3, the filling station mark VK allows a max. setting point of -10°C.
3. "Angewandte Chemie" 1936. Vol. 49, page 385.
4. For S3--VK the max. Noack Test value is 5.0.
5. The present sheet 7 supersedes the "Technical Acceptance Conditions for Aero-Engine Lub. Oils" issued by the RLMLC II 2e on 15.3.36. Copies still in existence should be destroyed.

Abbreviations: V--Vakuum K--Koln B--Bremen W--Wifo

Prepared by: Mineral Oil Section. Group GL/A-M II. This sheet is primarily intended for the suppliers. Thus there are two columns for S3 (to differentiate finished products from blended stocks)- see the other sheet 7 for military applications.

APPENDIX C

MOTOR OIL SPECIFICATIONS FOR COMPONENTS OF AVIATION MOTOR OIL S-3

	Low Viscosity Oil			High Viscosity Oil		
Code Designation	SS 607	SS 707	SS 807	SS 906	SS 1006	SS 1106
Specification Number	TL 147-560	TL 147-570	TL 147-580	TL 147-600	TL 147-610	TL 147-620
Appearance	The oil must be clear, free from undissolved water and mineral acids, and contain no solid impurities.					
Refractive Woex Max.	1.4995	1.4935	1.5010	1.4770	1.4860	1.4816
Density @20°C Max.	0.905	0.895-0.897	0.905	0.862	0.875	0.870
Viscosity @50°C	cs	cs	cs	cs	cs	cs
	57.5-62.0 °E	52.0-57.5 °E	51.0-60.0 °E	334-350 °E	300-380 °E	278 min. °E
@100°C Min.	7.6-8.2 cs	6.9-7.6 cs	6.8-7.9 cs	44-46 cs	40-50 cs	365 min. cs
	9.66 °E	9.35 °E	9.35 °E	42.3 °E	41.3-45.2 °E	38.0 °E
	1.80	1.77	1.77	5.63	5.5-6.0	5.1
Pole Height Max.	2.40	2.08	2.20	1.73	1.75	1.65
Slope Max.	3.80	3.66	3.75	3.05	3.05	3.00
Viscosity Index Min.	70	88	80	107	105	107
Cloud Point °C Max.	-15	-15	-16	-25	-20	-25
Flash Point °C Min.	225	225	222	225	295	280
Ignition Point °C Min.	265	258	258	263	325	330
Acid Value Mg KOH/gm	0.03	0.06	0.07	0.06	0.06	0.06
Saponification No. mg KOH/gm	0.10	0.17	0.17	0.2	0.1	0.2
Noack Evap @250°C wt %	9.0	14.0	12	8	1	5.5
Conradson wt %	0.2	0.25	0.30	0.2	0.45	0.25
Ash wt %						
Hard Asphalt wt %	0	0	0	0	0	0
Water Content wt %	0	0	0	0	0	0

APPENDIX C

MOTOR OIL SPECIFICATIONS
GROUND FORCE WINTER AND SUMMER OIL

	WEHRMACHT (SU) Summer	WEHRMACHT (WI) Winter
General Characteristics	The oil must be clear and contain no sediment	
Density @ 20°C Max.	0.91	
Evaporation Test	7-14%	20%
Viscosity		
Fngler @ -15°C	1800° F	550° F
Fngler @ 100°C	1.9-2.1	1.6° F
c.s. @ -15°C	18800 -	--
c.s. @ 100°C	10.8-12.8	--
Pole Height	2.1	2.0
Total Impurities	Free	Free
Hard Asphalt	Free	Free
Benzol-alcohol solution	Free	Free
Solid Impurities	Free	Free
Combustibles	Trace	Trace
Ash	0.10	0.07
Acid Value mg KOH/gm	0.25	0.25
Saponification No. mg KOH/gm	Free	Free
Water		200
Flash Point °C		-25
Pour Point °C		

APPENDIX C

MOTOR OIL SPECIFICATIONS
GENERAL COMMENTS ON TEST TECHNIQUE

GENERAL:

1. The tests should be carried out with officially authorized standard testing equipment, either in the possession of the supplier or in that locality (at suppliers expense) or at the State Chemical Institute, Berline-Polotzensee.

2. No additional payment will be made to the supplier for the testing, check testing, packing and shipping of the samples.

3. If the tests are not performed by the government institution preliminary recognition by the consignee of the testing equipment used is essential. The consignee will instruct the responsible testing department representative regarding test technique. The supplier will be responsible for his own equipment and test personnel. The consignee has the privilege of retesting any sample at the state institute.

4. Only firms authorized by the High Command may supply motor oil (winter or summer) to the German Ground Forces. Information of the suppliers production technique must be made known to the High Command and also to the state institute. Any changes in composition of the product must be authorized.

5. The motor oil must be supplied in sealed containers which can be readily opened for spot testing.

ACCEPTANCE:

6. A sample of 1.5 kgms is required in each of the tests. After removing sample the container will be properly sealed.

7. Tests will be made on each sample. If any one of the tests is unsatisfactory the sample will be rejected.

a. The density @ 20°C will be determined in accordance with German Industrial Standards DVM 3653. It is satisfactory to use a hydrometer.

b. The volatility will be determined by the Noack Method described in "Avg. Chemie 49,385 (1936)" and in "Alud Kohle 1942".

c. The viscosity will be determined by the Vogel-Ossag-Viscosimeter using a sensitive thermostat, calibrated capillaries, thermometers calibrated to 1/10°C, and calibrated stop watches. All the above apparatus should be calibrated by the State Physical Technical Institutes.

Capillaries will not be used interchangeably as erroneous results will be obtained. Additional calibration is required from time to time using calibrated oils as standards.

The minimum time of flow is dependent upon the diameter of the capillary as shown in the following table.

	K	t (s)
2-	0.8	30
	0.6	34
	0.4	42
	0.2	60
	0.1	85

A time of flow of 90 seconds should not be exceeded if possible. Measurements will be made at 20°C, 50°C and 90°C. The values for -15°C 100°C and VP are calculated from E20 and E90 according to the Uble Code Weather formula, or directly from the next issue of CTR tables. The measurement of the 50°C value is a control for the 20° C & 90°C values.

d. To estimate total impurities mix 5 gms of oil with 50 c.c. of normal (standard) gasoline in an iodine bottle. Stopper, and allow to stand for three hours in the dark and filter the normal gasoline solution through a pre-treated Jenaer IG 4 glass filter crucible mounted on a vacuum flash. The residue is washed with cold normal gas to free it of oil, and then with warm normal gas to dissolve possible paraffin and ceresin. It is then dried for ½ hour at 105°C, left to cool in a dessicator and then weighed. The gain in weight represents the content of hard asphalt, alcohol, soluble benzol and solid impurities. The contents of the crucible should then be washed with benzol on the vacuum flash until the filtrate is colorless; dry, cool and weigh as above; the difference of the two weights is the hard asphalt content. Wash crucible contents with a 1:1 benzol alcohol mixture, treat as above; difference in weight is the alcohol soluble benzol. The final residue represents the solid impurities.

Preparation for use of the I.G. 4 crucible

The crucible is cleaned with chromic acid ($+ H_2SO_4$), washed with water till neutral; 5 gms of pure cryolite powder are put in, the crucible is mounted on a vacuum flash and the powder is tamped down (fast)

under vacuum with a flattened glass rod. The crucible is then washed out with 20 ccs of a benzol-alcohol mixture and some normal gasoline. Air is then sucked through for 1 to 2 minutes and the crucible is dried for ½ hour in a desicator at 105°C. After adequate cooling it is weighed in a weighing glass. The weighing glass should not be used with a rapid analytical balance. Without such a balance, weighing glasses are to be used for all the above mentioned weighings.

Ash content is determined according to "German Industrial Standards" DVM 3657, with one alteration, namely that 100 gms of oil are weighed out. The difference between the solid impurities and the ash is the combustible matter.

e. The acid-value is determined according to DIN, DVM 3658. To be able to titrate dark colored oils without trouble, the DVM procedure is deviated from in the preparation of the solvent mixture. This is prepared according to Wizoff as follows: 33.8 gms analytical alkali blue 6 B are added to a mixture of 3 litres pure benzol and 1.5 litres 90% (at least) alcohol. The mixture is then agitated for a few hours, left to stand over night, and filtered. The solution is then ready for use.

f. The saponification number is determined according to DIN DVM 3659. Solvent mixture as for acid-value.

g. H_2O content is determined by a visual test on the sample after vigorous shaking up and pouring into a calibrated test tube. It is essential to carry out the heating test for presence of water.

High Command of the Army
Army Ordnance Office (Wa Chief Engineer)

APPENDIX 'D'

GREASE SPECIFICATIONS TABLE 1

Type	Rocker Arm Grease	Protective Grease (40L)	Axle Grease	Refrigeration Grease	Avia. Instr. Grease	Aviation Grease Blue
Specification Number	TL 147-730	TL 147-770	TL 147-740	TL 147-750	TL 147-750	TL 147-735
Appearance	(1)	(1)	(1)	(1)	(1)	(1)
Drop Point (Min.) °C	80	50	160	90	165	90
Color (2)	brown to black	dark brown to black	brown to dark green	brown	red/brown (distillate)	blue (dyed)
Acid Value (3)	--	neutral	--	--	--	--
Alkali Value (3)	--	neutral	--	--	--	--
Ash Content wt %(min)	2	0.5	2	4	1	3
Oil Separation wt %	4	--	1	1.5	1	1.5
Water Resistance	--	waterproof	--	waterproof	waterproof	waterproof
Homogeneity	homogeneous	homegeneous	homogeneous	homogeneous	homogeneous	homogeneous
Consistency	--	--	--	--	Penetrometer 280 ± 20	Penetrometer 250 ± 20
Water content(min) wt%	0.5	--	1	3	0.5	2.5
Storage Stability yrs	1	2	1	2	1	1
Cold behavior range (2)	0°C to-30°C (4)	(5)	0°C to-35°C (6)	(7)	0°C to-65°C (8)	0°C to-60°C (9)

CODE:

(1) Appearance should be structureless, smooth with short fibers.
(2) Test instructions may be obtained from the Air Ministry.
(3) No limit specified but reading should be recorded.
(4) (5) (6) (7) (8) (9) Calibration curves may be obtained from the G.A.F. Laboratory, Travemonoe, E 3L. The curves are attached in the order shown.

APPENDIX D

GREASE SPECIFICATIONS

General Comments on Greases Listed in Preceding Table

1. The Air Ministry must be informed on the types and quality of the raw materials, mixing proportions, operating conditions used in the manufac ure of the grease. The manufacturer is obliged to inform the Air Ministry of any change in the composition of the grease.

2. Contents of containers delivered to the Air Force and/or the Aircraft Industries must be made known.

3. The material should be packed in clean containers which conform to specific form, size and material of construction.

4. The manufacture of the grease must be done by firms authorized by the Air Ministry. If new or additional facilities are required the Air Ministry must be notified in advance.

5. The grease must conform to the listed specifications.

6. Only tests approved by the Air Ministry are to be used in testing the grease for quality and acceptance.

APPENDIX D

GREASE SPECIFICATIONS TABLE 2

Type	Protective Grease 40(Tp)	Protective Grease 40
Spec. No.	TL 6023	TL 6017
Appearance	Transparent and must not become liquid when stirred.	Transparent and must not become liquid when stirred
Drop Point(Min)°C	75	50
Color	Colorless	colorless
Acid Value mg KOH/gm	Mineral Acid--0 Organic Acid--0.5	Mineral Acid--0 Organic Acid--0.2
Alkali Value	--	--
Ash Content wt % (min)	1.0	0.5
Oil Separation	None permitted	None permitted
Water Resistance	Completely water resistant at 20°C with no oil separation.	Completely water resistant at 20°C.
Homogeneity	No change in homogeneity after 3 hrs heating at 150°C	No change in homogeneity after 3 hrs heating at 150°C
Consistency	Must be spreadable at 0°C as well as at 20°C.	Must be spreadable at 0°C as well as at 20°C
Water content wt % (min)	---	---
Storage Stability	---	---
Corrosion Stability	Rust proof for a minimum of 3 days at 40°C and 100% relative humidity	Rust proof for a minimum of 3 days at 40°C and 100% relative humidity.

COMMENTS:

A sample of 500 grams is required from each 100 kg batch. One sample has to pass the minimum requirement for each test listed above otherwise the whole batch is rejected. Additional comments regarding test techniques follow.

1. Drop point test done according to DIN DVM 3654.
2. Heat Resistance--A sample of grease is spread on a sheet metal container with raised edges and then heated for 3 hours at 50°C. The sample must be spreadable and show no marked changes.
3. Water Resistance--A lager of grease 10 cm long, 1 cm wide and about 1 mm thick is spread on a prepared glass strip 16 cm x 1.5 cm. The prepared glass strip is placed in a flask with 100 cc. distilled water so that the grease lager projects 5 mm above the water surface. If the lager has not changed after 48 hours the sample passes provided

that the water is clear. If the water is clouded or a slight change occurs on the greased surface the test should be continued for one week.

4. Ash Content--Done according to DIN DVM 3657
5. Free Acid--Done according to DIN DVM 3658
6. Corrosion Stability--Emery cloth polished steel rods, 50 mm x 10 mm are dried for 3 hours in a desiccator and then coated with grease by dipping for three minutes in molten grease at 100°C. The coated rods are hung so that surplus grease runs off and the sample reaches room temperature. The rods are next put in an air sealed chamber over warm water at about 50°C, so that a constant air temperature of 40°C is maintained. The sample passes when there is no trace of corrosion after 3 days.

APPENDIX D
GREASE SPECIFICATIONS TABLE 3

Type	Weapon Grease	Instrument Grease	Water Pump Grease	Lubricating Grease
Spec. No.	TL 6006	TL 6009	TL 6035	TL 6014
Appearance	Without lumps	Transparent	without lumps	Transparent and free of oil pockets
Drop Point °C (min)	145	160	95	130
Color	Transparent	Colorless	Salve color	Colorless
Acid Value mg KOH/gm	0	*0.10	Mineral Acid-0 Organic Acid-0.2	---
Alkali Value	0	---	Slightly alkaline	---
Ash Content wt %(min)	--	0.5	4	---
Oil Separation wt %	None permitted	0.5	5	0.5
Water Resistance	--	waterproof	---	---
Homogeneity	No change after 3 hrs @ 50°C	homogeneous	No change after 5 hrs @ 50°C	No change after 5 hrs @ 50°C
Consistency	Between solid and tallow consistency at 20°C	2.0 Kg @ 0°C (1)	---	0.3 Kg @ 50°C (1)
Water Content wt %(min)	---	0	4	---
Storage Stability	---	---	---	---
Unsaponitiable wt %	70°	---	---	---

CODE: (1) The consistency value is the smallest weight in Kg, which causes the extension of a 10 mm length strip in a period of 60 seconds. The details of the test follow.

a. Each sample must be kneaded in special apparatus for 200 strokes.
b. Fill the test cylinder with grease sample, taking necessary precautions to exclude air bubbles.
c. Place test cylinder in thermostat. A minimum of 45 minutes is required at 50°c and a minimum of 2 hours at -10°C.
d. Place the necessary weights on plunger to extrude grease.

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ITEM No. 22 & 30
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The Joint Chiefs of Staff,
by Col. R. H. Cravens
EWS

FISCHER TROPSCH
AND ALLIED PROCESSES

Fischkinds, O.
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RESTRICTED

FISCHER TROPSCH AND ALLIED PROCESSES

26 June 1945

Reported by:

W. HIRSCHKIND CWS, Hq ETOUSA
F. R. LOWDERMILK CWS, Hq ETOUSA

31 July 1945

CIOS Target Nos. 30/6.01, 30/5.01, 22/1264, 22/196
Miscellaneous Chemicals
Fuels and Lubricants

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

TABLE OF CONTENTS

Subject — Page No.

Kaiser Wilhelm Institute Fuer Kohleforschung Muelheim/Ruhr

1. Introduction..... 3
2. Organization..... 3
3. Scientific Developments..... 3
4. Laboratory and Pilot Plant Equipment of Note..... 4
5. Commercial Developments..... 4

Ruhr Chemie A.G., Holten-Sterkrade

1. General..... 5
2. Processes..... 7

Chemische Fabrik Holten, Holten-Sterkrade

1. General.....21
2. Operations.....21

Oxo Gesellschaft m.b.H., Holten-Sterkrade

1. General.....21
2. Process.....22

PERSONNEL OF INSPECTION TEAM

W. Hirschkind.....CWS, Hq ETOUSA
F. R. Lowdermilk.....CWS, Hq ETOUSA
L. C. Turnock.....CWS, Hq ETOUSA
G. B. Panero.....CWS, Hq ETOUSA

FISCHER TROPSCH AND ALLIED PROCESSES

KAISER WILHELM INSTITUTE FUER KOHLEFORSCHUNG MUELHEIM/RUHR

1. INTRODUCTION.

The Kaiser Wilhelm Institute was visited on 25 June 1945. Dr. H. Pichler, Assistant Director, and Dr. H. Koch, Department Head, were interviewed and all information received came from them.

The Institute was founded by leading industrialists of the Ruhr, principally coal operators, in 1912. In the same year the Director was chosen in the person of Dr. Franz Fischer, Prof. of the Royal Inst. of Technologie, Berlin. During the early part of 1913 the plans for the buildings were approved and, in June, 1914, the whole project was completed. It included a modern laboratory building with adequate shops, to which later equipment for high pressures and low temperatures were added together with pilot buildings and installations. The official opening took place in 1914 shortly before the outbreak of World War I.

2. ORGANIZATION.

Director.....Dr. Franz Fischer, 1912-1943
Dr. Karl Ziegler, 1943 - date
(residing at Halle, at present)

Dept. Heads.....Dr. H. Tropsch, 1921-1928 (died 1935)
Dr. H. Koch, 1927 - date (Dept. head since 1935)
Dr. H. Pichler, 1927 - date (Dept head since 1936, Asst. Dir. since 1943)

3. SCIENTIFIC DEVELOPMENTS.

Conforming to the wishes of the founders, Fischer started his work with the object of providing improved and new methods for utilization of coal in manufacture of coke, tar, oils, and gas. A treatise on coal tar distillation was the result of these first efforts. During the early twenties Fischer turned to the utilization of water gas with the following developments to his credit:

YEAR	PROCESS	PRESSURE ATM.	CATALYST	DEVELOPED BY
1922	Synthol. Oxygen cont. derivatives of hydrocarbons	100	Fe,CO,Ni	Fischer-Tropsch
1923	Synthane: Hydrogenated synthol	-	-	" "
1925	Benzine, some olefines	Normal	Fe,CO,Ni	" "
1936	Benzine, little olefines	5-20	CO	Fischer-Pichler
1937	" much olefines	5-20	Fe	" "
1938	Solid paraffins high melting	over 100	Ru	" "
1941	Iso paraffins	over 100	Ru	" "
1942-43	Naphthenes, aromatic, formic acid, fatty acids, alcohols, substitution of iron in catalysts for metals becoming scarce during the war.			

4. LABORATORY AND PILOT PLANT EQUIPMENT OF NOTE.

The pilot plant contains equipment for production and storage of water gas and synthesis gas. A storage tank for synthesis gas at 1000 atm was 1' diameter, 12' length. The converter units are 3" diameter by 10' length and water cooled. Compressors are installed for compression up to 1,000 atm. There are also units for catalyst testing by flow and static methods. Other equipment included:

Laboratory distillation columns with revolving ribbon agitator.

Furnaces with temperature control maintaining 1°C.

High pressure equipment built by Andreas Hofer, mechanic of the Institute.

Liquid air plant consisting of 4-stage compressor was built by Hofer with a capacity 0.7 liters of liquid air per hour; precooling with water raises the capacity to 14 liters per hour. Precooling by refrigeration increases the capacity still higher.

5. COMMERCIAL DEVELOPMENTS.

In 1934 Franz Fischer believed that the time was ripe to carry on large pilot plant and commercial developments of the processes of the Kaiser Wilhelm Institute and he put the matter before its governing body. This body subsequently asked a number of industrial concerns for proposals and bids. It was only natural that the bid of the Ruhr-Chemie A.G. was accepted. This concern was founded in 1928 by the principal coal operators of the Ruhr and adjacent districts for the

purpose of explcitation of processes and products incidental to the coal industry. The fixation of nitrogen was already carried out by this concern on a very large scale based on watergas and coke oven gas for the production of synthetic ammonia, nitric acids, and nitrates. As the result Ruhrchemie became the exclusive licensee of the Fischer-Tropsch and allied processes all over the world.

RUHR CHEMIE A.G., HOLTEN-STERKRADE.

1. GENERAL.

Ruhr Chemie is owned by approximately 80% of the coal and coal tar producers of the Ruhr represented by the Ruhr Kohlen Syndicate. It was organized about 1928 for the purpose of chemical exploitation of coal and coal products.

a. Organization.

Prof. Dr. F. Martin	Managing Director (Chemist)
Dr. Hageman	Director (Chemist)
Dr. Paul	Director (Mech. Engineer)
Mr. Weibell	Director (Business Mgr)

After the paralyzing bombing attacks of October and November, 1944, the German government requested evacuation of the managing personnel as well as some of the equipment and the more important documents and records. The latter were shipped to Reelkirchen (near Detmold). A few technicians in subordinate positions remained at Holten-Sterkrade. Among these were Dr. Spanier, one of the superintendents of the Nitrogenous Products Department; Dr. Lehreiber, Research Chemist, engaged in dehydrogenation work; Dr. Landgraf, General Manager of a subsidiary, Oxo Gesellschaft m.b.H., and Mr. Meby, Power Plant Engineer. Dr. Paul and Mr. Weibel, both directors, returned to the plant on or about June 20th under instructions from their principal to take any steps necessary for the rehabilitation of the plant. Both were interviewed on June 23rd.

b. Plant General.

(1) Estimated Value of Plant.

Buildings -	RM 20,000,000
Equipment	100,000,000
Total	120,000,000

(2) Number Employees.

Administration	200
Technical	350
Skilled Labor	700

Unskilled Labor	780
Control Staff	100
Research	150
Total	2280

(3) Principal Products.

(a) Nitrogenous Product (expressed as N_2) 55-60,000 tons/year representing 5% of Germany's prewar production.

(b) Hydrocarbon Products:

Paraffin	6000	tons/year
Lubricating Oil	6000	" "
Diesel Oil	19000	" "
Benzine	25000	" "
Propane/Butane	6000	" "
Total	62000	" " corresp to 3-4% of Germany's production
Catalyst production:	900	" "

(4) Power.

50% self generated, 50% purchased at RM .02/KWH

Fuel: 222,000 tons/year cost RM 11-12/ton

Water: From Rheinisch-Westphaelische Water Works - RM .06-.07/cbm. Hardness-12°

Steam Consumption: 1,500,000 tons/year at 18 and 35 atm Cost - RM 13/tons

c. Historical.

The first plants to be built by Ruhr Chemie after its organization were the synthetic ammonia and ammonia oxidation plant, of which the first units were completed in 1930. Linde plants for manufacture of the necessary nitrogen with oxygen as a by-product were built at the same time.

In 1935, after obtaining exclusive license for all processes originating at the Kaiser Wilhelm Institute, constitution of the first Fischer Tropsch Unit was started and completed during the following year. At the time of the bomb attacks, which closed down production the following plant units were in operation:

(1) Complete nitrogenous products plant manufacturing synthetic ammonia, nitric acid and nitrogenous fertilizers.

(2) Hydrogen plant from coke oven gas.

(3) Linde plant for manufacture of nitrogen with oxygen as a by-product.

(4) Complete low pressure Fischer Tropsch plant for manufacture of benzine and Diesel fuel.

(5) Complete medium pressure synthesis plant for manufacture of olefins.

(6) Complete lubricating oil plant for manufacture of lubricating oil from olefins.

(7) Complete plant for manufacture of catalyst for own and licensee's use.

In addition to these production units, there were a number of operating pilot plants for the following processes:

(1) Manufacture of benzine with high octane rating.

(2) Manufacture of isoparaffins directly from synthesis gas.

(3) Isomerization of paraffins.

(4) Substitution of the cobalt catalyst by an iron catalyst.

d. Bomb Damage.

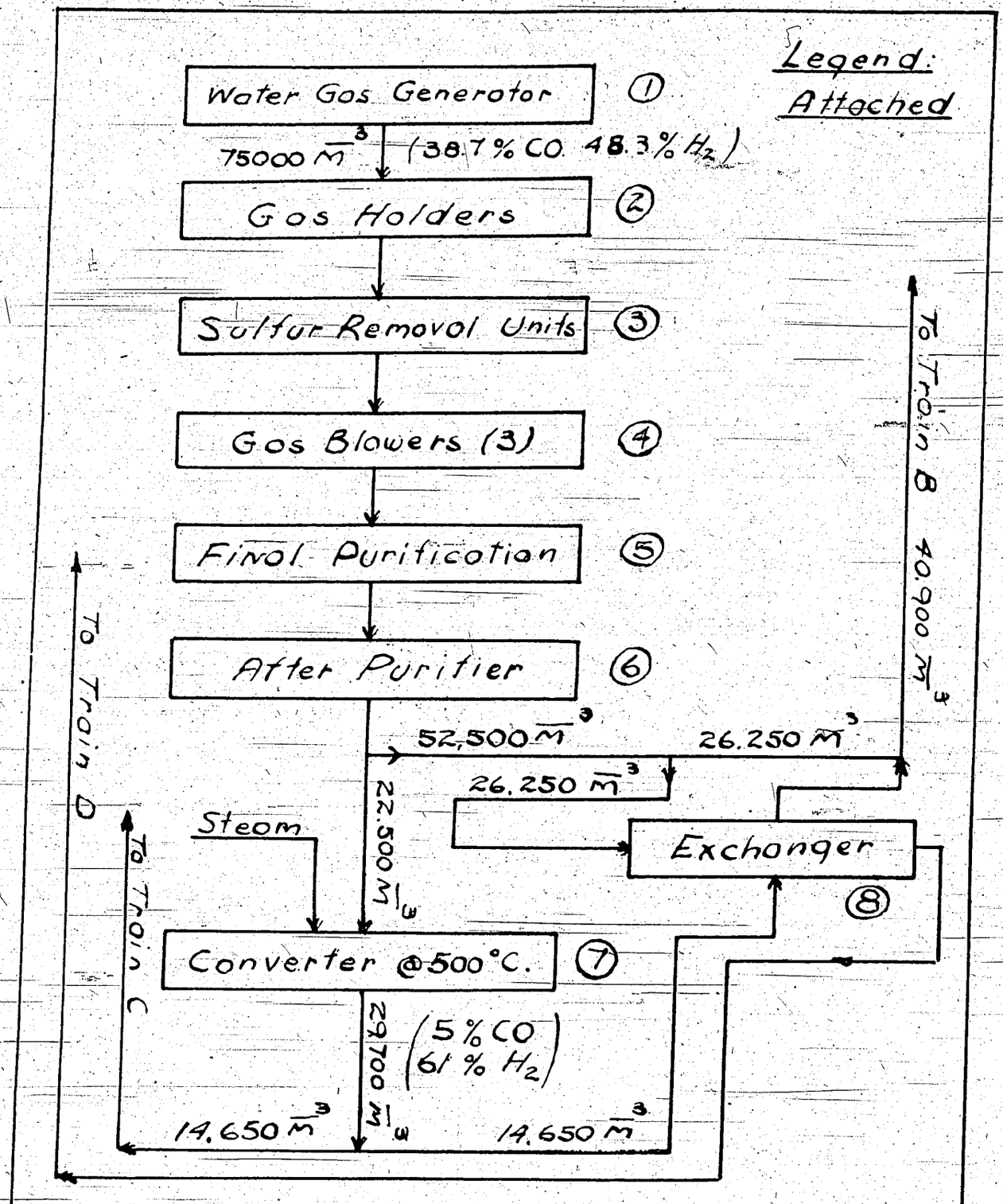
Bombing attacks during October and November, 1944, put an end to operations. Hardly a building was left undamaged. The equipment suffered less and some of it was evacuated on order of the government. The bomb damage is estimated at RM 46 - 50,000,000. Plans have been made for the resumption of operations on a limited scale. Provided that power and raw materials could be made available, the management indicates they could operate partially:

Oxygen and Nitrogen plant	-	within 1-2 weeks
Nitrogenous compound plant	-	" 4 months
Hydrocarbon plant	-	" 6-8 months

2. PROCESSES.

a. Water Gas, Normal, Purified, and Hydrogen Enriched. (Reference: Flow Sketch No. 1)

Water gas containing 38.7% CO and 48.3% H_2 was generated in the usual manner from steam and coke and was stored in gas holders. Sulfur



FLOW SKETCH NO. 1 - TRAIN A

WATER GAS PURIFICATION

HOURLY FLOW RATES

F.A.L.
7-22-45

LEGEND FOR TRAIN A.

(1) Water gas generator, capacity 75,000 cum/hr gas of 38.7% CO, 48.3% H_2.

(2) Gas holders, each 20,000 cu m capacity.

(3) Sulfur Removal Units, 8 towers (2 as spares), each packed with 60 T luxmasse, capacity 6 towers: 80,000 cu m/hr.

(4) Gas Blowers (3), 1 @ 80000 cu m/hr, 2 each @ 40,000 cu m/hr gas handling capacity.

(5) Final Purification Unit, 10 towers, gas heated to operate @ 200-250°C, with series flow through a pair of towers; towers each packed with 60 T of special material (70% luxmasse, 30% Na_2CO_3) and each of 10,000 cu m/hr gas handling capacity.

(6) After Purifier, 2 towers, operating @ 190°C, parallel flow, each of 40000 cu m/hr gas capacity.

(7) Converter, steel construction, for conversion CO to CO_2 H_2, using steam and I.G. catalyst (FeO, MgO, etc.) placed on 4 shelves. (Catalyst Life - 2 yr).

(8) Gas to Gas Heat Exchanger

.....

removal was effected by passing the gas in parallel flow through 6 towers, packed with luxmasse; two additional towers were held in reserve. The 6 towers in run have a purification capacity for 80000 cu m/hr of water gas. Gas from the towers entered booster blowers, 2 in run and 1 spare, each with a capacity of 40000 cu m/hr, and was delivered to a final purification unit consisting of 10 towers, each packed with 60 T of a mixture of 70% luxmasse and 30% anhydrous Na_2CO_3. These latter purification towers operated at 200-250°C (gas fired) with series flow through a pair of towers. Capacity per pair is 20,000 cu m/hr; 1 pair remains in stand-by condition. The gas next entered the final purification unit, which consists of 2 towers operating in parallel at 190-195°C and packed with reduced metals such as Fe, Cu, Ni, etc. This reduced sulfur content to 0.1 gr per 100 cu m of "synthesis gas." Capacity of the 2 towers is 80,000 cu m/hr of gas.

Gas from final purification was partly used without further treatment for blending with hydrogen enriched gas to form "synthesis gas."

Hydrogen enrichment was effected by subjecting a part of the normal purified water gas to the water gas reaction in a converter, steel construction, operated at 500°C and less than one atm pressure. The catalyst in this converter was a special I.G. type (Fe O, Mg O, etc.) held on 4 trays; its normal life is 2 years.

b. Normal Pressure Synthesis.
(Reference: Flow Sketch No. 2, Train B).

The "synthesis gas" fed to this operation was obtained by mixing normal purified water gas with hydrogen enriched gas to effect a H_2: CO ratio of 2:1 and was conditioned by diverting a portion in a gas to gas heat exchanger prior to entrance to converter (1).

Converter (1), all steel, is of box-type construction (See Fig 1) water cooled, and operated at 200°C (1°C) and 0.2-0.3 atm gauge.

It was packed between the vertical divider plates with 1-3 mm size particles of catalyst of following composition:

CO (as metal)	30%
ThO_2, MgO mixture	10%
Kiesel guhr	60%

Gases from converter (1) were cooled by water spray directly into a tower, which resulted in condensing an oil of 150-360°C boiling range from the gas stream. The oil and water layers were continuously withdrawn at this point. The non-condensed vapors then flow through a steam-heated, funnel heater to preheat them to 180°C prior to entering second converter (4) which was packed with same catalyst as in (1). Operating temperatures were raised from 180 to 200°C over the life of the catalyst, but were held within 10°C during conventional operation. Synthesis of a large portion of the residual gas into oil products was done here. A second condensation and separation of oils was carried out in condenser (5) in the same manner as above.

The remaining gases passed through towers packed with activated carbon which absorbed low molecular weight oils, C_3 and C_4 hydrocarbons, equal to 3% of the weight of the carbon, from the gas stream. Towers operated on stream in 1/2 hour cycles; during the off-stream cycle adsorbed materials were stripped by steam blowing the carbon. The residual gases were used as fuel.

Product was largely straight chain paraffins, with a large quantity of liquid, and small quantities of gases and solids.

c. Medium Pressure Synthesis.
(Reference: Flow Sketch No. 3, Train C)

This process operated at 11 atm abs on a synthesis gas with a H_2:CO ratio of 2:1, but portions of the hydrogen rich gas were introduced between the first and second converters and between the second and last converters. This process may be operated with either Co or Fe catalyst; Co catalyst was used at Ruhr Chemie. With Co the operating temperatures were 190-200°C and the product distribution fairly equal in both liquids and solids. With iron catalyst, the operating temperatures were 225-240°C and the products were largely liquids with small quantities of solids and gases and more unsaturates than were obtained with Co.

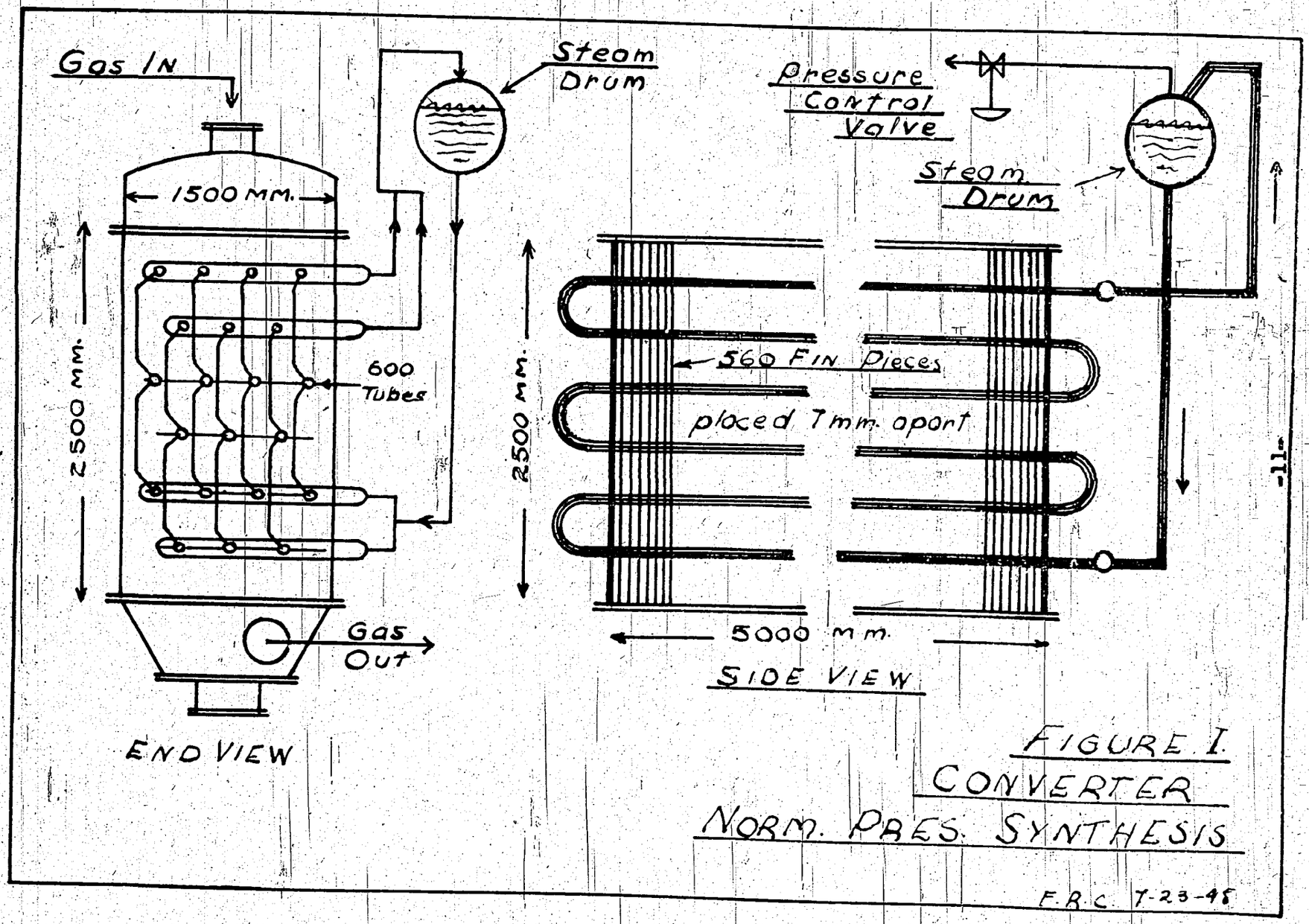
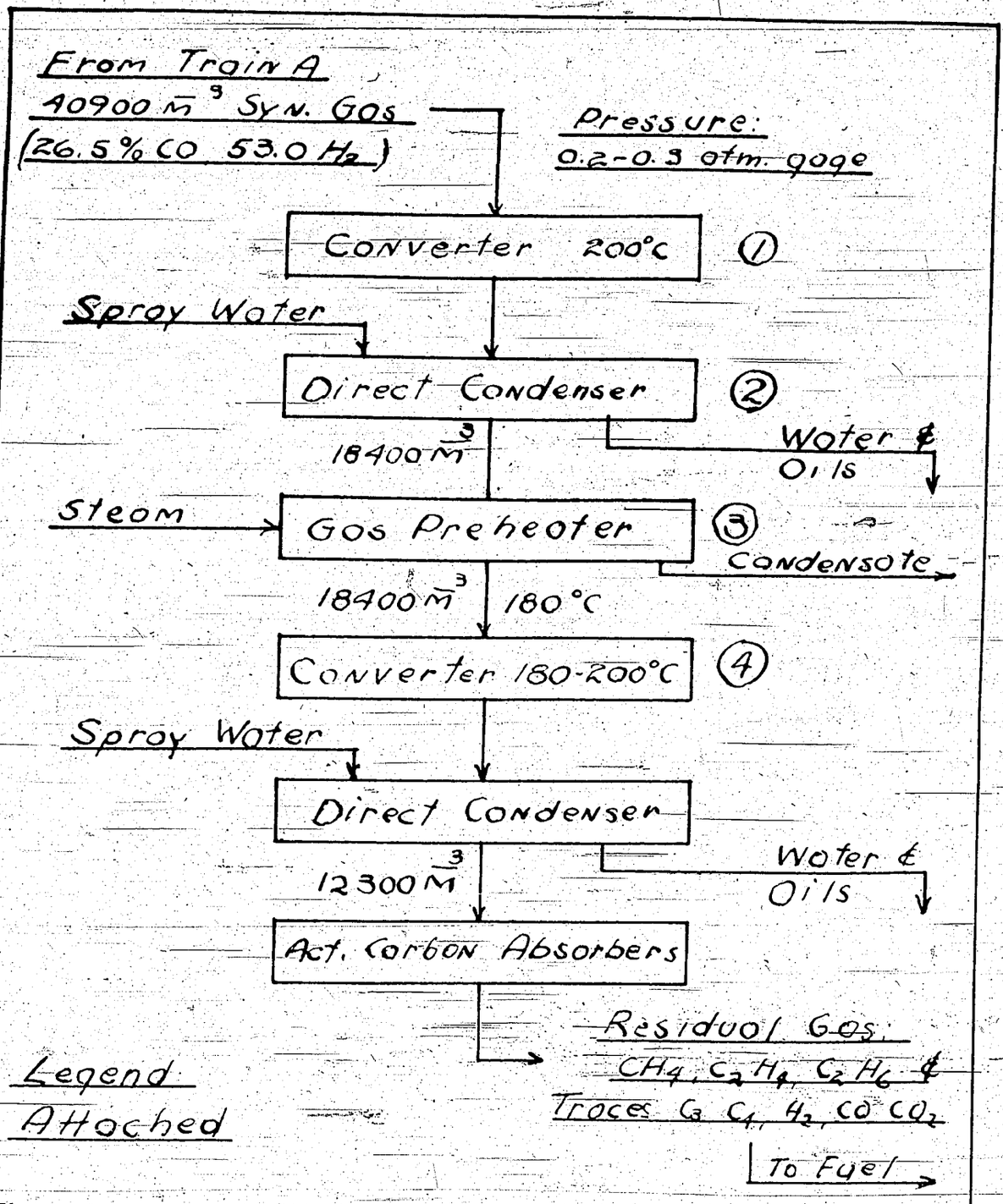


FIGURE I.
CONVERTER
NORM. PRES. SYNTHESIS

F.R.C. 7-23-45



FLOW SKETCH NO. 2 TRAIN B

NORMAL PRESSURE SYNTHESIS

HOURLY FLOW RATES

F. B. L.
7-22-45

LEGEND FOR TRAIN B. (p. 12)

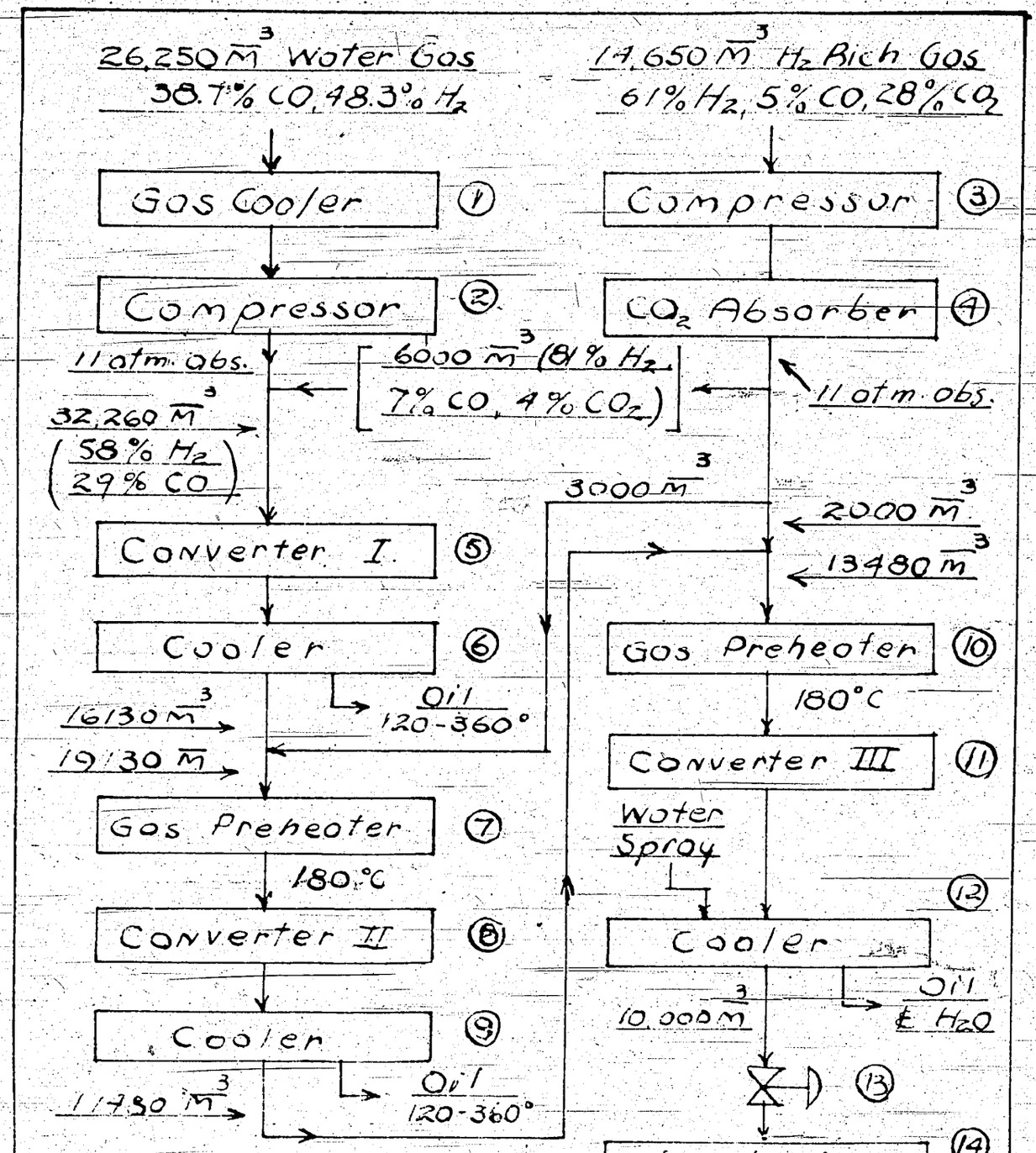
(1) Converter I, packed with catalyst consisting of 30% Co (as metal equivalent), mixture of 10% ThO_2 and MgO, 60% Kieselguhr, and water cooled for close (1°C) temperature control.
(2) Condenser, direct water spray type.
(3) Preheater, tubular, finned, condensing steam in tubes.
(4) Converter II, construction and catalyst same as in item (1).
(5) Condenser, same as in item (2).
(6) Absorbers (2) packed with activated carbon, cyclic operation, followed by steam stripping of adsorbed oils from carbon.

LEGEND FOR TRAIN C. (p. 14)

(1) Gas cooler, for water gas portion of feed.
(2) Gas compressor, discharging @ 11 atm abs.
(3) " " , for hydrogen rich portion feed gas.
(4) CO_2 Absorber, (CO_2 absorbed in water under pressure).
(5) Converter I, Tubular type, packed with catalyst consisting of 30% Co (as metal equivalent) 10% as a mixture of ThO_2 and MgO, 60% Kieselguhr, and water cooled with water for close temperature (1°C) control. (See Sk converter for medium pressure synthesis.
(6) Cooler and condenser, shell and tube, gas in tubes, water in shell.
(7) Gas preheater, shell and tube, steam in shell.
(8) Converter II, same as item (5) packed with same catalyst.
(9) Cooler and condenser, same as item (6).
(10) Gas Preheater, same as item (7).
(11) Converter III, same as item (5), packed with same catalyst.
(12) Cooler and condenser, direct type, employing water spray counter to gas flow in vertical tower.
(13) Expansion valve (reduction pressure to 1 atm abs)
(14) Activated carbon absorbers (2), operating in 1/2 cycles to adsorb as 3% of wt of carbon, oil constituents, which are desorbed by direct steam blowing during off-run cycle.

.....

Cooled water gas entered gas compressor (2) and was discharged at 11 atm abs to a mix-tee where hydrogen rich gas from compression and CO_2 removal units (3) and (4) was admixed to produce synthesis gas for Converter (1), where reaction was effected with conversion to the extent of 50% at 180-200°C, depending upon age of catalyst. This converter is constructed of steel, shell and tube design, water cooled, with special annulus - type catalyst holding tubes (see Fig. 2). Water cooling is used for a temperature control of 1°C. Catalyst of size and composition described in (b) above, was packed in the annular



TRAIN C.

FLOW SKETCH NO. 3

PRESSURE SYNTHESIS

HOURLY FLOW RATES (N.T.P.)

F. A. L.
7-22-45.

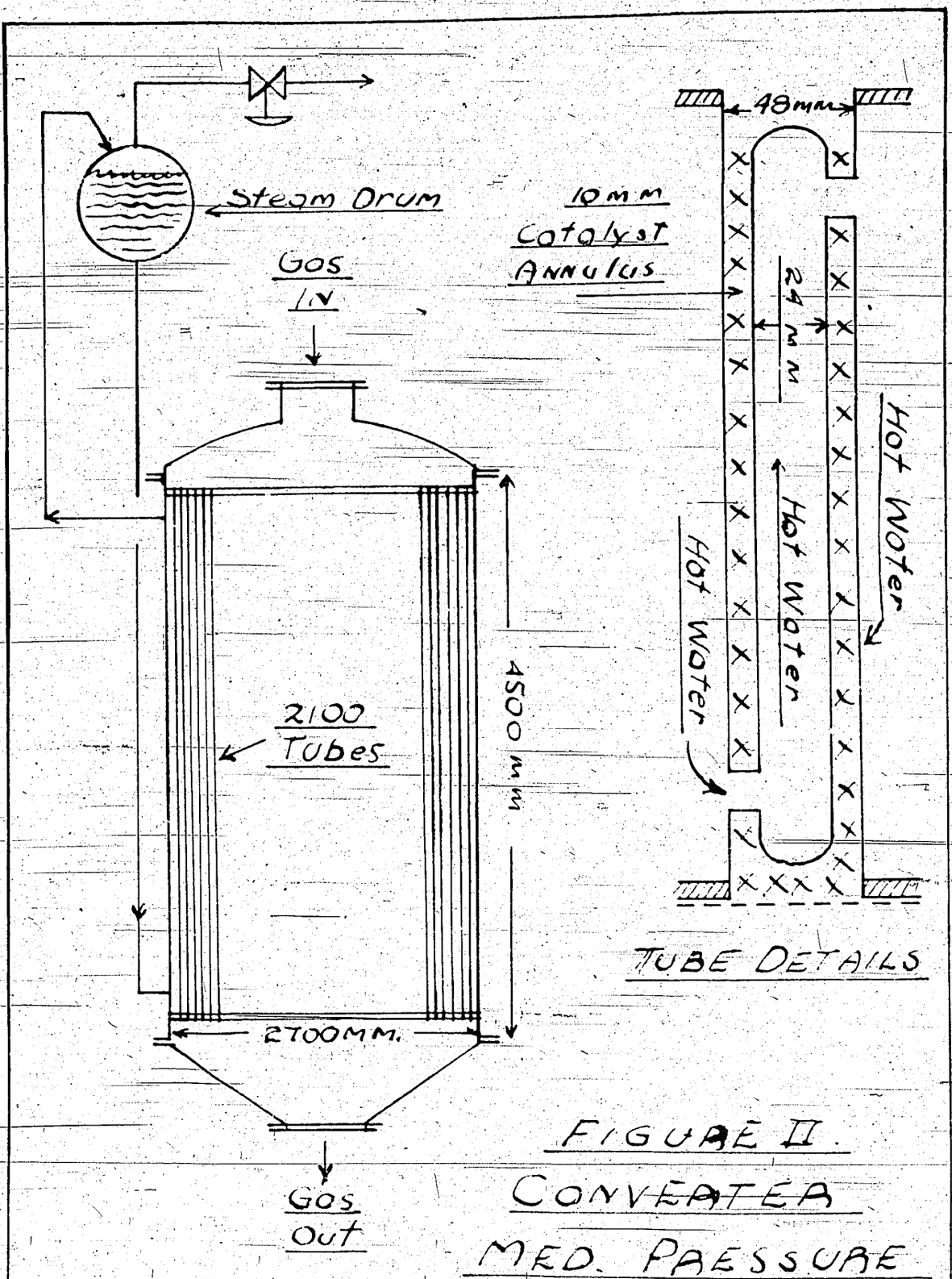


FIGURE II.
CONVERTER
MED. PRESSURE
SYNTHESIS

F.R. 7-24-45

section of the double concentric tube. All converters were of identical construction and were packed with same type catalyst.

Oil products of a boiling range 120-360°C were condensed from gas from Converter (1) in an indirect Condenser (6), the condensed oil layer being continuously withdrawn. Gases from Condenser (6) were refortified with additional hydrogen rich gas and the mixture then preheated to 180°C in steam heated preheater (7) prior to entering Converter II.

Converter II also was operated over the temperature range of 180-200°C to effect a 30% conversion of feed gas. Oil product of boiling range 120-360°C was condensed from the gas stream in condenser (9) from which it was continuously withdrawn. Exit gas was again fortified with additional hydrogen rich gas and the mixture preheated to 180°C in preheater (10) prior to entering Converter III.

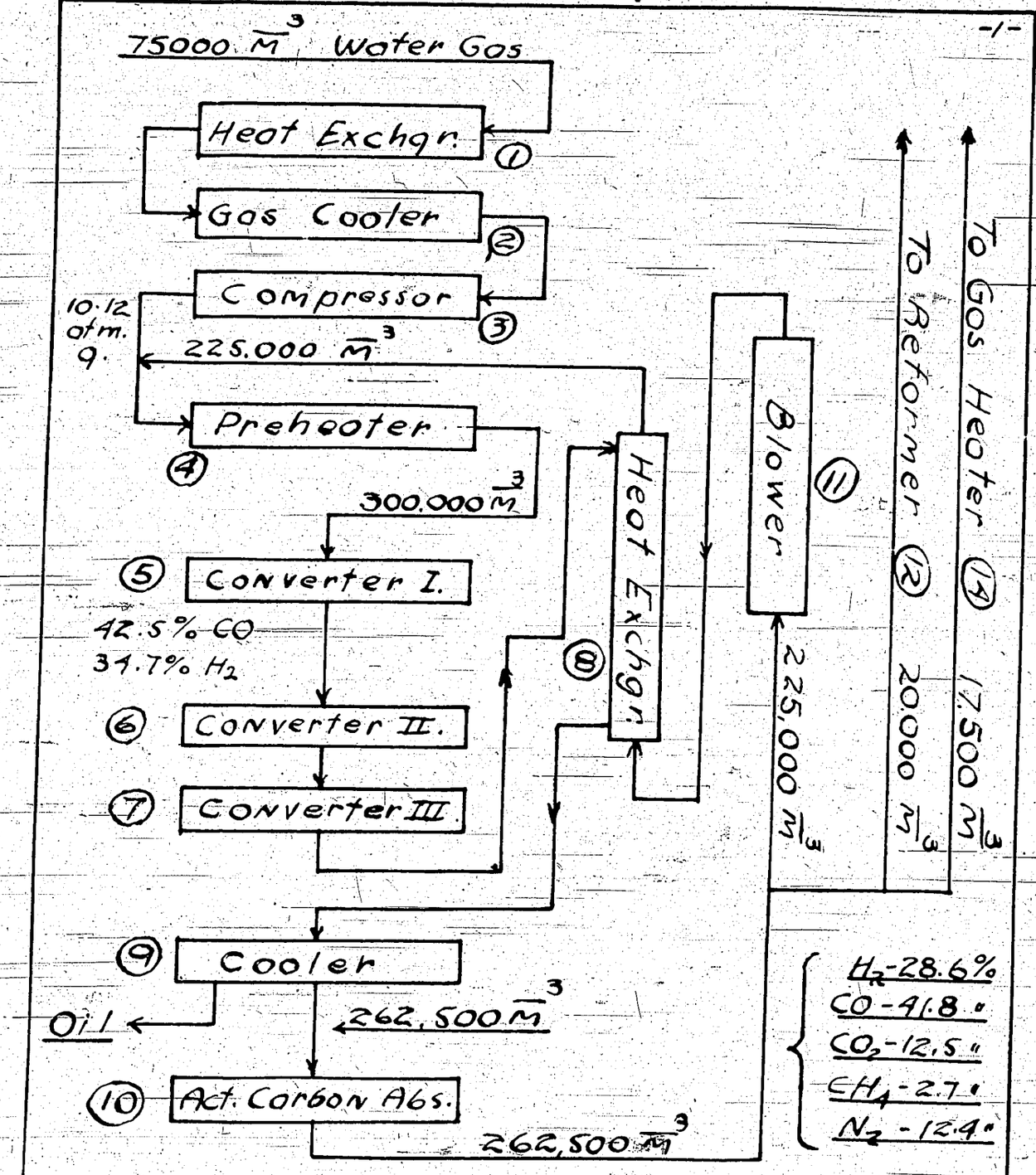
Converter III was operated similarly to the other converters to effect a 21.5% conversion of feed gas following which cooling, condensation, and oil separation steps, as previously described, were made.

Gases from the final cooler (12) passed through a reducing valve to cyclic-operating activated carbon absorbers, operated at atmospheric pressure, where low molecular weight oils and C_3, C_4 hydrocarbons were adsorbed, then desorbed by stream stripping as described (2) above. Residual gases were used as fuel.

d. Olefin Synthesis.
(Reference: Flow Sketch No. 4, Train D)

In this synthesis purified water gas was the feed stock, but the addition of three volumes of recycle gas to the feed gas prior to the conversion step resulted in a converter feed gas containing H_2: Co in the ratio of 1.00:1.23 together with CO_2, CH_4, N_2. Two converter trains were used, the first with three converters was operated at 10-12 atm gauge; the second was a conventional normal pressure train of two converters, operated at 1 atm gauge and 190-200°C and was employed to complete the synthesis from the residual gases from the first train. Catalysts in both trains were the cobalt type. The products from the first converter train contained more unsaturates and considerably less solid hydrocarbons than those from the second train (a normal pressure synthesis train, using CO catalyst).

Purified water gas was fed to the system through gas to gas heat exchanger (1), through gas cooler (2) and into suction of compressor (3) which discharged at 10-12 atm gauge to a mix-tee where 3 volumes of recycle gas mixed with the compressed water gas. The combined gas streams were preheated to 195-200°C in gas heater (4) and flowed in series to and through Converters I, II, III.



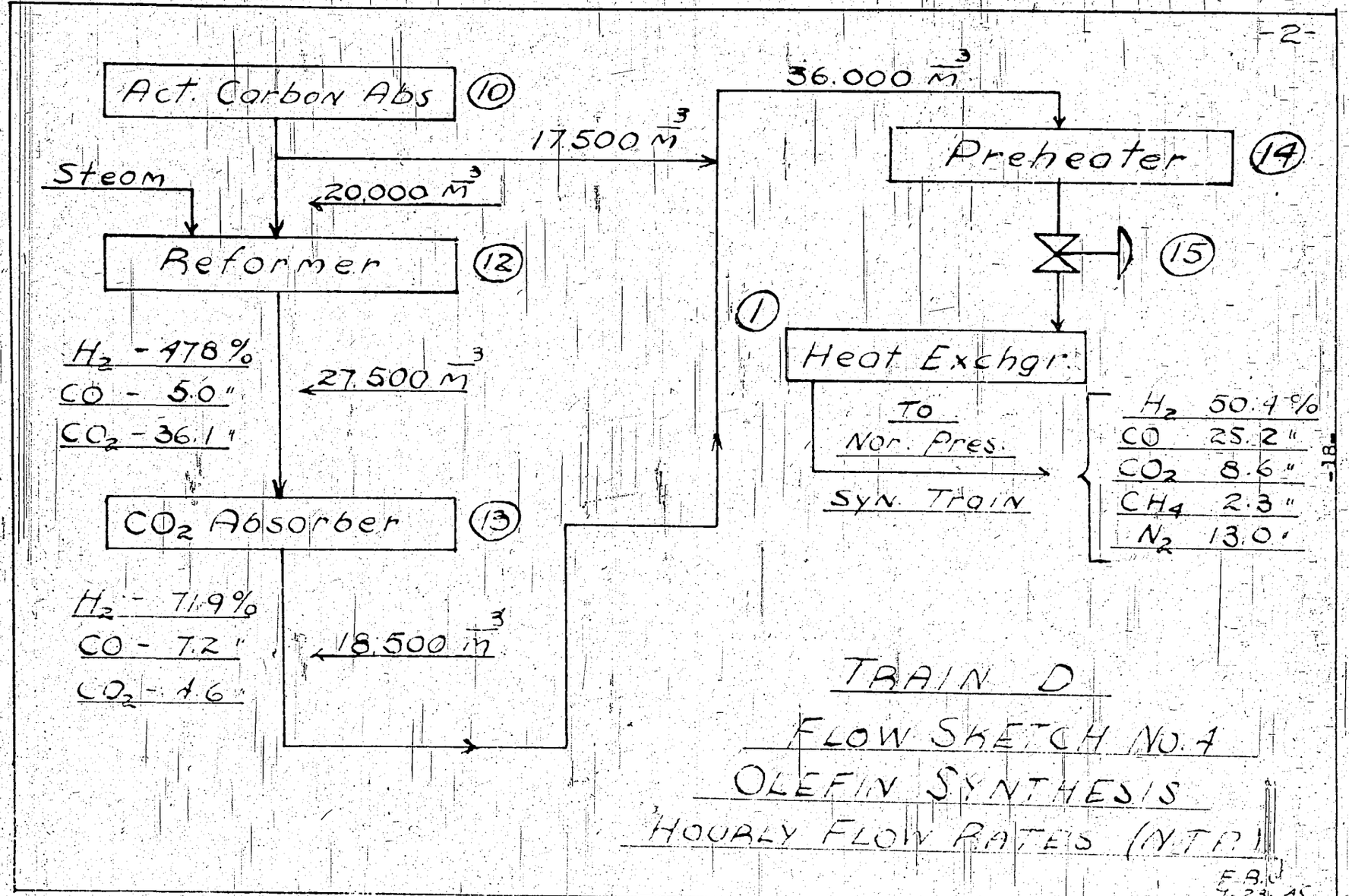
TRAIN D.

FLOW SKETCH NO. 4

OLEFIN SYNTHESIS

HOURLY FLOW RATES (N.T.P.)

FAL 7-19-45



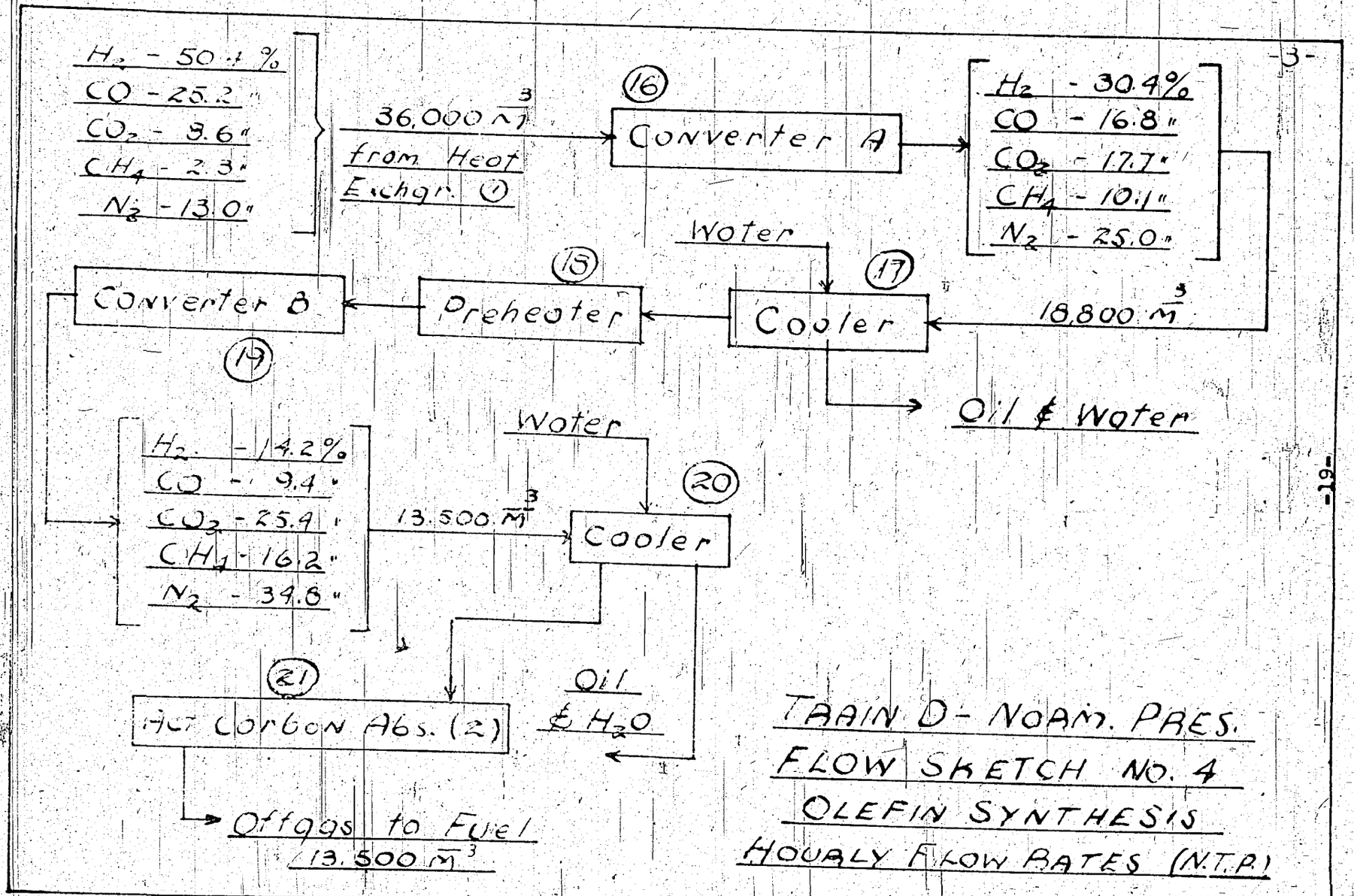
TRAIN D

FLOW SKETCH NO. 1

OLEFIN SYNTHESIS

HOURLY FLOW RATES (N.T.P.)

F.B.J.
7-23-45.



TRAIN D - NOAM. PRES.
FLOW SKETCH NO. 4
OLEFIN SYNTHESIS
HOURLY FLOW RATES (N.T.P.)

F.R.L. 7-23-45

LEGEND FOR TRAIN D.

(1) Gas to Gas Heat Exchanger.
(2) Gas cooler, shell and tube.
(3) Gas compressor, 20 atm max discharge pressure.
(4) Gas heater, shell and tube, steam in shell.
(5) Converter I, " " " (see sk "Medium Pressure Converter").
(6) Converter II, same as item (5).
(7) " III " " " "
(8) Gas to gas heat exchanger.
(9) Condenser and cooler, shell and tube.
(10) Activated carbon absorbers (2).
(11) Recycle gas blower.
(12) Reformer, (CO and steam reaction for H_2 production).
(13) CO_2 absorber (H_2O under pressure).
(14) Gas preheater, shell and tube.
(15) Pressure reducing valve.
(16) Converter A (See sk "Low Pressure Synthesis Converter").
(17) Cooler and condenser, direct water spray type.
(18) Gas preheater, tubular, finned, steam in tubes.
(19) Converter B, same as item (16).
(20) Cooler and condenser, same as item (17).
(21) Activated carbon absorbers (2).

.....

Converters were operated at 200-210°C, depending upon the age of catalyst. Converter construction and design were identical with those used for medium pressure synthesis (see Fig. 2). Space velocity was 80 cu m gas per cu m catalyst volume per hr.

Converter exit product passed through heat exchanger (8) counter current to recycle gas; thence to condenser and cooler (9) to effect condensation of oil from the gas stream; the condensed oil was continuously withdrawn from this unit. The exit gases were further stripped of oil products by activated carbon absorber units (10) which were operated as previously described.

Gas from absorbers (10) was split into three streams - (a) the largest portion (recycle gas) was delivered to recycle blower (11) which discharged it at 10-12 atm gauge through heat exchanger (8) to mix with incoming feed gas ahead of gas heater (4); (b) a second portion flowed directly to gas heater (14) for preliminary conditioning for the normal pressure synthesis; (c) a third portion was directed through reformer (12) where the catalytic water gas reaction occurs, resulting in hydrogen enrichment and CO_2 formation. CO_2 was scrubbed out in CO_2 absorber (13) and the scrubbed gas was mixed with portion (b) prior to passage through gas heater (14) and pressure reducing valve (15) then through heat exchanger (1) in counter current flow with hot, purified water gas.

The resulting conditioned gas was fed to a normal pressure synthesis train, which was identical in design and operation with that described in (2) above.

Yields were experienced as follows:

Train 1 - 10 gr liquifiable gas) per cu m feed gas
120 gr liquid products)

Train 2 - 30 gr total products " " " " "
160 gr liquid and gaseous products " " " " "

CHEMISCHE FABRIK HOLTEN, HOLTEN-STERKRADE.

1. GENERAL.

This concern is located adjacent to Ruhr Chemie and is owned jointly by the latter, I.G. Farben, and Th Goldschmidt A.G., each holding a 1/3 interest. Managing and technical personnel were not present although the general manager, Dr. Fuller, was expected to arrive shortly.

The plant has been badly damaged.

2. OPERATIONS.

The company purchased ethylene and propylene from Ruhr Chemie and converted them into the oxides by the chlorhydrin process. The capacity was 500 to 600 tons per month, 90% of which was ethylene oxide and 10% propylene oxide.

The finished materials were shipped to one of the Goldschmidt plants for conversion into the respective glycols.

OXO GESELLSCHAFT m.b.H., HOLTEN-STERKRADE.

1. GENERAL.

(a) The company was organized in order to exploit the discovery of Dr. Roelen of Ruhr Chemie, which consisted of the formation of aliphatic aldehydes by catalytic reaction of water gas on olefines followed by the reduction of the aldehydes to alcohols. I.G. Farben and Henkel and Co. were both interested in these alcohols for production of detergents by sulfonation. These two companies therefore joined with Ruhr Chemie in the formation of the new company, each participating 1/3. Since Ruhr Chemie furnished the raw materials, the plant was built adjacent to its property. The finished products were to be shipped to the plant of Henkel and Co. in Witten (Ruhr) to be converted into detergents by sulfonation.

Construction of the plant was about completed at the time of the severe bombing attack in October and November, 1944, but it had never operated.

(b) Bomb Damage.

The bomb damage was relatively slight probably not exceeding 15-20% of the investment. As was the case in the Ruhr Chemie plant, the equipment installed underground, presumably bombproof, consisting of tanks and pipelines suffered a good deal more than the tall reactors and distillation columns above ground. This was caused by extensive cave-ins which demolished the equipment.

(c) Organization.

The investment of the rather lavishly constructed plant was RM 11,000,000. The general manager, Dr. Landgraf, remained in Holten-Sterkrade after the bombing and is now engaged in rebuilding the plant.

Annual production: 10,000 tons higher aliphatic alcohols C_{12}-C_{18}

Estimated Cost of Product: RM .90-.95/kg

Market Price: RM 2.50/kg

Raw Materials: Quantities & Cost.

Power:	1,500,000 KWH /yr at RM .025-.03
Water:	200 cbm/hr
Watergas:	750 cbm/hr at RM .03-.035/cbm
Olefins:	1,250 kg/hr at RM .38-.40/kg
Hydrogen:	400 cbm/hr at RM .60-.70/cbm
Steam:	300 tons/day

2. PROCESS.

An olefin fraction from C_{11} to C_{17} was treated catalytically with purified water gas at medium temperatures under pressure to produce aldehydes which contain one more carbon atom than the parent olefins. The olefins may be derived either directly from the Fischer Tropsch synthesis or by Dubbs cracking of higher paraffins from C_{20} to C_{40} which were produced by Ruhr Chemie. The olefins should be predominately straight chain with the double bond at the end. The reason for this is that sulfonated alcohols derived from the other types of olefins have too low a melting point for the desired commercial uses although their detergent properties may not be impaired. The reaction was carried out batchwise, at temperatures of 125°C initially, 135°C at midstage, and 145°C at the finishing point, under 150 atm, using the standard Ruhr Chemie cobalt catalyst. The catalyst was finely ground to about 200

mesh and suspended in the olefin charge. The purified water gas, which was obtained from Ruhr Chemie, contained 38% CO, 48-49% H_2, with the balance being nitrogen and CO_2.

FLOWSHEET - (See Sketch Attached).

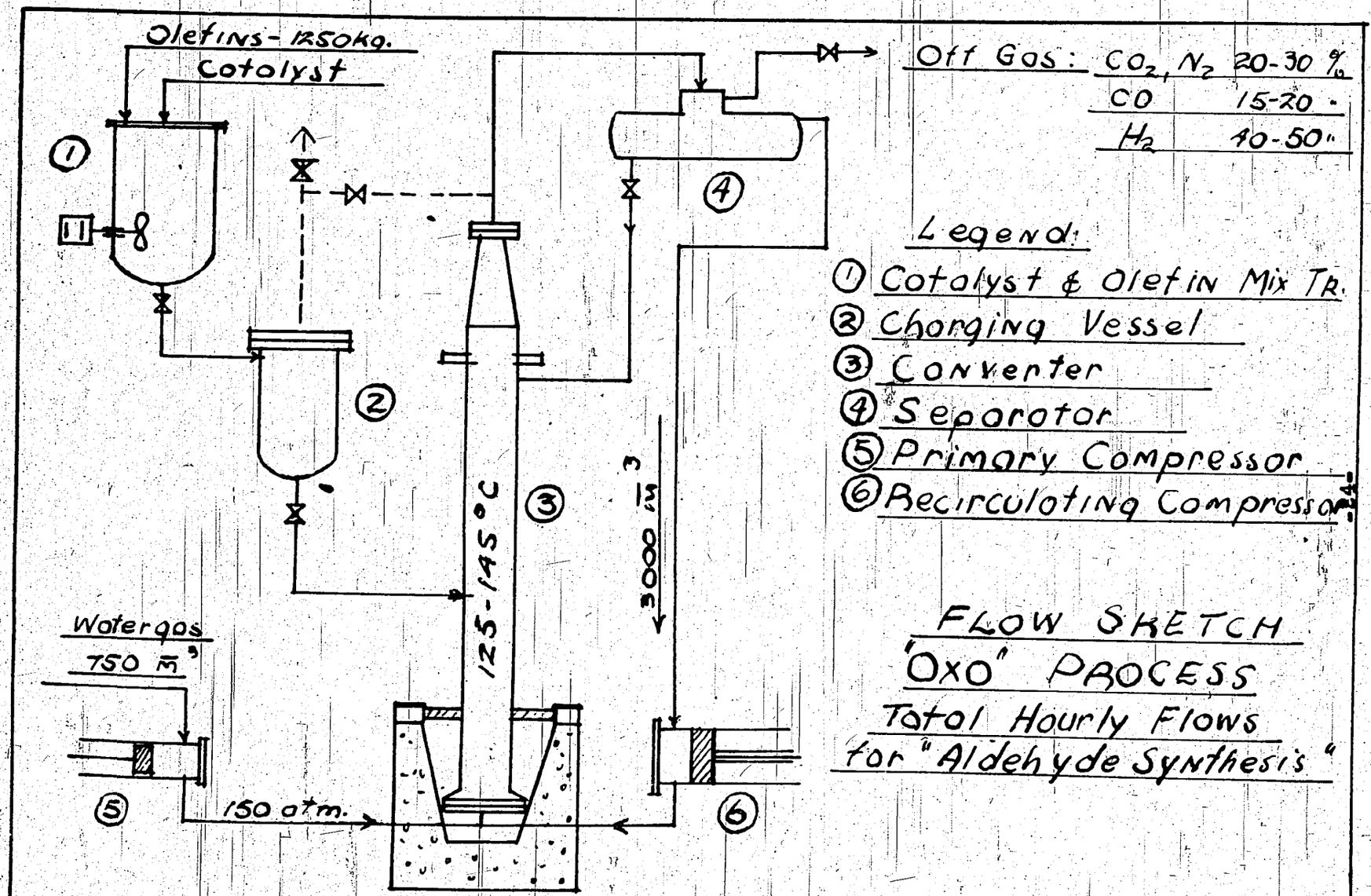
A batch charge of liquid olefins was mixed with the necessary catalyst in tank #1, transferred into charging vessel #2 after which the pressure was equalized with the system. The charge was then emptied by gravity into the converter #3, equipped with a bundle of bayonet heating and cooling tubes. Compressor #5 supplied water gas to the converter while its exit gas was largely recycled by compressor #6 and vented from separator #4 sufficiently to maintain adequate hydrogen and CO concentration within the system. The batch cycle was 1 hour, half of which was reaction time and the other half required for charging and discharging. Gas circulation was maintained during charging and discharging.

Batches of crude aldehyde were collected in storage and converted to alcohols by hydrogenation using the same catalyst and the same type of equipment as used for the aldehyde synthesis. The operation was also batchwise, with pressure of 150 atm and temperature of 180°C. Hydrogen of 90-92% purity (balance nitrogen) was used and recycled similar to the watergas in the aldehyde synthesis.

Of the 9 converter units installed, 5 were to be used for aldehyde synthesis and 4 for hydrogenation.

The crude alcohol was purified by vacuum distillation in batch stills and plate columns.

1 pound of olefin was expected to yield approximately 1 pound distilled alcohol.



Off Gas: CO_2, N_2 20-30 %
CO 15-20 "
H_2 40-50 "

Legend:

1. Cotalyst & Olefin Mix Tk.
2. Charging Vessel
3. Converter
4. Seperator
5. Primary Compressor
6. Recirculating Compressor

FLOW SKETCH
"OXO" PROCESS
Total Hourly Flows for "Aldehyde Synthesis"

F.A.L.
7-21-45.

Copy 1

ITEM No. 30

FILE No. XXXIII—40

NORDSTERN COKE-OVEN PLANT

GELSENKIRCHNER BERGWERKS A.G.

Neumann, R. and Bates, H. H.

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Dec. 1946

COMBINED INTELLIGENCE OBJECTIVES

SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

NORDSTERN COKE-OVEN PLANT

GELSENKIRCHNER BERGWERKS A. G.

Reported By

R. Neumann (Br)
H. H. Bates (Br)

on behalf of

BRITISH MINISTRY OF FUEL AND POWER

and

U. S. TECHNICAL INDUSTRIAL INTELLIGENCE COMMITTEE

CIOS Target No. C30/21
Fuels and Lubricants
21st April, 1945

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE.

G-2 Division, SHAEF (Rear) APO 413.

TABLE OF CONTENTS.

SUBJECT	PAGE NO.
DESCRIPTION OF PLANT	1
BY-PRODUCT PLANT	2
BOMB DAMAGE	4
FLOW SHEET	5

Personnel of Team.

R. NEUMANN, BRITISH, Ministry of Fuel and Power.

H.H.BATES, " " " " " "

A.R.POWELL, U.S. Petroleum Administration for War.

PERSONNEL INTERROGATED

Assessor Dütting
Dr. Ochs - Coke Oven Assistant to Assessor Dütting
Herr Eisenhüt - Manager of Plant

DESCRIPTION OF PLANT

The plant consists of 4 Batteries of Still 6 metres high ovens, No. 1 being built in 1928, No. 2 in 1930 and Nos. 3 and 4 in 1938 and was at that time the largest privately owned plant in Germany. In addition it had a number of novel features not found elsewhere.

Number of Ovens — Battery 1 - 42; 2 - 54; 3 - 42; 4 - 54

Coal Throughput — 6,000 tonnes per day

Coke Output — 4,400 tonnes per day

Coal Blend — 75% Fettkohle — 25% Flammkohle

Analysis of Blend

H_2O	10%
Ash Dry Basis	6%
Volatile Matter (Dry Basis)	26 - 27%
Sulphur	1.6%

Grading — 82% < 3 mm

Bulk Density — 750 Kgrm/cum.

Gross Coking Time — 22 hours

Coal Handling Plant

Tippler into receiving bunkers. The two coals are mixed on conveyor belt and pass through Carr Type Crushers before being elevated to the service bunkers.

Underfiring

During the week the underfiring is by means of producer gas generated in Demag producers. Before being used it is cleaned by means of Disintegrator Type Apparatus.

BY-PRODUCT PLANT

As will be seen from the attached flow diagram, the gas leaves the ovens in two parallel streams. Both streams are treated in a similar manner until after the ammonia scrubbers. After this point all gas sold is purified and treated in the high pressure benzole recovery plant whereas any gas used for underfiring the ovens passes unpurified through the low pressure benzole scrubbers. During the week all gas made is sold and therefore the low pressure benzole scrubbers are not used. During weekends, however, a portion of the gas is used for heating the ovens.

The high pressure benzole scrubbing system (8 atmospheres) was installed solely on account of the gas pressure in the grid being 8 atmospheres.

Primary Coolers

Gas leaving ovens at 100° is cooled to 35°C by Water Tube Coolers.

Exhausters

Each stream has its own pair of exhausters - one of each pair electrically driven, the other being steam turbine driven.

Secondary Coolers

Further water tube coolers reduce the gas temperature to 20°C.

Ammonia Recovery

The indirect system of Ammonia recovery is used - the apparatus consisting wholly of tower scrubbers. In the first scrubber through which the gas passes a strong ammonia liquor is circulated to remove cyanogen compounds. The liquor is dephenolated by means of benzole washing before being distilled. The phenol is removed from the benzole by means of caustic soda, the sodium phenolate being sold. The "devil gas" from the saturators is treated by the "Lurgi" Contact process and 8 - 10 tons per day H_2SO_4 are obtained. It is estimated that some 60% of the H_2S in the gas is removed along with the ammonia liquor. It was intended to change part of the production from Sulphate of Ammonia to 25% Concentrated Ammonia liquor and the necessary plant was on order.

H_2S Removal

By means of iron oxide purification material. Partly in deep boxes and partly in tower purifiers. Purification is down to 0.3 grms H_2S/100 cubic metre.

Compressors.

Rotary machines by A.G. compressing to 8 atmospheres.

High Pressure Coolers.

Water tube coolers re-cooling the gas to 20°C.

Benzole Scrubbing.

Tower scrubbers are used in both high and low pressure benzole scrubbing systems. Creosote oil is used but no mixing of the oils used in the high and low pressure system takes place. It was stated that the oil used in the low pressure system (unpurified gas) thickened at approximately twice the rate (per unit of gas) of the thickening in the high pressure system.

Final Purification.

After leaving the high pressure benzole scrubbers, the gas is passed through a set of "Lurgi" Active Carbon absorbers where the last traces of benzole and all organic sulphur is removed. The set consists of 6 chambers each containing 10 tonnes carbon-4 absorbing and 2 steaming, drying and cooling. Spent active carbon is removed from the bottom of each chamber daily and fresh added at the top. The amount of the daily change is such that the life of the carbon is 6 months.

The plant which was reported as having been transferred to "Alma" was a similar set of absorbers to those mentioned above.

Benzole Plant.

The benzole plant is also divided into two sections, the oil from the low pressure scrubbers being kept separate from that from the high pressure scrubbers. The crude benzoles from the two plants are mixed with the light benzole from the active carbon plant although it was intended that the two crudes would be separated as soon as the Ethylene and Methane extraction plant was in operation and only the H_2S free benzole from the high pressure plant would be used for that purpose.

No benzole restification was carried out and the crude benzole - 96% distilling at 180°C was sold to Gelsenberg-Benzin A.G.

Ethylene and Methane Extraction Plant.

This plant was very nearly complete and would have been in operation within one month from February 28th 1945, when the plant was put out of action by bombing.

The process which was designed by Karl Still was new and was in the nature of an experiment.

1,700,000 cubic metres per day of the gas leaving the high pressure coolers at 20°C after being cooled by the return gas in heat exchangers was to be further cooled to 4°C and scrubbed with sulphur free crude benzole at 8 atmospheres pressure. The rate of benzole circulation was to be 500 cubic metres per hour. The Ethylene and Methane rich benzole was to be distilled and the vapours compressed to 42 atmospheres before fractionation into Ethylene, Methane and Treibgas.

The intense cooling was to be attained by ammonia refrigeration plant.

The Ethylene was to be boosted to Hüls for rubber synthesis. The methane and Treibgas were to be compressed into cylinders for any suitable purpose.

The estimated daily yields were as follows:-

Ethylene	24 tonnes
Methane	28 "
Treibgas	7½ "

The following were estimated consumptions:-

Steam	26 tonnes per hour.
Power	2060 K.W. per hour.
Fresh Water	845 cubic metres per hour.
Recirculation Water	735 " " " "

BOMB DAMAGE.

It was stated that before February 28th 1945 when the plant was completely put out of action, that on several occasions slight bomb damage had occurred but was repaired in a short time. The raid on February 28th, however, was so severe that it was impossible to bring the plant into operation again.

Coal Handling Plant.

Although the bunkers were standing, the conveyors were completely destroyed.

Batteries.

The two old batteries(1 and 2) were intact but battery No.4. had received a direct hit and would require re-building from pad level. No.3. battery had near misses and it was estimated that the end 10 ovens would require re-building from pad level and the remainder from sole level.

1050 S.P.B.Co Ltd 22·7·46 50035

FLOW DIAGRAM OF NORDSTERN COKE

COAL

BATTERIES

COKE SCREEN

COKE

GAS PRODUCER PLANT

COOLERS

COOLERS

GAS FOR UNDERFIRING

EXHAUSTERS

EXHAUSTERS

COOLERS

COOLERS

SEPARATOR

CRUDE TAR

WATER

AMMONIA SCRUBBERS

AMMONIA SCRUBBERS

AMMONIA LIQUOR

AMMONIA LIQUOR DEPHENOLATION PLANT

BENZOL WASHER

SODIUM PHENOLATE

CAUSTIC SODA

BOOSTERS

L.P. BENZOL SCRUBBERS

AMMONIA STILLS

WASTE LIQUOR TO EMSCHER CANAL

SULPHURIC ACID

OXIDE PURIFYI... MATER...

PURIFI...

GA... HOLD...

SATURA...

AMMONI... SULPH...

W DIAGRAM OF NORDSTERN COKE OVEN PLANT.

WATER

CAUSTIC SODA

SULPHURIC ACID

OXIDE PURIFYING MATERIAL

CREOSOTE OIL

LURGI ACTIVATED CARBON

AMMONIA SCRUBBERS

AMMONIA SCRUBBERS

BOOSTERS

L.P. BENZOL SCRUBBERS

PURIFIERS

GAS HOLDER

GAS HOLDER

COMPRESSORS

H.P. COOLERS

H.P. BENZOL SCRUBBERS.

ACTIVATED CARBON ABSORBER

BOCHUMER - VEREIN

S. FREE GAS SUPPLY

RUHRGAS A.G.

ONIA LIQUOR

AMMONIA LIQUOR DEPHENOLATION PLANT

BENZOL WASHER

AMMONIA STILLS

SATURATORS

LURGI CONTACT PROGRESS

DEVIL GAS

SULPHURIC ACID

ETHYLENE METHANE PLANT

L.P. BENZOL PLANT

H.P. BENZOL PLANT

WASTE LIQUOR TO EMSCHER CANAL

SODIUM PHENOLATE

AMMONIUM SULPHATE

CRUDE BENZOL

Apparatus.

Quite a lot of apparatus was intact although the buildings were completely destroyed. The low pressure benzole scrubbers and ammonia washers were badly holed and shaken from their foundations. The exhausters and high pressure apparatus was intact. The gasholders were destroyed. The steelwork and mains were a tangled mass and this in itself was sufficient to put the plant out of commission for some time.

The Ethylene and Methane plant was destroyed

Classification Cancelled,
by authority of
The Joint Chiefs of Staff,
by Col. R. E. Gruber.
1 WH

ITEM No. 22
FILE No. XXX-63

COPY No. 3

RESTRICTED

STYROFLEX

A PLASTIC PRODUCED BY

NORDDEUTSCHE SEEKABELWERKE

Prentice, T. M.

RESTRICTED

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

RESTRICTED

STYROFLEX

A PLASTIC PRODUCED BY NORDDEUTSCHE SEEKABELWERKE

Reported by

Lt. Cmdr. T.M. PRENTICE, USNR
NavTecMisEu

CIOS TARGET NO. 22/1405
Miscellaneous Chemicals

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

RESTRICTED

TABLE OF CONTENTS

Subject	Page No.
Summary	3
Introduction	3
Composition	3
Manufacture	3
Properties	4
Uses	5
Form in Which Manufactured	5
Finished Product	6

SUMMARY

This report describes a clear transparent plastic known as Styroflex derived from POLYVINYL BENZOL manufactured by the Norddeutsche Seekabelwerke. The Physical, chemical, and electrical properties are listed.

STYROFLEX

A PLASTIC PRODUCED BY NORDDEUTSCHE SEEKABELWERKE

Introduction

"Styroflex" is a trade name for a clear transparent flexible plastic manufactured by the Nordeutsche Seekablewerke. The company developed this product for use as an insulating material between the core and the shield of co-axial submarine cables. This use is described in U. S. Naval Technical Mission in Europe Technical Report No. 272-45, entitled "Submarine Cables." The purpose of this report is to describe the material more fully, list its physical, chemical, and electrical properties and to discuss other uses to which it has been put.

Composition

Chemically "Styroflex" is believed to consist of Polyvinyl Benzol.

Manufacture

The material from which "Styroflex" is produced is an I.G. Farben product known as "polystrol" which is delivered to the Norddeutsche Seekabelwerke in granular form about the coarseness of sand. This material which arrives in drums and is poured into machines which melt and mix it and then extrude it in the form of a band about 3/8 inches thick and 1 3/4 inches wide. This band is colorless, transparent and hardens on cooling to a tough brittleness. It is tougher than glass but can be shattered by a sharp blow. Sample (A) shows the material in this form. This particular sample is discolored but otherwise typical. This is known as "Polystrol".

"Polystrol" can be remelted and molded into any shape and is suitable for a variety of uses. This company uses it to form the head and screw-cap for flexible tubes for paste products. For this purpose the polystrol is colored and rendered opaque.

To produce "Styroflex", the bands of polystyrol are remelted and extruded. It comes from the die in tubular or cylindrical form. The continuous tube is drawn out and flattened on the machine and then cut at the bends so as to form two flat sheets which are then trimmed to uniform widths, cut into strips of the desired width and wound on spools ready for packing for shipment or for further use in the factory.

As described by Dr. Boos, the Technical Director of the plant, the secret of the manufacture of "Styroflex" from polystyrol is in the handling as the material is drawn out into sheets as described above. It is understood from his description of the process that the material does not change chemically but only mechanically. The "Styroflex", whether in the form of sheets, strips or strands, is flexible and no longer brittle as is the polystyrol. This change takes place during the process of remelting and extrusion.

Properties

Sample (B) is a booklet giving the properties of "Styroflex" in German and containing samples of the material in sheet and strand form in various thicknesses and diameters. The translation into English is as follows:

"Physical, Chemical and Electrical Properties of Styroflex"

Ultimate tensile strength	Approx. 700 kg/cm^2 (9950 lbs/in^2)
Extensibility	Approx. 3.0 to 4.0%
Ultimate compression strength	Approx. 950 kg/cm^2 (13500 lbs/in^2)
Specific gravity	1.05
Capacity to resist heat	60-70 "Martensgrade" standard
Combustibility	High (VDE standard)
Capacity to resist radiant heat	First Quality (VDE standard)
Water absorption after 7 days	0
Odorless and tasteless	
Resistant to	Acids, bases, alcohol, mineral oil, vegetable and animal oils.

Soluble in	Gasoline, benzol, ether, turpentine, chloride, ethyl esters and ketone
Surface resistance	3 million megohms
Surface resistance after 4 days at 80% moisture	3 million megohms
Surface resistance after 24 hours in water	3 million megohms
Internal resistance	3 million megohms
Loss angle tangent delta for frequencies up to 1000 kc.	2×10^{-4}
Dielectric constant	2.5
Breakdown voltage	Approx. 100 kv/mm.
Permability to water vapor	Between 3 and 4×10^{-8} $\frac{\text{grams}}{\text{cm. hr. mm. mercury}}$

Uses

"Styroflex" is used in strip form for the insulation of electric cables for both land and underwater use. It is used as the dielectric in condensers. It is also used in combination with metal foil for the manufacture of tubes to contain paste products. It is believed that "Styroflex" and "Polystyrol" might be applicable to a variety of uses because of their resistance to acids, bases, alcohol and various oils.

Form in Which Manufactured

The booklet, Sample (B) lists the standard dimensions in which "Styroflex" is produced as follows:

Foil - From 0.02 to 0.15 mm. thick
Variation - average 10%, maximum 15%
Measurements to within 5/1000 mm.
in thicknesses over 0.10 mm.
Minimum width - 2.0 mm.

Thread - From 0.10 to 5.0 mm. in diameter
Variation - average 5%
Variation - maximum 10%
Measurements to within 1/1000 mm.
Other sizes are made on special order only.

Finished Product

Sample (C) is a finished roll of "Styroflex" 0.02 mm. in thickness.

Prepared by:

T. M. PRENTICE,
Lt. Comdr., USNR

Copy 1

B.I.O.S. FINAL REPORT No. 626
ITEM No. 30

DRYING, BRIQUETTING AND LOW-TEMPERATURE CARBONISATION OF BROWN COAL IN LURGI-SPÜLGAS RETORTS.

Bardgett, W.

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Bureau of Mines
DEC 1946

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BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

DRYING, BRIQUETTING AND LOW-TEMPERATURE CARBONISATION OF BROWN COAL IN LURGI-SPÜLGAS RETORTS.

at

BRAUNSCHWEIGISCHE KOHLEN BERGWERKE

SCHWELWERK OFFLEBEN near HELMSTEDT

Reported by

H. BARDGETT, Fuel Research Station, Dept. of Scientific and Industrial Research.

on behalf of

MINISTRY OF FUEL AND POWER.

BIOS Trip No: 2153

B.I.O.S. Target No. C30/343

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

32, Bryanston Square, London, W.1.

TABLE OF CONTENTS

SUBJECT	PAGE No.
PERSONNEL INTERROGATED	1
DATES OF VISITS	1
CONDITION OF TARGET	1
INTRODUCTION	1
DESCRIPTION OF PROCESS	2
COAL HANDLING	2
DRYING PROCESS	3
BRIQUETTING	4
CARBONISATION	5
GAS PRODUCERS	7
COKE TREATMENT	7
FIRE AND EXPLOSION PRECAUTIONS	8
BY-PRODUCT RECOVERY	8
TAR	9
LIGHT OIL	9
LIQUOR	9
DATA OBTAINED FROM PLANT PERSONNEL	11
DATA OBTAINED FROM MILITARY GOVERNMENT. HANOVER.	13
DATA OBTAINED FROM N.G.C.C.	14
COSTS	14
FIG.1. FLOW - SHEET OF PROCESS	
FIG.2. DIAGRAM OF LURGI - SPULGAS RETORT	

B.I.O.S. TRIP 2153

PERSONNEL OF TEAM

J.P. POSTLETHWAITE.	Ministry of Fuel & Power.
H. BARDGETT.	Fuel Research Station, D.S.I.R.
H.F. BONDY.	Ministry of Fuel & Power.

BRAUNSCHWEIGISCHE KOHLEN BERGWERKE

SCHWELWERK OFFLEBEN near HELMSTEDT

Personnel interrogated.

Dip. Ing. Voelmicke.	Manager
Dr. Knothe.	Chemist
Herr Wand.	Works Foreman

Dates of visits 4th and 5th May 1946.

Condition of target. Undamaged.

INTRODUCTION.

The information contained in this report was obtained as a result of interrogation of the above mentioned personnel of the B.K.B. and of discussions with Major E.E.J. Farmer, the representative at Helmstedt of the North German Coal Control, to whom the investigators are indebted for much valuable assistance during the visit.

The installation at Offleben consists of plant for the drying, briquetting and low-temperature carbonisation of the briquetted brown coal from adjacent open-cast workings. The Company controlled three local brown-coal mines, Treue, Wulfersdorf and Viktoria. The border line between the British and Russian zones of control passes near the site of the Offleben plant and at present the two mines first-mentioned are in the British zone whereas the Viktoria mine is in the Russian zone. The Company owned, in addition to the Offleben plant, several neighbouring works, Treue, Buddenstedt and 1st Bismark, producing brown coal briquettes for industrial and domestic use, and a power station at Harbke. Electric power for the Offleben plant is supplied from the Harbke power station, which is in the Russian zone, and steam supplies are from the briquetting factory at Treue.

The total carbonisation capacity of the plant is approximately 3000 tons (metric) of briquettes per day, while the full briquetting capacity is only 2500 - 2600 tons of briquettes per day. At times when the carbonisation plant was running to full capacity the additional briquettes required were drawn from the adjacent Treue briquette factory.

The brown coal, dried at the plant from a moisture content of about 48 to 12 - 13 per cent. by means of burned gas produced in the process, is briquetted in extrusion presses and then carbonised in Lurgi-Spülgas retorts. The installation includes 10 retorts and 7 driers. At the time of the visit 3 retorts and 2 driers were in use.

The coal used is mainly from the Wulfersdorf mine and is selected from the upper portion of the 14 metre thick seam, as this gives a higher yield of oil than is obtainable from the lower portion.

DESCRIPTION OF THE PROCESS

A flow-sheet for the complete process is shown diagrammatically in Fig. 1.

Coal Handling.

The lump coal, containing 48-50 per cent. moisture, is received at the works in drop - bottom wagons and is discharged to storage bays of capacity 5,000 tons. From the bays the coal is elevated by a rubber belt conveyor discharging on to grates consisting of horizontal rollers spaced to give a gap of 16 mm. A section of this conveyor is mounted on a weighbridge and the weight of coal carried on the belt is determined by periodically noting and recording the weight of the loaded section of the belt. This method of weighing was said to be accurate within ± 2 per cent. On discharge from the belt to the roller grate any tramp iron is removed by a magnetic separator. The fine coal below 16 mm. passes through the grate; the oversize, from which any stones etc. are first hand-picked, passes to a hammer-mill in which it is crushed to below 20mm. Fine coal passing through the grate, together with the crushed coal from the mill discharges on to a conveyor belt leading to the drying plant.

The installation for handling the coal includes two of the roller grates, made by A.W.Mackensen, Magdeburg, and two hammer-mills made by M.I.A.G., Brunswick. The latter are each of capacity 110 tons per hour and contain two horizontal shafts, rotating in opposite directions, driven by 100 K.W. motors. One mill is normally a stand-by.

It was stated that every 1½ hours it was necessary to open up the mill to remove accumulations of wet dust from the inner surfaces, a procedure which entailed stopping the mill for a period of ½-¾ hour.

In normal circumstances the coal handling system worked for 18 out of each 24 hours.

Drying process.

Drying of the coal from 48 to about 12-13 per cent. moisture is effected by means of hot inert gases in a drying plant having a circulating heating gas system - Umlauftrockner - designed by Büttner Werke A.G. of Uerdigen-Krefeld. The system is such that, as a result of rapid evolution of steam from the coal, the latter is not merely dried but is also considerably reduced in size.

The equipment includes seven driers for supplying the partially dried coal to the ten carbonisation units. At the time of the visit two driers were in use in conjunction with three retorts.

The drying system is shown diagrammatically in Fig. 1. Coal from the coal handling system is discharged to a cylindrical hopper through the centre of which passes a vertical pipe, of diameter 1,000 mm., made of special cast iron. The coal to be dried is fed from the base of the hopper into the vertical pipe and is carried upwards by a stream of hot inert heating gas. Gas produced by carbonisation of the briquettes, after passing through the by-product recovery system, is burned in a firebrick-lined combustion chamber. The burner used in the chamber is of Balcke-Bochum design and is such that a swirling motion is imparted to the gas-air mixture supplied for combustion. The temperature in the chamber is 1,000-1,100°C. and the products of combustion, containing 7-8 per cent. CO_2 and not more than 0.5 per cent. oxygen are drawn into the bottom of the vertical pipe and carry the coal upwards. The temperature of the gases entering the pipe is regulated by admitting to the stream leaving the combustion chamber a proportion of cooler gas from a later stage in the system. The mixture of gases enters the drying pipe at a temperature of about 700°C.

Coal of size over 4½mm. is removed from the stream by means of a 4½ mm. vibrator screen, the oversize from which is crushed in a roll-mill and returned to the drier. Fine coal carried forward in the gas is separated firstly in cyclones, the gas from which then passes through a Lurgi electrostatic precipitator, operated at 50,000 volts, in which the very fine particles are removed. A fan, 1640 mm. diameter, near the top of the vertical drier pipe provides the necessary suction of about 200 mm. water gauge at the fan inlet. The fan is driven by a 175 KW. motor and the roll-mill for crushing the over 4½ mm. coal by a 30 KW. motor.

One-third of the total volume of gas is drawn off at a position prior to the electrostatic precipitator and recirculated for the purpose of controlling the temperature of the inlet gas to the drier. The volume of gas passing through the precipitator is 40,000 M^3 per hour. This is discharged to atmosphere via chimneys extending 10 metres above the roof. The temperature of the gas entering the chimneys is about 96°C. The chimneys are of metal lagged externally with a layer 120 mm. thick of slag wool to minimise condensation of water and consequent corrosion. Originally the chimneys were lined with rubber but in most cases the lining is now perished. One chimney lined with rubber is, however, still in working condition after about 7 years use.

The amount of dust lost by discharge to atmosphere was said to amount to 30 kg. per hour from each of the seven drying units. When the plant was started in 1936 the driers were not equipped with chimneys, but these were fitted in 1939 to remove the fine dust from the vicinity of the plant.

The speed of the heating gas stream in the main drier pipe was said to be 30-35 metres per second. A section of the lower portion of one of these pipes, which had been removed after seven year's use, was inspected. This section had carried gas at a temperature of 700°C. and although cracked in several places was otherwise in reasonably good condition.

The volume of gas from the carbonisation system used in the drying plant amounts to approximately 40 per cent. of that produced and is about 200 M^3 per ton of briquettes. This represents a heat input for drying of about 16 therms per ton.

The dried coal from the cyclones, together with that from the electrostatic precipitator, of size 0 - 4 mm. is discharged to a conveyor leading to the briquetting plant. Arrangements are not provided for intimately mixing the separate products from cyclone and precipitator.

Briquetting.

Briquetting of the coal, dried to 12 - 13 per cent. moisture and of particle size 0 - 4 mm., is effected in extrusion presses at a pressure of about 10 tons per sq.in. (1500 atmospheres). There are 11 presses of similar type, six of which were produced by Maschinenfabrik Buckau-Wolff, of Magdeburg-Buckau, and five by Zeitzer Eisengiesserei und Maschinenbau, Zeitz. Each is operated

electrically and has four pistons and the briquette produced is in the form of a block of four roughly cylindrical briquettes joined together. The overall length of the block is approx. 8 ins. (206 mm.) and each of the four portions is approximately $2\frac{1}{2}$ x 2 in. and $2\frac{1}{2}$ in. in depth. As coal is fed to the press in front of the pistons it is pressed forward against the briquettes previously made, and the briquettes issue from the press in a continuous strip which is carried forward as each new set of briquettes is pressed. The strips fall on to a conveyor belt and in doing so they break into the separate blocks.

The stroke of the pistons is about 180 mm. The power necessary for briquetting was said to be 42 KW. per ton of briquettes.

At the time of the visit three presses were in operation and it was said that three presses were necessary to briquette the output from two driers.

Carbonisation.

The retorting plant consists of 10 Lurgi-Spülgas low-temperature carbonisation units, each of capacity 300 tons of briquettes per day. Each unit consists of two separate vertical shafts placed one on each side of central combustion chambers supplying the heating gases.

The arrangement of one half of a unit is shown in Fig. 2. in diagrammatical form.

Each half unit consists essentially of two portions mounted one above the other. The upper portion serves for drying the briquettes and it is considered essential that the whole of the moisture should be removed in this section before the subsequent carbonisation, otherwise serious disintegration of the briquettes would occur during carbonisation. The dried briquettes pass from the upper to the lower portion of the unit in which the carbonisation is effected and in which the coke is cooled before discharge.

Both upper and lower portions of a half unit are rectangular, the overall cross-sectional dimensions of a complete unit being 8750 x 6400 mm., the half units being 3650 x 6400 mm. The outer casing of both drying and carbonisation portions consists of sheet metal 5 mm. thick, the drying section being lined with brickwork of thickness 125 mm. The carbonisation section is lined with a layer 20 mm. thick of slag wool for insulation purposes, and with refractory brick (chamotte) of better quality than that used in the drying section. The thickness of the brick lining in the carbonisation section is 250 mm. on one side and 375 mm. on the other.

Heating of the briquettes in both the drying and carbonisation sections is by means of the products of combustion of the gases produced on carbonisation which are recirculated and burned in combustion chambers, after recovery of the by-products. Combustion of these gases takes place in two separate combustion chambers, one for each section. In the drying section heating gases from the combustion chamber enter the retort at a temperature of 250 - 280°C., are distributed via a series of metal channels and are then recirculated in this section by means of a fan. A portion of the gaseous products, mainly steam, is bled to atmosphere from the upper portion of the drying section. The casing of the fan used for recirculating the gases in this section of the retort is lined with lead to minimise corrosion.

Heating gas burned in the combustion chamber for the lower section enters the retort at a temperature of about 700°C., is distributed by firebrick-lined channels and passes upwards to the gas offtake near the top of the carbonisation section. The gases, consisting of a mixture of the volatile products of carbonisation with the recirculation gases, leave the retort at a temperature of 210°C.

The bottom portion of the lower section of the retort is used for cooling the coke before discharge. Cool recirculation gases enter the coke discharge chambers, are distributed through channels and, after passage through the coke, enter the combustion chamber supplying heating gas for the carbonisation.

Briquettes are charged to the upper section by means of a tipping bucket conveyor which feeds into a moveable distributor hopper from which the retort hoppers are charged. Each half unit of a retort has a separate charging hopper.

The coke extraction mechanism consists of two slotted plates, the slots in which are made to register to allow the passage of coke. The process is continuous the throughput being regulated by varying the rate and extent of movement of the plates. Below the discharge plates are the coke chambers from which the coke is discharged.

The metal gas offtakes are brick-lined and the collecting main, in which much of the liquor condenses, is lead-lined to minimise corrosion. These lead linings require renewal about every four years.

Temperatures of the briquettes in either the drying or carbonisation sections are not determined, the necessary control being effected by determining and controlling the temperatures of the gases. The maximum temperature reached by the briquettes in the

carbonisation zone was said to be not over about 500°C.

Of the 500 M^3 of gas produced per ton of briquettes, 60% is used in the carbonisation process. This is equivalent to about 24 therms per ton of briquettes.

Gas producers.

The process is practically self-supporting from the point of view of gas necessary for drying and carbonisation and it was said that normally there was little surplus gas. Two Lurgi gas producers are installed for the purpose of supplying the necessary gas when starting up the plant and to supply auxiliary gas should this at any time prove necessary. Originally when the plant was built twelve producers were installed but ten were subsequently removed elsewhere when it was found that they were unnecessary.

The producers are of internal diameter 2.6 metres, are water-jacketed and are fitted with mechanically operated grates. They each gasify 1 ton of brown-coal briquettes per hour and produce per hour 2,000 M^3 of gas of calorific value 1,500 k.cal. per M^3. The briquettes are charged intermittently. The producer installation includes a direct liquor-spray cooler, an electrostatic precipitator and a dust separator packed with Raschig rings for cleaning the gas. The tar produced, amounting to 70 per cent. of that obtainable by Fischer assay of the briquettes, is mixed with the tar from the carbonisation process.

Coke treatment.

Coke is discharged from the coke chambers, of which there is one for each half unit of a retort, on to a vibrator conveyor plate which is completely shrouded to exclude air. The temperature on discharge is about 150°C. This conveyor plate delivers on to a rubber belt which elevates the coke to the 'Ageing' and cooling plant. The rubber belt is completely enclosed, gas from the coke being drawn off by a fan and exhausted to atmosphere via a dust separator. From this belt the coke is fed into one end of a cylindrical metal drum approximately 8 ft. in diameter and 90 ft. long. The drum is mounted at a small angle to the horizontal and is rotated electrically. It contains a helix, attached to the inner surface, for moving the coke. Air is drawn into the drum by means of a fan located at the opposite end. Water to the extent of 1,400 litres per hour for each drum is supplied to the drum at the coke inlet end. The fan discharges to atmosphere via a dust separator.

One coke cooling drum serves to deal with the coke from three retorts. At positions where corrosion is worst the drum is lined with a cheap metal lining which can readily be replaced. The life of the main body of the drum was said to be about 9 years.

The coke on discharge from the cooling drum contains about 5 per cent. of moisture. This 5 per cent. moisture coke was a special product required by Brabag, Magdeburg. In this relatively dry condition the coke is very liable to heat up and ignite and it is necessary to transport it in special air-tight wagons. For normal consumption the coke receives additional quenching by water and after 4 hours draining contains 20 per cent. moisture.

Coke is screened into three sizes 0 - 10, 10 - 20, and over 20 mm.

The briquettes break down in size considerably during carbonisation, as is indicated by the screen analysis given later. The maximum size of coke being produced at the time of the visit was about $1\frac{1}{2}$ in.

Fire and explosion precautions.

Owing to the highly reactive nature of dry brown coal and of the coke produced, the design of the plant is such that air is not allowed to come into contact with either the finely divided dry coal or the hot coke, the conveyors etc. being enclosed. Provision is made so that if the plant is to be shut down, or if the CO_2 content of the circulating gases in the coal drying part of the installation becomes too low, steam can be admitted in order to prevent any heating up. Careful control is maintained, the instruments for recording temperatures, pressures and CO_2 and O_2 contents of the circulating gases being all located on central illuminated panels. During the early days of running the plant, before experience had been gained, a bad explosion had occurred at a time when the drying plant was being shut down.

By-product recovery.

Gas leaving the carbonisation retort at about 200°C. passes successively through a combined dust remover and primary direct cooler in which the temperature is reduced to 130°C., a Lurgi electrostatic tar precipitator working at 60,000 volts, two indirect condensers in series cooling the gas to 30°C., an exhaustor and a Feldwasher for recovery of light-oil. Gas leaving the washer is then recirculated to the drying and carbonisation sections of

the plant, without further purification. Hydrogen sulphide is not removed from the gas.

Cooling in the primary cooler is by means of a spray of liquor, dust being removed by means of a horizontal cage with metal bars which dip into oil as the cage is revolved. The indirect condensers contain 800 tubes of 90 mm. diameter inclined slightly to the horizontal. There are two such condensers to each retort.

Light-oil is removed in the Feldwasher by scrubbing the gas with middle-oil produced in the carbonisation process. The rate of circulation of middle-oil is 10 - 12 M^3 per hour through each washer. There is one Feldwasher to each retort.

Tar.

The tar as produced contains a considerable quantity of dust which is removed by centrifuge. The dust recovered was sold for use in making fire-lighters.

Light-oil.

The benzolised wash-oil is pumped by centrifugal pumps to overhead tanks and passes through a separator to remove water. The oil then goes through heat exchangers to a distillation column heated by steam at 120°C. The distillate is refluxed and the light-oil condensed and collected in tanks, the recovered wash-oil being recirculated to the Feldwasher.

The benzolised wash-oil contains 2.8 - 3.0 per cent. of light-oil boiling to 180°C. The light-oil recovered has a boiling range of from 60 - 220°C. and contains 80 per cent. boiling to 180°C.

Liquor.

Recovery of phenols from the liquor is not practised although it had been intended to instal plant using the Phenolsolvan process. The quantity of phenols and cresols discarded per annum in the liquor was said to amount to 1,500 - 2,000 tons, when the plant was operating to full capacity.

Laboratory tests with the Phenolsolvan process applied to the liquor produced gave the following results. The liquor was washed three times with 10 per cent. Phenolsolvan solution, which consists mainly of butyl acetate together with some propyl and amyl acetates.

Crude phenols in liquer

		per cent
(1)	As determined by phenolsolvan	1.27
(2)	" " " bromide, bromate method	0.8

Phenols in washed liquer

As determined by bromide, bromate method	0.007-0.008

Fractionation of crude phenolic oil

	per cent
181° - 230°C.	58.9
230° - 246°C.	9.0
over 246°C.	32.1

Re-fractionation of 181 - 230°C. fraction

Phenol	50.8
o.cresol	7.4
m.cresol	8.5
p.cresol	14.2
xylenol	15.8
neutral oil	3.3

DATA OBTAINED FROM PLANT PERSONNEL

Production per day	Present	Total Capacity
Coal received	1,700-1,800	4,500-5,000
Briquettes produced	950-1,000	2,500-2,600
Briquettes carbonised	950-1,000	3,000

Briquettes as charged
Analysis

	Per cent
Moisture	12
Volatile matter	45
Ash	7 - 9

Fischer Assay at 525°C.

Coke	50
Tar (including light oil)	12
Liquer	7
Gas + losses	19

Coke Screening

0 - 10 mm.	55
10 - 20 "	30
over 20 "	15

Coke

Analysis of average coke

Moisture	3.7
Volatile matter	17.0
Ash	18.8
Carbon	72.2
Hydrogen	2.3
Sulphur	4.4
$N_2 + O_2$	1.6

The coke was described as being very uniformly carbonised, the volatile matter content of each of the sized grades being similar.

Liquid Products

The total yield of tar and oil amounts to 10 per cent. by weight of the briquettes carbonised, of which 7, 1½, and 1½ per cent. respectively are tar, middle-oil and light-oil.

Tar

Specific gravity at 50°C.	0.940
Distillation to 320°C.	40 per cent
Phenols content	30 - 35 " "
Paraffins	15 - 20 " "

The tar produced was not distilled but was sold to Brabag., Magdeburg for treatment.

Middle-oil

Specific gravity at 20°C.	0.950
Initial boiling point	205°C.
Distillation to 320°C.	80 per cent
Phenols	15 - 20 " "
Sulphur	3.5" "
Viscosity at 20°C.	1.8° Engler
Setting point	-8°C.

Light-oil

Specific gravity at 20°C.	0.840
Phenols	5 per cent.

Analysis of the light-oil was not available but it was believed to consist mainly of unsaturated hydrocarbons as 90% of the oil could be extracted by 80% sulphuric acid. After refining by treatment with H_2SO_4 and NAOH the spirit rapidly discoloured and within 4 hours of treatment there were indications of gum formation.

Gas

The process produced very little surplus gas, practically the whole of that produced being used for supplying heat to the process.

Analysis of scrubbed gas.

Calerific Value K.Cal/NM³		per cent
	CO_2	15.0
	H_2S	2.5
	O_2	0.7
	C_nH_m	0.9
Gross: 2031	CO	16.7
Nett: 1857	H_2	18.2
	CH_4	6.5
	N_2	38.3

DATA OBTAINED FROM MILITARY GOVERNMENT, HANOVER.

Raw Brown Coal

	per cent
Moisture	48 - 50
Ash	6.
Calerific value, K.Cal/kilo.	2800

Briquettes

Moisture	15
Ash	10
Calorific Value K.Cal/kilo	4800

Coke

Moisture	3 - 6
Ash	18
Carbon	72
Hydrogen	1.98
Sulphur	1.29
Nitrogen & Oxygen	1.33
Sulphur in ash	3.0
Calerific value K.Cal/kilo	6267
Fusion point of ash	1400 - 1500°C.

Middle-oil

Creosote	16 - 18
Sulphur	3.5
Setting point	12°C.

Prices

Coke		
To Magdeburg	(6% Moisture)	7.1 RM/ 1000 kilo.
Industry	(20% ")	10-13 " " "
Household	(" ")	17 " " "
Tar		
Ex. Works		76.25 " " "

DATA OBTAINED FROM NORTH GERMAN COAL CONTROL.

HELMSTEAD SUB-DISTRICT

(1) Production per day, Tonnes.		Present	Normal
Fine coke	0 - 10 mm.	350	1000
Middle "	10 - 20 "	200	600
Rough "	20 - 30 "	50	200
	Total	600	1800
Tar		70	210
Middle Oil		15	45
Light Oil		15	45
		100	300

(2) Consumption

Coal per day	tonnes	1750	5000
Briquettes per day	"	1000 - 1100	2900 - 3000
Electricity per hour	KW.	3200	6900

(3) Labour

Workers employed	390	525
Employees	50	52

(4) Distribution at present

(a) Tar and oil to Hutte Brunswick and Haltermann of Hamburg on instructions from North German Oil Control.

(b) Coke to Brunswick and Hanover etc. on instructions from the North German Coal Control.

(5) Distribution Normal

(a) To Brabag. Magdeburg. (contract per year)

Tar (including oil)	100,000 tonnes
Coke (all sizes)	300,000 "

(b) To Hanover Area

Coke	200,000 "

COSTS.

According to information obtained from Military Government, Minden, the capital cost of the installation when erected in 1935 was as follows:-

	RM.
Buildings	4,832,500
Equipment	14,045,794
Sundry installations (including workshops, admin. offices etc.)	1,199,100
Total	20,077,394

The following costs, RM., are calculated per tonne of briquettes carbonised.

	1945.	1938.
Total carbonisation cost	18.12	12.65

including overheads and depreciation equivalent to 5RM. per ton. of briquettes treated.

Credits.		
Tar	8.51	7.06
Coke	6.71	4.88

These are based on prices per ton. of Tar and Coke of 76.25 and 11.55 RM. in 1945 and 72 and 9 RM. in 1938.

The process was not economic and even in 1938 operated at a slight loss.

The following additional data relating to costs of mining the raw coal and of producing briquettes in the factories other than at Offleben were obtained.

	1945.	1938.
Mine costs.		
Cost of 1 ton. of raw coal	3.65	2.70
Selling price	4.39	4.08
Profit	0.74	1.38
Briquette factories.		
Total cost of 1 ton. of briquettes.	12.19	9.74
Selling price 1 ton. of briquettes	14.03	13.72
	1.84	3.98

In 1945 and 1938 the quantities of briquettes produced were respectively 729,700 and 988,870 tonnes.

The briquetting costs at the Offleben plant were lower than those at the adjacent briquetting factories, on account of the fact that gas used for drying the coal was available at Offleben but had to be purchased by the briquette factories. The total briquetting cost at Offleben, including the cost of coal, labour and power but excluding overheads, was quoted at 7.13 RM. per ton. of briquettes.

The power used for briquetting at Offleben was said to be 42 KW. per ton. of briquettes. One kilo. of raw brown coal was said to be used in producing 1 KW. of power.

M.M.P.LTD

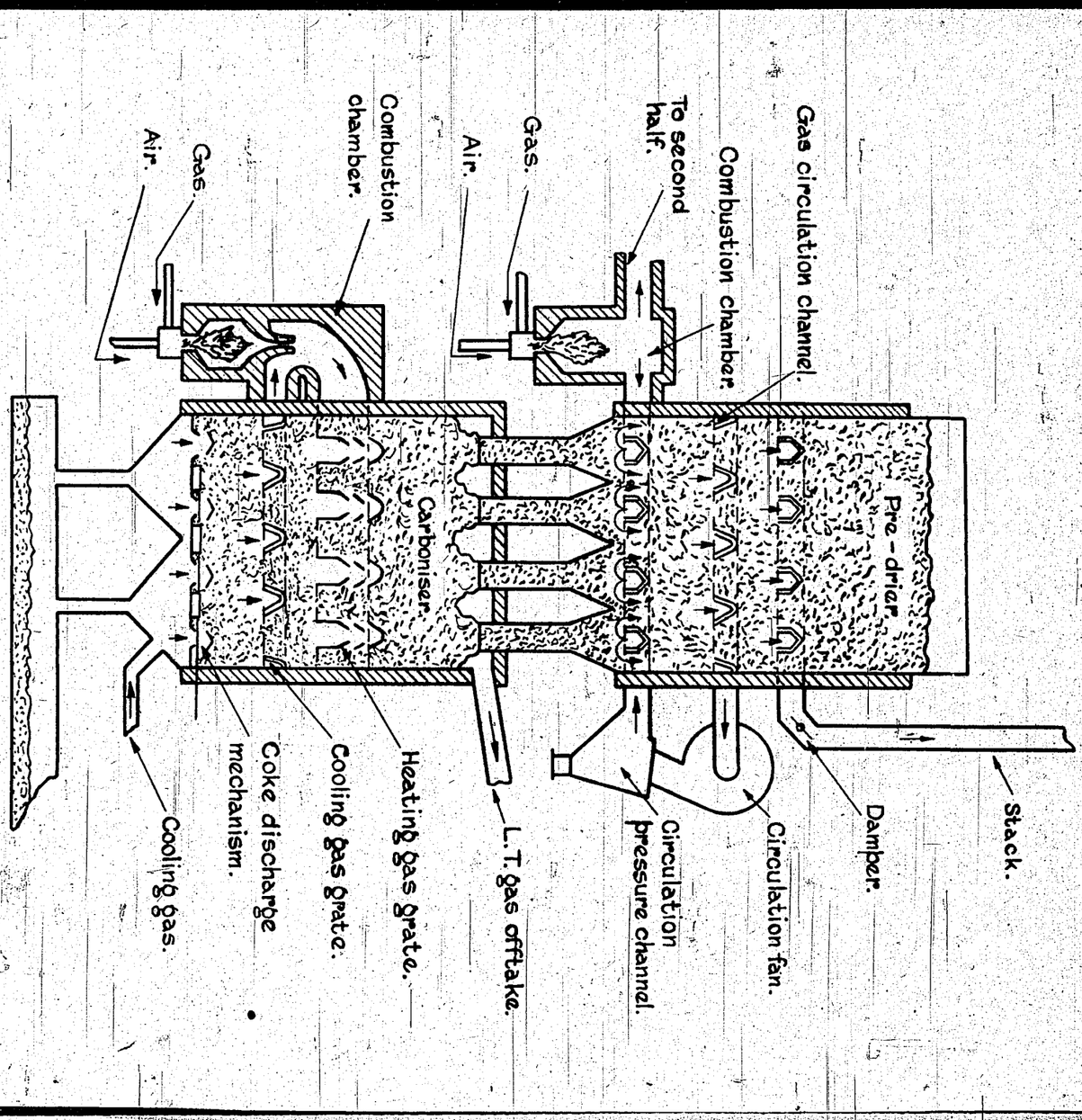

FIG. 2.

DIAGRAM OF LURGI-SPÜLGAS RETORT.

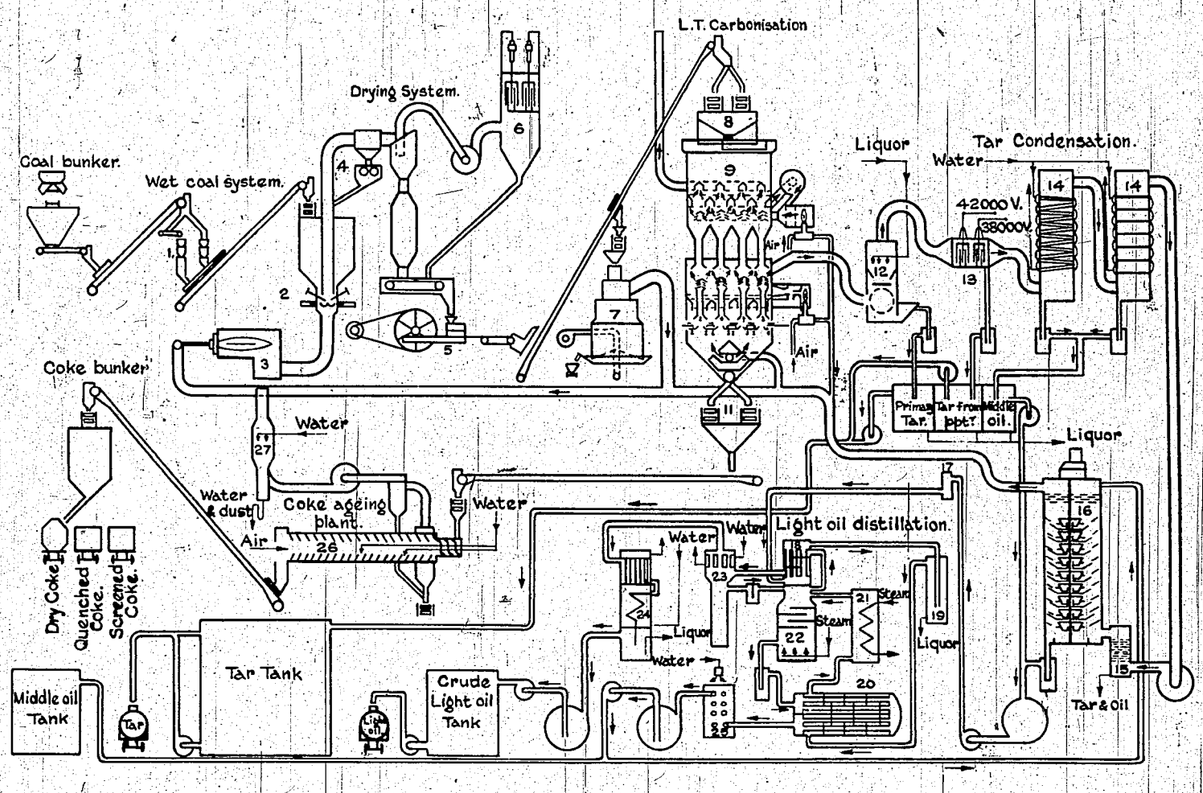

FIG. 1.

B.K.B. HELMSTEDT, SCHWELWERK OFFLEBEN FLOW-SHEET OF PROCESS.

1. Raw coal breaker.
2. Raw coal discharge.
3. Gas heating.
4. Circulation drier.
5. Briquette press.
6. Electrostatic ppt.r (dust)
7. Gas producer.
8. Briquette hopper.
9. Drying section.
10. Carbonising section.
11. Coke discharge.
12. Primary cooler and dust separator.
13. Electrostatic ppt.r (tar)
14. Condensers.
15. Liquor separator.
16. Feldwasher.
17. Wash-oil tank.
18. Heat exchanger (steam-oil)
19. Liquor separator.
20. Heat exchanger (oil-oil)
21. Oil heater.
22. Flash column.
23. Reflux condenser.
24. Light-oil cooler.
25. Spray cooler.
26. Coke cooling drum.
27. Dust separator.

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B.I.O.S. FINAL REPORT No. 596

ITEM No 30

GESELLSCHAFT FÜR TEERVERWERTUNG VARZINER-STRASSE DUISBURG-MEIDERICH, RUHR.

Bardgett, N.

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BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON-H.M. STATIONERY OFFICE

GESELLSCHAFT FÜR TEERVERWERTUNG

VARZINER-STRASSE

DUISBERG-MEIDERICH, RUHR.

Reported by

H. BARDGETT, (British),

on behalf of

Fuel Research Station,

D.S.I.R.

B.I.O.S. Target Number : 30/7.04

Fuels and Lubricants.

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE,
32, Bryanston Square, London. W.1.

TABLE OF CONTENTS.

SUBJECT.	PAGE NO.
Personnel Interrogated.	1
Date of Visit.	1
Condition of Target.	1
Organisation.	1
Output.	1
Continuous Vacuum Distillation of Tar.	2
Plant Operational Data.	3
Production in the Year 1943.	
(a) Working up of tar.	4
(b) Purchases.	4
(c) Production.	5
Price List of Products.	
1. Hydrocarbons.	7
2. N_2 - O_2 and S-Containing Compounds.	8
On Homologues of Indole in Coal Tar - The Recovery of Skatol from Tar.	10
Production of Pseudocumene, Mesitylene and P-Ethyltoluene.	11
Fig. 1. Flow-sheet of Continuous Tar Distillation.	
Fig. 2. Distillation Characteristics of Tar-Oil Fractions.	
List of Documents Obtained.	

PERSONNEL OF TEAM.

MR. H. BARDGETT. British Ministry of Fuel & Power.

DR. F.H. REED. U.S. Bureau of Mines.

DR. L.D. SCHMIDT. U.S. Bureau of Mines.

List of Documents Obtained.

1. "Über homologe Indole im Steinkohlenteer
   Nachtrag: Zur Gewinnung von Skatol aus dem Steinkohlenteer.

2. Herstellung von Pseudocumol, Mesitylen und p-Äthyl-Toluol.

3. Erzeugung im Jahre 1943.

4. Preisliste uber die von der Gesellschaft fur Teerverwertung m.b.H., Duisberg-Meiderich hergestellten Praparate aus Steinkohlenteer, gultig ab. 1 Jan. 1943.

5. Stetige Destillation des Vielestoffgemisches-Steinkohlenteer Sonderabdruck. Z.V.D.I. Beiheft "Verfahrenstechnik" Nr.1, 1944, S13-29 by Von Obering Dip. Ing. W.Fischer V.D.I.

Translations of items 1 to 4 above are included in this report.

Gesellschaft für Teerverwertung.

Varziner Strasse, Duisberg-Meiderich, Ruhr.

Personnel interrogated: Dr. Christian Seidler, Director.
Ober. Ing. Walter Fischer.

Date of visit: 31st July, 1945.

Condition of target: Described as 40 per cent. destroyed by air-raid and shell bombardment, but at time of the visit the damage appeared to be somewhat less than this. Work was not in progress, but it was hoped that production of road-tar would commence on the following day.

Organisation.

Gesellschaft für Teerverwertung is an association formed by twenty-three tar-producing companies in the Ruhr area for the centralised treatment of tar. The share-holding of the 23 companies is proportional to the quantity of tar supplied for processing, Gelsenkirchener Bergwerke A.G. (G.B.A.G.) being the Company with the largest holding. Sixty per cent. of the tar produced in the Ruhr is treated by the Company either at the works at Duisberg-Meiderich or at two other works at Rauxel and Alsdorf, and in addition tar is received from the Hermann Goring works at Watenstedt near Brunswick and Linz (Danube). The works at Rauxel and Alsdorf distil the tar only to the stage of intermediate products which are sent to Meiderich for subsequent treatment and production of chemicals.

Output.

Details were obtained for the year 1943 relating to the quantity of tar treated at each of the three works of the Company, and of the quantities and cost price of raw materials purchased, together with the quantities of products made and their selling price. These data are appended, together with a list, showing selling price, of special products made by the Company on a small scale for technical use. The Company could not give an estimate of the amounts of such special products prepared by them; their preparation was not regular but was as necessary to fulfil orders. A list of special commercial products made in larger quantities, together with their prices, is also given.

A total quantity of about 806,000 tons of tar was distilled during 1943 at the Company's three works.

Information was asked for concerning the methods of preparation of some of the special chemical products. At the time of the visit members of the chemical staff who could provide the detailed methods were not available, but Dr. Seidler will have detailed descriptions prepared of any methods required. Descriptions of the methods of preparation of Skatol (4-methylindole) prepared by Dr. O. Kruber, and of pseudocumene, mesitylene and p-ethyltoluene prepared by Dr. Schmitt were obtained and are appended.

Continuous vacuum distillation of tar.

Although several processes and plants have been proposed and described for the continuous distillation of tar in Germany, only a few, for example, those of Raschig (1) and of Koppers, (2) are stated to have achieved any practical importance.

Fischer of Ges. für Teerverwertung has worked over a period of some ten years on the design of a continuous system incorporating a pipe still, and a plant of capacity 10 to 12 tonnes of tar per hour, erected by Koppers, has been in satisfactory operation at Duisberg-Meiderich since 1942. A pilot scale installation had previously been tried in 1938.

The process, described as the Teerverwertung-Koppers process incorporates a pipe still consisting of a nest of tubes of internal diameter 80 mm. erected on the sides and top of a combustion chamber heated by coke oven gas. Tar for distillation is first de-hydrated in a separate gas-fired still at atmospheric pressure at a temperature of up to about 120°C., the water content being reduced from 5 to 0.3 per cent. before entry to the pipe still. The heated tar from the latter passes first to a vapourising column which contains a series of horizontal perforated plates, but which is unpacked. The tar at atmospheric pressure and at 320°C. enters

(1) B. Neumann: Z. angew. Chem. Bd. 34 (1921) S 193-95.
(2) O. Eisler, Z. Zamrzla & W. Weinkopt: Glückauf Bd. 72 (1936) S 184-91

the column where the pressure is reduced to 80 - 100 mm. and the temperature to 280°C., and spontaneous vapourisation takes place. Pitch is run off hot from this column. The vapours then pass successively through three columns fitted with reflux condensers. Rectification takes place in these columns without any additional supply of heat, the products being respectively anthracene oil, wash-oil, and naphthalene oil. Carbolic oil is condensed out following the last column. These three columns are packed with Raschig rings. Fig. 1 shows a flow sheet of the process and Fig. 2 the distillation characteristics of the fractions obtained.

It is claimed that the process gives 3 - 5 per cent. higher yield of oil than normal, and owing to good rectification the increase is in the form, not of high boiling anthracene oil, but of lighter oils.

The anthracene oil was described as of Avenarin quality and was stated to give a quite satisfactory fuel oil after separation of the solid residues. The wash-oil was described as of Solvay quality. The naphthalene oil containing the bulk of the naphthalene was said to be practically free from phenol.

Plant operational data.

After the plant had operated for several months, the following operational data were obtained:-

Yield of de-hydrated tar:	12 tonnes per hour.
Heat consumption	
(a) Coke-oven gas	29.4 Nm^3 per tonne
(b) Steam	30.0 kg. " "
Current consumption	
(a) For vacuum plant	2.5 KWH " "
(b) For pumps	1.5 KWH " "
Water consumption	1.5 M^3 " "

The heat consumption of the pipe-still, of coke-oven gas equivalent to 112,000 Kcal. per tonne, was considered to be extraordinarily low and was stated to be approximately equal to the theoretical requirement. Unavoidable heat losses were said to be made up by the utilization of a portion of the heat of the pitch.

Steam is used for heating up pumps, armatures, etc., for blowing through pipe lines and for heating a special additional arrangement designed to increase the amount of phenol in the carbolic oil.

Fischer stated that during a period of two years' operation there had been no formation of coke in the tubes of the still. The pitch column had been cleaned out only once during this period, while it had not yet been necessary to clean out any of the fractionating columns.

A paper by Fischer in Z.V.D.I. Beiheft "Verfahrenstechnik Nr.1. 1944 deals with theoretical considerations underlying the continuous distillation of tar under vacuum and gives data relating to the spontaneous vapourisation temperatures of components in tar mixtures. The paper also describes the Teerverwertung-Koppers process in use at Meiderich. A reprint of this paper is among the documents obtained during the visit. Figs. 1 and 2 are reproduced from the paper.

Production in the year 1943

(a) Working up of tar.	tonnes	tonnes per month.	R.M. per tonne.
Meiderich	292,887	24,400	
Rauxel	473,978	39,500	
Alsdorf	39,872	3,300	
	806,737	67,200	52.39
(b) Purchases			
Welheim Oil	21,735		
Hollandöl	21,247		
L.T.tar	4,078		
Rhein steel oil	2,013		78
Anthracene Oil-filtered	337		75
Napthalene-crude	334		78.50
	49,744		
Benzol fore-runnings	1,418		150
Middle Oil	225		80.60
Motor Benzole - crude	3,869		281.10
Tuluol fraction	2,733		
Light oil	2,795		135
Residues from air-blowing	5,214		61
	16,254		
Carbolic Oil	19,058		138
Phenol - crude	3,361		320
Phenol - crude (in the form of phenate solution.	3,464		
	25,883		
Middle and heavy oil	3,674		78
Wash-oil - diluted	1,196		
Retort pitch	2,466		45.60

N Oil from Pott-Broche or pitch-hydrogenation process at Ruhröl A.G., Bottrop-Welheim.

(c) Production	tonnes.	tonnes per month	R.M. per tonne
Pitch	149,734	12,478	46.50
Prepared tar	16,025	1,335	61
Fuel oil III	637	53	61
Eisenlack (iron lacquer?)	804	67	165
V.f.T.Marine oil	106,183	8,849	76
Impregnating oil	1,504	125	73
Marine oil + added pitch	2,071	173	64
Solvay-wash oil	5,880	490	82.50
90's wash oil	881	73	74
I.G. and hydrogenation oil	9,649	804	89
Anthracene oil-filtered	4,174	349	75
Fuel oil	604	50	76
Motor oil	1,304	109	79
Fluxol	1,524	127	87.50
Naphthalene wash oil	89	7	97
Naphthalene - pure	2,831	236	155.50
" - hot pressed	1,015	85	108
" - washed, Centrifuged)	13,576	1,131	99
" - washed	131	11	130
" - crude	334	278	78.50
Anthracene residues	7,688	641	35
Phenol	5,013	418	795.55
Cresols	7,733	644	310 to 452.50
Xylenols	118	10	329
Clear resins	1,425	119	236
Dark "	2,063	172	123
Motor benzol	2,959	247	309.50
Benzol - purified	853	71	325
Benzene - pure	210	18	387
Toluene - pure	2,725	227	433.50
Xylene - pure	674	56	402.50

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Marine oil + added pitch	2,071	173	64
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90's wash oil	881	73	74
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Fuel oil	604	50	76
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Naphthalene - pure	2,831	236	155.50
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Benzol - purified	853	71	325
Benzene - pure	210	18	387
Toluene - pure	2,725	227	433.50
Xylene - pure	674	56	402.50

	tonnes	tonnes per month	R.M. per tonne
Solvent benzol	1,559	130	232.50
Heavy benzol	1,864	155	133
Pyridene - pure	2	0.17	661.40
" - special type I	152	13	1009.50
" - " " II	195	16	1009.50
High-boiling bases	122	10	76.50
Ammonium sulphate	431	36	394
Low-cooled oil (gas-holder oil, Meider oil, etc.)	3,078	257	93 to 190
Insecticides	493	41	102.50 to 126
Burning oil	5,025	419	119
Cold tar (Kaltteer)	775	65	96.50
Paints (railway colours & Synova paint)	2,004	167	218 to 369.50
Insulating lacquer (varnish)	33	3	144.50
Other tar products (soft pitch, adhesives, base mass, putty, etc.)	2,971	248	46.50 to 190
Quinoline - crude	9	0.75	
α - methyl-napthalene - commercial	2	0.17	
β - " " "	1	0.08	
Diphenyl - commercial	7	0.58	
Diphenylene oxide - commercial	4	0.33	
Crude quinoline II	20	1.7	
1.3.5 - Xylenol - commercial	5	0.42	

Roofing-board, 15 metres, = 75,745 rolls = 1,136,175 sq. metres
= 94,681 sq.metres per month.

Price List of Products prepared from Coal Tar By Gesellschaft für Teerverwertung m.b.H., Duisberg-Meidreich (current from 1st January, 1943)

1. Hydrocarbons	R.M. per kg.
Acenaphthene	pure 10
"	commercial 3
Anthracene	pure 10
Chrysene	" 25
" (synthetic)	" 100
Diphenyl	" 10
"	purified 6
Dicyclopentadiene	" 12
"	commercial 7.50
1.6 Dimethylnaphthalene	pure 30
2.6 "	" 20
2.6 "	purified 10
2.7 "	pure 75
2.3 "	" 50
Fluorene	" 10
Fluoranthene	" 20
Indene (D.R.P.345,897)	" 5
1 - Methylnaphthalene	" 12
1 - "	purified 6
2 - "	pure 4
Phenathrene	" 20
"	commercial 5
Pyrene	pure 20
" (white)	" 30
m - Xylene	" 3
O - "	" 15
Pseudocumene	" 20

2. Nitrogen - , Oxygen - and Sulphur-containing Compounds.

		R.M. per kg.
Quinoline	pure	12
"	commercial	3
Isoquinoline	pure	25
Quinaldine	"	60
Lepidine	"	200
Pyridine	"	9
"	purified	4
α- Picoline	pure	10
2.4 - Dimethylpyridine	"	75
2.6 - "	"	75
Sym. (2.4.6.) Collidine	"	60
Acridine	"	50
"	purified	35
* Indole (D.R.P. 223,304, 454, 696	pure	100
2 - Methylindole (methylketol)	"	200
3 - Methylindole (Skatol)	"	300
7 - Methylindole		
Thionaphthene (D.R.P. 325,712, 350,737)	"	100
Diphenylene oxide	"	6
Carbazole	"	10
"	commercial	3
Xylenol 1.3.4. (D.R.P. 447,540)	pure	20
" 1.4.5 (" ")	"	30
" 1.3.5	"	5
" 1.3.5	commercial	3
" 1.2.4	pure	10
0,0' - Dioxydiphenyl (0 - Diphenol)	"	20
" " "	commercial	10
0 - Oxydiphenyl (0 - Phenylphenol)	pure	25
" " "	commercial	12

* The sale of Indole is arranged through I.G.Farbenindustrie A.G., Lohmuhlenstr., Berlin.
The quantity of the above materials which can be supplied is limited.

The following commercial products can be supplied in large quantities without restriction : -

		R.M. per kg.
1. Methylnaphthalene	Commercial	3
2. "	"	2
Diphenyl	"	2.50
2.6 - Dimethylnaphthalene	"	3
1.6.- "	crude	3
Fluorene	commercial	5
Phenanthrene	crude	3
Fluoranthrene	"	6
Pyrene	"	6
Chrysene	"	6
Diphenyleneoxide	commercial	3
1.3.5 - Xylenol	"	3
0,0' - Dioxydiphenyl (0-Diphenol)	crude	5
Quinoline	"	2
Acridine	commercial	25
Heating-bath oil		2

On homologues of indole in coal tar.

Supplement: The recovery of skatol from tar.

Among the four homologues of indole - 2-, 3-, 5- and 7-methyl-indol - that I have up to now shown to be present in coal tar, skatol occupies a special place by virtue of the replacement of the β-hydrogen atom in the pyrrol ring by a methyl group. It is alone among the four isomers in being unable to form a β-carboxylic acid when its sodium derivative is treated with carbon dioxide. I isolated the 2-, 5- and 7- methyl indoles in the form of β-carboxylic acids from the mixture of their homologues. α-carboxylic acids could not be detected, and the very poor yield in the preparation of 7 -methyl-n-benzoylindole shows how little the imide hydrogen atom is replaced when indole is fused with sodium. The skatol must consequently accumulate in those fractions of the indole oils that remain unattacked when fused with sodium. The oil set after repeated fusion followed by distillation, crystallizing to form large glittering plates that turned out to be skatol.

The starting material was 9 kg. of carefully fractionated indole oil, of which 90 per cent. boiled between 267 and 270° and had a specific gravity of 1.07 at 15°. This oil was fused in portions of 3.5 kg. with sodium, 500 gm. each, in the manner already described. After the fused product had been treated with carbon dioxide, the oil that had for the time being remained unattacked - about 2.1 kg. - was made up by the addition of some original oil and passed on to the next fusion. After the seventh repeat 1.31 kg. of the original material remained unattacked, the homologues of similar boiling point, 5- and 7- methylindole having, from what has already been said, been separated from it largely in the form of their β-carboxylic acids. This 1.31 kg. of oil set after distillation under a reduced pressure to a crystalline mass.- The solid that had separated - 375 gm. - was removed on a suction filter after the whole was cooled in ice. After two recrystallizations from the same quantity of ligroin 210 gm. of pure skatol in the form of glittering crystalline plates was obtained; it was characterized by its melting point, 96°, by a mixed melting point and by its intense nauseating odour. Further amounts of the pure substance were obtained by working up the mother liquors, and also by fusing with sodium the oil, 935 gm., that had been separated from the crystals at the first cooling and filtered off by suction.

The total yield of skatol finally amounted to 380 gm.

Of course, the portions subjected to fusion, which were originally intended to serve a different purpose from that described above, could have been greater, and this would have shortened the procedure greatly. In any case, it can be seen from the experiments carried out, even if they do seem time-consuming, that, of the four indole homologues newly observed in coal tar, skatol is relatively easy to come by provided sufficient quantities of the necessary starting material, properly fractionated in the first place, are available, for

the purification of this methyl indole can be effected without previously converting it to a carboxylic acid, although, of course the compounds accompanying it and having a similar boiling range must be removed beforehand as carboxylic acids.

Duisburg-Meiderich, 11th November, 1926. (Signed) Dr. O. Kruber.

Production of pseudocumene, mesitylene and p-ethyltoluene.

A fraction having the boiling points: 15% - 161.5 - 162°; 90% - 163° was made from solvent naphtha II. As this fraction still contained considerable quantities of constituents resinifiable by means of sulphuric acid, it was washed three times in succession with 10 per cent. by weight each time of 80 per cent., 90 per cent. and 98 per cent., neutralized and redistilled. Thus 14.4 kg. = 22.5 l. were obtained with boiling points: 6% - 162.2 - 162.5°; 90% - 163.6°; 95% - 164.2°, and was sulphonated at 70° with an equal volume of concentrated sulphuric acid. After cooling 22.5 l. of water was added with cooling, when, after the lower acid layer was drawn off, a solid sulphonic acid crystallized out. It was filtered on a suction filter and re-dissolved in an equal volume of 50-per cent. sulphuric acid. The 6.2 kg. of solid sulphonic acid thus obtained was treated with three times its volume of saturated potassium chloride solution and the potassium salt thus obtained was re-dissolved in an equal volume of water. On blowing air through this solution, 1.16 kg. of pure pseudocumene was obtained with the boiling point 10% - 167.1-167.6°; 90% - 167.7°; 95% - 167.9°.

The mother liquor from the re-dissolving of the solid sulphonic acid in 50-per-cent. sulphuric acid was treated with three times its volume of saturated sodium chloride solution, the separated salt was re-dissolved in half a volume of water and the large plates of the sodium salt that crystallized out were air-blown with sulphuric acid. In this way 0.4 kg. of p-ethyltoluene were obtained.

A further quantity of ethyltoluene was obtainable from the liquid sulphonic acid from the sulphonation of the original material. It was treated for the purpose with five times its volume of saturated salt solution. After re-dissolving and air-blowing a further 230 gm. of ethyltoluene was obtained with the boiling range 2% - 159.6-160.0°; 53% - 160.4°; 90% - 160.8°.

When the original material was sulphonated, 1.37 kg. remained unconverted, and it was ascertained that this consisted mostly of mesitylene. It was sulphonated with an equal volume of sulphuric acid at 70°, converted into the potassium salt by precipitation with potassium chloride solution, and re-dissolved. On air-blowing 450 gm. of mesitylene, with a boiling point of 10% - 163.0-163.3°; 30% - 163.4°; 70% - 163.5°; 90% - 163.8°, was obtained.

A further quantity of mesitylene was obtainable by working up the mother liquor obtained from the potassium pseudocumene sulphonate. If the purification of the pseudocumene salt is not taken so far, the solid potassium salt contains both mesitylene and pseudocumene, but these can easily be separated owing to the difference in the temperatures at which their fission occurs. Mesitylene-sulphonic acid is easily split at 110°, whereas pseudocumene sulphonic acid goes only at 145-145° (sic). P-ethyltoluene-sulphonic-acid too, is decomposed only at about 150°, so that it is easily separated from any mesitylene that may happen to be mixed with it, by air-blowing, and obtained with a good degree of purity.

Duisburg-Meiderich, 19th May, 1933. (Signed) Dr. Schmitt.

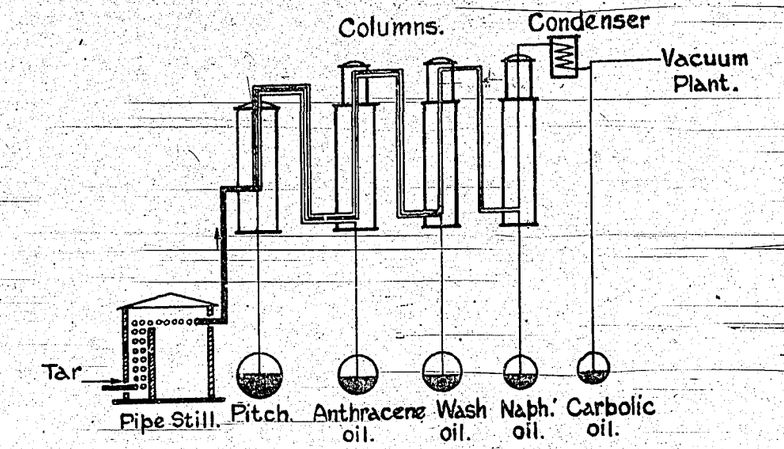

FIG. 1. FLOW SHEET OF CONTINUOUS TAR DISTILLATION BY THE TEERVERWERTUNG-KOPPERS PROCESS.

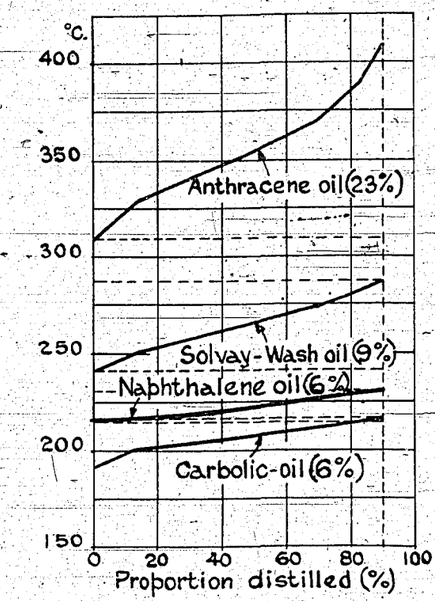

FIG. 2. DISTILLATION CHARACTERISTICS OF TAR OIL FRACTIONS OBTAINED IN THE TEERVERWERTUNG - KOPPERS PROCESS.

Copy No. 1

B.I.O.S. FINAL REPORT No. 617
ITEM No. 31

TOP STRIPPING EQUIPMENT USED IN OPEN CASTE COAL WORKING

(Bell, W. G., Cooke, V. R., Chamberlain, H. L., and others)

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BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

TOP STRIPPING EQUIPMENT USED IN OPEN CAST COAL WORKINGS

Reported by

Members of M.O.S. Tube Mission Team.

B.I.O.S. Trip No. 993 September, 1945.

BIOS Target Numbers: C31/959, C31/1121.

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE
32, Bryanston Square, London, W.1.

CONTENTS

		Page
Introduction		1
Section I - C31/959	Kabelbagger Roddergrube A.G. Abteilung Vereinigte Ville - Knapsack	1
Section II - C31/1121	Gewerkshaft Hurtherberg Vereinigte Stahlwerke, Hermulheim	3
Summary		4
Fig.1		5
Fig.2		6

MEMBERS OF TEAM

Mr. W. C. Bell	M.O.S.
Capt. V. R. Cooke	M.O.S.
Mr. H. L. Chamberlain	M.O.S.
Mr. J. C. Cooper	M.O.S.
Mr. L. T. Maddocks	M.O.S.
Mr. S. M. Tate	M.O.S.
Mr. E. G. Saunders	M.O.S.
Mr. W. A. Smith	M.O.S.

TOP STRIPPING EQUIPMENT USED IN OPEN CAST COAL WORKINGS.

Introduction:

Reference to special equipment for this purpose was first noted in the G.(T)and C.W.files at 21st Army Group HQ. where mention was made of baring or top stripping equipment in use at the Fortuna Mine, Knapsack. It was decided that while this subject was not included in the terms of remit to the Tube Mission, the equipment should nevertheless be investigated as a Target of Opportunity. The matter was of interest to team members in terms of the open cast quarrying of ironstone connected with British Steel and Tube plants.

Various German officials were questioned, and the quarry workings finally located in the Knapsack and Hermulheim area. The equipment was not in operation at the time of the visit, but a series of photographs were taken. Unfortunately a later accident destroyed this film, and recourse has had to be made to the sketches shown in the Appendix.

SECTION I.

Kabelbagger Roddergrube A.G.

Abteilung Vereinigte Ville - Knapsack

The open cast quarrying of brown coal is on an extensive scale in this area, and in the quarry examined, the depth of coal face was fully 70 metres with an overburden of 15-20 metres, consisting of soft blue clay next to the coal measure, and gravel on the surface. A notable feature of the quarry is its width. The top stripping or baring machine is an elaborate form of dragline with facilities for depositing the spoil about 750 ft. across the quarry from the baring face. The coal is won by vertical cutting machines operating the full length of the vertical face, and loading direct into railway wagons, situated on the floor of the quarry.

The stripping machine made by Adolph Bleichert, Leipzig, has been in operation for about 20 years, with only one rope change necessary in this period.

Fig.1 illustrates the method of stripping employed. It will be noted that to all intents and purposes, the stripping operation is carried out by a dragline bucket of orthodox design, and that the especial feature is the facilities for discharging this bucket by a conveyor system, depositing the spoil on the spoil bank side of the quarry. A feature of this discharge arrangement on the far side is that the actual point of discharge from the bridge can be varied, giving an even discharge which avoids hill and dale formation of the spoil. The extent of the transverse is the main point of interest, as this is very much greater than anything possible in the U.K. using draglines operating on the swinging mast principle from the baring side.

The manning cost of this machine at Knapsack is low. There is one man in the main operating cab on the baring side, and one man acting as a bottom man on the spoil banks. In addition, a mechanic and electrician engaged in the general maintenance of all equipment operating within the quarry area, are available in the event of minor breakdown.

The bucket capacity is 10 cubic metres, but the customary load is 6 cubic metres in order to avoid spillage. 275 trips are made in an 8 hour shift with the normal break for a meal, giving over 200 cubic metres of overburden removed per hour. Actual coal production is of the order of 30,000 tons per day.

It was learned that the personalities of the firm operating this equipment are :

Herr Niehmur, Director.
Herr Brunner, Director and Chief Engineer.

SECTION II.

Gewerkshaft Hurtherberg

Vereinigte Stahlwerke, Hermulheim

The location of this target is a few miles from the machine at Knapsack reported in Section I. In this case, the top stripping machine was designed and supplied by Algemein Transport Gesellschaft, Leipzig, and the impression was formed that Director Ries of this firm is a recognised authority on this type of equipment, and perhaps the best designing engineer for the purpose in Germany.

Fig.2 indicates the method of operating, and it will be seen that this machine is more adapted to conditions and practice in the U.K. than the type of equipment dealt with in Section I.

Description:

The machine consists of a digger similar in type to the 100 Ruston Bucyrus machine used in this country, feeding on to a continuous belt, which, carried on a structure as shown in sketch, can drop the spoil wherever required. Baring operations were being carried out in two stages, as shown, and dealing with an overburden about 20 metres total thickness. Operating on this principle, the capacity was stated to be 10,000 cubic metres overburden removed per 24 hours, but it was admitted that using the hopper-collector as shown, there was no reason why two diggers should not operate to feed the same conveyor equipment, and on this basis it was considered that the capacity of the handling equipment could be considerably increased.

The important feature of the set up is that the top soil can be removed separately, the spoil side of the quarry kept level, the top soil replaced, and the area under cultivation again within one year.

Examination of the working area afforded proof of this statement, and on the spoil bank side, the line of the land under cultivation was within 200 yards of the actual line on which spoil was being deposited.

Summary:

It is considered that the equipment seen on these targets, particularly the machine dealt with under Section II, is of particular interest to Ironstone Companies in the U.K. operating open cast quarries, particularly where land restoration problems arise.

It is realised that the open cast workings inspected presented an easier proposition than ironstone quarrying in the U.K., particularly in terms of the relatively soft and shallow overburden to be removed, and in the absence of limestone stratas in the overburden. On this latter point, the German operators expressed the view that any massive limestone formation encountered could be dealt with by preliminary "pop blowing" from the working face.

The most knowledgeable German personality on both these machines is perhaps Herr Fetcher who is employed at the Benrath West Mine. Fetcher lives only a few yards from Military HQ. in Knapsack, is wellknown to this HQ. and can be readily located by them.

Further investigation of this target by mechanical and mining engineers engaged in ironstone operations is strongly recommended.

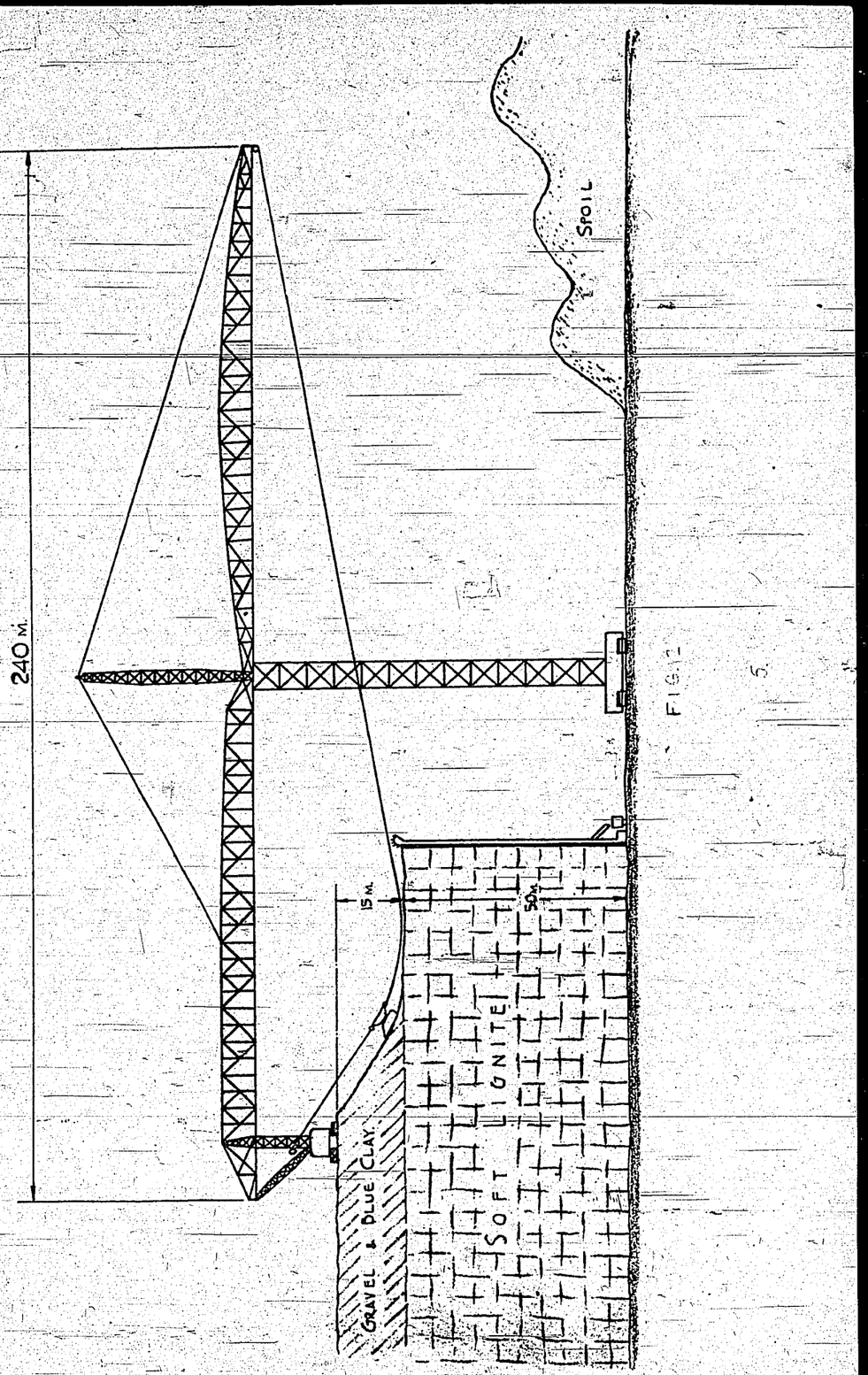

FIG 2

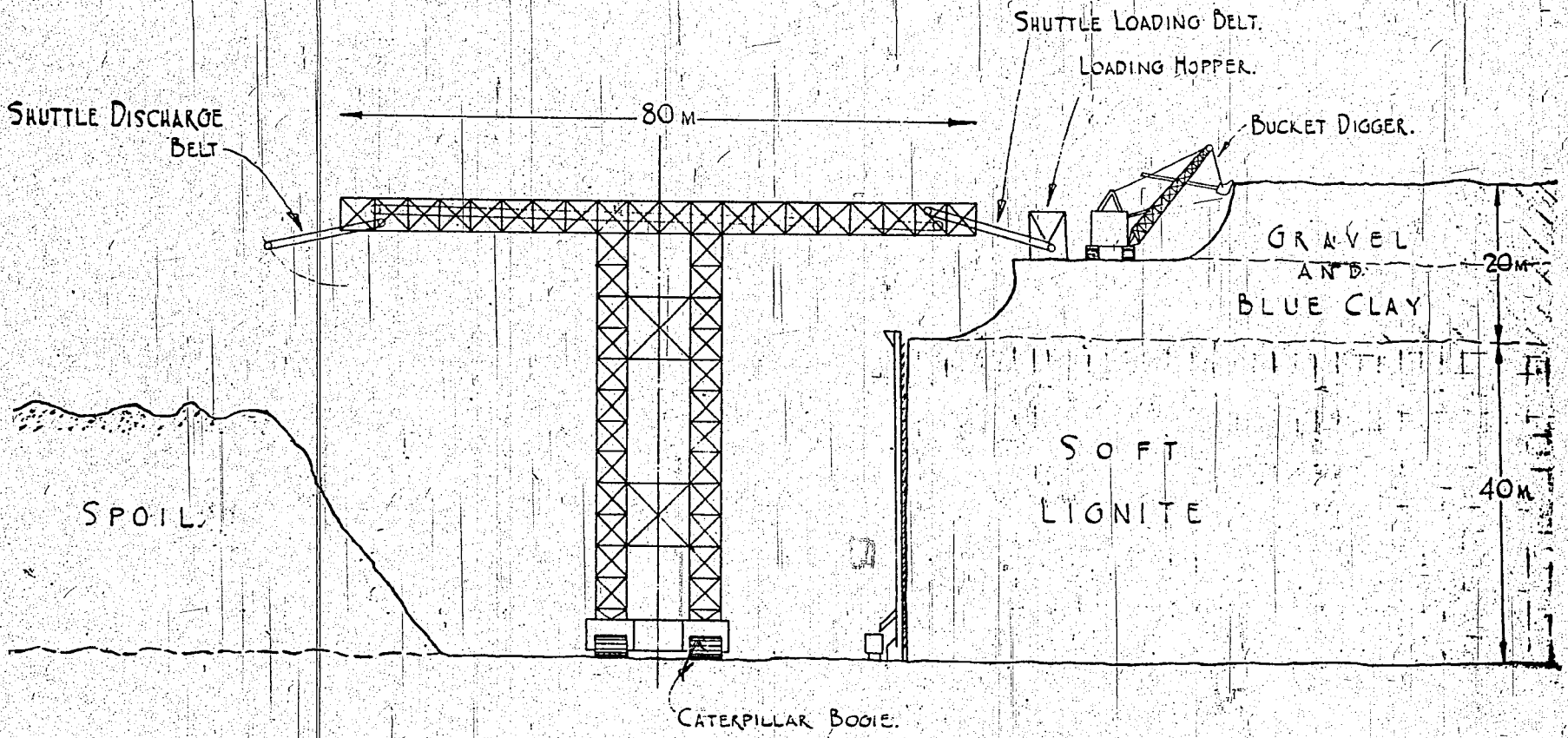
SHUTTLE LOADING BELT.
LOADING HOPPER.
SHUTTLE DISCHARGE BELT
80 M
BUCKET DIGGER.
GRAVEL AND BLUE CLAY
20M
SOFT LIGNITE
40M
SPOIL
CATERPILLAR BOGIE.

FIG. 2.

Copy No. 1

B.I.O.S. FINAL REPORT No. 670
ITEM No. 21

GERMAN METHODS OF MANUFACTURE OF COPPER TUYERES FOR BLAST FURNACES.

Booth, J.C.H., Reay, J.L.

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LONDON—H.M. STATIONERY OFFICE

GERMAN METHODS OF MANUFACTURE OF COPPER TUYERES FOR BLAST FURNACES

Reported by

J.C.H. Booth, M.O.S.
J.L. Reay, M.O.S.

B.I.O.S. Trip No. 2033 March, 1946.

BIOS Target Nos:

21/14(b) C21/449
C21/942 C21/734
C21/1338

C.I.O.S. Black List Item No. 21

METALLURGY

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

TABLE OF CONTENTS.

1. Object of Visit.
(page 3).

2. Works Visited.

(a) August Thyssen, Hamborn.
(page 4). 21/14(b)

(b) Hundt und Weber, Siegen.
(page 5). C21/449

(c) Sieglander Kupferwerke, Siegen.
(page 9). C21/942

(d) Dango und Dienenthal, Siegen.
(page 10). C21/734

(e) Rhein-Emscher Armaturenfabrik,
Baerl. C21/1338
(page 15).

3. Conclusion.
(page 18).

At the request of the authors the Association of Bronze & Brass Founders' team visited in December 1945, the works of Hundt und Weber and Dango und Dienenthal, both situated at Siegen. At the works of the former, a novel method of producing copper tuyeres by a centrifugal casting process was seen and proved to be of sufficient interest to the trade to warrant a closer investigation. The Association of Bronze & Brass Founders accordingly arranged for the authors to visit Germany for this purpose, and the opportunity was taken to examine in detail the various German methods of manufacture of copper tuyeres.

AUGUST THYSSEN.
HAMBORN, GERMANY.

Target No.1.

Date of investigation - 29th March 1946.

OBJECT OF VISIT.

To obtain details of copper tuyeres in common use in German Blast Furnaces.

PERSONNEL INTERVIEWED.

Dr. Risser - Chief Engineer.
Mr Schumacher - Blast Furnace Manager.

This firm, as probably the largest blast furnace operators in Germany, have wide experience in the use of different makes of copper tuyeres, and the names of the makers appearing in the latter pages of this report were obtained from the persons interviewed.

Generally speaking the water available for the use of blast furnaces in the Ruhr is of poor quality, and for this reason the opinion was expressed that forged copper tuyeres stood up to conditions better than either statically or centrifugally cast tuyeres. At times the water was found to be so contaminated that additional filtering arrangements had to be made in the pipe lines at the furnace.

A double pressure tuyere was described and parts of it were seen on the plant. Full particulars of this tuyere together with the results of the experiments made with it have already been obtained by a Blast Furnace Team, with a member of which it has been discussed.

HUNDT UND WEBER.
SIEGEN, GERMANY.

Target No.2.

Date of investigation - 1st and 2nd April 1946.

OBJECT OF VISIT.

Investigate the centrifugal method of casting copper tuyeres.

PERSONNEL INTERVIEWED.

Mr Weber - Owner.
Mr Kolb - Assistant to Weber.
Mr Mertens - Foundry Manager.

This plant had already been visited on the 10th December 1945 by the Association of Bronze & Brass Founders' team, and in their report a description of this process is included. The description with a few additions and slight modifications is repeated here so as to make this report complete.

This firm has developed a special technique for manufacturing electrolytic copper tuyeres which are cast on vertical centrifugal machines. The following method was adopted :-

A cylindrical cast-iron casing was fixed to the horizontal table after being lined with a loam mixture to give the desired tapered form to the outside of the tuyere. The loam mixture was originally two thirds loam and one third iron filings which gave a semi-permanent mould which with patching produced up to 30-40 castings. At the time of the visit however, owing to iron filings being no longer obtainable an ordinary loam lining was used which, though satisfactory, needed constant patching. A loose ring which fitted into a recess at the top of the casting covered the top edge of this lining and protected it from crushing by the plate carrying the core. The ring itself was recessed to locate the plate which was spigotted. This top plate had four holes 7/8" diameter through which the four vent tubes of the core passed. The vent tubes were screwed and the core held to the top plate by nuts, - see figure 1 giving details of assembly.

When casting, the mould was allowed to attain full speed and then all the molten copper was poured through a funnel with a vertical 2½" diameter pouring spout. The metal was poured quickly and skimmed with a wooden pole. For a tuyere 17" o.d., x 11" i.d. at top, 14" o.d. x 7" i.d. at bottom, x 19" long, a speed of 350 r.p.m. was used. This gave a taper to the inside wall of the tuyere.

The firm had obtained a licence for electrolytic copper cathodes which were melted in an oil fired furnace of a special design. The copper was de-oxidised with 10% Phosphor Copper at the rate of 500 grammes to 125 kilogrammes of copper. It was noted that the phosphor copper was added with the charge which was melted without the use of charcoal or a flux.

(b) Coremaking Method.

A machined tapered core barrel (made from light gauge steel sheet) was employed. At the large end there was an endplate with four holes to locate the vent tubes. The size of the endplate varied with the size of the core being manufactured. An ordinary loam former was struck up on this tapered barrel and dried. The former was then covered with a coating of heavy grease.

For these cores a special loam consisting of :-

4 parts shredded flax
4 " sand
¼ " coal dust

was used. The ingredients were mixed in a mill having two sets of contra rotating knives. Only the minimum amount of water was added so that the loam as used was very stiff.

After a certain amount of loam had been applied over the former the steel vent tubes were laid in with one end projecting through the endplate. These vent tubes were 5/8" o.d. with holes drilled at approximately 3" intervals through the length. At one end there was a flange approximately 1.1/4" diameter which located the tubes in the holes of the endplate. These flanges also enabled the core to be correctly located during assembly prior to casting.

After the tubes were properly bedded in the loam they were tied together with iron binding wire. One wire was tied through the tubes at the central position and then wound spirally up and down the length of the core and

finally fastened at the initial anchorage. Each tube was then individually tied 3" from the faceplate end to the spirally wound wire. The tubes were further secured by nails being put through each tube in two places so as to project either side of the tube. More loam was then added and the core finally strickled to shape and size.

The whole assembly was then put into a drying oven and when dried the core was withdrawn from the former, facilitated by the grease. While still hot the core was painted with a preparation called Hexolith. The heat of the core dried this cement wash almost immediately and then two coats of blacking with a small quantity of Hexolith added were applied and dried with a gas burner. A number of oblique saw cuts for gas venting were made across the top of the core where it jointed with the top of the casting box and the core was then ready for casting.

After casting and dressing the tuyere body was machined on the faces of the open end and the bore cleaned up for a few inches towards the nose. A steel ring which had previously been machined and tapped for the water inlet and outlet was then brazed on to the body of the tuyere the faced end of which fitted into slots machined into the steel ring. The outside diameter of this ring was ½" smaller than the bore of the tuyere and the outside diameter ½" larger than the outside diameter of the tuyere. After brazing, for which a gas fired furnace was used, the tuyere was placed in the lathe again and the steel ring turned down to the finished sizes. Finally the tuyere was tested under water pressure.

In this foundry were observed some statically cast copper slag notch coolers. In appearance they were very much inferior to the British product and the method of manufacture was not investigated.

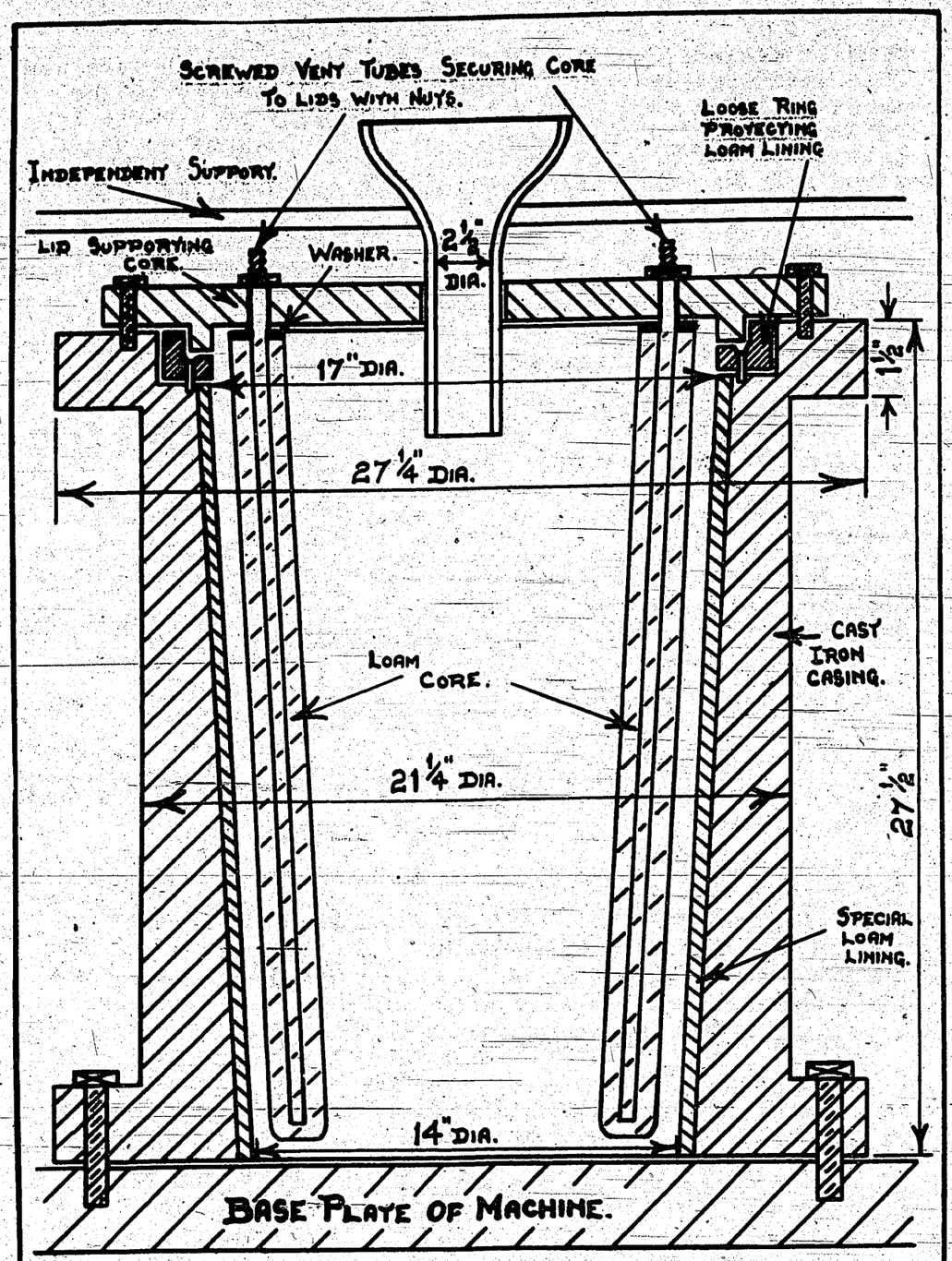

FIG. 1 ASSEMBLY FOR CENTRIFUGALLY CAST COPPER TUYERE.

SIEGLANDER KUPFERWERKE.
SIEGEN, GERMANY.

Target No.3.

Date of investigation - 1st April 1946.

OBJECT OF VISIT.

To examine methods of forging copper tuyeres.

PERSONNEL INTERVIEWED.

Mr Herling - Manager.
Mr Schliefenbaum - Manager.

Works undamaged and in production. In addition to a sand foundry in which there was nothing of interest to note, this firm forged copper tuyeres from blanks cast in cast iron chills. The blanks which formed the nose of tuyeres were first worked on a small power hammer and completed by hand forging over dies placed on a mandrill. The walls of the tuyere were then built up by brazing copper sheet to the nose and a steel top was brazed to the copper sheet.

The method was ponderous and slow, and the finished tuyere which was very rough in appearance did not seem to be altogether satisfactory judged from one seen on test which was leaking badly at the brazed joints.

DANGO UND DIENENTHAL.
SIEGEN, GERMANY.

Target No.4.

Date of investigation - 1st April 1946.

OBJECT OF VISIT.

To examine manufacturing methods of copper tuyeres.

PERSONNEL INTERVIEWED.

Herbert Dango - Director.

Works badly damaged and not in production.

They specialised in the manufacture of tuyeres for blast furnaces. The nose was forged and the body built up of copper or steel sheet. They claimed that their forged tuyeres were superior to the static or centrifugally cast types, and prior to the war were exported to the U.K. and other continental countries.

This firm closed their foundry prior to the war and obtained all their castings from Saar Metallwerke at Saarbrucken 3, with whom they were associated.

As the plant was not working particulars were obtained from Mr Herbert Dango of their manufacturing methods.

Alloy. Cathode copper or electrolytic copper ingots only were used and the metal was de-oxidised with phosphor copper before pouring.

Melting. The copper was melted under charcoal in a gas-fired crucible furnace of usual design.

Casting. The mould consisted of a copper water cooled bedplate on which was laid a hinged mould of cast iron. In the wall of the mould there was a downgate opening into a tangential ingate. A hollow blank was made by inserting a sand or steel core, (see figure 2). The moulds were coated with graphite before casting. Quick chilling made it unnecessary to remove the sunk head and the top sunk surface was used to form the open end of tuyere, the nose portion being formed from the bottom of the blank.

Preheating. This was accomplished in a conventional gas fired muffle furnace where the blanks were preheated to 900°C for one hour.

Forging. Originally the tuyeres were made as hand forgings, but were later hot pressed on a 450 ton vertical Schuler press. Most tuyeres were formed in three operations, split cast iron dies being used for the outside and a steel mandrel for the centre. The lower platen moved upwards in the forming operation against the adjustable top platen. In practice the hollow preheated blank was laid on top of the mandrel and was then pushed by the action of the press down into the die.

Fabrication. Unless the tuyere was short only the nose was made as a forging, the rest of the length being made up of formed copper sheet brazed to the nose. The Tuyere was finally finished by brazing on a cast steel head. - (see figure 3 giving dimensions of a typical forged nose, and figure 4 showing fabricated tuyere.)

The life of a forged tuyere was stated to be one year as compared to a sand cast life of three to six months. It is interesting to note however, that Schumacher of August Thyssen, Hamborn, gave the life of a forged tuyere as 4/6 months.

They also make welded mild steel tuyeres using normal methods, whilst during the war a number of aluminium tuyeres were made on account of the copper shortage. No figures regarding life were obtained for these types of tuyeres.

DANGO UND DIENENTHAL.
SIEGEN, GERMANY.

Target No.4.

Date of investigation - 1st April 1946.

OBJECT OF VISIT.

To examine manufacturing methods of copper tuyeres.

PERSONNEL INTERVIEWED.

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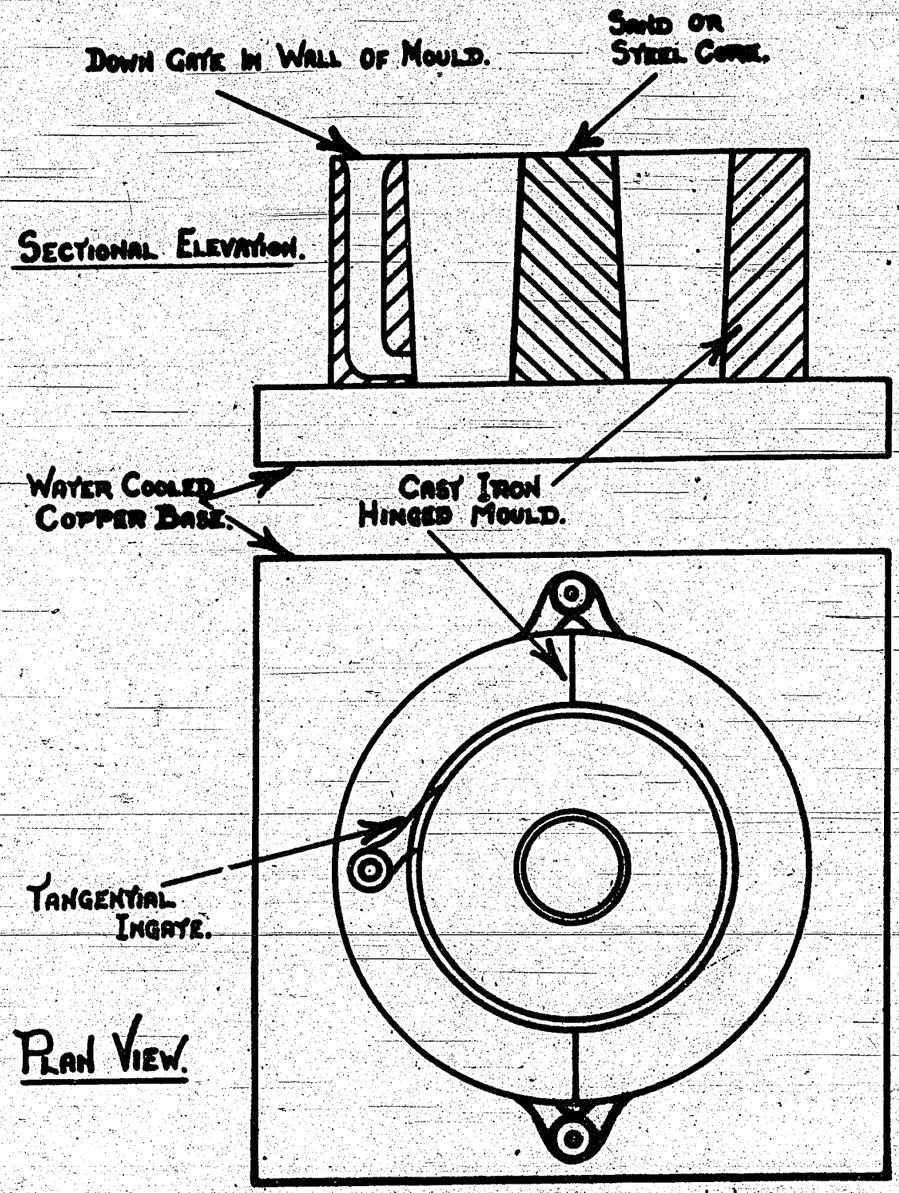


Fig. 2 Chill Mould for Casting Blank Prior to Forging.

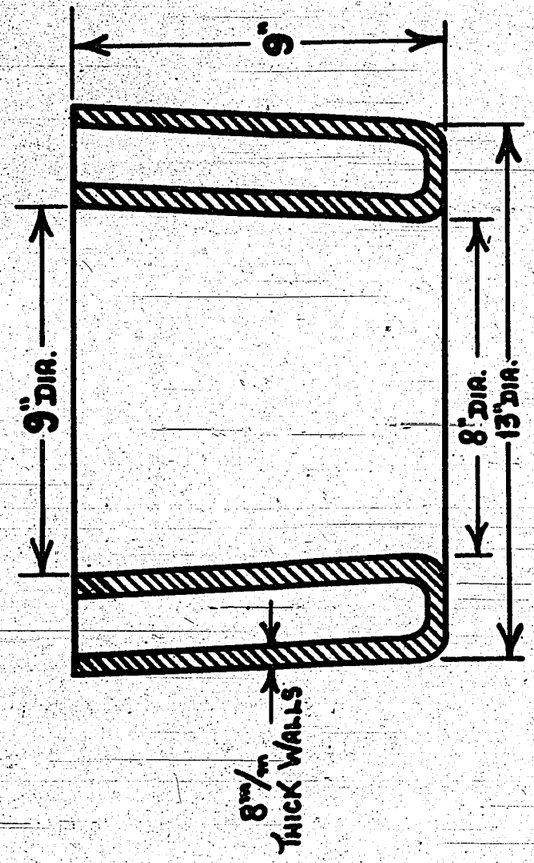
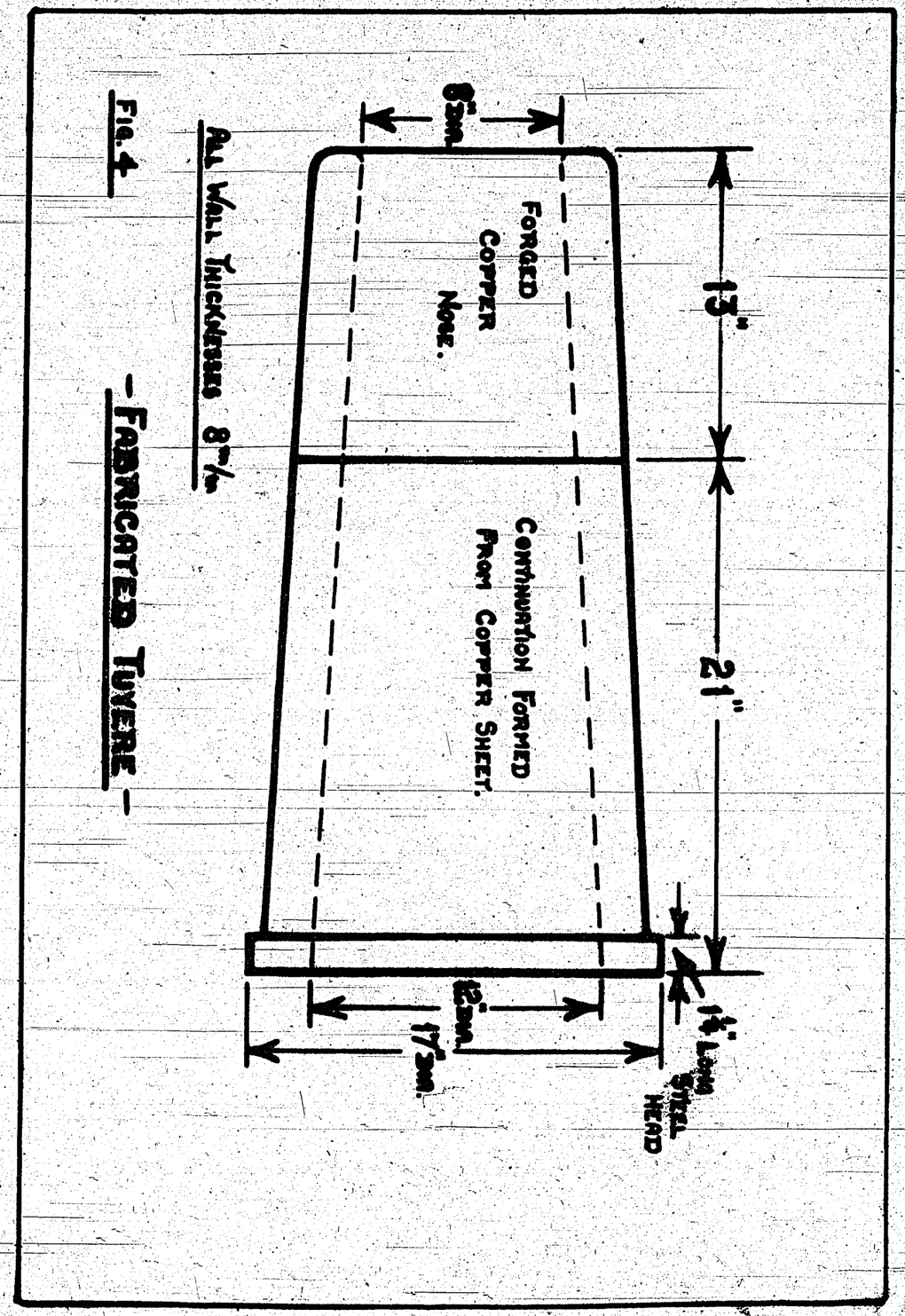


Fig. 3. Forged Tuyere Nose.



RHEIN-EMSCHER ARMATURENFABRIK.
BAERL KREIS MORS, GERMANY.

Target No.5.

Date of investigation - 3rd April 1946.

OBJECT OF VISIT.

To examine methods of manufacturing copper tuyeres.

PERSONNEL INTERVIEWED.

Berhardt Schoer - Tuyere Shop Foreman.

This works was undamaged but owing to power difficulties was not in production at the time visited.

Tuyeres made by this firm were known in Great Britain before the war and had a reputation of being of good quality. Although not in production the plant was therefore inspected.

Copper tuyeres were made from cast blanks bought from an outside foundry. The blanks were preheated in a gas fired muffle furnace before each operation and worked to shape on a specially built machine which consisted of a central revolving table on which the blanks were secured in a hydraulically operated clamp, flanked on either side by revolving steel discs which could be operated either horizontally or vertically.

The blanks were first slit open by the discs rotating horizontally to a distance determined by the bore of the tuyere (figure 5). The split blanks were then placed under a hand operated hammer and flattened until the split portion was almost closed again.

Operation 3 was to place the blank in a recessed ring on the machine and with the discs working vertically to increase the diameter of the top of the blank to the required size (figure 6). Operation 4 repeated operation 3 for the lower part of the blank. With the discs operating horizontally for operation 5 (figure 7), vertically for operation 6 (figure 8) and again horizontally for operation 7 (figure 9), the body of the tuyere was finally forged to shape, the nose still being solid.

The forging was next taken to the machine shop which was well equipped and the nose of the tuyere drilled or punched out according to the size. The top faces were machined and the inner and outer diameters turned to a sufficient distance towards the nose to make them true and concentric. The tuyere was then returned to the forging machine and the outside rolled once more.

A steel top as seen on other makes of tuyeres was then either pressed or brazed on to the body of the tuyere.

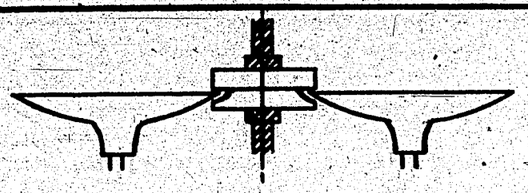


FIG. 5.

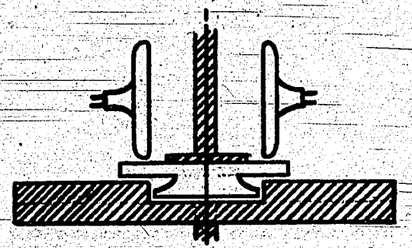


FIG. 6.

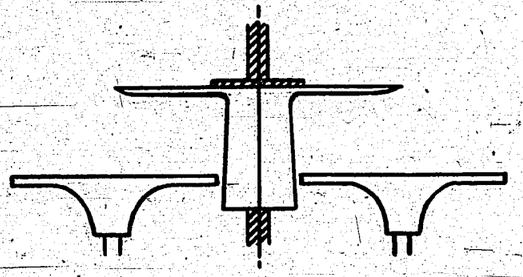


FIG. 7.

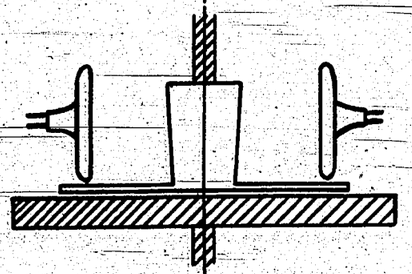


FIG. 8.

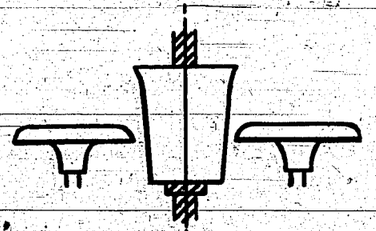


FIG. 9.

RHEIN-EMSCHER ARMATUREN-METHOD OF ROLLING COPPER TUYERE.

CONCLUSION.

While tuyeres manufactured by the forging process are undoubtedly satisfactory, the cost of production is excessive, and it is considered that it would not be an economic proposition to manufacture forged tuyeres in this country. The centrifugal process as seen at Hundt und Weber's works at Siegen is however of considerable interest, and it is recommended that one casting machine together with its equipment should be brought over for operation on an experimental basis.

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FINAL REPORT No. 324
ITEM No. 30

BAND C . REFINERY AT PORTA (Nr. MINDEN)

Brookbank, J., Green, J. A., & Kenyon, W. D.

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LONDON—H.M. STATIONERY OFFICE

BAND OIL FILTER
OIL REFINERY AT
PORTA (Nr. MINDEN).

Reported By

T. BROCKBANK
J.A. GREEN
W.D. WEMYSS

on behalf

of

MINISTRY OF FUEL AND POWER

BIOS Target No. C30/361
Fuels and Lubricants

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE
32, Bryanston Square, W. 1.

TABLE OF CONTENTS

Subject	Page No.
Makers of Filter.....	1
Patent and Construction Designers.....	1
Description of Filter.....	1
Operation of Filter.....	3
Situation of Press in Tunnel.....	3

Personnel of Team

T. Brookbank, Ministry of Fuel and Power

J.A. Green, " " " " "

W.D. Wemyss, " " " " "

MAKERS OF FILTER

Buckan. R. Wolf. Magdeburg.
Model 36. Bandzellen Filter.

PATENT AND CONSTRUCTION DESIGNERS

FIRMA EDELEANU

FRANKFURT a/MAIN.

DESCRIPTION OF FILTER

The filter consists in the main of two parts. The tank and cover which is liquid sealed, and the conveyor (sectional trough) band, within the tank, which resembles a chain grate stoker.

Size. Tank 32' 0" long 15'0" wide 5'8" deep, the cover being 6' 0" high, both being ¼ plate welded.

Weight. Estimated 25 tons.

Working Details from plate.

DAMPFEINTRITT.
HOCHSTER BETRIEBSDRUCK.
1 Kg/cm^2
HEIZRAUM 140/INHALT.

Tank. Rectangular, to size above, with a sealing trough 8" deep x 5" wide all round, Sketch 'A', the cover being immersed in the liquid seal and fastened with 12 light clamping screws. The filter cake end of the tank is formed to a hopper shape which allows the wax from the troughs to fall into a screw conveyor, driven by a 24 H.P. electric motor. The tank is divided into two sections - one forming the hopper, the other being fitted with a connection for withdrawing surplus feed.

Frame for Conveyor. The frame for carrying the conveyor band is built up within the tank.

Cover. Dome shape, with landing edge double rolled to give 2" thickness of edge about 9" up, which forms a stiffening bead. Cover is fitted with 6 circular 12" dia. port lights on either side and two rectangular windows 2'6" x 14" at both ends and sides. 8 - 12" diameter glazed inspection covers with quick release screws staggered on top of cover, and at either end is fitted a lifting lug.

Conveyor. The conveyor is formed by 40 troughs, Sketch 'B', 11'9" long, 1'5½" wide by 8½" deep made out of ¼" plate, linked together to form a complete chain.

Troughs. Each trough is fitted with 8 sectional filter pads cloth covered, and held in position by means of an ⅛" plate strap around the perimeter of the trough. Total filter area 646 sq. ft. The troughs are fitted with a trailing edge which overlaps the straight front face of the following trough. The troughs are tapered at the bottom and the ends blanked but fitted with a 1¾" pipe nipple for a flexible connection to the suction slipper pads. This connection would presumable be in rubber or leather.

On the underside of each trough at each end of it are fitted wheeled carriers which form the links of the chain and which carry the weight of the troughs plus contents, and outboard of these carriers are fitted suction slippers.

Suctions Slippers. The suction slippers, Sketch 'C', which permit the finished product to be drawn from each tray are connected to the 1¾" nipple fitted to the troughs as described above.

The slippers are of cast iron, hollow formed, and fitted with 2 spring loaded pistons. On the underside of the slipper is formed a 4¾" x ⅝" suction port.

The slippers are linked together by means of a heavy cloth fabric which seals the suction and blow ports of the main suction or blow header as the slippers ride over the suction or blow ports. (Contact is metal to metal).

The slippers are also fitted to the wheel carrier brackets to keep them in mesh.

When the slippers pass over the suction and blow ports, the spring loaded pistons are pressed down by means of fixed pressure beams to ensure close contact.

Drive. The carrier links being connected together form an endless chain, which is driven by 2 Hexagon flat faced sprockets 44" across flats which revolve on the main driving shaft, the opposite end being driven by the rotating chain.

The main drive consists of a 60 h.p. Electric Motor which drives the main shaft through a chain of reduction gears.

Operation of Filter

Feed. The oil feed is by means of a 6-way distribution header (Sketch 'D') equally spaced to give equal distribution of feed oil over the trough surface.

Wash liquid. The wash liquid is distributed across the troughs by means of five serrated weirs, Sketch 'D', spaced at suitable intervals over the troughs.

Filtrate. As the troughs are carried along, the suction slippers pass over the suction ports in the main suction header, where the liquid now lying in the bottom of the troughs after having passed through the filter pads, is drawn off.

Filter Cake. At the sprocket end of the chain the troughs now part company to pass round the sprocket. At this point the wax falls out of the troughs or alternatively is blown off when the suction slippers now come in contact with the blow ports on the blow header, Sketch 'D', which is fitted on the underside of the conveyor frame and immediately after the troughs are again linked together by the trailing edge again overlapping the straight face of the following trough.

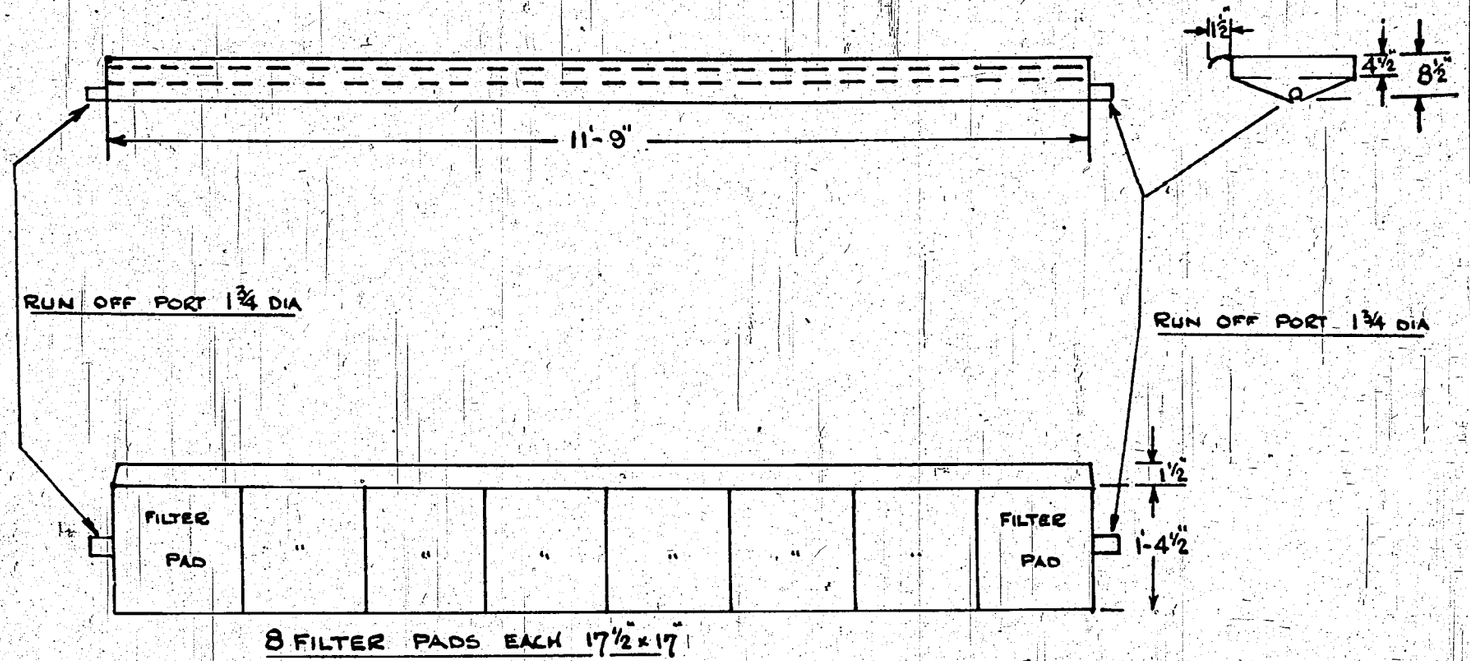
The filter cake leaving the troughs falls into the hopper section of the tank thence into a scroll conveyor which evacuates the filter cake into a main scroll conveyor connected to the remaining filters in the unit.

Suction Ports. 15 suction pipe connections are fitted to the suction headers on each side of the filter.

Blow Ports. The view of the blow ports being obstructed, little information can be given, but they are presumed to be similar to the suction ports.

Situation of Press in Tunnel

The four filters are situated at the end of the dewaxing plant tunnel on a platform some 20'0" high. The access is now considerably restricted by the erection of chiller and other units, hence to remove the filter, it will be necessary to cut the tank and cover up into small convenient units for handling past the obstructions and also for transportation.

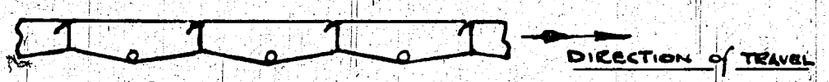


TROUGH FOR "WOLF" BAND FILTER

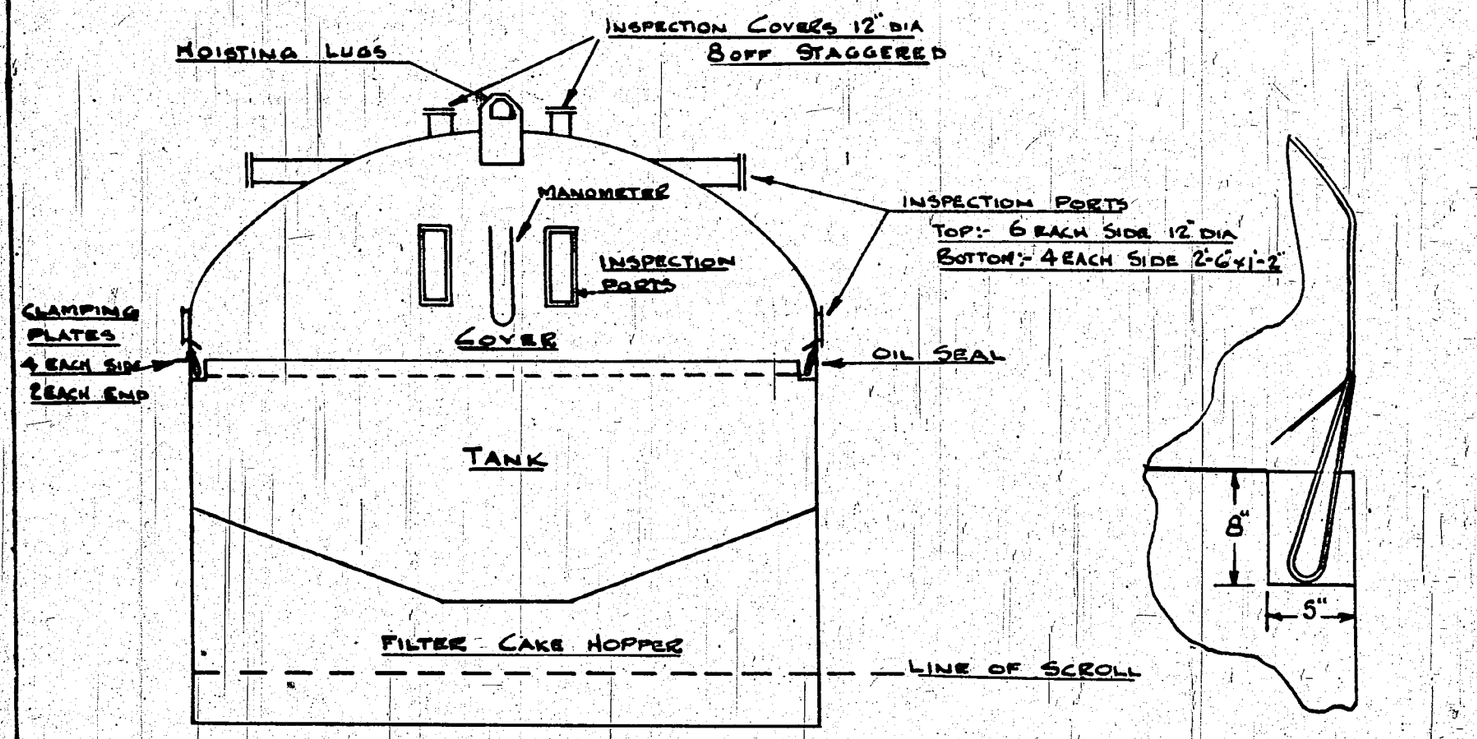
40 OFF — M.S. PLATE

"PORTA" WOLF BAND FILTER

SKETCH:- "B"



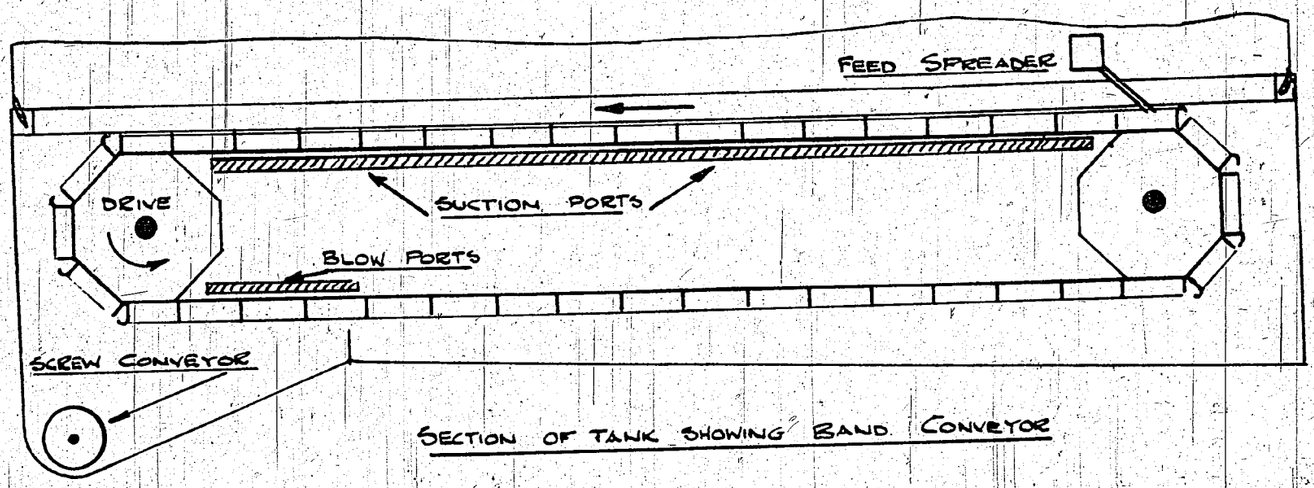
METHOD OF LINKING TROUGHS



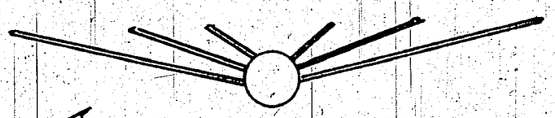
ENLARGED SECTION OF OIL SEAL

SKETCH OF (PORTA) BAND FILTER TANK

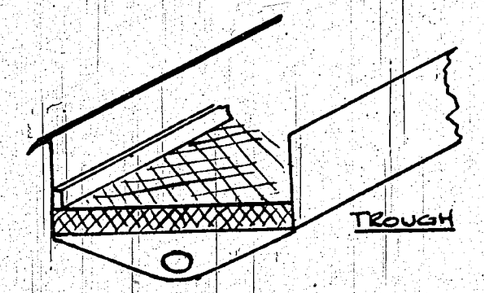
SKETCH "A"



SECTION OF TANK SHOWING BAND CONVEYOR

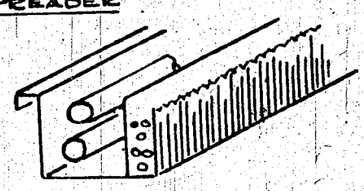


FEED SPREADER



8 SECTIONAL FILTER PADS

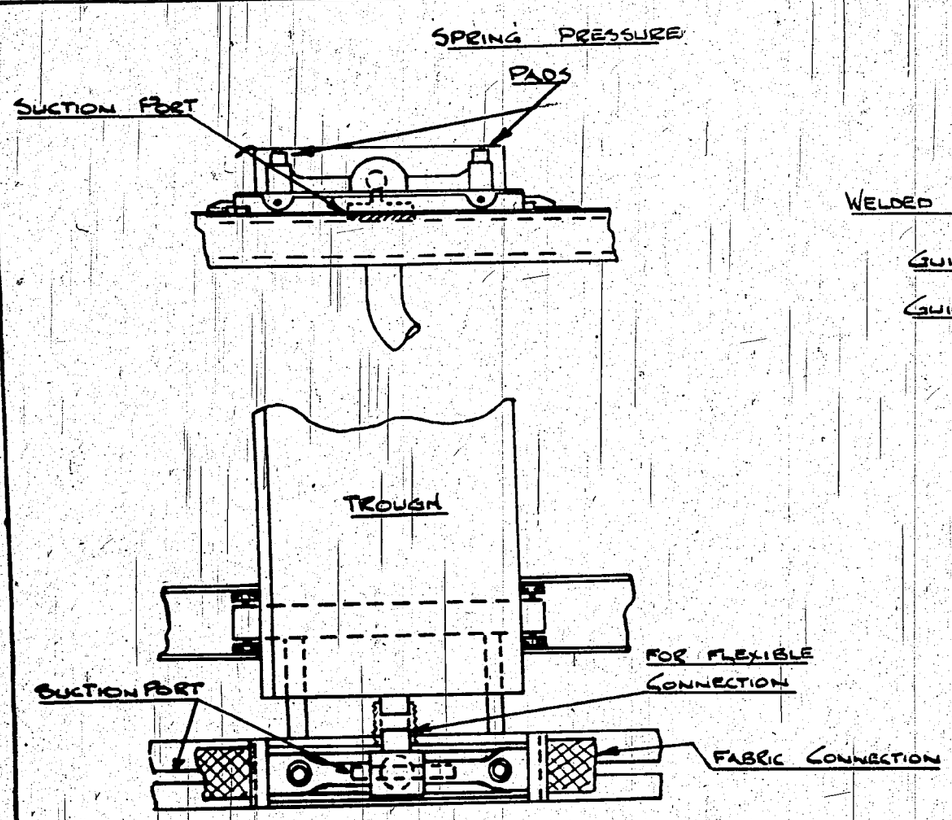
17½" x 17"

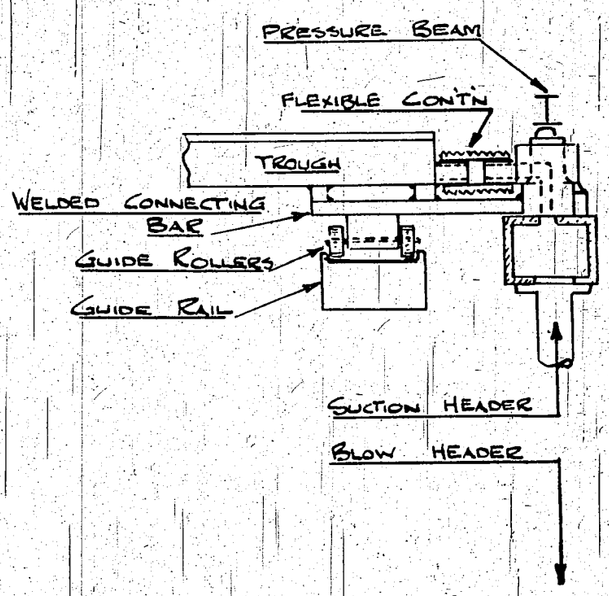


WASH DISTRIBUTION TROUGH

"PORTA" WOLF BAND FILTER

SKETCH D





SUCTION HEADER ASSEMBLY

"PORTA" WOLF BAND FILTER

SKETCH "C"

Copy No. 1

B.I.O.S. FINAL REPORT No. 715
ITEM No. 22

THE MICROANALYTICAL METHODS EMPLOYED IN THE ANALYTICAL LABORATORIES OF I. G. FARBEN, ELBERFELD-WUPPERTAL, GERMANY

Braun, W.

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BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON - H.M. STATIONERY OFFICE

The Microanalytical Methods Employed in the Analytical Laboratories of I.G. Farben, Elberfeld-Wuppertal, Germany.

Reported by:

W. Brown. M. of S.

B.I.O.S. Trip No. 1552

BIOS Target No. 22/1(m)

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

32, Bryanston Square,

London, W.1.

TABLE OF CONTENTS

	Page
Introduction	3.
Description of Laboratory and Equipment	3.
The Automatic Microanalytical Estimation of Carbon and Hydrogen in Industrial Laboratories	4.
The Gasometric Estimation of Nitrogen in Organic Compounds	5.
The Estimation of Chlorine in Organic Compounds	5.
The Estimation of Sulphur in Organic Compounds	6.
The Estimation of Oxygen in Organic Compounds	6.
The Estimation of Acetyl Groups in Organic Compounds	7.

The Microanalytical Methods Employed in the Analytical Laboratories of I.G. Farben, Elberfeld-Wuppertal, Germany

Introduction

A visit to this laboratory was undertaken with the object of discovering what advancement had been made in quantitative organic microanalytical methods in Germany during the "War years". Dr. Karl Tettweiler, who was in charge of the microanalytical laboratory was most helpful and gave every assistance.

Nothing of an outstanding nature was discovered although some of the modifications are interesting and worthy of note.

Description of Laboratory and Equipment

The micro-laboratory was a large room on the ground floor of the building. The heating of this room, which was maintained day and night during cold weather, was thermostatically controlled at 20°C.

The microchemical balances, three in number, were at the far end of the laboratory, mounted on the usual concrete shelves with dust-proof cases and illuminated by tubular fluorescent lamps.

All the tubular furnaces for the various combustion tubes, which were all electrically heated, were automatically switched on one hour before the usual time for commencing work, so that all the combustion tubes were heated up to constant temperature by the time that the staff arrived, and work could be commenced straightaway.

The various tubular furnaces were thermostatically controlled at the optimum temperature for the particular estimation for which it was used.

The combustion tubes were all of clear quartz, and all the movable heaters were automatically moved along the tube at a uniform rate, either by means of a clockwork arrangement or by means of a revolving screw driven by a small motor. Each apparatus had an arrangement whereby a bell rang when the movable heater reached the long burner so as to call the attention of the analyst who then proceeded with the next step in the estimation.

Most of the modifications used in this laboratory are based on the time saving and automatic methods recommended by Dr. Zimmermann of I.G. Farben, Ludwigshafen. In these methods the personal element is reduced to a minimum in order that anyone of laboratory assistant grade, having average ability, can quickly become proficient in carrying out the analyses. Dr. Tettweiler had four such assistants, but in order to keep a check on results, every analysis is carried out in duplicate.

The methods, which will be briefly described, are to be found in the journals to which references are given. These journals are now available in this country.

The Automatic Microanalytical Estimation of Carbon and Hydrogen in Industrial Laboratories W. Zimmermann, Mikrochemie 31, 149 (1943)

The principal features of the apparatus used for this estimation are the automatic movable heater, and the automatic wiper which is used for wiping the absorption tubes before weighing.

The apparatus, which is electrically heated and thermostatically controlled, consists of the usual heating mortar, long heater, movable heater and pre-heater.

The quartz combustion tube has the usual dimensions and filling, except that pumice stone impregnated with pumice is preferred to silver wire.

The movable heater is pulled along the combustion tube by means of a cord which is attached to the minute hand of an alarm stop-clock. The alarm bell rings when the movable heater has reached the long heater. The analyst then completes the final quick heating of the tube with a bunsen burner.

Oxygen only is used for sweeping out the combustion tube. The absorption tubes, which are the Pregl type, are removed from the apparatus, the ends cleaned out with the usual wire and cotton wool, wiped on the automatic wiper for 30 seconds, and immediately weighed filled with oxygen.

The automatic wiper consists of a small motor with an elongated spindle. Some pieces of chamois leather are attached to the spindle; these rotate and sweep over the surfaces of the absorption tubes which are held in a metal frame that fits into the box enclosing the wiper.

Two control absorption tubes, which lie alongside the absorption tubes during the combustion, are used as tares. These are filled with the same absorption reagents and are given the same treatment, except that no gases are pased through them.

Several automatic movable heaters have been described by American authors, but the automatic wiping of the absorption tubes is quite novel.

Automatic movable heaters do not find favour with some micro-analysts owing to the variable behaviour of different compounds on heating. Dr. Tettweiler, who has used this type of apparatus for some time, claims that it works equally well with any type of compound.

The Gasometric Estimation of Nitrogen in Organic Compounds
H. Gysel. Helv. Chim. Octa 22, 1088 (1939)

The method used here differs from the usual one in that it is not necessary to remove the combustion tube from the furnace each time a fresh substance is to be analysed. The substance is weighed into a boat, covered with a layer of copper oxide and manganese dioxide, and introduced into the combustion tube on the front end of a roll of oxidised copper gauze which has a little platinum scoop to hold the boat.

The arrangement of the copper oxide and reduced copper filling of the combustion tube differs only slightly from the usual method. In order to prevent air entering the combustion tube and oxidising the hot reduced copper during the introduction of the boat, carbon dioxide is led into the combustion tube from the nitrometer end, that is, in the reverse direction. When the oxidised copper gauze with the boat has been pushed into the combustion tube and the stopper replaced, the current of CO_2 is again reversed and led in through a side arm on the tube about 1 cm. from the stopper.

The source of CO_2 is either a Kipp's apparatus or a vacuum flask containing cardice.

This method is a definite improvement on Pregl's and has much to recommend it, particularly in laboratories where many nitrogen analyses are carried out.

The Estimation of Chlorine in Organic Compounds

The substance is decomposed in the usual way either in a combustion tube or by the wet oxidation method of Zacherl and Krainick. In both cases the halogen is absorbed in a bead tube containing dilute caustic

soda with hydrogen peroxide. This bead is jointed to the combustion tube. After the combustion the sodium halide is washed out with glacial acetic acid, and the solution titrated potentiometrically using Ag/AgCl electrodes.

The potentiometric titration method for chlorine gives results which are as accurate as the gravimetric method, and the time for an analysis is cut almost by half.

The type of combustion tube used for this estimation is that described by Wurzschmitt and Zimmermann, Zeitschrift für Analytische Chemie, 114, 6450. (1938). This tube differs in having two separate gas streams. A stream of air enters at one end of the tube and a stream of oxygen enters by a fused-in side tube just in front of the long heater. Almost immediately following this is a fused-in diaphragm with a 2 mm. hole. These extra parts ensure better mixing of the organic vapours with oxygen; the function of the air stream is merely to wash the mixture through.

The Estimation of Sulphur in Organic Compounds

Wurzschmitt and Zimmermann's combustion tube is also used for the estimation of sulphur. The bead tube is replaced by a tube having a roll of silver gauze fixed into it. The silver reacts with the SO_3 to form silver sulphate which can be dissolved off and titrated potentiometrically with standard sodium chloride solution. The part of the tube containing the silver gauze is maintained at a temperature of about 600°C. (just below the melting point of silver sulphate). Chlorine does not react with silver under these conditions and therefore does not interfere.

The Estimation of Oxygen in Organic Compounds
J. Unterzaucher, Berichte, 73, 391 (1940).

In this estimation the substance is decomposed in an atmosphere of oxygen-free nitrogen, and the products of the thermal decomposition passed over carbon at 1120°C. where any oxygen is quantitatively converted to carbon monoxide. The carbon monoxide is then led over iodine pentoxide where the well-known reaction takes place:

$$5\,CO + I_2O_5 = 5\,CO_2 + I_2.$$

The liberated iodine is driven on by heat into a removable bead tube where it is absorbed. The iodine can either be titrated with thiosulphate or by Leipert's volumetric method.

The nitrogen is freed from traces of oxygen by passing it over carbon at 1120°C., and the carbon monoxide removed by iodine pentoxide, the iodine being absorbed by KOH.

The success of this method depends on the quality of the iodine pentoxide, and a method that yields a highly active preparation is given by M. Schutze, Berichte, 77, 484 (1944).

This method gives fairly accurate results but is liable to give trouble occasionally due to blanks being exceptionally high.

The Estimation of Acetyl Groups in Organic Compounds
E. Wiesenberger, Mikrochemie, 30, 241 (1942).

The method consists of splitting off the acetyl group in the presence of alcohol, distilling off the ester into standard sodium hydroxide solution; and after saponification titrating back the unused soda.

Any small differences in the methods summarised above, from those given in the literature, are modifications due to Dr. K. Tettweiler.

WB/RP.

Copy No. 1

B.I.O.S. FINAL REPORT No. 171
ITEM No. 22

CATALYTIC HYDGROGENATION OF ACETYLENE TO ETHYLENE, DR. ALEXANDER WACKER GES. FUR ELEKTROCHEMISCHE INDUSTRIE, BURGHAUSEN.

Cambrau, A.

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LONDON—H.M. STATIONERY OFFICE

Catalytic Hydrogenation of Acetylene to Ethylene

Dr Alexander Wacker Ges. fur Elektrochemische Industrie, Burghausen

September, 1945

Reported by

A. Cambron - Canadian Investigator

BIOS Trip No. 1155

BIOS Target No 22/1

" " " 22/42 (b)

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32 Bryanston Square, London, W.1.

CONTENTS

Para	Subject	Page
1	Introduction.....	1
2	Description of Plant -	
	Part I - Design of Catalytic Converter..	1
	Part II - Operating Procedure.....	1
	Part III - Recovery of Ethylene.....	2
	Appendix 1	
	Appendix 11	

SUMMARY

The process in use at Gendorf, which was an I.G. development, could not compete with other sources of ethylene when the latter is obtainable from petroleum refinery operations or when alcohol may be produced from cheap molasses. It provides, however, a convenient alternative source of ethylene when other sources are not available.

Personnel of Team

A. Cambron

1. Introduction

The ethylene plant at Gendorf has been the subject of a previous investigation, the results of which were reported by R.M. Hunter in C.I.O.S. Report No XXV-17. The present investigation was undertaken particularly to obtain additional information on the design and operation of the catalytic reactor, on the purification of the acetylene, and on catalyst stability and life.

The personnel interviewed included Major M.G. Rogers, U.S. Army, Control Officer at the Plant, Dr Gruber, Plant Superintendent, and Dr Ekers Berger, works chemist in charge of ethylene production.

2. Description of Plant

Part I - Design of Catalytic Converter

A detailed drawing of the catalytic converter is attached as Appendix I. The converter consists of a cylindrical steel case, 2.8m diameter and 6.5m high, through which the reacting gas flows downwards through three 23cm layers of Pd catalysts. The catalyst consists of Pd deposited on 0.5cm silica gel, and is evenly distributed on wire mesh trays (45). The temperature of the gases emerging from the first and second catalyst beds is brought down to 250 - 300 C by a water spray in the gas stream, the amount of water being regulated by hand in order to maintain the gas temperature within the set limits. The gas temperature is determined by thermo couples in the catalyst bed and in the gas stream. Another feature is a spiral baffle placed under each water spray in order to force the gas into a circular path, thus improving the contact between the gas and the evaporating spray.

Part II - Operating Procedure

In operation two catalytic converters are converted in parallel as shown in the flow diagram attached as Appendix II. When a unit was started the acetylene-hydrogen mixture was by-passed through steam jacketed line D in order to raise the temperature of the system to reaction level. After the exothermic reaction had started, the gases were recirculated through line C. The

recirculation rate through blower B was 35,000 cu m per hr. When the system had reached equilibrium, 500 cu m of gas, calculated on a steam free basis, were removed per hour as product, and an equal volume of acetylene along with a slight excess of hydrogen were fed continuously into the system. The excess of hydrogen varies inversely with the activity of the catalyst, and is regulated to maintain the concentration of acetylene in the product below 0.8%. The activity of the catalyst gradually decreased on prolonged use. When excess hydrogen was no longer effective in maintaining the concentration of acetylene in the gas product below 0.8%, the off gas from the two parallel converters was passed through a third converter, (III), as shown in the attached flow diagram.

The catalyst life was 3 to 4 months. The decrease in activity being due mainly to the deposition of acetylene polymers on the catalyst. Reactivation of the catalyst is done at Gendorf by passing steam and air over it at 600°C. The preparation of the catalyst was done at Ludwigshafen, (I.G. Farben).

The hydrogen used in the process was obtained mainly by hydrolysis, but some hydrogen was produced by the water-Fe reaction. Since the presence of sulphur was particularly bad in causing poisoning of the catalyst, both the hydrogen and the acetylene were subjected to a special treatment to remove this impurity, and to remove any PH_3 present in the acetylene. In addition to the usual hypochlorite, water and caustic soda scrubbing, it was found necessary to pass the acetylene feed through active charcoal absorbers to remove higher polymer and prevent fouling of the catalyst.

Part III - Recovery of Ethylene and Yield

The composition of the crude ethylene product was reported to be as follows:-

50 - 60%	Ethylene, by volume
3%	Ethane
4 - 6%	C_3 and higher
0.8%	Acetylene
diff.	Hydrogen

The recovery of the ethylene was done by liquifaction and fractionation in a Linde plant. The hydrogen residue was returned to the hydrogenation plant.

The composition of the final ethylene product, which was piped to another part of the plant for conversion to chlorohydrin or for chlorination, was as follows:-

96% - Ethylene
2% - Ethane
1% - Nitrogen
1% - Hydrogen

The reported yield of ethylene, based on acetylene, was 84% in the hydrogenation step, the remainder being converted to ethane or to higher polymers.