

The amounts of hydrogen and nitrogen in these gases was approximately:-

	H <sub>2</sub>		N <sub>2</sub>	
	%	M <sup>3</sup> /hr	%	M <sup>3</sup> /hr
40 ats. coal L/D	61	2,700	6.0	260
40 ats. gas washing	61	11,700	6.0	1,150
20 ats. Sat'n L/D	71	1,650	15.4	355
20 ats. splitting	68	1,490	13.8	300
		<u>17,540</u>		<u>2,065</u>
H.O.L.D. L/D gas	71.4	3,700	6.8	350
Gas washing 7 ats. L/D	24.6	<u>330</u>	4.0	<u>54</u>
		4,030		404
Coal rich gas	5.86	410	1.9	135
Vapour Phase rich has	7.3	<u>370</u>	2.5	<u>125</u>
		780		260

Total dissolved H<sub>2</sub> = 22,350 m<sup>3</sup>/hr = 16.5% on make up

Total N<sub>2</sub> = 2,729 m<sup>3</sup>/hr = 2% on make up H<sub>2</sub>

Total m.u. hydrogen 135,000 m<sup>3</sup>/hr

The Linde plant treated the lean gases for recovery of hydrogen and at the same time provided a purge of nitrogen; the bulk of the nitrogen purge was via the Linde Plants. Out of 22,000 m<sup>3</sup>/hr of hydrogen lost by solution in products, 15,000 m<sup>3</sup>/hr or about 70% was recovered. The gas fed to the Linde plants contained about 60% H<sub>2</sub>. Precooling of the gas after compression was done by ammonia which had to be imported and not by liquid propane.

Compression

About 2/3 of the total make up hydrogen was required by the coal stalls at a pressure of 700 ats, the remaining third was used at 325 ats. pressure. The 325 ats. compressors for H<sub>2</sub> gas were 13 in number. They were the usual horizontal machines driven by 5000 volt 4000 kW motors. Each compressor had a suction rate of 16,200 m<sup>3</sup>/hr and a delivery of 10,000 m<sup>3</sup>/hr. (Stages 1 - 3 up to 28 ats. Stages 4 - 6 28 - 325 ats.). Four machines compressed the hydrogen from the Linde plants to 325 ats. There were nine 325 - 700 ats. machines for the coal stalls. These were 10,000 m<sup>3</sup>/hr nominal capacity at 300 ats. suction pressure. At 325 ats. suction pressure they would produce about 98,000 m<sup>3</sup>/hr.

Fuel Gas

The water gas plant produced about 25,000 m<sup>3</sup>/hr of water gas of calorific value 2,000 te cal/1000 M<sup>3</sup>. This was mixed with surplus lean gas and Linde residual gas to provide a fuel gas with a calorific value of 3500 - 4000 te cal/1000 M<sup>3</sup>. The distribution of this fuel gas in the factory and to the coke ovens at Nordstern is illustrated below. The specific consumptions for the coal stall preheaters, coal drying and sludge recovery have been calculated. These figures can only be regarded as a reasonable approximation as gas balances are never very accurate. The mixing and control of the fuel gas system was at the water gas plant.

	August 1942	Feb. 1943	May 1943	Sept. 1943
N. Water Gas m <sup>3</sup> /hr	25,600	26,658	23,590	24,110
Lean Gas "	10,000	10,238	6,480	3,181
Linde residue "	6,940	3,366	6,132	8,163
H <sub>2</sub> S gas "	120	227	266	497
Total	<u>42,660</u>	<u>42,600</u>	<u>36,349</u>	<u>36,779</u>
Calorific Value te cal/1000 M <sup>3</sup>	3,774	3,610	3,690	3,977
Te/hr cal available	161,000	153,000	134,000	147,000
	Te cal/hr	Te cal/hr	Te cal/hr	Te cal/hr
To stalls te/cals/hr	57,700	52,000	50,000	43,500
To Stills	27,500	30,300	27,600	25,600
Kiln ovens	11,000	10,500	10,600	9,300
Coal grinding	3,500	4,500	4,300	3,800
Coke ovens (Nordstern)	39,600	23,000	23,000	35,000
Boilers	18,400	31,400	14,700	26,600
Total	<u>157,700</u>	<u>151,700</u>	<u>130,800</u>	<u>143,800</u>
Raw Coal tes/hr	85.5	81.6	81.5	74.0
a.m.f. coal tes/hr.	74.5	71.0	70.6	64
Feed to kilns tes/hr (calc)	24.1	22.9	22.8	20.5
Te cal/to coal ground	41	55	53	52
Te cal/to kiln feed	455	460	465	452
Fuel gas to Stalls te cal/to a.m.f coal	770	730	710	680
Fuel gas to Stills - do -	370	430	390	400

Water Gas and Hydrogen Sets (cont'd)

	August 1942	February 1943	May 1943	September 1943	Date ?	Date ?
<b>Alkacid</b>						
H. gas m <sup>3</sup> x 10 <sup>6</sup>	81.4	74.57	82.93	78.92	79.61	82.91
H <sub>2</sub> S gms/m <sup>3</sup>	3.95	3.91	3.89	3.813	3.96	4.1
After rough wash. gms/m <sup>3</sup>	.536				.432	.533
After final wash. gms/m <sup>3</sup>	.102	.130	.159	.180	.100	?
L.P. Steam used tes.	16,850				13,247	12,705
tes/1000 m <sup>3</sup>	.207				.166	.153
Electric Power KWH/1000 m <sup>3</sup>	2.53				2.33	2.23
<b>Hydrogen Plant</b>						
Gas to Plant x 10 <sup>6</sup> m <sup>3</sup>	80.49		81.92	77.80		81.75
Regen Gas x 10 <sup>6</sup> m <sup>3</sup>	8.53		9.45	9.40		8.13
Ratio cat.gas/inlet <sup>3</sup>	1.53		1.35	1.35		1.33
L.P. Steam t/1000 m <sup>3</sup> inlet gas	.461					.426
Electric Power KWH/1000 m <sup>3</sup> inlet	2.99					3.56

**B. Linde Plants**

The gases produced from the stalls comprised dissolved hydrogen equivalent to about 1% of the total make up hydrogen, hydrocarbon gases made in the process and nitrogen from the make up gas. CO<sub>2</sub> and H<sub>2</sub>S are also present. It was desired to recover the maximum amount of propane and butane, for use as motor car fuel (Treibgas) and iso-butane for iso-octane manufacture at Sololven.

The gases dissolved in the stalls catchpot products at pressure, and in the gas washing oil were fractionated in the usual way by letting down the oil in stages. This gives hydrogen rich fractions (lean gases) and rich gases containing the bulk of the higher hydrocarbons C<sub>3</sub>+C<sub>4</sub>. A sample flowsheet is shown here for the gas system. (Note this flowsheet is not balanced in every respect in particular the N<sub>2</sub> content of gas to the Linde Units.) For convenience this flowsheet is placed at the end of the report.

Summary of gas system

(a) Lean gases

60-70% hydrogen.

coal stall	40 ats. let down	} To Linde fractionation and fuel gas.
gas washing	40 ats. let down	
saturation	20 ats. let down	
Splitting	20 ats. let down	

Purge of circulating gas

(b) Gas washing 7 ats. let down To fuel gas  
H<sub>2</sub> 24.6% N<sub>2</sub> 4.0% CH<sub>4</sub> 42.6%  
17% C<sub>2</sub>H<sub>6</sub>

(c) Gas from H.O.L.D. and kilns To compression for Hydrogen make up  
70% H<sub>2</sub>

(a) Rich gases

5 - 8% H <sub>2</sub>		} To petrol light ends and propane, butane, recovery
Coal Stall	1 at. let down	
Gas Washing	"	
Saturation	"	
Splitting	"	
Coal Stall & Vapour Phase Stall dissolved gases-ex stills	"	

table it will be seen that the efficiency of the generators as regards coke and steam consumption was not better than is possible in this country. The normal nitrogen content of H. Water Gas appeared to be about 1.7%. H<sub>2</sub>S removal was done by the alkacid process using DLK liquor (aqueous solution of impure potassium dimethyl glycine). The process was conducted in plant constructed of aluminium. Three plants were used, one for H. Water Gas, a second one for the recovery of H<sub>2</sub>S from lean gases before the Linde plant and a third for recovering H<sub>2</sub>S from rich gases before the propane-butane recovery plant. For some reason unknown the alkacid plant running on H. Water Gas had suffered from corrosion caused by the accumulation of iron sulphide in the liquor which resulted in contact corrosion of the aluminium. The H<sub>2</sub>S content of the purified H. gas seems to be rather high - .102 - .108 gms/M<sup>3</sup>.

Conversion of the CO to hydrogen was said to be done with standard Brown Oxide catalyst (iron oxide plus chromium oxide). CO<sub>2</sub> removal was done by water scrubbing at 25 ats. pressure and the final CO removal with ammoniacal copper carbonate at 325 ats. pressure.

Water Gas and Hydrogen Sets

	August 1942	February 1943	May 1943	September 1943	Date ?
Total BWG 10 <sup>6</sup> m <sup>3</sup>	100.4	92.48	100.41	96.28	
HWG	81.4	74.57	82.93	78.92	
N. Gas	19.0	17.91	17.48	17.36	
Generator : Hrs. run.	11,842	15.6	16.1	15.6	
Generators run	8,480	8,830	8,381	8,530	
Output/Gen/hr/ m <sup>3</sup>	56,100	49,800	55,200	53,300	
Dry coke. tes	.559	.540	.550	.523	
Coke/1000m <sup>3</sup> BWG	76,609	67,520	77,809	74,513	
17 ats. Steam made tes.	46,678				
Export	23,887				
Used in generators	2,044				
" in turbines					
Total L.P. Steams (2.5 ats.)	123,163				
to generators tes.	1.23				
Steam/1000 m <sup>3</sup> BWG					

1943 Productions & Consumptions

	Tar Oils tes.	Raw Coal tes.	Petrol VT. 707 tes.	Treibgas tes.	1-C <sub>4</sub> tes.
January			28,940	7,106	1,124
February			26,490	6,726	1,275
March	2,549	60,971	32,130	7,673	1,681
April			29,560	6,553	1,842
May	1,999	60,707	31,080	7,363	1,793
June	2,390	57,500	28,580	7,100	1,764
July	2,336	54,825	27,450	6,370	1,864
August	2,194	46,907 <sup>□</sup>	22,570 <sup>□</sup>	5,270	1,086
September	2,433	53,263	27,100	6,829	1,548
October	2,411	59,095	27,350	7,306	2,011
November	-	-	-	-	-
December	2,623	57,723	27,170	6,385	2,015
			<u>308,330</u>	<u>74,681</u>	<u>18,003</u>
Total May- Dec.	<u>16,386</u>	<u>390,020</u>	<u>191,300</u>	<u>46,623</u>	<u>12,081</u>

□ Air raid - 72 bombs.

Note : The figures for consumption and production in November are missing. The figures for petrol made Jan-Aug. have been checked from another document. From another document the total production for 1943 was found to be 335,000 tes.

1944	Tar Oils tes.	Raw Coal tes.	Petrol VT. 707 tes.	Treibgas tes.	1-butane tes.
January	2,590	63,269	30,040	7,754	1,860
February	4,779	48,792	26,040	6,170	1,665
March	4,545	62,401	31,470	7,801	2,030
April	4,668	58,016	30,330	6,997	-
Total :	<u>16,582</u>	<u>232,478</u>	<u>117,900</u>	<u>28,632</u>	

The figures quoted for raw coal are believed to refer to wet coal. The figures available in the document for ash and moisture in the coal have not been examined in detail but reference to other data suggests that the average a.m.f. coal on raw coal was 8%. Making allowance for the tar middle oils, it can be calculated that the yields of aviation petrol for the periods May-Dec. 1943 and Jan-April 1944 were 52.1% and 51.4% respectively. These figures cover all losses in storage and handling. From the average figures given in records for new formed oilm 6% containing 64% petrol and middle oil, the petrol yield is calculated to be 54.6% on the a.m.f. coal. This calculation allows nothing for losses in refining the liquid phase petrol, for handling losses on finished petrol, and for the reduced yield of petrol in the vapour phase stalls after the introduction of 7846 catalyst. (This reduction on account of 7846 catalyst is of the order of 1.5% on the coal).

IV. HYDROGEN PRODUCTION

A total of 135,000 m<sup>3</sup>/hr of hydrogen was required. 100,000 m<sup>3</sup>/hr was made from coke via water gas, 15,000 m<sup>3</sup>/hr was made from stalla lean gas by fractionating in a Linde Plant. 15-18,000 m<sup>3</sup>/hr. were imported from Huls and and 5,000 - 6,000 m<sup>3</sup>/hr. of gas from H.O.L.D. and the sludge kilns containing 60-70% hydrogen was put straight to the compressors.

A. Water Gas

The plant had 20 Demag generators, each with an output of 8,600 m<sup>3</sup>/hr. of blue water gas. 15-16 generators were usually on line. Each generator had an overhead bunker, the bunkers were fed by a crane travelling along the top of the building. The generators were fitted with vertical dust cyclones delivering to horizontal waste heat boilers generating steam at 17 ats. pressure. It was not possible to see the generator control gear, this had been either removed or in some cases destroyed by bombing. It was noted that the ash extractors were tiled to reduce wear. The plant was very well laid out. The generators were in a building but all the auxiliary equipment was outside. Although oxygen was produced in the Linde plants there was no evidence from records that it was used on the water gas generators; we were told that it was put to atmosphere and some was bottled. From the figures given in the following

The annual productions and consumptions are estimated to be as follows:-

1940	Raw Coal	Petrol	Treibgas
	tes.	tes. +	tes.
January	10,280	4,446 (4446)	-
February	13,232	7,680 (7675)	0,750
March	15,570	8,223 (8195)	1,377
April	16,745	9,395 (9402)	2,604
May	13,636	7,589 (7606)	1,667
June	20,170	12,060 (12074)	1,950
July	25,421	14,044	3,258
August	29,217	15,206	4,424
September	25,746	12,694	3,805
October	12,097	7,627	2,088
November	17,840	9,159	2,581
December	22,011	13,027	3,451
	<u>221,965</u>	<u>121,150</u>	<u>27,955</u>

+ 354 Fremd benzin.

Figures in brackets were obtained from a different set of documents. The petrol make was also checked from a graph. Figures for tar treated are not yet available.

1941	Raw Coal	Petrol	Treibgas	
Total	tes.	tes.	tes.	
January	23,939	11,806 (11823)	2,968	
February	20,133	12,007 (12028)	2,948	
March	32,078	16,189 (16208)	4,511	
				Aromatised
				part petrol made
				Aroma- CV 2 BL and
				tised CV 2B.
				tes.
April	33,992	15,592 (15634)	3,757	2,619
May	32,308	14,950 (15000)	3,805	?
June	36,031	15,337 (15392)	4,270	5,598
July	42,303	18,090 (18172)	4,281	7,178
August	40,338	18,042 (18127)	5,379	5,086
September	38,613	18,840 (18928)	4,080	907
October	48,576	22,318 (22318)	5,203	991
November	40,173	19,000		?
December	50,027	24,500		?
Total :	<u>438,511</u>	<u>206,671</u>		

Note: The figures for coal consumption were checked by reference to two separate documents. Figures in brackets are from another set of documents. The catalyst used, averaged for the year was -

FeSO <sub>4</sub> catalyst (22.8% Fe)	1.1%	on coal.
Bayer Masse (29.6% Fe)	1.6%	
Na <sub>2</sub> S	0.3%	

1942.

Figures for 1942 were not readily available for abstracting on site.

For the year October 1941 to September 1942 inclusive, the output was given as 294,495 tes. In a report we obtained the monthly output of petrol was given in tonnes/hr. for the period November 1941 to August 1943. From these figures it appears that the output in 1942 was -

January	24,600	(some aromatised petrol made)
February	20,300	
March	23,300	
April	23,900	Check
May	24,400	Output Oct/41-Sept.42 294,495
June	29,400	Output Oct-Dec. 1941. 65,818
July	27,600	228,677
August	29,800	Output Oct-Dec. 1942. 89,000
September	28,600	
October	29,400	(29,278) Output 1942. 37,677
November	29,200	(29,367)
December	30,400	(30,268)
Total:	<u>320,800</u>	tes. The difference is probably accounted for between petrol actually made and petrol despatched.

The purge of solids required to maintain the concentration of solids in the pasting oil at about 10%, was obtained by diluting the H.O.L.D. and concentrating the solids by centrifuging. The concentrate was treated in rotary kilns, (Kugelovens) for recovery of the oil associated with the solids, the whole of the asphalts in the oil were lost as coke and in effect only 80% of the oil was recovered. The oil lost amounted to 4.5% on the a.m.f. coal, 2.5% of this was accounted for by asphalts. The recovered oil was recycled to pasting oil. The vaporised oils from the hot catchpot were condensed by cooling and the liquid product and the circulating gas were separated in the high pressure separator. The oil was let down in stages (40 ats., 1 at.) to fractionate the dissolved gases. The oil was separated by distillation into "A" middle oil boiling up to 335-345°C, which was treated in the vapour phase, and heavy oil which was used as diluent for fugal feed and for paste making. The conditions of operating the coal stalls were such that the new formed oil from the coal contained only sufficient heavy oil to balance the loss of heavy oil in sludge recovery. About 10% of petrol on the a.m.f. coal was made in the coal stalls. Up to August 1942 this petrol was taken out of the "A" middle oil and it was refined separately to remove phenols and bases by treatment over 5058 catalyst (W.S<sub>2</sub>). After August 1942 the whole of the petrol and middle oil was treated in the saturation stalls over 5058 catalyst. Some petrol was made in the saturation stalls by splitting of the middle oil. The main object of this stage was to remove phenols and nitrogen compounds. No phenols were separated from the liquid phase petrol.

The ammonia liquor from the coal stalls and the vapour phase stalls was treated with CO<sub>2</sub> to recover H<sub>2</sub>S. The H<sub>2</sub>S together with H<sub>2</sub>S from the alkacid scrubbery of rich gas, was converted to 88% sulphuric in a large Vanadium catalyst plant. Part of the H<sub>2</sub>S was added to the splitting stalls feed to maintain the partial pressure of H<sub>2</sub>S over the catalyst.

The saturation stall product was distilled together with the product from the splitting stalls. The bottoms from the vapour phase stills ("B" middle oil) consisted of saturated middle oil boiling from 155°C to about 300°C and recycle middle oil from the splitting stalls. This constituted the feed to the splitting stalls. The splitting stalls contained 6434 catalyst (WS<sub>2</sub> on activated Terrana Earth) and 5% of the saturated middle oil was converted per pass to petrol of end point 155-160°C. This catalyst is not tolerant to more than traces nitrogen compounds and ammonia.

After October 1942 the shortage of tungsten had become acute and it was necessary to replace the 5058 in one converter in each saturation stall by one containing only 25% of WS<sub>2</sub> (7846 = 8376). This had an adverse effect on the life of the splitting stall catalyst and the yield of petrol was reduced by about 2% on the middle oil processed.

From April 1941 to January 1942 part of the petrol was made over catalyst 7019 under conditions which gave petrol with 50% aromatics and of similar properties to D.H.D. petrol. At 300 ats. this catalyst proved to have a poor life and the experiment was not repeated. Dr. Jacob said that at this time there was not the demand for aromatics that there had been earlier.

The use of the Welheim process for making aromatic petrol at 700 ats. over a different catalyst had been given consideration but it was decided that it would involve too great an expenditure on new plant.

### III. PLANT PRODUCTIONS AND CONSUMPTIONS

An air raid on October 1st 1940 destroyed part of the distillation plant tank farm and the output of the plant was reduced to 50% for nearly three months. From February to June 1942 it was necessary to export part of the middle oil as fuel oil, the reason for this is not known at present.

In June, July and August 1943 the output was reduced by a shortage of hydrogen due to the elimination of H<sub>2</sub> and by a raid on August 12th. The hydrogen limitation was partially relieved by reducing the hydrogen partial pressure at the stalls and, therefore, the losses of hydrogen by solution in the products. The plant did not produce any petrol after June 13th, 1944 when it was destroyed by bombing. Two attacks on September 11th and September 27th destroyed the work done to get the factory back into production and no further attempt was made. As late as March 1945 the sludge plant Kugel ovens were being used for cracking petroleum residues in order to make some petrol. From February 1944 to the time when the plant shut down in June, there was some increase in the quantity of tar oil hydrogenated but the increase was only from 30,000 tes/year normal to 55,000 tes/year.

between iron sulphide, hydrogen chloride and hydrogen sulphide is unfavourable and protection of the converters from hydrogen chloride attack is not obtained as it is at 300 ats. The major effect on petrol yield of working at 700 ats. pressure is on the amount of unconverted coal; at Scholven this was 8-8.5% on the coal, at Gelsenberg with similar coals it was 4%. This reduction in unconverted coal has a two-fold effect, (a) a direct increase in oil make (b) a reduction in losses in sludge recovery which more than compensates for the effect of increased catalyst used as explained above. The oil recovery obtained in the Scholven kilns was only 75% against 78% for Gelsenberg because Scholven had more asphalts in H.O.L.D. The total solids to be purged were 13-14% and 11.3% and the sludge losses 6.0 - 6.2% and 4.6% on the a.m.f. coal respectively for Scholven and Gelsenberg. The overall effect of using 700 ats. was to increase the yield of aviation petrol by about 4% on the coal i.e., from 49% to 53%. There was apparently no reduction in gas make when operating at the higher pressure. We think that this is due mainly to the absence of H.Cl. and in part to the higher converter temperatures employed. The average new formed oil make on the coal stalls was 69% on the a.m.f. coal, 64.5 - 65% was petrol and middle oil. The effect of pressure on the throughput of the coal stalls was considerable the throughput of ash, moisture free coal being increased from 0.25 tes/M<sup>3</sup>/hr at 300 ats. to 0.37 at 700 ats. The output of oil per unit of reaction volume (or liquefaction leistung) was increased from 0.16 tes/M<sup>3</sup>/hr to 0.26 tes/M<sup>3</sup>/hr. Because of a limitation on the maximum weight of converter forging which could be obtained, the 700 ats. converters had only 75% of the reaction volume of the Scholven converters. At Gelsenberg the reaction volume per stall (36 M<sup>3</sup>) had been made nearly the same as that at Scholven (39 M<sup>3</sup>) by using four converters in place of three. Scholven could not use four converters because of a heating limitation. Gelsenberg could only make use of the full reaction volume by the use of paste interchangers and this necessitated reducing the effective coal concentration in paste from 46% to 40%. The major operating difficulty at 700 ats. was burst preheater tubes. This was largely overcome by modifications to the method of operating the stalls and changes in the manufacture of preheater tubes.

The plant started up in June 1939 with a capacity of 150,000 tes/petrol. The plant was enlarged in steps and after October 1942 it was capable of producing 340,000 tes/petrol and 100,000 tes. of propane and butane for motor car fuel, this figure also includes 16-20,000 tes/yr. of iso-butane for Scholven.

There were 6 coal stalls plus one under construction, 4 saturation stalls and 3 splitting stalls. The stalls went into operation as follows :-

Coal Stalls	1. Aug. 1939	Saturation	303	June 1939
Nos. 701-706	2. Dec. 1939	"	302	Oct. 1939
	3. Marc. 1940	"	304	Nov. 1939
	4. May 1940	Splitting	301	Jun. 1940
	5. Feb. 1941	"	305	Mar. 1941
	6. ?	Saturation	306	May 1941
		Splitting	307	?

The annual petrol production figures are estimated with fair accuracy to be:-

1940	121,000 tes.	
1941	206,000 tes.	part aromatised.
1942	321,000 tes.	Some middle oil exported.
1943	335,000 tes.	
1944	118,000 tes.	(4 months only)

## II. GENERAL SCHEME OF OPERATION

Hydrogen was produced mainly from coke via water gas. Of the total production of 135,000 m<sup>3</sup>/hr. hydrogen, 100,000 m<sup>3</sup>/hr were produced from coke. 15,000 m<sup>3</sup>/hr were recovered from lean gases by the Linde Process, 15-18,000 m<sup>3</sup>/hr were imported from the rubber works at Hula and 5000 m<sup>3</sup>/hr of 60-70% hydrogen were obtained from coal stall let down gas. No coke oven gas was used for hydrogen manufacture and there was no methane steam plant. Sulphur removal was done by the alkacid process. The coal used was a washed bituminous coal containing 8% carbon (a.m.f. basis) and 4-5% ash (dry basis). The coal containing about 8% water was ground and dried in one operation before mixing with the pasting oil to make a 46% coal paste. The coal paste was treated in six coal stalls at 700 ats. pressure, on the average five stalls were available, one stall being with maintenance for cleaning accumulated solids from preheaters, converters and hot catchpot. The average coal throughput was 72 tes./hr a.m.f. coal but when six coal stalls were running this rose to 86 tes/hr. The products from a coal stall were divided into two parts by the hot catchpot. (a) Heavy oil let down (H.O.L.D.) containing the coal ash, catalyst and unconverted coal. (b) Vaporised oil and liquor made in the reaction.

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Capt. C.M. Cawley	

I. INTRODUCTION

The plant was visited between April 12th & 16th. The team consisted of Messrs. E.L. Baldeschwieler, C.M. Cawley, C. Cockram, G. Von Elbe, L.L. Hirst and F.M. Williams.

The following notes represent the results of interrogating plant personnel and some information extracted on the site from documents for the purpose of getting a general picture of the operations carried out at Belsenberg. Notes taken by Major H.G. Simpson (C.A.F.T.) and data on engineering matters obtained by Major J.F. Ellis during a later visit, have been incorporated. A considerable amount of information was segregated from the mass of documents found on the plant and from the documents sent to London it should be possible to work out detailed flowsheets for the process.

This plant had been hydrogenating bituminous coal at 700 ats. pressure. 2,000 - 4,000 tes/month of tar oils were also used. The middle oil and liquid phase petrol produced from coal were saturated over 5058 catalyst and the product was split over 6434 catalyst. The Vapour phase operations were carried out at 300 ats. pressure and appeared to be perfectly standard. There was no D.H.D. plant for aromatising petrol and no manufacture of iso-octane. Up to 335,000 metric tons (tes) of aviation petrol of 55% volatility at 100°C and 72 clear octane number had been produced in one year.

It is evident that the Germans went to 700 ats. operation on coal stalls mainly with the object of making the process simpler to operate and more reliable. They were able to dispense with H.Cl as a catalyst and, therefore, did not need to remove H.Cl from the products by alkali scrubbing. They could replace tin which was in short supply by a cheaper catalyst, iron sulphate and Bayer Masse, a residue from the aluminium industry. The reduction in asphalts brought about by using 700 ats. made the sludge recovery easier; the kilns ran for 6-12 weeks without decoking compared with 4 weeks at Scholven. The substitution of tin and chlorine by a much larger amount of inferior catalyst was disadvantageous because the quantity of ash to be purged from the system via the sludge treatment plant was increased by 2.2% on the coal and the loss of oil in the sludge plant was increased by 0.8% on the coal on this account. It is thought that tin and chlorine cannot be used at 700 ats. pressure because the equilibrium between



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Visit to  
GELSENBERG HYDROGENATION PLANT

Reported by C. Cockram on behalf  
of British Ministry of Fuel and  
Power and U.S. Technical and  
Industrial Intelligence Committee.

CIDS Target No. 30/4,08

Dated August 8th, 1945.

Combined Intelligence Objectives Sub-Committee

G-2 Division SHAF (Rear) AFO 413.

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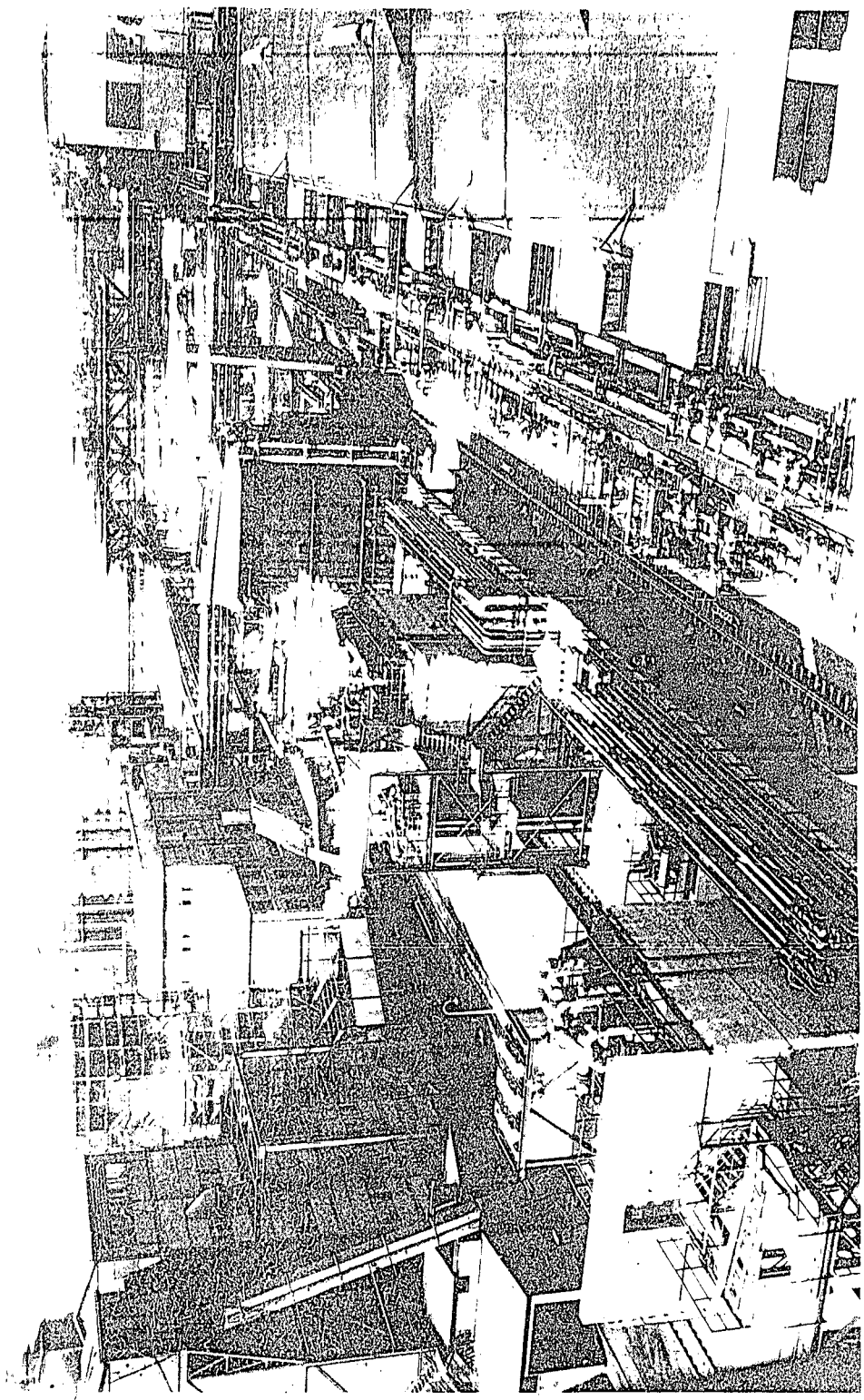
# GELSENBERG HYDROGENATION PLANT

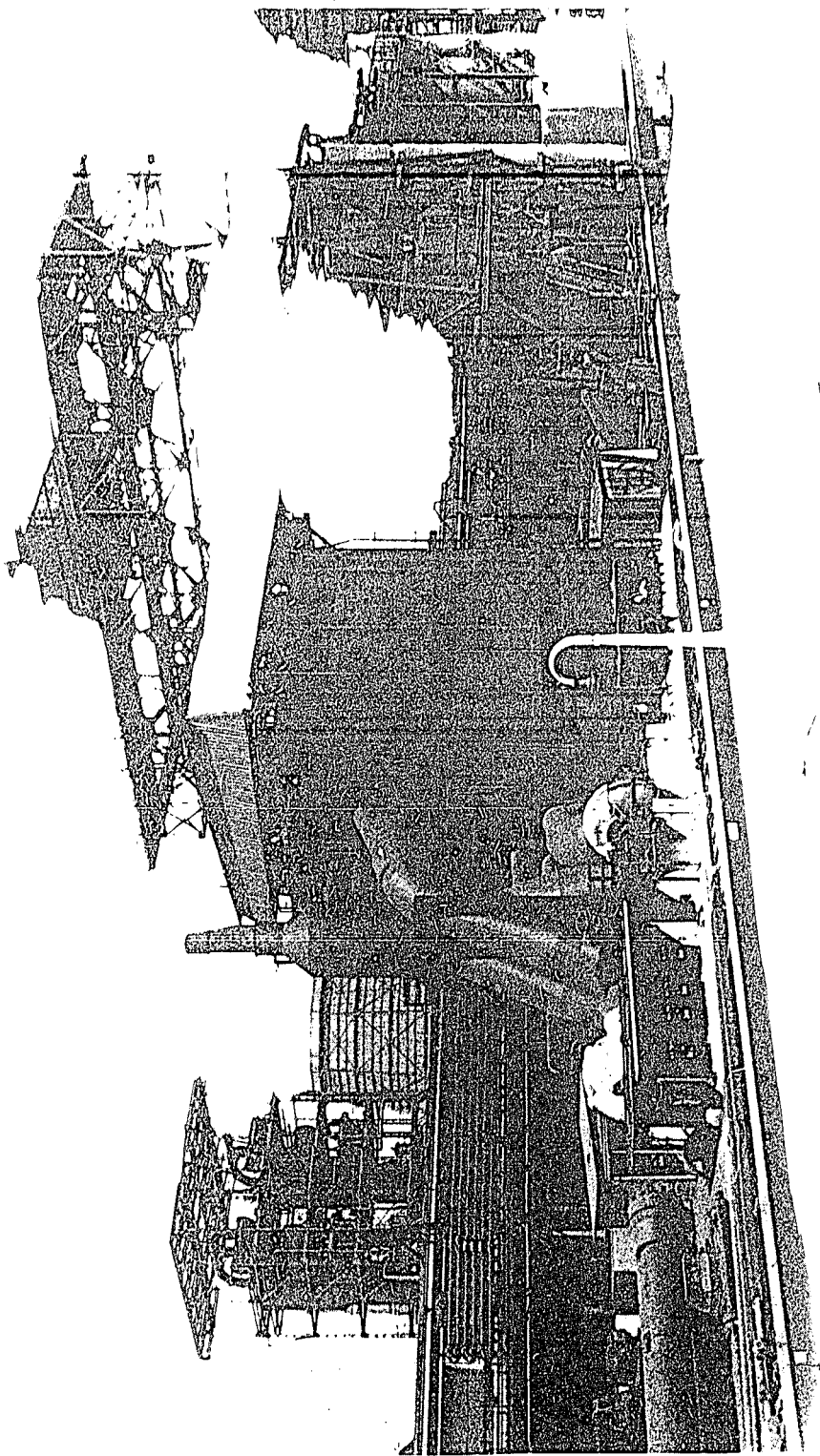
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COMBINED INTELLIGENCE OBJECTIVES  
SUB - COMMITTEE





PITCH STALL

experimented with various combinations of wide and narrow tubes for the Autoclaves but had not reached any finality although one or two people thought that they could achieve the object by making the velocity in the Autoclave much greater and in order to obtain the necessary reaction time to make the Autoclave much longer. In order to get better average running of the extraction unit they had lately run with only half the Autoclave in service with the other half out of the heater being cleaned. Because the reaction volume was halved the preheater had something in hand and the actual reduction in throughput was only from 18 to 17 M<sup>3</sup>/hour and they were able to get more running days/year.

They had been bothered by corrosion in the pitch stall interchangers due to Chlorine but had overcome this by the addition of Sodium Carbonate to the feed. The addition was less than theoretical.

They had trouble with Vapour Phase interchanger glands as a result of the air raids and had found that the rapid shut-downs on air-raid warnings had given rise to cracking of the welds between the tubes and tube-plates.

For all hot joints on the stalls they used Elastic-lens-rings, and for the V.2A tubes used the normal high temperature lens rings.

The N.10 lines between the converters on the Vapour Phase stalls were internally lined with 14 mm. of asbestos rope wound on to thin V.2A tubes and inserted in the tubes. They had done this to reduce the operating temperature of the tubing. They had, however, tried a length of unlined tubing between the preheater and the first converter and had found it satisfactory, but this is to be expected because the tube temperature conditions here are milder than in the last pass of the preheater.

Vapour Phase converters have only one grid at the bottom and have a number of cooling gas inlet coils spaced along the length of the basket.

Drawings showing the fitting of branches to interchanger covers by screwing and welding were seen and the views of the engineering staff taken. They did not like this method of construction and were doing all they could to change the design.

VIII. SUMMARIED CAPITAL COSTS FOR WELHEIM

Capital costs of Plant at 31.12.44. (From Major J.F. Ellis)

	P.M.
Hydrogen Production	21,628,361
Conversion	23,373,479
Refinery	7,051,233
By Products	1,141,730
Services	9,881,501
General	6,948,762
Pott-Broche Extraction	8,734,594
	<hr/> 78,759,660
Inventory, starting up	2,252,428
	<hr/> 81,012,088

VII. NOTES ON ENGINEERING MATTERS (BY MAJOR J.F. ELLIS)

This plant was visited on June 5th, 1945 and the experiences of the staff with Hydrogenation at 700 ats. were obtained. They claimed that they had had no trouble with steel tubes in their preheaters except for the occasional failure which they had no cause to believe lay with the steel, but rather with the actual running conditions or some local peculiarity.

They had originally started off with N.8 steel in the cold parts of the pitch stall preheaters and V.2A in the very hot parts. This had given them absolutely no trouble. They later developed a 700 ats. Vapour Phase Plant and used at first N.10 for the hot parts of the preheater. When they later extended the plant they put the V.2A in one of the Vapour Phase stalls because the temp. conditions were more severe there. N.10, however, has shown itself equally suitable for the severe Vapour Phase conditions. Since then they have run trouble free on both types of stall, and in fact have run N.10 steel under the more arduous Vapour Phase conditions without any cause for complaint. From their experience it can be said that they have not had any cause to think that N.10 is unsuitable for the hot zones of preheaters to stalls on plants working at 700 ats.

Until 1943 they were troubled with the building up of deposits in the pitch stall preheaters and this meant that they had to blow down and clean the preheater periodically because of the high P.D. and not because of the low "K". They then started to inject about 700 L/hour of water at the inlet of the preheater and this has enabled them to improve running very considerably.

They were bothered a lot at first with the coking of the H.C.P. and finally overcame this by installing a stand-pipe in the base of the pot so as to hold a volume of 200 - 250 Litres of oil in the base of the pot. The inlet gas and oil pipe was so altered that the oil fell into the burden in the base of the pot and did not fall on the wall. The depth gauge legs for the lower point were cut short just above the top edge of the stand-pipe about 100 mm. Into the burden on the cone they injected 7,000 M<sup>3</sup>/hour of circulating gas. They claimed that they had increased the life of these pots from 50 to 180 days, that the H.O.L.D. let-down gas rates were not increased and that because of the gas injection they had increased the interchange of the feed side of the interchangers by 17°C.

They have had considerable experience with both the single and double cone joints and are definite that the single cone joint is more difficult to make and to break. The breaking of the joint takes a much longer time than for a double cone joint and also needs considerably bigger forces to make it. They had made schemes for the modification of the single cone joints to double cone joints but had not actually altered any of the vessels.

All the converters were insulated with 63 - 64 mm. of asbestos cement or Schammote-stein. They were originally fitted with a thickness of 90 mm. but they found that the wall temp. was so low that they felt it safe to decrease the thickness. On doing this they had found no appreciable change, and the normal wall temperature was quoted as 90 - 160°C. They said that only in one case had they ever recorded a wall temperature of 300°C and that was when they had had a rapid blow-down and had collapsed the lagging.

They have had trouble with the pump-bodies of some of their pumps but have overcome this by modifying the shape of the forging so that unfavourable changes of section do not occur, and so that the forging can be more easily heat-treated. They had also had a lot of trouble with their 3-throw pumps and had had to reduce the pump speed from 135 to 90 R.P.M. after which their troubles were much reduced.

They had experienced difficulty with the packing on the Pitch pumps and had to reduce the operating temperature from 130 to 105 C. For the lubrication of the Pitch pump rods they injected 150 L/hour of clean oil to each ram and for the Middle Oil injectors they injected 10 - 30 L/hour to each ram. The oil was normally only injected on the suction stroke but if the glands were getting bad they raised the pressure to enable oil to enter on the delivery stroke. The oil in both cases was the heating oil they made on the plant.

The pitch stall feed contained 12% solids (insols in a 80-20 TETRAHYDRONAPHTHALENE Cresol Mixture).

They had tried Nitrided rams but as the quality was variable they had changed to normal case-hardened steel.

They had abandoned the use of interchangers on the extraction plant because they became fouled too easily and the extra fuel consumption had been paid for by the longer runs they were able to make. The Extraction Preheater showed little sign of incrustation, but the Autoclaves had to be cleaned every six weeks. They had

We formed the impression that the molybdenum was added either as ammonium molybdate or as molybdic acid. Samples of unreduced catalyst have been obtained and it should be possible to check this point. Frese had said earlier that the catalyst consisted of Bleich Erde + HF with 0.4% MO, 2% Cr, 4% zinc sulphided to 7-10% sulphur content and activated in hydrogen. The foreman was not clear about the addition of zinc oxide. The analyses referred to on page 13 in this typescript confirm that zinc oxide was used and that sulphur was added to the mix.

References were found to catalyst made with 60% AT5 paste and 40% Terrana. We think that this refers to the quantity of Terrana which was added to the mix after the addition of H.F. and was required to produce a clay of the right characteristics.

The foreman gave us the following information :-

The general procedure was to put 80 Kgs. of earth into a pan mixer, add distilled water and stir for 1/4 hr. Add 4 l of  $\text{CrO}_3$  solution, stir 1/4 hr; add 1-2 Kg. HF solution (sometimes 3-4 Kgs was added), 15-20 Kgs. terrana and 4-5 litres of ammonium molybdate solution. 8 litres of ammonia were added and the whole lot was stirred for one hour. Some more Terrana earth was added to make a clay of the right consistency for pressing. The mix was put through a pair of rolls which fed into a horizontal extruder to produce a cylindrical block  $5\frac{1}{2}$ " diam. x 1'6" high. These blocks were transferred to two vertical extrusion presses which were driven mechanically. The clay was extruded through dies and the rods were cut off with a wire. The 10 x 10 mm pellets were dried for three days at 70°C and the dry pellets were transferred to an oven where they were heated in hydrogen at 270-300°C. The reduction was a batch process, 8 hrs. heating up to 300°C, 8 hrs. at temperature, 8 hrs. cooling. Before discharging hydrogen was displaced by  $\text{CO}_2$  and solid  $\text{CO}_2$  was put into the catalyst containers. The reduction furnace was a makeshift affair. Three horizontal tubes 10" diameter and about 12' long were heated in a firebrick setting. HP hydrogen was let down to the tubes and exhausted to atmosphere after scrubbing the hot gas with water to remove  $\text{H}_2\text{S}$  (Smell! and ammonium sulphide chokes). Several pyros were located in the bottom of the tubes. Three mild steel mesh containers for the catalyst were put into each tube. It was noted that these containers were covered with a heavy deposit of sulphide scale. The pellets were screened before reduction.

According to the foreman, this catalyst or modifications, has been made for four years. As molybdenum became more scarce the percentage in the catalyst was reduced. Frese said that 0.4% MO is the best, more molybdenum gives a better life but lower aromatics in the petrol. Lower molybdenum gives more aromatics, but a shorter life and lower leistung. They were experimenting on pellets in the form of rings.

Frese gave a figure for the cost as 2000 RM/te compared with 5058 at 10-12,000 RM/te. The catalyst requires a hydrogen partial pressure > 500 ats. Some batches have given trouble with pressure drop.

VI. HYDROGEN PRODUCTION

Like Scholvan, hydrogen was produced partly by Linde fractionation of coke oven gas and the remainder, about 1/3 of the total, was produced from water gas. The output of the Linde units was 20,000  $\text{m}^3/\text{hr}$ , this had been achieved. The Linde plant was operated at 18 ats. pressure with  $\text{CO}_2$  removal at this pressure. The purified gas was then compressed to 25 ats. in a separate building and then transferred for compression to 325 ats. The 325 ats. gas was then compressed in three machines to 700 ats. In this building were also housed the four 0.700 ats. compressors for hydrogen from water gas. The plant was originally built for water gas hydrogen only and three Pintsch generators were installed, the number was increased to four. Frese estimated that four generators were required to make 12,000  $\text{m}^3/\text{hr}$ . of hydrogen. He also gave the following data:

For 12,000  $\text{m}^3/\text{hr}$   $\text{H}_2$  -

Coke 12 tes. equivalent to 0.66 tes/1,000  $\text{m}^3$  B.W.G.  
 3 ats. Steam 15 tes. " " 0.83 tes/1,000  $\text{m}^3$  B.W.G.  
 Condensate to jacket boilers 7.5 tes.  
 H. Water gas 15,000  $\text{m}^3/\text{hr}$ . (% of B.W.G. = 83)  
 N. water gas 3,000  $\text{m}^3/\text{hr}$ .

The coke consumption is unusually high.

$\text{H}_2\text{S}$  was removed by an alkacid plant with oxide boxes as standby.  $\text{CO}_2$  and CO were removed at 25 ats. The CO plant operated with liquor at - 3°C and it was necessary to use copper formate, copper carbonate used by the I.G. at 250-300 ats. has not the required solvent capacity for CO. The reason for removing CO at 25 ats. was that hydrogen was required at this pressure for the hydrogenation of naphthalene to tetralin for the original Pott-Broche process.

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TABLE V (cont'd)

	January	February	March	April	May	June	Total
Reaction Volumes - m <sup>3</sup>		36	72	36	72	72	
Liquid phase		12	13.5	13.5	28.6	28.6	
Vapour phase							

Note: The figures for input and products probably represent receipts and despatches. The period must be considered as a whole in estimating yields under balanced conditions.

TABLE VI

Summary of Operations on Stalls 54a & 17.  
700 at. Vapour Phase

1944	Feed	Feed		P + MC converted	Petrol made	% of oil converted	Petrol Leistung	% Petrol to 195°C in make up oil
		tes/m	% make up oil					
January	54a	11,815	54	6,270	4,909	78.3	0.480	18.7
February	"	10,936	47	5,228	4,140	79.1	0.440	23.6
March	"	11,362	51	5,482	4,384	80.0	0.436	22.7
April	"	6,577	45	3,222	2,700	83.7	0.379	24.4
May	54a + 17	12,400	50	6,890	5,050	78.0	0.336	21.8
June	54a + 17	14,300	58	8,250	6,660	81.0	0.335	21.9
Totals:				34,942	27,843	Av 79.5%		

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## V. CATALYST MANUFACTURE AT WELHEIM

The plant in which the vapour phase catalyst was made was undamaged. The building attached to it was completely wrecked but as far as we could ascertain this building was not used for the manufacture of this catalyst. The plant was built in a peculiar way, such that the flow of materials and intermediate products required much more labour than need have been used with a better design.

The building had three stories, movement of material to the top was done by a hoist. The bottom floor contained bunkers for unreduced catalyst, and the raw material store. This store contained barrels of Chromic acid, zinc oxide and Terrana earth and carboys of hydrofluoric acid. The middle floor contained a pan mixer fitted with fume hood and canvas curtains, a Baker Perkins type mixer and a roll and screw extrusions press for working the clay like mixture of raw materials.

The top floor had three rectangular air drying chambers for drying pellets and three vacuum ovens which had been used for some other purpose.

There were two extrusion presses for making pellets. The presses had vertical cylinders  $5\frac{1}{2}$ " diam. and 1'6" high, a piston driven by a rack and pinion, extruded the clay first through a  $1/4$ " square mesh screen and then through the die plate with round holes on the top of the cylinder. The screen and die plate were attached to a hinged cover which was held down by a quick acting clamp after the preformed block of clay had been inserted in the cylinder. The die plates and wires for cutting off the pellets had been removed.

Outside the building was an oven for reducing the dried pellets.

A foreman explained the sequence of operations and he gave us the amounts of various materials used. Later we got separate confirmation from Frese who could only outline the process. He said that elemental sulphur was added to the mix. He thought that the molybdenum was put in as the thiomolybdate but we found no evidence for this.



TABLE IV.  
Vapour Phase Stall Operations  
(Abstracted from monthly balances.)

	April 1944	May 1944	June 1944
Hours run	528	744	720
Reaction Volume m <sup>3</sup>	13.5	20	27.7
Feed Leistung t/m <sup>3</sup> /hr.	.922	.818	.717
Petrol Leistung t/m <sup>3</sup> /hr.	.379	.336	.335
Feed t/hr.	12.46	16.61	19.86
Make up t/hr.	5.6	8.31	11.57
L <sup>o</sup> Petrol to 195° in make up	1.37 (24.4%)	1.81 (21.8%)	2.54 (21.9%)
Oil in C.G.P. t/hr.	11.46	14.70	17.7
Petrol	5.11	6.8	9.27
MO	6.35	7.9	8.43
Gas	1.96	2.37	2.49
Liquor	.16	.50	.32
Total	13.58	17.57	20.51
Yields % on P + MO converted			
Petrol	83.6	78.0	81.0
Gas	32.0	27.2	21.8 <sup>a</sup>
Liquor	2.6	5.7	2.8
Total	118.2	110.9	105.6
H <sub>2</sub> used	6.1	5.7	5.7
Ratio $\frac{n-C_4}{1-C_4}$	5.2 : 1	2.6 : 1	2.6 : 1 <sup>a</sup>
Consumptions & Productions for balanced stocks, (Calc'd)			
P + MO consumed t/month	3,230	6,490	8,250
P made t/month	2,700 <sup>†</sup> (2,700) <sup>†</sup>	5,050 <sup>†</sup> (5,075) <sup>†</sup>	6,660 <sup>†</sup> (6,676) <sup>†</sup>

<sup>†</sup> Presumably average figures for two stalls, one running only part of the month.

<sup>a</sup> Because of the air raid on 21.7.44. they had to use the analyses for the month of May for calculation of gas balances.

<sup>†</sup> Figures shown on the balance sheets.

TABLE V

Production figures for 1944. (taken from file of correspondence with Pier.)

	January	February	March	April	May	June	Total
Input							
Pitch	8,658	10,917	17,782	4,679	8,248	7,767	58,051
Tar Oils	2,307	3,123	5,130	1,277	5,460	5,667	22,964
Total	10,965	14,040	22,912	5,956	13,708	13,434	81,015
% Pitch in make up oil							71.5%
Products							
Aviation Petrol VI 330	4,431	3,961	4,098	2,486	4,772	6,094	25,842
Fuel Oil	3,968	6,056	9,958	2,270	6,266	4,749	33,267
Treibgas	1,226	1,284	1,179	431	1,063	1,038	6,221
Yield of liquid products on make up oil (excluding HOLD purge)							<u>65,330</u>
H <sub>2</sub> used - 10 <sup>6</sup> m <sup>3</sup>							
Linde	10,107	10,896	14,267	3,800	11,399	12,071	62,540
Water Gas	3,958	4,295	6,482	3,790	5,755	5,830	30,110
							<u>92,650</u>
							= 7,700 tes. or 9.5% on make up oil.

This flowsheet shows a yearly production of

		<u>% on feed</u>
Aviation Petrol	60,000	22.8
Fuel Oil	119,500	45.4
HOLD to briquetting	37,500	14.2
Treibgas	18,400	7.0
Total:	<u>235,400</u>	<u>89.4</u>

The hydrogen used is given as 32,000 m<sup>3</sup>/hr. or 2.67 t/hr. representing 7.3% on the make up oil. Frase had quoted a figure of 7.6% overall hydrogen consumption.

From this flowsheet it can be calculated that the yield of aviation petrol from the liquid phase petrol plus middle oil fed to the vapour phase stalls is 7%. This agrees with figures calculated from the monthly balances found on the plant.

TABLE III

Pitch Stall Operations. (Abstracted from monthly balances.)

	April 1944		May 1944		June 1944	
Hours run	264		744		720	
Reaction volume m <sup>3</sup>	36 <sup>†</sup>		72		72	
Fresh Feed Leistung t/m <sup>3</sup> /hr.	0.63		0.256		0.26	
	t/hr	% on m/u	t/hr	% on m/u	t/hr	% on m/u
Feed	36.64		28.83		32.19	
Make up (m/u)	22.73		18.43		18.66	
C.CP	15.28		14.58		14.89	
Petrol	1.82	8.0	1.55	8.4	2.11	11.3
MO	4.58	20.2	4.52	24.5	5.88	31.5
Fuel Oil	8.88	39.1	8.51	46.1	6.90	37.0
Purge HOLD	3.70	16.3	2.02	11.0	1.93	10.3
Gas	2.21	9.7	1.60	8.7	1.82	9.7
Liquor	1.32	5.8	0.49	2.7	1.1	5.9
Total	22.51	99.1	18.69	101.4	19.74	105.7
H <sub>2</sub> used	1.56	6.85	1.31	7.1	1.34	7.2
$\frac{n-C_4}{i-C_4}$	4.9 : 1		7.3 : 1		6.9 : 1	
(Calc. balanced)						
(Production: Tss/m.)						
Petrol	480	(478) <sup>‡</sup>	1,151	(1,152) <sup>‡</sup>	1,520	(1,521) <sup>‡</sup>
MO	1,210	(1,208)	3,360	(3,370)	4,230	(4,233)
Fuel Oil	2,340	(2,329)	6,350	(6,344)	4,960	(4,975)

<sup>†</sup> Believed to be one stall on line, this requires confirmation.

<sup>‡</sup> Figures in brackets are those given on the balance sheets.

Products	tes.	% on feed	tes.	% on feed
Fuel oil	94,772	48.6	88,392	43.9
Middle oil	9,402	4.8	11,905	5.9
Petrol	31,422	16.1	33,907	16.9
Triebgas	17,769	9.1	11,777	5.8
	<u>153,365</u>	<u>78.6</u>	<u>145,981</u>	<u>72.5</u>

In addition some 28,000 tes/yr. of heavy residues from the pitch stall, i.e. H.O.L.D. were sold for briquetting.

Specific Consumptions per te. Fuel oil, petrol and middle oil.

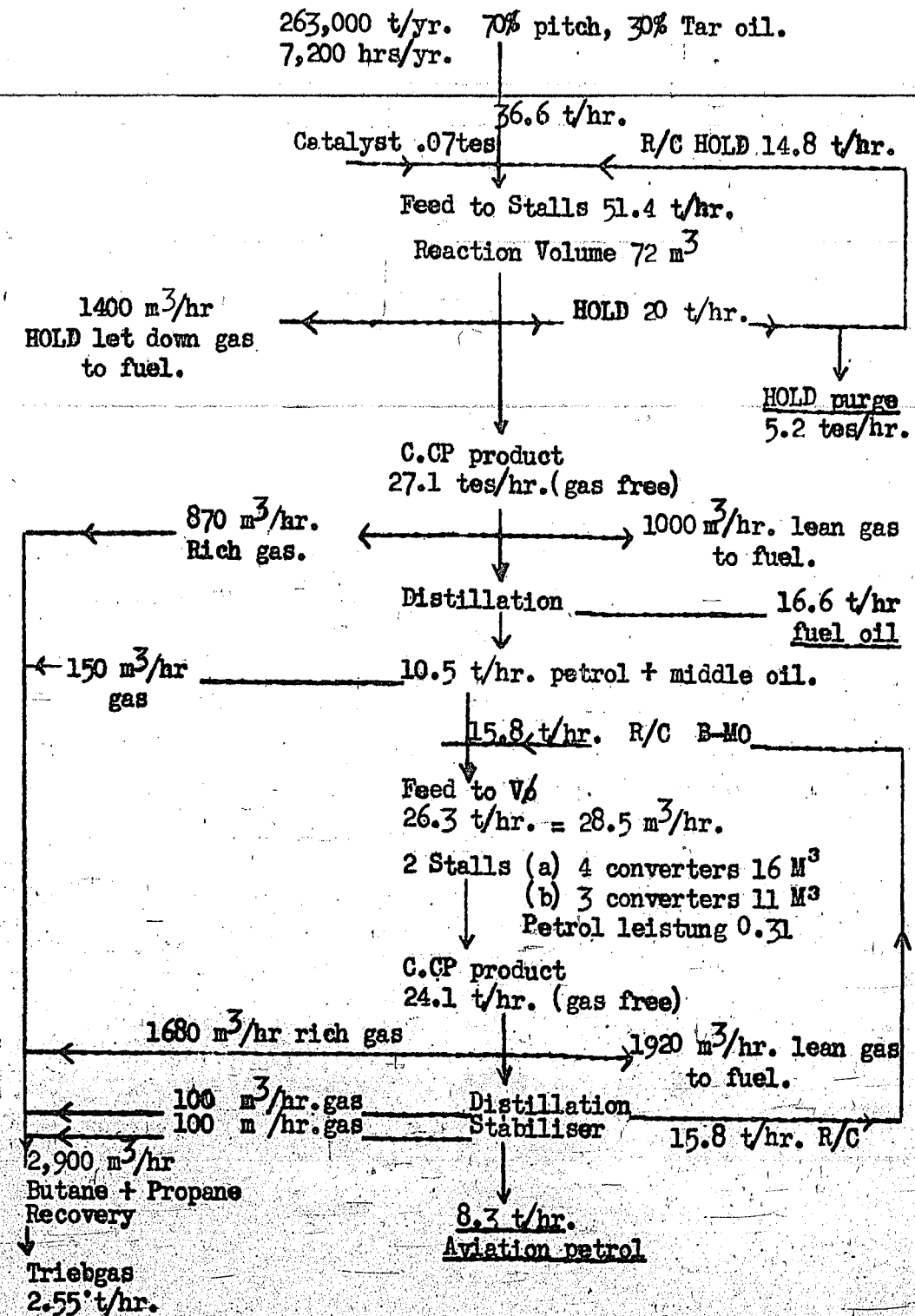
Electric Power	KW.	1,436	1,620
Steam	tes.	4.3	4.3
Fuel gas	m <sup>3</sup>	1,050	1,420
Water	m <sup>3</sup>	16.2	2211

Flowsheet III was prepared by Frese, it represents what they expected to achieve in 1944. Tables III and IV represent calculations made from the monthly plant balances. They confirm Frese's claim that the yield of petrol from the middle oil plus petrol fed to the vapour phase stalls is 80%; this compares with a yield of 75% on petrol treated by the I.G. D.H.D. process.

Table V taken from a file of monthly letters to Dr. Pier shows that given freedom from air raids, they would have achieved the flowsheet figure of 60,000 tes. of petrol. These figures appear to represent the quantities of pitch and tar oils received each month and the despatches of finished products, they do not represent balanced conditions for each month but taken as a whole they present a fair picture of operations. Table VI summarises data for the Vapour phase stalls, taken from other documents. These show that the Vapour phase stalls, were capable of producing over 6000 tes/month of petrol.

FLOWSHEET III

For Pitch and Tar Oil Hydrogenation. (Frese)



Some experiments were done with I.G. 7019 catalyst but this catalyst was considered to be inferior to Welheim catalyst. The analyses recorded are of interest.

7019 analysis dated 7.3.40:

Active charcoal	81.8%	
Ash	18.2%	
Ash - Sulphur	0.8	
Cr <sub>2</sub> O <sub>3</sub>	10.6	7.3 Cr.
Al <sub>2</sub> O <sub>3</sub>	6.8	3.5 AL.

7019 analysis dated 30.7.41:

Given as activated charcoal + 7% Cr; 1% Va;  
1.7% Fe; 4.5% Al.

Total ash 18 - 23%  
Sulphur 2% on catalyst.

Experimental Work at Welheim

Experiments were made at 300 ats. and at 700 ats. with 7019 catalyst using pitch middle oil and coal middle oils. These were done in a 5.5 l. converter at a throughput of 1 Kg/1/hr.

(a) Pitch Middle Oil

	300 ats. - 500°C			600 ats - 500°C	
Petrol FBP	206	198	161	185	163
% at 100°C	13.5	33.5	56	26	60
% Aromatics + olefines	71	66	62	60	59
Octane number clear	78	79	79.5	76.5	78.0
CFR.MM. + 0.5 TEL.	84	84.5	85.5	82.5	85.5

(b) Coal Middle Oil

	600 ats. - 500°C		
Petrol FBP	213	174	155
% at 100°C	13.5	25.5	53
% Aromatics + olefines	55	54	52
Octane number clear	70.5	75.5	76.5
+ 0.5 TEL	79.0	83.0	84.5

Experiments had been done on the treatment of low temperature tar pitch over the Welheim vapour phase catalyst at 700 ats. One interesting feature of the process is that the removal of ash from the pitch by filtration was facilitated by heating the pitch with about 0.1% of sulphur at 100 - 150°C before filtering. The addition of too much sulphur made filtration too difficult. Reports on this work are available. High temperature tar pitch was not a suitable feed stock.

IV. FLWSHEETS AND PRODUCTION DATA FOR HYDROGENATION

Until the end of 1943 Welheim were exporting middle oil which was in excess of their vapour phase stall capacity.

Table II shows the yearly consumptions and productions for 1942 and 1943. These were abstracted from the annual production reports which were available from the time the plant commenced operations. These reports were left with the U.S.S.B.S. team, to be sent to C.I.O.S. London, when they have finished their survey.

TABLE II

	Year 1942	Year 1943
	tes.	tes.
<u>Input</u>		
Hard Pitch	146,562	159,889
Hard Pitch distillate	21,364	19,308
Tar oil	12,577	12,854
Redistillate	5,358	2,933
Other miscellaneous oils	9,360	6,352
Total:	195,221	201,336

% Hard Pitch in make up feed: 75                      79

(b) VAPOUR PHASE STALLS

When the plant was first started up there were no vapour phase stalls. The Stinnes company had pursued an active research policy to develop catalysts of their own and they succeeded in producing a catalyst for use at 700 ats. pressure which could be operated at 500°C and would give a petrol of high aromatic content. Petrols with over 60% aromatics were produced in semitechnical units. They were very bitter about the patent position. As they expressed it, the I.G. having patented the periodict table of elements as catalysts, the Stinnes people had to be content with a patent covering operations at 700 ats. pressure.

The petrols made by this process were superior to those made by the IG, DHD process, particularly in respect of their rich mixture rating. They also obtained in one step, instead of three, 5058 → 6434 → DHD, a higher yield of petrol. The ideal feed for aromatising is one containing naphthenes and the minimum of paraffins, the middle oil produced from pitch is particularly good from this point of view. It was clear from what Frese told us and from correspondence that Welheim had carried out their schemes with a considerable amount of opposition from the I.G. and in particular from Dr. Pier. The success of their large scale operations led to the decision to put in the process for the extensions to Blechammer.

In May 1939, the first vapour phase stall was put into operation (1 conv. 4.3 m<sup>3</sup> reaction volume) and this confirmed their experiments, except that petrols made on the plant had a lower aromatic content than those made in the laboratory. Another converter was added making a total of 10.5 m<sup>3</sup> reaction volume. Later, the number of stalls was increased to two, each with three converters with a reaction volume of 13-14 m<sup>3</sup>/stall. The date when the second vapour phase stall was put on line has not been ascertained, but it was probably in May 1944. Until the second stall was available Welheim exported middle oil to other hydrogenation plants or for use in diesel oils.

The average operating conditions were :-

Temperatures	480 - 502°C.
Inlet gas	2,500 - 2,500 m <sup>3</sup> /te.feed
Cooling gas	250 - 600 m <sup>3</sup> /te.
Petrol leistung (including petrol in feed)	0.35 - 0.4 of petrol FBP 165°C.
H <sub>2</sub> consumption	.076 to .085 te/te.petrol

Detailed information about operating conditions can be obtained from the stall record books which were sent to London.

Frese said that they obtained a petrol leistung of 0.7 Kg/l/hr. in semi-technical units but they were not able to get over 0.4 Kg/l/hr on the stalls because of heating limitations.

The catalyst was stated to have a life of at least 300 days. The catalyst had been varied in composition from time to time depending on the availability of molybdenum. Catalysts of the following composition had been made and used :

K.534	0.6% MO	2% Cr.	5% Zn.	5% S	} All made with <sup>+</sup> 60% AT 5 paste 40% Terrana.
K.536	0.7% MO	2% Cr.	5% Zn.	5% S	
K.413	0.4% MO	2% Cr.	5% Zn.	5% S	

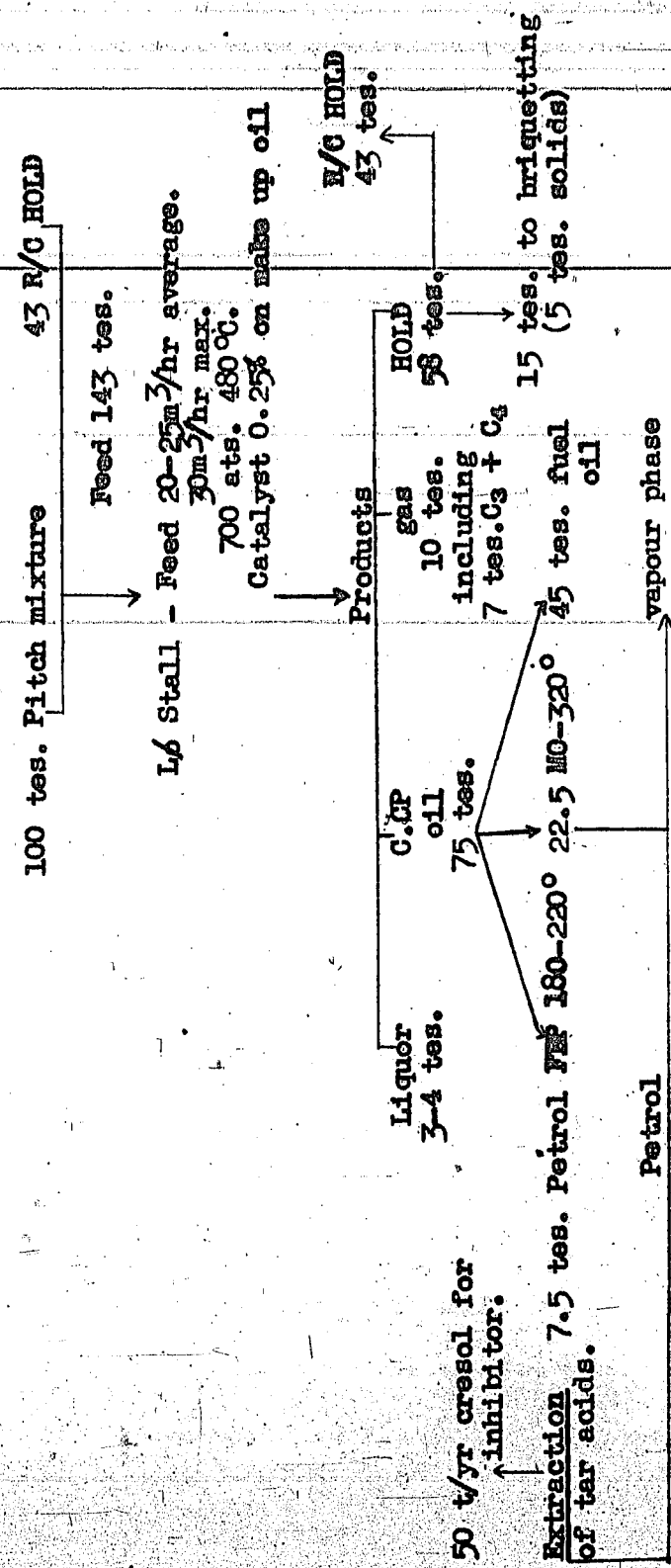
A higher molybdenum content gave a higher leistung, and catalyst life but 0.4% molybdenum gave a satisfactory performance and improved yield of aromatics.

A description of the method of making these catalysts is given in Section V - Catalyst Manufacture.

+ See notes on catalyst manufacture.

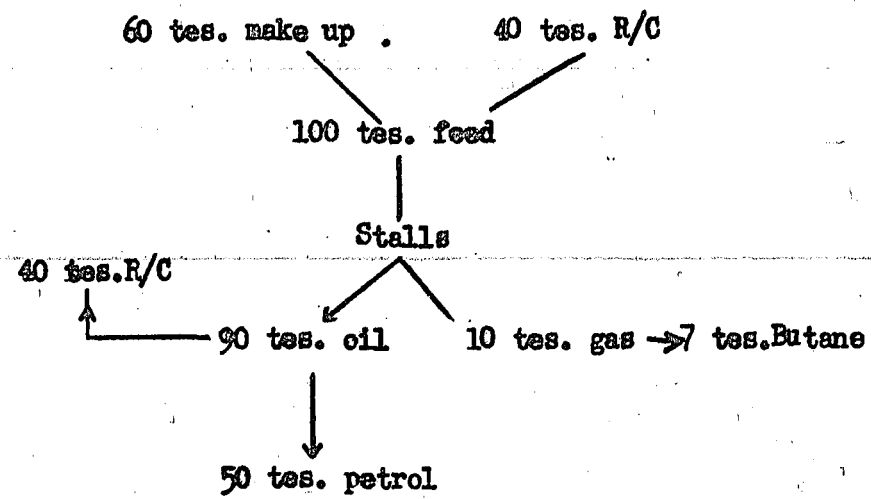
FLOWSHEET II (Verbally from Dr. Frese)

Hydrogenation of Pitch.



FLOWSHEET II (Cont'd)

Vapour-Phase Stalls



Calculated Petrol yield 3.5% on P + MO fed to vapour phase or excluding L<sub>6</sub> petrol, petrol from MO =  $\frac{35}{45} = 78\%$  of the middle oil.

### III. HYDROGENATION PLANT

#### (a) PITCH STALLS (700 ats.)

There were two stalls operating on pitch, each with four converters with a total reaction volume per stall of 36 M<sup>3</sup>. The converters were of various sizes - 18 m x 1,000 mm; 12 m x 900 mm and 9 m x 1,000 mm. The general layout of the converter bays, preheaters, coolers, control rooms, injection and circulation building followed the plan of the stalls at Scholven and Gelsenberg. The 700 ats. injectors, as at Gelsenberg, were driven hydraulically. They had the standard arrangement of a central drive with an H.P. cylinder at either end of the hydraulic ram. All the other machines, circulators and small pumps were electrically driven. A photograph at the end of the report shows the general layout of the stalls, the Pott-Broche extraction unit is in the background.

The stalls were designed to treat 250,000 t/yr. of pitch - tar oil mixture. Frese said that they had tried 80% of pitch (70° melting point) in the blend but this proved too sticky and they preferred a 70 : 30 blend. Liquid pitch was brought in insulated wagons from nearby distillation plants. The bulk of the pitch came from Meiderich and Rauxel. It was loaded at ca. 250° and was usually over 200°C when discharged. There was storage for 5-6,000 tes. of liquid pitch on the site in addition to storage for the diluent oil.

The liquid pitch, diluent oil and recycle HOLD were pumped to mixing tanks where the catalyst was added. This catalyst amounted to 0.25% on the make up oil and consisted of iron sulphate on grade (Brown coal water gas generator coke). The iron hydroxide was precipitated with caustic soda. The catalyst contained up to 10% Fe; normal 6-7%. Chlorine in the pitch amounted to 0.1% and caustic soda was added to the feed to reduce the chlorine in the liquor produced by hydrogenation to less than 10-20 m.gms. per litre. Frese gave us the following rough flowsheet of operations (See Flowsheet II). A more detailed flowsheet (III) prepared by Frese is given later. 40-45% of the pitch-tar feed was converted into fuel oil which could be blended with petroleum oils because of its low asphalt content. Some 10-16% of the feed was purged as heavy oil let down (HOLD) and was sold for briquetting.

The cold catchpot product from the pitch stalls was cut into three fractions, Fuel oil, middle oil to 320-330°C. and liquid phase petrol. The petrol was cut to an end point which was varied over the range 180-220°C. This petrol was extracted with caustic for the recovery of tar acids. The washed petrol was blended back with the middle oil for treatment in the vapour phase stalls; petrol in the petrol + middle oil amounted to 21-25% by weight. The tar acids recovered from the petrol were fractionated to give a cresol fraction; 60 tons/yr. of this fraction was made for addition to aviation petrol as a gum inhibitor. We were told that Welheim was the only producer of inhibitor and that no attempt was made to separate individual cresols.

There was no gas washing plant for stall circulating gas at Welheim. The liquid and vapour circulating systems were common and the 700 ats. vapour phase stalls provided the purge of hydrocarbon gases from the pitch stalls in excess of that dissolved in the pitch product. In this respect operations differed from Scholven and Gelsenberg.

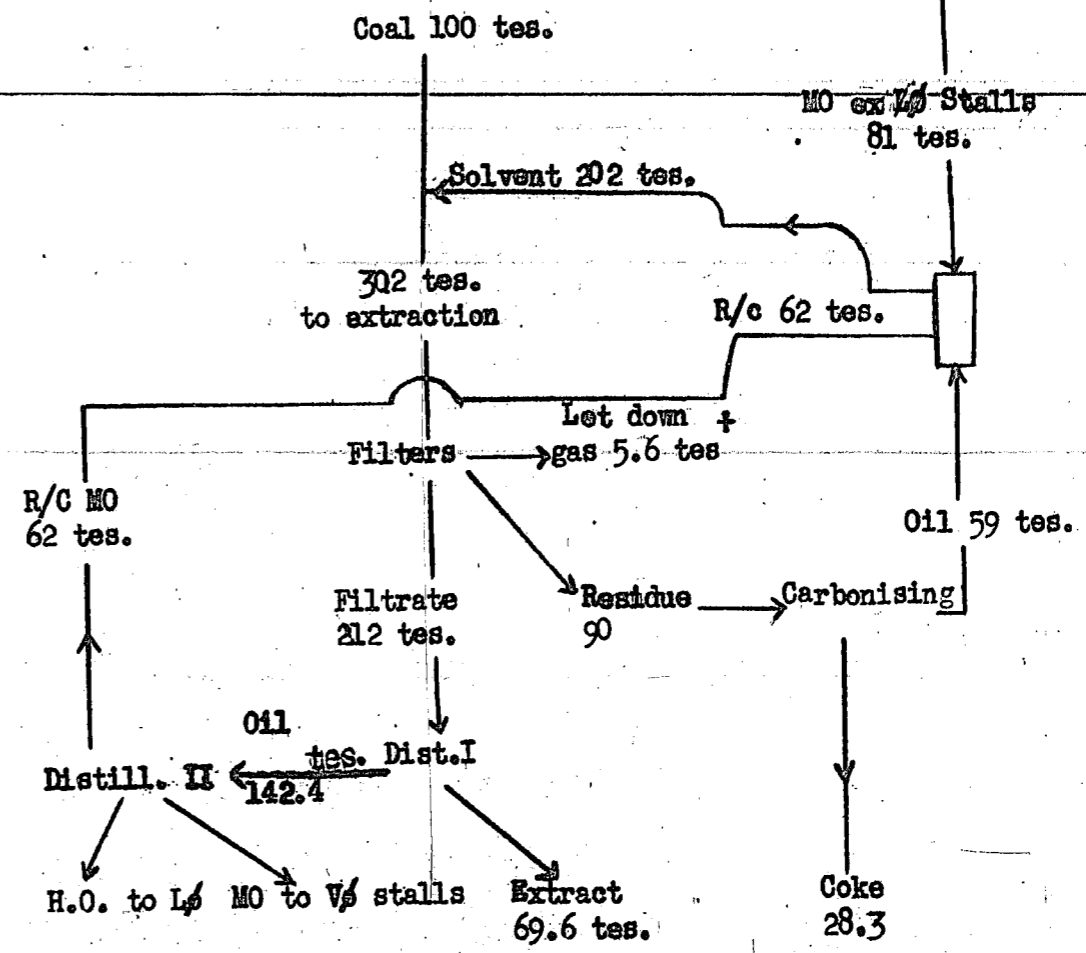
Some analyses of the coal used and the products obtained were noted.

	Coal	Extract	Residue
C.	86.2 to 86.7	89.1 to 89.3	86.3
H.	5.1 - 5.3	5.4 - 5.5	4.0
N.	1.7 - 1.8	1.9 - 2.1	1.45
S.	1.3 - 1.5	0.8 - 0.9	1.5
Cl.	.14 - .20	.08	0.45

Frese made a particular point about the low ash content of the extract and that it made a superior coke to bituminous coal tar pitch coke for this reason. He also said that it was blended off with pitch before coking. Some ash analyses were noted where the ash content was as high as 0.1% on the extract.

Frese also said that Prof. Wilke at Oppau had been experimenting with the extract as a fuel for pulverised coal motors. It had been possible to reduce the ignition point of the extract from 400° to 200°C by the addition of NO and NO<sub>2</sub>.

**FLOWSHEET I**  
For Pott-Broche Process.  
(From Dr. Frese)



Coal fed	5.3 t/hr.	
Solvent	10.7 t/hr.	% on coal
Extract	3.7 t/hr.	69.6
Heavy Oil made	0.5 t/hr.	9.4
Coke	1.5 t/hr.	28.3
Hours/year 7,200 = 26,600 t/yr. extract.		

† As given on the flowsheet, the filtrate or residue should be correspondingly reduced to make a balance.



The solution was filtered in batch filters containing ceramic elements (Kurtzen filters). There were three filters in use. A fourth which was of different design and had a subsidiary rotary filter mounted on top appeared to have been used for development work. The filters comprised a circular tank with a conical bottom fitted with a plough for removing the cake when it had been blown off. The filter elements (estimated to be 20 per filter) were inserted through the top cover. Each element consisted of a splined rod over which were fitted the ceramic rings  $4\frac{3}{4}$ " O.D.  $3\frac{1}{2}$ " I.D. x  $1\frac{1}{2}$ " deep. The rings were separated by klingerit washers and the whole assembly was bolted up solid. The length of ceramic element was 79". The ceramic elements looked like a coarse sandstone but they were obviously moulded. Each ceramic had two iron wires spot welded round it to reduce the chance of bursting while blowing off the cake which was formed on the outside. The filters had pressure gauges marked with a red operating line - feed 2 ats. middle oil for washing the cake 8 ats, CO<sub>2</sub> for blow back 6 ats. We were told that the normal feed pressure was 5 ats.

The solution was let down from the extraction unit and stored in lagged tanks from which it was pumped to the filter tank, after building up the cake the solution was replaced by middle oil to wash the cake. The washed cake was blown off with CO<sub>2</sub> and was removed by the ploughs onto a conveyor beneath for transport to two rotating retorts where it was carbonised. The vent gases from the filters were scrubbed with cold middle oil to reduce the losses of middle oil as vapour. We were told that they could do 3 - 4 filtrations per hour and that the ceramic elements were discarded after 5,000 filtrations.

The recovery of oil from the residue was stated to be 90 - 95% leaving 0.2% of extractable oil in the coke. The filtered solution was distilled, first under vacuum to remove middle oil and heavy oil together and the distillate was refractionated to separate the heavy oil which was produced from the coal during extraction and which amounted to about 8% on the coal. 60% of the middle oil was recycled to the process and 40% was added to the vapour phase stall feed. The make up middle oil was liquid phase middle oil from pitch hydrogenation.

It was stated that it was not possible to extract more than 80% of the coal substance without getting into difficulty in the filtration stage and the extract was too hard. The extract normally produced had a melting point of 220 C. The extract was carbonised at the coke ovens and was stated to yield 65 - 70% coke, the rest was gas.

The plant was designed to treat 125 t/day of coal but examination of the records indicated that this had not been achieved for any reasonable period.

The following flowsheet (Flowsheet I) was prepared for us by Dr. Frese who also gave us much of the information about the plant. Frese' figures have been converted to a basis of 100 tes. of coal.

Actual yields of extract taken from records for 1943 and 1944 are given below. Only in one month (May 1944) did they succeed in making extract at the rate of 20,000 tes. a year.

TABLE I

	Coal treated tes/month	Extract tes/month	Yield %	Tes. coal per hour
December 1943	2,239	1,494	66.8	3.0
January 1944	3,250	1,679	51.6	4.37
February	2,104	1,086	51.6	3.04
March	2,834	1,160	41.0	3.8
April	1,813	960	52.8	2.52
May	3,043	1,800	59.0	4.08
June	1,594	727	44.3	2.2
Total:	16,877	8,886	Aver. 52.7	

From these figures it is clear that they had considerable difficulty in achieving flowsheet outputs.

and 10,000 - 12,000 tes/yr. was exported to other hydrogenation plants or for use as diesel oil. 12,000 to 17,000 tes/yr. of butane were exported for use in cars. Some 28,000 tes/yr. of pitch stall residues (H.O.L.D.) were sold for briquetting.

During 1944 it was intended to make 120,000 tes/yr. of fuel oil and 60,000 tes/yr. of petrol. In May and June the vapour phase stalls did achieve this output by drawing on middle oil stocks, but the pitch stall did not approach flowsheet output.

60 tes/yr. of cresols were separated from liquid phase petrol and were used as gum inhibitor for aviation fuel. We were told that Welheim was the only source of inhibitor.

From an economic point of view the Welheim plant must have been a poor proposition. The overall hydrogen consumption on the pitch and tar oils treated appears to have been about 7.5% when making 60,000 tes/yr. of petrol and 120,000 tes/yr. of fuel oil. Nevertheless the plant made a useful contribution to Germany's fuel oil supplies and it made use of pitch for which no other use could be found.

The plant was destroyed by an air raid on October 31st 1944, another raid on November 6th caused 6,000 tes. of liquid pitch to flow over the site and attempts to get the plant back into operation were abandoned. It was noted that of the original tanks and gas holders above ground, only one small tank was not damaged beyond repair. Prior to these raids efforts were being made to convert the Pott-Broche extraction unit for the purpose of cracking petroleum oil residues. It was said that there were 300,000 tes. of these residues in Germany.

The Plant employed:-

Process men and women and maintenance	1,027
Labourers	539
Cleaners, firemen, etc.	126
Prisoners of war	176
Supervisors, Staff	250
	<u>2,118</u>

In Feb. 1944 they also had 446 contractors men and 51 labourers on new buildings.

II. THE POTT-BROCHE EXTRACTION PROCESS

By this process coal was heated to 400-420°C with a solvent at 100 ats. pressure, the solution was filtered to remove the ash and undissolved coal and the solvent was then removed by distillation. No gas was added to the coal paste.

The plant started up using tetralin as a solvent, part of the tetralin was dehydrogenated to naphthalene which was rehydrogenated at 25 ats. pressure. This was the reason for adopting the scheme for CO removal by copper liquor at -3°C and 25 ats. for the production of make up hydrogen gas. It was found that middle oil containing 7% cresols could be used as the solvent and it reduced the cost of the process.

It was the intention to hydrogenate the extract at 700 ats. pressure but the development of the process was slow and bituminous coal tar pitch was used as an alternative feed stock for the hydrogenation plant. This proved to be a more satisfactory material than the coal extract and operation of the Pott-Broche process was continued for the purpose of making high quality electrode carbon of low ash content. Dr. Frese suggested that the refractory nature of the extract was due to the high temperatures used in the extraction process.

The coal was ground and mixed with middle oil in the ratio: 1 of coal to 2 of middle oil. This was pumped to the extraction system which was built in the form of a square preheater with circulating flue gas. There were 40 vertical preheater tubes arranged around the outside of the heater and 48 soaking tubes in the centre. The number of working tubes in the soaking section had been reduced to 24. The time of contact was stated to be 1 to 1½ hours. The soaking section was referred to as the autoclave.

No gas was circulated with the paste but some gas was made in the process. To prevent gas locking, the rising legs of the hairpins were 180 mm. diameter and the downcomers were 120 mm. diameter. The pressure was 110 inlet, 100 ats. exit at the start of a run and the tubes were cleaned when the pressure drop increased to 40 ats. and the inlet pressure reached 150 ats. The temperature in the soaking section was 433°C. The feed rate was stated to be 16 m<sup>3</sup>/hr. of paste.

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VIII. Capital Costs	" 29

PERSONNEL OF TEAM

Mr. E. L. Baldeschweiler	U.S. Petroleum
Mr. L. L. Hirst	Administrator for War
Mr. G. VenElbe	U.S. Bureau of Mines
Major C. Geckram	Ministry of Fuel and Power
Major F. A. Williams	
Capt. C. M. Gawley	

NOTES ON VISIT TO RUHROL A.G. (MATHIAS STINNES)

WELHEIM, MARCH 10TH - 13TH, 1945

I. INTRODUCTION

These notes represent the results of interrogating plant personnel and some information extracted on the site from documents, for the purpose of getting a general picture of the operations carried out at Welheim. Notes from Major H. G. Simpson (C.A.F.T.) on operation of the plant and notes on engineering matters from Major J. F. Ellis, who visited the plant some time later, have been incorporated in this report. Director Dr. Frese gave us a considerable amount of information.

This plant was occupied with two processes. It was primarily built to operate the Pott-Broche process for extracting coal with a solvent, which was originally tetralin and cresol but later liquid phase middle oil from the hydrogenation of pitch was used. It was the intention to hydrogenate the extract in the liquid phase at 700 ats. pressure but the difficulties of filtering the extract were not solved until late 1943. Also it was found that the extract was not as easily hydrogenated as bituminous coal tar pitch. There was a surplus of 700,000 - 800,000 t/yr. of pitch in Germany and it was decided to hydrogenate pitch instead of extract. This was started in 1938 but schemes for hydrogenating Pott-Broche extract were still under consideration as late as August 1943. The Pott-Broche plant continued running and the extract was carbonised for the production of high quality electrode carbon for the steel and aluminium industries.

The main product of the hydrogenation of pitch was fuel oil (100,000 t/yr) which was suitable for mixing with petroleum fuel oils. The liquid phase middle oil made was converted to petrol in one stage at 700 ats. pressure. The catalyst had been developed by the Stinnes concern, the particular interest in this catalyst is that it gives a petrol with ca 50% of aromatics in one stage with a yield of 80% on the petrol plus middle oil treated. This petrol has a rather better rich mixture rating than D.H.D. petrol. The output of petrol was 30 - 35,000 t/yr. Until the end of 1943, The vapour phase stalls had not sufficient capacity to treat the whole of the liquid phase middle oil

RESTRICTED

Visit to

BOTROP-WELHEIM HYDROGENATION PLANT.

Reported by G. Cockram on behalf  
of British Ministry of Fuel and  
Power and U.S. Technical and  
Industrial Intelligence Committee.

CIOS Target No. 30/4. 11

Dated August 8th. 1945.

Combined Intelligence Objectives Sub-Committee.

G-2 Division SHAEP (Rear) APO 413.

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ITEM NO. 30

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**BOTROP-WELHEIM HYDROGENATION PLANT**

*Cockram, C.*

REC'D. FEB 14 1946

TIC L.F. & L. S-C.

~~RESTRICTED~~

**COMBINED INTELLIGENCE OBJECTIVES**

**SUB - COMMITTEE**

11. Description of Lurgi Process for manufacture of Synthetic Lubricating Oils.
12. Flowsheet of Lurgi Synthesis-Gas Plant.
13. Comparison of 8 processes of synthesis gas manufacture.
14. Flow diagram of gas-recirculation system of Lurgi hydrocarbon synthesis process.
15. Flow diagram and material balance of Lurgi H.P. gas-recirculation process of hydrocarbon synthesis, using catalyst.
16. Flow diagram and material balance of Lurgi H.P. gas-recirculation process of hydrocarbon synthesis using Fe catalyst.
17. Flow diagram and material balance of Lurgi H.P. gas-recirculation process of hydrocarbon synthesis, with special catalyst for high benzol yield.
18. Flow diagram of experimental hydrocarbon synthesis plant with iron catalyst.
19. Drawing of 500 mm. H.P. contact vessel.
20. " " catalyst reduction furnace.
21. " " 3 mm. H.P. contact vessel.
22. " " lamellar-type contact vessel.
23. Diagram of experimental H.P. synthesis plant for Ruhr-Benzol.
24. Drawing of experimental H.P. tubular oven.
25. Flowsheet for semi-technical experimental plant.
26. Experimental converter (70 atm. water pressure)
27. Lurgi-Hoesch negotiations on Kreislauf process.
28. Recovery of alcohols from A K - water (Hoesch)
29. The Lurgi Recirculation Process.

30. A plant for the removal of organic sulphur from synthesis gas.
31. High-temperature recirculation with cobalt catalyst.
32. Lurgi Process for Synthesis Gas production.
33. Project for converting the Wanne-Eichel Works to the Lurgi recirculation process.
34. Preparation of Paraffin Wax from Water Gas.
35. Medium-Pressure Synthesis Scheme for Japan (Rumoi)
36. Two-stage process with cobalt and iron catalysts using water gas.
37. Iron catalyst synthesis.
38. Medium-Pressure Synthesis Scheme.
39. Large-scale work on the Lurgi recirculation process at Hoesch-Benzol.
40. Engineering Drawings for the Lurgi Pressure Generator and Plant at Böhlen.
41. Angaben über Sauerstoff Anlagen.

The above documents Nos. 13 to 41 and others of related subject matters have been placed in CIOS Bags Nos. 2708, 3499, 3500 and 4132 for safe-keeping and future reference, and have been reproduced in Series A, B and E microfilms of the Liquid Fuels and Lubricants CIOS Team and been deposited with the Secretary of the Interior, Washington, D.C., and with British Ministry of Fuel and Power, London.

3955 Preparation of hard coal in generator gas manufacture  
 3953 Carbonization apparatus for oil shale  
 3948 Lubricating medium  
 3945 Production of sulfur by reduction of (ore) roasting gases.  
 3937 Improving the corrosion resistance of protective coatings.  
 3933 Sulfur recovery.  
 3927 Coolant and lubricant for metal working.  
 3914 Preparation of fuel oil from carbonization tar.  
 3913 Preparation of Diesel oil and fuel oil from coal  
 carbonization tar, shale tar, etc.  
 3896 Production of higher paraffins and olefinic hydrocarbons,  
 addition of dilute synthesis gas.  
 3893 Apparatus for thermal conversion of gases under pressure.  
 3887 Synthesis of hydrocarbons with iron catalysts.  
 3879 Ship deck paint  
 3874 Gasification of fuels in shaft ovens.  
 3859 Separation of organic sulfur from gases under pressure.  
 3840 Preparation of de-poisoned city gas by hydrogenation  
 3810 Recovery of phenols.  
 3781 Recovery of hydrocarbon mixtures.  
 3767 Gasification of fuels in shaft ovens.  
 3761 Carbonization of high ash fuels.  
 3757 Carbonization of oil shale and similar high ash fuels  
 3738 Production of hydrocarbons by CO hydrogenation.  
 3734 Preservation of wood.  
 3707 Separation of organic sulfur from gas under pressure.

APPENDIX D.

Documents and Papers.

1. File:-  
Carbonization of Bituminous Coal,  
Krupp-Lurgi Reports  
1937 to Sept.1941.
2. File:-  
Ringwaltzen Press. Tech. Reports to 1942.
3. File:-  
E.T.4. Projected Plant at Hoesch using air circulation  
1939-1944.
4. File:-  
Fischer-Tropsch Miscellaneous Projects at different  
Works including Japan.
5. Prints:-
  - a) Recovery systems of Fischer-Tropsch plants,  
different arrangements.
  - b) Flow Diagrams of Fischer-Tropsch yields  
by three recirculation systems.
  - c) Miscellaneous Drawings of Experimental Plant.
6. Stenographer's Notebook
7. "Ergebnisse der Steinkohlenschwelung"
8. "Krupp-Lurgi-Kammer-Schwellanlagen"
9. Drawing of "Krupp-Lurgi-Steinkohlenschwelofen".
10. Directors of Lurgi Personnel 1945.

APPENDIX C.

a. Department of Process Development in Erbstadt.

Director:- Dr. Ing. H. Ley  
Sulphur & Sulphur Compounds.  
Phosphorus & Phosphates  
Clay Products.  
Metallurgi:- Preparation and Smelting; for  
example, processing lean iron Ores.

RECENT METALLGESELLSCHAFT-LURGI PATENT  
APPLICATIONS OF POSSIBLE INTEREST TO  
PETROLEUM INDUSTRY.

b. Department of Surface Phenomena.

Director:- Dr. Ing. L. Schuster.  
Chemical treatment of metal surfaces for  
purposes of corrosion prevention and facilitat-  
ing shaping.

Metallgesellschaft-Lurgi Serial Nos.

4262 Insecticides  
4261 Carbonization and gasification of fuel and oil shale.  
4257 Sulphur from Coking Ovens.  
4243 Powder coking  
4230 Cracking oils, tars, etc.  
4228  
4227 Oxidation of aliphatic hydrocarbons.  
4223 Thermal treatment of liquid and gaseous hydrocarbons.  
4222  
4201 Process for oxidation of aliphatic hydrocarbons.  
4190 Applying synthetic rubber to a solid base  
4186  
4184 Combustion of H<sub>2</sub>S and O<sub>2</sub>  
4182 Tar Cracking  
4181 Shaft gasifier  
4124  
4111 Isotope diffusion  
4107  
4103 Preparation of alumina.  
4100 Carbonization process  
4090 Procedure and apparatus for mixing gases and liquids.  
4087 Synthesis of hydrocarbons with iron catalyst at atmospheric  
pressure.  
4085 Reduction (2 stage) of iron catalysts for CO hydrogenation.  
4075 Removal of acids from synthetic fats.  
4057  
4032 Production of hydrogenation catalyst.  
4011 Process for reduction of iron catalyst.  
4010 Oxidation products of predominantly paraffinic hydrocarbons.  
3997 Process of making synthesis gas of low methane content.  
3991 Process for reduction of iron catalysts.  
3961 Carbonizing oven. Damping oven with opening in side wall.  
3960 Oven for carbonization of fuels.

c. Department of Research and Service in Frankfurt am Main,  
Bockenheimer Anlage 45

Director:- Dr. Ing. habil. J. Fischer.

d. Analytical Department

Frankfurt am Main, Bockenheimer Anlage 45.

Director:- Dr. phil. Th. Becker.

e. Department for Colloid Chemistry (Z.F.K.)

Director:- Dr. phil. Miedel  
Research in Rubber, especially Revertex  
Present Activity:- Plasticizer for Buna  
(Naftolen)



APPENDIX B.

The activities of the Lurgi Companies are as follows:-

LABORATORIES OF METALLGESELLSCHAFT A.G.

Lurgi-Gesellschaft für Chemie und Hüttenwesen:  
Construction of Sintering Plants (Dwight-Iloyd)  
" " Roasters  
" " Sulphuric Acid Plants.  
Apparatus for the Cellulose Industry.  
Various Process for Lead and Zinc Smelters.

MAIN RESEARCH (Z.F.)

Lurgi-Apparatebau G.m.b.H.  
Cottrell Electrical Precipitators for Gas Purification  
Mechanical De-dusters.

Metal Laboratories. (Z.F.M.) in Niedernhausen/Taunus.

Director:- Prof. Dr.phil. E.Schmid.

Lurgi-Gesellschaft für Wärmetechnik m.b.H.:-

a) Fuel Technology

Construction of low-temperature Carbonization Plants.  
Gas Plants for Municipal Gas and Producer Gas.  
Motor-Fuel Plants (Fischer Process)  
Garbage-Disposal Plants.

a. Metals Department  
Director:- Dr.E.Schmid (whereabouts unknown)

b. Alloys Department  
Director:- Doz.Dr.phil.habil.G.Wassermann  
(whereabouts unknown)

c. Technology.  
Director:- Dr.phil.habil.k.Löhberg  
(whereabouts unknown)

b) Steam Engineering.

Evaporating Plants.  
Drying Plants.  
Crystallization Plants.  
Plants for Treatment of Vegetable Oils and Fats.  
Distillation Plants.  
Extraction Plants.

Fields of Activity:- Aluminium Alloys.

- a. Castings (especially Silumin)
- b. Forging Material (amongst others development of a copper-free forging alloy having the properties of Dur-Aluminium)

c) Recovery of Solvents (Active Carbon).

Zinc Alloys.

- a. Castings
- b. Forging Material.

Bearing Metals  
Lead Alloys.

Chemical-Metallurgical Laboratories (Z.F.C.)

Principal Offices:- Erbstadt.

Director: Doz.Dr.Ing.G.Roesner.

The chemical research reports of Metallgesellschaft were stated to be located at the laboratory in Erbstadt, near Friedberg, 20 miles north of Frankfurt. Documents relating to metals and gasification, among other subjects, were stored temporarily in Niedernhausen, near Wiesbaden, but were bombed out and shipped to Böhlen. It was suspected that these documents were lost in transit, but no definite information was available.

#### XII. RECOMMENDATIONS.

1. All Lurgi experimental data and reports relating to their work on the Fischer-Tropsch process should be examined, and Dr. Herbert and others should be interrogated further as indicated by a study of these data.

2. The Lurgi pilot plant at Hoesch Benzin (Dortmund) should be inspected and all pertinent local data regarding its operation should be picked up.

3. Details of the wax oxidation plant designed for Hubba and Fahrenholz should be obtained from appropriate Lurgi officials.

4. The Metallgesellschaft laboratory at Erbstadt should be visited and Dr. Roesner and other officials there should be interrogated in detail about their research on protective coatings and other problems.

5. The Metallgesellschaft-Lurgi pending patent applications and recent patents should be examined.

6. Complete data should be obtained regarding the iron catalyst tests conducted by Ruhland, including the details of preparing all of the catalysts involved.

#### APPENDIX A.

#### ORGANIZATION OF THE LURGI COMPANIES. (As of May 2, 1945)

The activities of the Lurgi Companies are carried on by four different Lurgi concerns:-

- 1) Lurgi-Gesellschaft für Chemie und Hüttenwesen
- 2) Lurgi-Apparatebau Gesellschaft
- 3) Lurgi-Gesellschaft für Wärmetechnik
- 4) Lurgi-Werkstätten G.m.b.H.

The capital of the Lurgi Companies is held 100% by the Metallgesellschaft, A.G., a company with a capital of 70,000,000 RM. The name Lurgi is derived from the original field of activity "Metallurgie". The Companies are directed by the following individuals:-

Director of all Lurgi Companies: Dr. Oetken who at the same time is a member of the Board of Directors of the Metallgesellschaft:-

Dr. Gähre  
Dir. Klencke  
Dr. Siecke  
Dir. Behlert  
Dr. Oetken.

#### Lurgi-Apparatebau Gesellschaft:

Dir. Räder  
Dir. Gies  
Dr. Oetken

#### Lurgi-Gesellschaft für Wärmetechnik m.b.H:

Directors:-  
Dr. Oetken  
Dr. G. Müller  
Dir. Klan.

#### Principal Technologists:-

Low-temperature  
Distillation of Fuels: Dr. Hubmann  
Production of City &  
Synthesis Gas: Dr. Danulat  
Synthesis of Motor Fuels: Dr. Herber  
Power Plants: Dipl. Ing. Müller  
Dr. Ruesping.

#### Lurgi Werkstätten G.m.b.H.

Dr. Räder  
Dir. Wätcher  
Dr. Oetken.

#### IX. PARAFFIN WAX OXIDATION.

Lurgi had started the construction of a plant for the oxidation of Fischer-Tropsch wax by the firm Hubba & Fahrenholz at Magdeburg. This plant was not entirely finished but had operated on a small scale until the supply of Fischer-Tropsch wax was cut off by bombing of the synthesis plants. Presumably more information on this subject could be obtained from Lurgi files, or by questioning of the officials concerned, but lack of time prevented this course being followed on the present trip. Lurgi did not have any contractual agreement with Ruhrchemie regarding the oxidation of Fischer-Tropsch wax.

#### V MISCELLANEOUS RESEARCH.

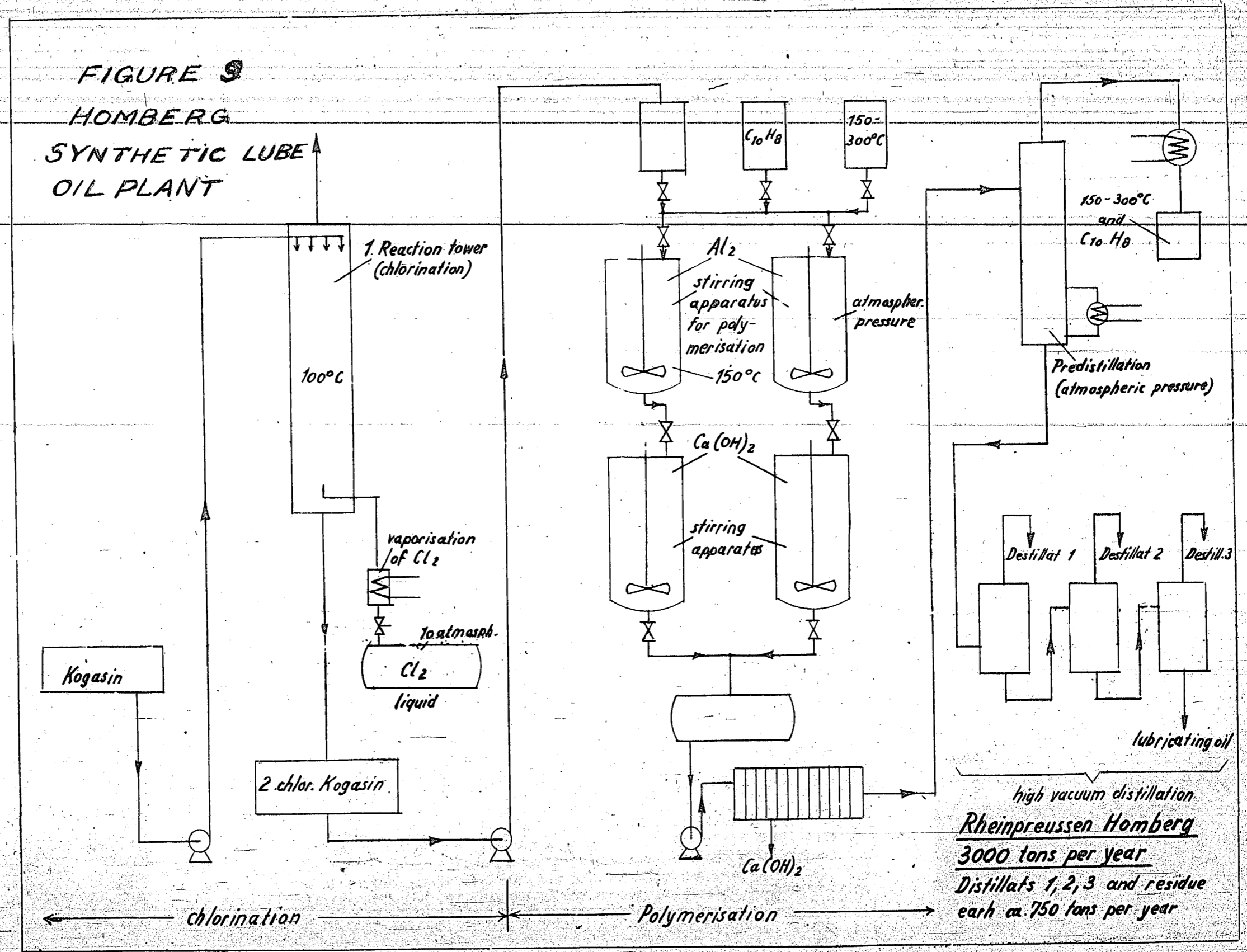
According to Dr. Herbert the research on protective coatings for metals, mentioned in the attached outline of activities, related to treatment with phosphate solutions (similar to Parkerizing). Details could be obtained from Dr. Roesner at the Metallgesellschaft laboratory in Erbstadt but time did not permit getting in touch with him on this trip.

Dr. Herbert also stated that the indicated work on rubber softeners consisted mainly in routine evaluations of solvent extracts from Rhenania Oessag conducted at the Lurgi Frankfurt Laboratory for some outside firms who were using such extracts. No further enquiries were made on this subject.

#### XI. PATENT ACTIVITIES:

The Patent Department of Metallgesellschaft and Lurgi had been moved to Castle Koenigstein at Kronberg, and was functioning there until shortly before the arrival of American troops. Certain business departments of Metallgesellschaft had also moved to the Castle and additional activities were housed in the Kronberg-Hof. The patent files were inspected superficially with Mr. H.C. Heine, head of the Patent Department. They include about 800 pending patent applications and a fairly complete file of United States and German patents for research purposes. The German patents were believed to be complete up to the time when the German Patent Office ceased to function, and would be a useful collection of recent German patents in the fields of Metallgesellschaft and Lurgi activity. Unfortunately these files were badly disarranged in transfer to the Reichsbank at Frankfurt, but are presumably intact and available there if needed. From Lurgi indexes for recent years a list of their patent applications which appeared to be of particular interest to the oil industry had been prepared. This list is attached for the use of anyone who may desire to obtain these files from the Reichsbank.

**FIGURE 9**  
**HOMBERG**  
**SYNTHETIC LUBE**  
**OIL PLANT**



(1) Compressors and Blowers:-

Gutthoffnungshütte, Oberhausen,  
Maschinenfabrik Sauer & Co. Ltd.

(2) Refrigerating Apparatus

Linde Werke, Hiltropstraße

(3) Engine Machines

DeV Lyndt, Berlin Tempelhof.

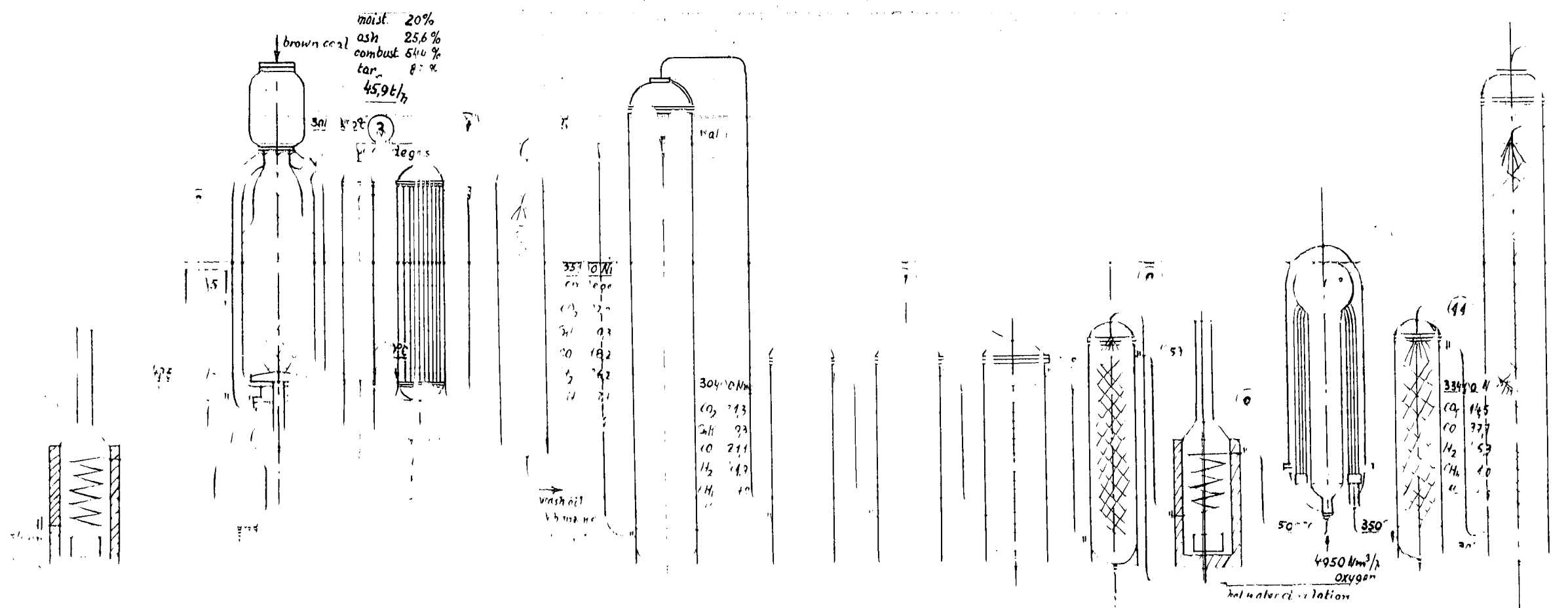
(4) Aluminum Packing for the Regane

Dr. Kolbel, Augsburg

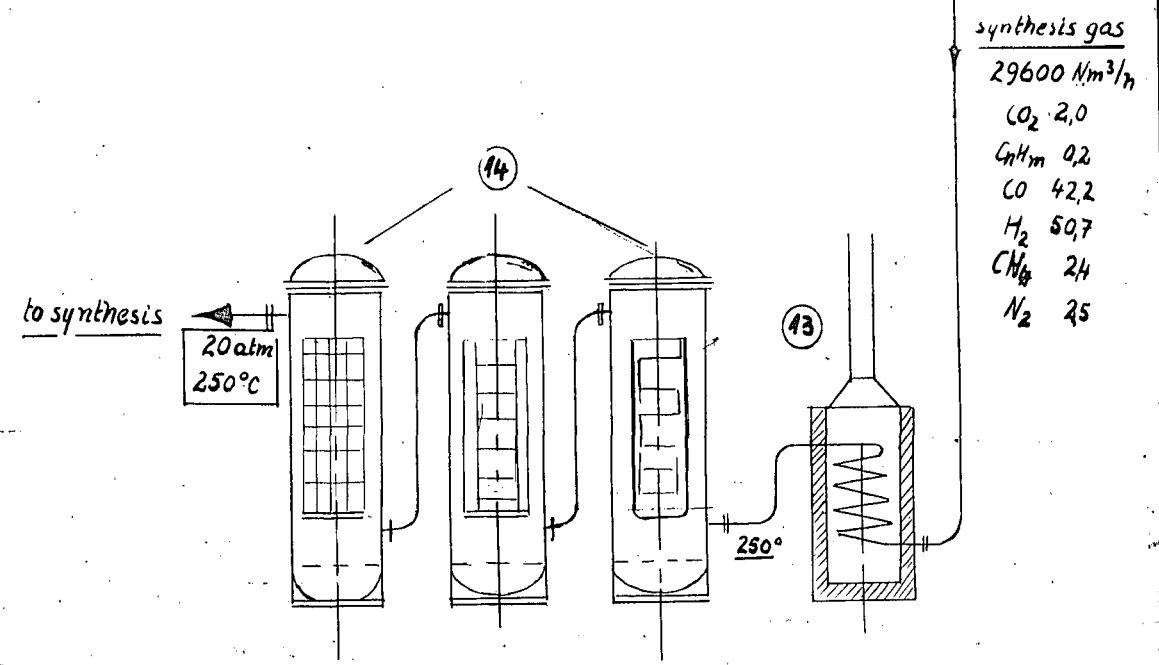
III SYNTHETIC LUBRICATING OILS

Lurgi built the equipment for the Rheinpreussen synthetic lubricating oil plant at Homberg. Dr. Herbert said that this was the only plant of its kind in Germany, but he had no personal knowledge of the quality or uses of the oil made there. However he had been told by Dr. Kolbel, in charge of this work at Homberg, that they considered the oil a superior lubricant for steam engine and marine use only. A flow diagram for this Rheinpreussen process as furnished by Lurgi in response to the request of Capt. Chaffee, is reproduced as Figure 9, page 43. The operation of this plant has been described in detail in CIOS report No. 2, Target 30/5.05.

Lurgi also built the atmospheric and vacuum stills for the synthetic lubricating oil made by the Ruhrchemie aluminum chloride polymerization process at Oberhausen. These stills were designed by Messrs. Morlock and Siebert of the Lurgi organization in 1940 or 1941. Dr. Herbert believed that the capacity of the Ruhrchemie plant was 20-25 tons of lubricating oil per day and that the operation was entirely successful. He did not know where the products were tested or used. Preliminary fractionation tests on the overall product were made at the Lurgi carbonization laboratory in Hedderheim, but Dr. Herbert doubted if any samples of the oil would still be left at this laboratory since it had been badly damaged by bombing. It seems probable that additional information about these oils could be obtained from Lurgi files or by interrogation of Morlock and Siebert. However these possibilities were not pursued since it was assumed that first-hand information would be obtained from Ruhrchemie by other CIOS teams.



- ① steam superheater
- ② gas producer
- ③ spray cooler
- ④ tube cooler
- ⑤ benzene scrubber
- ⑥ water wash tower I
- ⑦ extraction of H<sub>2</sub>S
- ⑧ saturator
- ⑨ gas superheater
- ⑩ cracking chamber for methane
- ⑪ spray cooler
- ⑫ water wash tower II
- ⑬ superheater
- ⑭ extraction of organic sulphur



synthesis gas

29600 Nm <sup>3</sup> /h
CO <sub>2</sub> 2.0
CH <sub>4</sub> 0.2
CO 42.2
H <sub>2</sub> 50.7
CH <sub>6</sub> 24
N <sub>2</sub> 45

Fig. No. 8.

Lurgi Gasification Process.

Ffm. 4.12.45 Dev.

the Societa Italiana Carburanti Sintetici project, which was to use the Lurgi Gasification process, was placed with Messer, not because their estimate of power consumption was lower than that of Linde (0.536 as compared with 0.58 Kw. per Nm<sup>3</sup> of O<sub>2</sub>), but because Linde was unable to undertake the contract.

For the purpose of estimating the cost of oxygen production, Lurgi use a figure of 0.65-0.68 KwH per Nm<sup>3</sup> for total power consumption on the air-separation plant excluding power for the subsequent compression of the oxygen to the pressure used for gasification, (A figure was obtained later at Boshlen of 1.2 KwH including the power for compression to 23 atm.). The total cost of producing oxygen in plants containing units of from 1000 to 2000 Nm<sup>3</sup> per hour capacity was estimated as 2.2 to 2.5 pfg. per Nm<sup>3</sup> of which cost, 40% is for capital depreciation, 40% for power and 20% for wages, maintenance and chemicals. The cost would be less for larger units; thus, in connection with a recent project requiring 17,000 Nm<sup>3</sup> per hour, Linde had proposed a plant consisting of 6 units, one a stand-by, and had estimated that the total cost of production would be 1.5 pfg. per Nm<sup>3</sup> of which 0.4 pfg. would be for capital depreciation.

The oxygen used for pressure gasification was usually of about 95% purity. This concentration could be reduced by addition of air when gas of low calorific value was required. The plant at Hirschfelde was operated on "rich air" containing 70-75% of oxygen.

A short description of the Linde-Frankel Process for air separation was given as follows:-

All the inflowing air to be separated is compressed in Turbo-compressors (made by Guttehofnungshütte, Oberhausen) to 4.5 or 5 atm. After removal of carbon dioxide by means of aqueous caustic, the compressed air is introduced into the separating equipment which comprises oxygen and nitrogen-regenerators filled with aluminum packing, and the rectifier. A smaller part of the low-pressure air coming from the first compression-stage is further compressed from 4.5 atm. to 200 atm. following which the highly-compressed air is cooled to minus 45°C. before entering the expansion machine. The high-pressure compressors have 5 stages. The cold required for the process is provided in expansion machines (Heylandt, Berlin-Tempelhof) in which a portion of the compressed air from the high-pressure compressors is expanded from 200 atm. to 4.5 atm. with energy recovery. For plants of very large capacity, Linde has employed other process method; for example, expansion of the outlet cold nitrogen from the separating equipment in turbines. The manufacturers for the machines and apparatus of the plant follow:-

2. Pressure Gasification with Cracking of the Crude Gas (without Regenerators);
3. Atmospheric Gasification with addition of CO<sub>2</sub>;
4. Atmospheric Gasification with addition of CO<sub>2</sub>;
5. Pressure Gasification with Cracking of Crude Gas (with Regenerators);
6. Pressure Gasification with Cracking of Residue Gas (Koppers);
7. Pressure Gasification with CO<sub>2</sub> addition; and
8. Winkler Gasification with Prior Low-Temperature Distillation;

were made by Lurgi and it came to the conclusion that the economically most advantageous method for preparing the Synthesis Gas from the lignitic brown coal was to employ oxygen at a pressure of about 23.5 atm. and, after washing the produced gas with water to remove about one-third of the CO<sub>2</sub> and some of the H<sub>2</sub>S followed by oxide treatment to remove the remainder of the latter, the resultant gas is heated to about 145° C. and saturated with water. Thereafter, the gas is preheated to 500°C. and by partial combustion with oxygen, without regenerative heating, is heated to 1300°C. in a methane-converter in the absence of a catalyst, thereby to convert methane content thereof to CO and H<sub>2</sub>. By heat exchange, the outlet gases from the methane converter are reduced to 350°C. at which temperature, and under pressure, they are scrubbed with water and cooled to about 30°C.; the hot scrubbing water is circulated to the above 145°C water-saturator step where some of its heat is employed to preheat and saturate gas that enters the 500°C. preheater for the methane converter. After another water-scrubbing step, for the substantial removal of CO<sub>2</sub>, the formed crude Synthesis Gas has then the following composition:-

CO <sub>2</sub>	-	2.0%
C <sub>2</sub> H <sub>6</sub>	-	0.2
CO	-	42.2
H <sub>2</sub>	-	50.7
CH <sub>4</sub>	-	2.4
N <sub>2</sub>	-	2.5

The Synthesis Gas is then preheated to 250°C. and passed to

the apparatus for removal of organic sulphur whereupon it is ready for delivery to the synthesis.

In this scheme of Synthesis-Gas production, there is consumed per m<sup>3</sup> thereof, 0.317 m<sup>3</sup> of oxygen. It was stated to be advantageous that the high-temperature methane cracking reduced the amount of gums in a synthesis gas; it was considered important by Lurgi to make provision to remove these compounds from a synthesis gas by, for example, active carbon to protect the catalyst.

Dr. Danulat in written statements, has emphasized the importance of the slagging generator for solving the above technical problem; its use would provide both the cheapest plant and lowest operating costs, but he indicated in December 1942 that such equipment was only in the development stage and could not be recommended for the Fischer-Tropsch plant at V aldarno, Italy. He also stated that the above pressure-generator operation, coupled with thermal cracking without regenerative heating, is even more favorable than the Winkler gasification process employed for the same technical problem, and emphasized that such operation had for Lurgi the advantage that it could make the company a competitor of the Winkler process in the field of ammonia synthesis and hydrogenation. Dr. Danulat emphasized that the development of a slagging pressure-generator was a necessary development for Lurgi to undertake immediately. During one of the interrogations, he stated that such a generator would be built along the lines of the blast furnace.

The accompanying Fig. 8 shows diagrammatically a flow-sheet and apparatus as recommended by Lurgi for preparing Synthesis Gas having a H<sub>2</sub>/CO ratio of 1.2 as is employed in combination with the Fischer-Tropsch iron catalyst.

#### Oxygen Production

The pressure-gasification plants erected at Hirschfelde, Boehlen and Bräx are equipped with air-separation plants of the standard Linde type erected by Ges. fuer Linde's Eismaschinen, A.G. of Munich. Lurgi have not carried out any research on problems connected with the separation of air into components and could supply no detailed information concerning recent developments in the use of expansion turbines.

The Messer Co. of Frankfurt-am-Main have erected a number of air-separation plants of the Linde type using normal heat exchangers, but were believed to have carried out some work on the design of expansion turbines. The contract for the air separation plant of



### Synthesis Gas.

In the Fischer-Tropsch synthesis of hydrocarbons from CO and H<sub>2</sub> as usually practised with the cobalt-thoria catalyst, the employed Synthesis Gas preferably has a H<sub>2</sub>/CO ratio of 2. The lesser the content of inerts, the better. As above indicated, the pressure-generator gas product, after removal of carbon dioxide, contains hydrogen and carbon monoxide in some instances in a little higher than the said ratio of 2 and in the other instances, somewhat less; it also contains somewhat over 20% of methane. Such large proportion of methane would seriously limit the output of any Fischer-Tropsch hydrocarbon plant as it acts as a diluent and does not take part in the synthesis.

However, the pressure-generator with the advantages of continuous operation, as distinguished from the intermittent production of water-gas sets and also the facts that it operates satisfactorily on less expensive fuels and that it produces a gaseous product having a higher H<sub>2</sub>/CO<sub>2</sub> ratio than does the usual water-gas generator, can be employed for the manufacture of Synthesis Gas.

As hereinbefore mentioned, the actual pressure existing in the pressure generator influences the nature of the produced gas. At lower pressures, less methane is formed. In the presence of carbon, pressure displaces the CO/CO<sub>2</sub> equilibrium in the favor of carbon dioxide. If also the gasification process is carried out at a relatively low temperature, the equilibrium is further displaced in favor of a reduced formation of carbon monoxide. The pressure-gasification thus enables the formation of carbon monoxide to be restricted from two sides; that is, through the temperature and pressure of gasification. Since the required reduction of carbon monoxide formation is not very great, it is adequate to reduce the pressure from the usual 20 atm. to about 5 to 10 atm./sq.in.

Practical tests have shown that a carbon monoxide-hydrogen gas mixture suitable for the Fischer-Tropsch synthesis with cobalt-thoria catalyst can be prepared by the gasification of low-temperature bituminous coke (10-20 mm grain size) when employing a gasification pressure of about 8.5 atm. and both steam and oxygen as the gasification media. The formed crude gas has the following composition:

CO <sub>2</sub>	-	29.3%
CO	-	21.8%
H <sub>2</sub>	-	44.0%
CH <sub>4</sub>	-	3.3%
N <sub>2</sub>	-	1.5%

After removal of the carbon dioxide in the water-scrubbing step under pressure, the crude-gas yields a Synthesis Gas having the

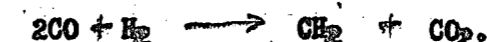
following composition:-

CO <sub>2</sub>	-	1.0%
CO	-	30.7%
H <sub>2</sub>	-	61.6%
CH <sub>4</sub>	-	4.6%
N <sub>2</sub>	-	2.1%

It should be further recorded that by increasing the addition of steam in the pressure-gasification, the formation of carbon monoxide may be so extensively reduced that a water-scrubbed gas containing 70-75% of hydrogen and only 15% of carbon monoxide is produced. This gas is well-suited for use in the synthesis of ammonia and hydrogenation.

The pressure under which the Synthesis Gas is produced has the advantage also that the hydrogen sulphide and some organic sulphur is removed at the same time as carbon dioxide is separated by the water-scrubbing step. The purification of the Synthesis Gas of sulphur compounds is thus importantly cheapened.

The iron catalyst prepared by Lurgi for the preparation of hydrocarbons from Synthesis Gas requires in such gas a H<sub>2</sub>/CO ratio differing from the requirement of the cobalt-thoria catalyst; the preferred synthesis pressure with the iron catalyst is 22 atm. and the H<sub>2</sub>/CO ratio in the employed gas is 1.2 with recirculation over the catalyst. The equation which seems best to explain the reaction with the iron catalyst is as follows:-



The high pressure of the Synthesis Gas required for the iron catalyst is easily supplied directly by the generator for the high-pressure gasification with oxygen. However, when the generator is normally operated at a pressure of over 20 atm. and a high temperature, as above discussed, the methane (inert) content of the produced gas and the H<sub>2</sub>/CO-ratio are too high. The CO content of the produced gas can be increased by employing higher temperatures or lower pressures, but the possible increase in temperature is limited by the fusion-point of the ash in the employed fuel and any sharp decrease in pressure would require provision of means to compress the produced gas up to the above-requires pressure of synthesis. This was the problem presented Lurgi in connection with the design of the SICS-plant project at Valdarno, Italy. The fusion-point of the ash of the employed coal was relatively low. Analyses of the various possibilities available to the technology for its solution, such as:-

1. Pressure Gasification with Cracking of Residue Gas (Bawag);

Results of Pressure-Generator Operations on Various Fuels. (Contd.)

	<u>Brown coal.</u> (Lausitz)	<u>Brown Coal</u> Middle Germany)	<u>Lean Coal</u> Fines (Ruhr)
6. Oxygen Requirement Nm <sup>3</sup> /Nm <sup>3</sup> Gas	0.15	0.145	0.198 *
7. Steam requirement Kg/Mm <sup>3</sup> gas	0.01	1.05	1.40

\* For comparative purpose, it is noted that the Winkler Generator employs slightly more than twice as much oxygen per m<sup>3</sup> of gas produced.

If, in the case of the above bituminous coal, the pressure is raised to 30 atm., the calorific value of the gas is raised from 4100 to 4300 Kcal/Nm<sup>3</sup>

Recovery of Liquid Hydrocarbons.

The crude gas leaving the pressure-generator at 300-350°C. is cooled in two steps. The first step is performed in a direct-spray cooler wherein the temperature is reduced to 120-140°C. and the second step in a tube-cooler that reduces the gas temperature to 30°C. This condenses much of the tar and water.

From the above description of the process, it is manifest that this gasification method is broadly a counter-current process which naturally protects somewhat the original hydrocarbons in the coal and makes it possible to recover, as tar, up to 85% of the tar content of the coal, as determined analytically.

The total tar is different from that obtained by atmospheric-pressure low-temperature distillation by the high proportion of hydrocarbons boiling up to 200°C. which is increased about 20%. The increased pressure has little effect on the other characteristics (Creosote, Paraffins, etc.) since the produced gas acts as a Spilgas. Only the high-boiling resins and asphalts are in part decomposed.

Dust and water content of the tar are similar to those of a good low-temperature tar. The gasoline fraction has an octane number of 90-100, and after refining, is a good motor fuel. The good quality of the tar makes a preliminary low-temperature carbonization of bitumen-containing fuel superfluous.

The benzene of the light-oil fraction is recovered by scrubbing the gas cooled to 30°C., or the like, with a scrubbing oil while it is still under pressure. The concentration of benzene in the scrubbing oil is 6-8 percent by volume.

Purification of the Gas.

After the above oil scrubbing, the gas passes, under pressure, to a water-scrubber wherein it is treated with about 700-800 m<sup>3</sup> of water per hour per 6000 m<sup>3</sup> of clean gas. The scrubbing water is then expanded in Pelton pumps, aerated and recirculated. This removes carbon dioxide and a large amount of the hydrogen sulphide. Residual hydrogen sulphide amounting to about 5 g. per 100 m<sup>3</sup> is removed in iron oxide.

If the employed coal has a high content of sulphur, the gas issuing from the water scrubbing will contain less than 20 g/100 Nm<sup>3</sup> of organic sulphur; it can be removed by the Ruhrchemie process employing alkaline iron oxide.

Heat Balance of the Process.

A. Heat Input	in %
1. - Coal	89.9
2. - Steam (saturated)	10.1
	<u>100%</u>

B. Heat Output	
1. Chemically combined heat in the town gas.	62.2%
2. In tar and benzene	14.3%
3. Phenols in gas liquor	0.3%
4. Carbon in ash	0.4%
5. Steam from cooling jacket	0.6%
6. Sensible heat of gas & heat losses.	16.2%
7. Losses in steam superheater and aeration tower.	5.4%
	<u>100%</u>

The efficiency of fuel conversion to gas is thus about 85.2%

Production cost of the city gas using brown coal was stated to be about 2.4 pfg. per m<sup>3</sup> as compared with coke-oven gas at 1.8 pfg. delivered to the distributor; that is, if the dried brown coal costs 6.5 RM per ton.

ring so-formed and supported as to expand against the valve seat under the influence of the gas pressure. It was stated that the Brück generators, which have not been seen, have an internal hydraulic ram in the coal pocket for opening the upper valve. The hardened-steel cone valve is of V<sub>2</sub>A steel. Incidentally, it was also stated that bituminous coal has been gasified successfully, but optimum conditions had not been established, and Dr. Oetken stated that a true coking coal had never been employed in the pressure generator; however, Ruhr anthracite and the so-called "gas flammkohlen" can be used. Too strongly coking coals are precluded from use.

In the operation of the generator, the carbon dioxide content of the raw gas issuing therefrom is the control index of operating conditions. The temperature of gasification as measured by Seger cones in the fuel bed is 1050-1100°C.

The outlet temperature to the generator is controlled by the moisture content of the brown coal - with 20% water the temperature is 300-320°C. The velocity of the outlet gases is maintained at about 2.5 meters per second so as to prevent precipitation of tar and dust in the upper part of the generator.

The super-heated steam continuously flowed into the lower part of the generator is prepared by combustion of the high-pressure gas discharged from the generator's coal-pocket during its charging. A special relief holder is provided for its storage.

#### Coal Capacity of a Generator.

A pressure-generator having a 5 m<sup>2</sup> cross-section when operating at 20-30 atm. can gasify:-

100 to 150 tons of dried brown coal per 24 hrs, or  
60 to 70 tons of bituminous coal.

This corresponds to a capacity per generator of  
2750-3500 Nm<sup>3</sup> city gas per hour, or 20,000,000 Nm<sup>3</sup>  
per year.

Normal operating rates can be reduced to one-third of capacity and temporary fluctuations are possible without giving rise to any operational difficulties, thus making operation very elastic.

#### Gas Yield of a Generator.

The attainable gas yield depends on the characteristics of the fuel. Fuels high in moisture and ash naturally give a lower yield;

the same obtains for the brown coals since the higher yields of liquid products - tar, oils and gasoline - reduce the amount of gasifiable carbon of the coal.

1 ton dry brown coal (20% moisture, 10% tar)  
gives 680-580 Nm<sup>3</sup> gas having a gross  
heating value of 4300-4500 Kcal/m<sup>3</sup>.

1 ton lean coal yields 1570-1670 Nm<sup>3</sup> having a  
gross heating value of 4100-4300 Kcal/m<sup>3</sup>.

1 ton lean coal yields 1570-1670 Nm<sup>3</sup> having a  
gross heating value of 4100-4300 Kcal/m<sup>3</sup>.

#### Results of Pressure-Generator Operations on Various Fuels:

	Brown Coal (Lausitz)	Brown Coal. (Middle Germany)	Lean Coal Fines (Ruhr)
<b>1. Composition of the Fuel</b>			
Combustible Substance %	67.5	76.3	88.4
Water %	27.4	14.8	6.6
Ash %	5.1	8.9	5.0
Tar (Fischer Method) %	10.2	12.6	
Gross Heating Value Kcal/kg.	4730	5260	7600
Grain Size mm.	2.10	2.10	3.10
<b>2. Operating Pressure at.</b>			
	20	20	20
<b>3. Throughput Kg/m<sup>2</sup>h</b>			
	750	770	310
<b>4. Composition of Purified Gas.</b>			
CO <sub>2</sub> %	3.0	2.3	1.0
C <sub>n</sub> H <sub>m</sub> %	0.5	0.9	0.3
O <sub>2</sub> %	0.1	0.2	-
CO %	22.8	22.0	27.9
H <sub>2</sub> %	48.7	50.7	52.4
CH <sub>4</sub> %	22.6	21.8	16.9
N <sub>2</sub> %	2.3	2.1	1.5
Density Gas (air = 1)	0.448	0.435	0.432
Gross heating value Kcal/Nm <sup>3</sup>	4280	4500	4100
<b>5. Gas Yield Nm<sup>3</sup>/to coal</b>			
	760	680	1690

product, as prepared from brown coal at 20 atm. pressure with oxygen is as follows:-

	Crude Gas	Purified Gas.
CO <sub>2</sub>	30.6% by vol.	3.0% by vol.
C <sub>n</sub> H <sub>m</sub>	0.6	0.5
O <sub>2</sub>	0.1	0.1
CO	16.5	22.8
H <sub>2</sub>	34.0	48.7
CH <sub>4</sub>	16.3	22.6
N <sub>2</sub> , etc.	1.9	2.3

The oxygen consumption is relatively low and because of the exothermic heat of reaction of the produced methane, hot spots within the fuel bed are avoided. This in turn makes it possible to control slag-formation and consequently to maintain uninterrupted operation in a closed generator without any disturbances.

Normally, only solid fuels that are weakly-coking or non-coking are employed in the process; for example, lean bituminous coals, and all brown coals. Easily coking coals should be given a pre-treatment to destroy some of the coking properties. The water content of the employed fuels should not be over about 25%.

The preferred sizes of the fuel are about 5-25 mm. and especially about 5-15 mm. The fuels should be practically free of dust; that is, no constituents less than 2 mm. Throughputs of up to 1000 kg. per hour per m<sup>2</sup> of generator cross-section have been reached with fuel consisting of grain sizes of 2-8 mm.

The heating value of the produced gas depends on the reactivity of the fuel. At 20-25 atm., a gas having a gross heating value of 4300-4600 Kcal/Nm<sup>3</sup> is obtained when using brown coal. Bituminous coal or coke at the same pressure gives gas of a heating value about 200 Kcal. lower. The high conversion of carbon to carbon dioxide does not mean any loss of heat because, in the process, Hydrogen and methane are formed and the involved reactions converting CO to H<sub>2</sub> or CH<sub>4</sub> are always accompanied by the production of the inerts CO<sub>2</sub> and H<sub>2</sub>O; and the high conversion of chemical energy is thus not influenced by large CO<sub>2</sub> formation.

The pressure-generator gas has the following characteristics:-

- Relative Density (Air = 1) less than 0.5;
- Oxygen content " " 0.3% by vol.;
- It is substantially free of H<sub>2</sub>S, NH<sub>3</sub>, Naphthalene and tar;
- Its content of organic sulphur when employing high-sulphur coals is less than 20g/100 Nm<sup>3</sup> and by strong aeration of the water employed for removing the CO<sub>2</sub> it can be further reduced.

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In consequence of this scrubbing of the crude gas with water under pressure, the effluent gas contains not more than 5-10 grams H<sub>2</sub>S/100 Nm<sup>3</sup>; complete purification thus requires only a small oxide-purification installation. Cooling the gas under pressure also provides it with a very low partial-pressure of water vapour.

#### Installations.

The following plants have been built by Lurgi, all of which are built around the high-pressure generator:

- Hirschfelde near Zittau: This was the first commercial plant and had a capacity of 5 million m<sup>3</sup> per year of City gas.
- Böhlen near Leipzig: Two plants having a total capacity of 150 million m<sup>3</sup> per year.
- Bráx in Czechoslovakia: This plant was designed for a capacity of 100 million m<sup>3</sup> per year.

The first plant at Böhlen had 5 generators built in 1939 and to this the second plant, built in 1942, added 5 more. The Bráx plant was built in 1943.

#### The Generator.

The generator is a pressure vessel lined with bricks and is furnished with a water-cooled jacket. Despite the high pressure of operation, the coal-charging and ash-discharging pockets are so designed and provided with valves that the generator is continuously operative during both the charging and the ash-removal steps.

The outside shell of the generator is of ordinary steel and is 50 mm. thick, whereas the inner shell of the water-jacket is boiler steel about 25 mm. thick. One of the important points in design for successful operation is the refractory lining; it must be just thick enough to permit reaction-heat to be sufficiently rapidly carried away to prevent slagging of the coal ash. In those generators having a diameter of 2.5 meters (O.D.3.m), it is about 250 mm. thick. The latest generators have the above-stated diameter, but that of the Hirschfelde installation was 1 meter. Dr. Danulat said that the only limits, however, to diameter was transport considerations.

The rotatable grate that supports the fuel-bed is formed of cast chrome-nickel steel; its speed of rotation determines the amount of ash delivered to its discharge-pocket.

The valves employed for sealing these pockets from the generator during their operation are provided with rubber sealing-

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Gas formed by the process of coal distillation on a mixture of 75% gas coal + 25% Fetco coal has the following analysis:-

Carbon dioxide	-	4.5%
C <sub>n</sub> H <sub>2n</sub>	-	2.5%
C <sub>x</sub> H <sub>y</sub>	-	0.5%
Oxygen	-	0.2%
Carbon Monoxide	-	3.5%
Hydrogen	-	23.5%
Ethane	-	13.6%
Methane	-	46.1%
Nitrogen	-	5.0%
Density	-	0.805
Gross Heating Value	-	8137 Kcal/m <sup>3</sup>

The gas driven off the low-temperature coke by heating the same to 1000°C according to the laboratory method of Bauer has the following analysis; it amounts to about 225 m<sup>3</sup> per ton of said coke:-

C <sub>x</sub> H <sub>y</sub>	-	0.3%
Carbon Monoxide	-	7.4%
Hydrogen	-	77.2%
Methane	-	12.5%
Nitrogen	-	2.0%
Gross Heating Value	-	3840 Kcal/m <sup>3</sup>

The Krupp-Lurgi metallic coking cells were developed for coking at low-temperature of between 500-600°C. both coking and weakly-coking coals. Most any such coals as exhibit ability to form coke can be processed in them. Non-coking coals preformed into egg-shaped briquettes by means of bituminous binders, and the like, have also been processed to advantage and the coking time is reduced to about 65% of that required when the cells are charged with the same coal in unbriquetted condition. This increased capacity in many cases covers the cost of the briquetting.

The philosophy behind the development of this type of coking oven or cell has been stated as follows:-

The forming of a good- low-temperature coke depends upon a sufficiently rapid inflow of heat into the cells, between temperatures of 350° and 450°C., to bring all portions of a charge to the melting point about simultaneously. The good heat conductivity of the metal walls and the relative thinness of the charges makes this possible.

The more rapidly the temperature is raised, the more quickly all constituents that take part in the forming of the coke exert their effects and the less opportunity the divers constituents have to exhibit any possible distinctions in their reactivities such, for example, as differences in pre-principal- and post-gasification temperatures and to

decompose either too early or too late to make a substantial contribution to the cementation of the coal particles. By bringing all parts of the coal charges simultaneously to the molten state no highly viscous plastic seams are formed through which gases can escape only by exerting high pressures. Consequently, such coals as in the horizontal high-temperature coke ovens exhibit damaging expansion pressures against their heating walls, can be coked in the metallic cells without any serious pressures developing. Such highly expanding coals as those having 19.5 to 21% volatile-matter content have been coked in these oven-cells without grinding or any reduction in bulk density and without any resultant deleterious effect on the metal walls of the coking cells.

Reference is made to a Report by Drs. Hall and Powell on CIOS Target 30/5.02 for further data on the low-temperature carbonization ovens.

#### VII. HIGH PRESSURE GASIFICATION.

In the high pressure gasification process developed by Lurgi Wärmetechnik, solid fuel is continuously gasified at from 20-30 atm. pressure with oxygen and super-heated steam. The generator is of special construction and is designed as a pressure vessel having spaced double walls that provide a water jacket and those portions of the generator that are subjected to pressure are protected from overheating by a lining of refractories. Double-valved charging and discharging pockets at the top and the bottom of the generator serve respectively to charge the fuel and discharge the granular ashy residue in much the same fashion employed in gas-producers operating at atmospheric pressure or slightly thereabove. The charged fuel rests in the generator on a variable-speed rotary grate; the oxygen and steam are flowed upwardly through the grate and the fuel bed. The reaction between the fuel, steam and oxygen proceeds with decreasing temperature from its hottest part immediately above the ash-bed to the upper part of generator and passes respectively through the phases of combustion, gasification, distillation and drying of the fuel.

Gasification of coal under pressure with oxygen produces a gas-product having a relatively high content of carbon dioxide and methane along with carbon monoxide and hydrogen. With increasing pressures up to about 20 atm., the proportions of methane and carbon dioxide increase in their contents in the crude gas, whereas the hydrogen and carbon monoxide contents decrease. Since the crude gas leaving the generator is at about 20 atm. pressure the carbon dioxide is to a great extent easily removed by scrubbing with water leaving a residue-gas of high calorific value (4000-4600 Kcal/Nm<sup>3</sup>) that is, substantially equivalent to coke-oven gas. A typical crude gas and its purified

Normal Distillation  
of the Tar.

About 1% water  
3-4% Crude Gasoline  
15-20% Acid Oils  
37-47% Fuel Oil  
30-40% Pitch

Normal Distillation  
of the Tar.

About 2-4% Water  
0.8-1.2% Crude light oil  
4-6% Middle oil  
7.10% Heavy oil  
20-25% Anthracene oil  
4-6% Naphthalene  
55-60% Pitch

The values in the following table show the characteristics of the low-temperature coke obtained in the Krupp-Lurgi cells when operating on a high-volatile coal of low-ash content.

The Coal:-

Approximate analysis:-

Water 8.2%  
Ash 3.24%  
Volatile matter 22.4%

Screen analysis:-

0 to 1 mm. 38.7%  
1 to 3 mm. 34.4%  
3 to 6 mm. 23.2%  
6 to 10 mm. 3.7%  
Bulk Density Modst Coal 0.846 to 1 m<sup>3</sup>

The Low-Temperature Coke:-

Approximate analysis:-

Ash 3.78%  
Vol. matter 9.1%

Ultimate analysis:- (ash-free basis)

Carbon 91.45%  
Hydrogen 3.76%  
Sulphur 0.85%

Heating Value (ash-free basis) - 8240 Kcal/kg (gross)

Ignition point (air) 396°C.  
Porosity 50.4%

Drum Test:-

Over 40 mm. 78.5%

Size of the Crushed Coke:-

Over 40 mm. - 77.2%

40-20 mm. - 9.6%  
20-10 mm. - 3.8%  
10-6 mm. - 2.0%  
Below 6 mm. - 7.2%

A typical low-temperature tar obtained from mixed coals showed the following characteristic analysis:-

First Drop - 97.5°C  
to 200°C. - 3.0%  
200° to 230°C. - 12.5%  
230° to 270°C. - 13.5%  
270° to 300°C. - 10.0%  
300° to 360°C. - 21.5%  
60.5%

Specific Gravity - 1.058  
Viscosity 20°C. 35° E.  
50°C. 4.5° E.  
Pour Point (Stockpunkt) minus 1° C.

The low-temperature tar is of such character that after careful de-watering it can be used as a heating oil without further refining and it is claimed that 50-60% of the total tar yield can be easily used for fuel in slow and moderate speed Diesel motors. About 10% of the Diesel oil fractions are carbolic oils having a relatively large content of meta-cresol. The high-boiling fractions of the tar are especially valuable, because of their high hydrogen content, as raw material in high-pressure hydrogenation plants.

Water-gas produced from the coke is said to contain CO and H<sub>2</sub> in the ratio of 1:1.5 instead of in the ratio of 1:1.25 obtained from high-temperature coke.

The underfiring requirement per kilogram of coal is 500 kg. cal. which is equal to 900 BTU per pound of charged coal.

The coking time is about 6 hours for the substantially 3.5 in. thick cell charges.

remained unaltered and that no alteration in the carbon content of the steel has been determined.

Heating of the metal ovens and their individual coal cells is effected by circulating hot products of combustion through these heating spaces that alternate with the coking spaces. These circulated hot gases enter the ovens at a temperature of about 600-620°C. at one face and suffer a drop in temperature of about 40° to 60°C. at their outlets at the opposite face; the gases are then returned to the inlet side of the ovens and there reheated by admixture with hotter combustion-products before returning to the coking chambers. Thus, all required combustion for heating purposes is performed out of contact with the metallic walls and the gases coming into contact with them are limited to an oxygen content of less than 0.8 to 1.05%; by means of oxygen-recording apparatus the oxygen content of the combustion-products is constantly supervised in order to protect the cell walls. Also there has been no evidence that sulphur compounds in the heating gases have attacked the cell metal.

In the heating-gas system, the recirculating fans for the heating gases are of special construction and the movable parts such as the shaft and fan-blades are formed from special steel which it is claimed has been satisfactory. The construction, manufacture and identity of the employed steels should be determined by investigators. The cells are provided, in the heating system, with guide-plates (Führungsblechen) adapted so to direct the hot gases that the lower wider portions of the single cells with their larger content of coal are heated to a higher temperature than the upper portion but in accordance with the vertical taper of the cell to produce uniform heating from the top to the bottom thereof and a uniform carbonization throughout the cells. Cooler combustion-products that are outlet to the heating spaces are displaced from the heating system in a quantity equivalent to the volume of fresh-combustion products that are added for reheating purposes. The hot purged gases still have a considerable heat content that can be employed for a wide variety of purposes.

The low-temperature coke is discharged from the cells as large flat plates of uniformly rugged structure throughout. It was stated that in the drum test 76.5% of the coke was over 40 mm. and produced only a minor amount of breeze on crushing. The coke has 8-10% volatile matter and depending of course on the characteristics of the treated coal a heating value of 8000-8400 kcal/kg. based on the ash-free coke. It is smokeless burning; also depending upon the characteristics of the coal, the coke yield is 75 to 85% by wt. of treated coal. The statement is interesting that, in contrast to high-temperature coking procedure, the best low-temperature coke made in the metal ovens is that prepared from the

larger sized coal rather than from the finely ground.

The following table shows the size composition of gas or gasflammkohle suitable for use in the Krupp-Lurgi cells:-

0 to 1 mm	30 to 40%
1 to 3 mm	38 to 40%
3 to 6 mm	20 to 30%
6 to 10 mm	5%

#### Other Products

The low-temperature tar is rich in oils and is practically free of dust and has a low content of pitch.

The distillation gas has a gross heating value of 7800-8200 Kcal/m<sup>3</sup> and a relatively low hydrogen content. Depending upon the coal, the gas-yield is 70-120 Nm<sup>3</sup>/ton.

The recovered benzine amounts to 0.9-1.3% by wt. of the carbonized coal. It has a high content of aromatics and consequently is good motor fuel of high octane number and after washing by known methods shows good stability in storage.

The ammonia content is so low that no provision is made for its recovery; the gas is merely water-washed for its removal.

The following table gives a comparison between yields obtained in the Krupp-Lurgi metal cells and in high-temperature coke ovens processing one ton of the same dry Ruhr-Gas-Fettkohlen mixture having 27% volatile matter:-

#### Carbonization.

<u>Low-Temperature.</u>	<u>High Temperature</u>
820 kg. Coke (dry)	750 kg. Coke (dry)
90 Nm <sup>3</sup> Gas	320 Nm <sup>3</sup> Gas
8000 Kcal/Nm <sup>3</sup> (gross)	4600 Kcal/Nm <sup>3</sup> (gross)
11 kg. Crude Gasoline	11 kg. ammonium sulphate.
60 kg. Tar usable as heating oil.	9 kg. Crude light oil
	34 kg. tar.

Characteristics of a Typical Spülgas-Process Tar (Cont.)

Carbonization Temperature (Gas entering carbonization zone)	Creosote & Asphalt Contents of Tar. (Excluding Light Oil).
650°C	Creosote Asphalt 14.5% wt. 2.3% wt.
590°C	14.8% wt. 1.6% wt.
495°C	15.2% wt. 1.8% wt.

The low-temperature tar obtained by the carbonization of lignite and bright brown coals is mostly distilled to give fuel oil, Diesel oil and impregnating oils; it is also used as a source for pure phenols. Considerable quantities of the tar are used as raw stock for high pressure hydro-genation by the I.G.Farben methods to produce motor fuel, Diesel oil and lubricating oil.

The low-temperature tar from the Spülgas process can be, by hydrogenation, to the extent of 80% converted into gasoline. It can, at a pressure of 250 atm. and 400-450°C. and with only 10% loss, also be converted into -

Gasoline	20%
Diesel Oil	39%
Lubricating Oil	17.5%
Paraffin	13.5%

The largest Spülgas Plant built by Lurgi has a throughput of 12,500 tons per day of brown coal. The coke from this particular plant is used primarily for the production of steam in an adjacent power plant. The remainder of the coke is gasified to produce water gas and hydrogen which is used for the hydrogenation of the low-temperature tar.

Cold Extraction Methods for Tar Refining.

Experimental work has been done by Lurgi on cold extraction methods for tar. Plants were under construction at Hirschfelde and Altenburg. The process had been applied only to brown coal tar and involved:-

- (1) Extraction of the tar with amyl and butyl acetates to remove phenols;

- (2) Extraction of the 200-350°C. fraction with methyl alcohol giving 50/50 phenols and neutral oil, the latter being a Diesel oil of 35 cetane number and was suitable for blending with Fischer-Tropsch oil.

Dr. Herbert explained that this research work had been done in his laboratory on Mouson Strasse in Frankfurt.

Low-Temperature Carbonization by Indirect Heating.

In association with Friedrich Krupp A.G., Essen, Lurgi developed a new type of oven for distilling at low-temperature static vertically-disposed beds of coking or weakly-coking coals in small or nut sizes, or of briquetted fine coals, by indirect contact with hot combustion gases. This new distillation oven is based on employment of metal-walled cells as the coking chambers. As the basic unit has been used successfully, a group of metallic cells or an oven, each comprising six rectangular-coking spaces that are disposed in alternation with seven similar heating spaces; the structure is thus reminiscent of the well-known high-temperature horizontal coke-oven in its configuration. Each such unit is entirely surrounded by a housing through which is circulated horizontally along the heating spaces for the cells extraneously produced combustion-products that are maintained at an elevated temperature. The fuel to be carbonized is charged into the top of the cells as is common practice in the horizontal coke oven but the formed coke is withdrawn from the bottom thereof by gravity. A charging machine that moves over the tops of the units is provided with a device for stamping the charged fuel; this device can be used, if necessary, to assist in discharge of the coke.

The coking spaces of the metallic cells have each an average width of about 85 mm; they are about 75 mm. wide at the top and 100 mm. at the bottom. In height and length they are respectively 2.1 and 3.1 meters and the six coking spaces comprising an oven unit have coal capacity of 2.75 metric tons.

The ovens are formed of normal heavy cast steel parts that are welded in a special manner (not learned by the investigators). This subject and technology should be further investigated. Some years of operation in large-scale, it is claimed, have occasioned no bending or warping of the cell walls and periodic metallurgical tests made on them have shown that their original structure has



The shafts of the retorts, as above-mentioned, are open at the top and some of the circulated gases from the drying zone are continuously discharged therefrom - they consist principally of combustion products and steam and contain little free oxygen. The retort shafts are operatively divided into three zones and the fuel moves freely from the one to the other. There are no closing devices or moving parts in or between the zones so that the retorts are relatively compact. The gas off-take must be large to minimize the amount of dust carried into the condensing equipment.

The preferred size of carbonization zone has a capacity not in excess of 250-350 tons per day. Larger size installations present problems of distribution of materials and heat so that there may result unequal carbonization throughout the charge. For larger capacities, it is advisable to build several unit retorts having each the above capacity. It has proven feasible to supply at least two retorts from the same combustion apparatus and with the same off-take mains.

In the cases of younger coals which disintegrate on heating, and the formed small-sized coke has only low market value, the fuel is briquetted in, for example, a Krupp ring-roll press; the produced briquette coke is of lump size and is a more desirable product. In the briquetting, after the fuel is ground and dried by contact with hot combustion products at a temperature of 900-1000°C. to a moisture content of 6-10%, it is introduced into the ring-roll press as a powder and briquetted at a pressure of 30,000 to 35,000 lbs./sq.in. These briquettes are then used as the charging stock to the Spilgas retorts. They either retain their shape during the carbonization process or break into a few large pieces. It is claimed that the resultant product is a dense, abrasion-resistant fuel. This method of briquetting is claimed to be more economical than by briquetting after carbonization.

In large plants of the "Spilgas" type, it is customary fractionally to condense the tar. The gases leaving the retorts are first freed of dust and then cooled to about 100°C. and the heavy tar removed by Cottrell precipitators. The gas is next cooled to 20-30°C. and the light-tar fraction condensed and finally the light oil is removed in an oil scrubber. Thereafter, circulating gas is returned to the carbonizing ovens for recirculation through the fuel charge.

Successful operation of Spilgas plants depends upon uniform distribution of heat over the treated fuel, and to effect this result, maintenance of definite particle size and the treatment of a fuel that is either non-caking or only weakly so, are important factors.

The following data are given as characteristic Material and Heat Balances for a Lurgi Spilgas plant operating on brown coal briquettes containing 16% water and 14.8% tar, as determined in the Fischer-Hempel assay.

Input	Lb.	B.T.U.	% Heat
Brown coal Briquettes	1.000	9876.6	99.92
Air for combustion	0.425	8.1	0.08
Total :	1.425	9884.7	100.00

Output.

Semicoke	0.445	5627.5	56.96
Surplus gas *	0.253	676.7	6.84
Refined tar	0.116	2086.9	21.10
Light oil (below 200°C)	0.023	434.0	4.39
Liquor	0.031	2.9	0.03
Residue	0.009	122.2	1.23
Gas loss	0.001	12.4	0.13
Heat in cooling H <sub>2</sub> O		225.9	2.28
Waste gases	0.487	185.3	1.88
Loss	0.010	510.9	5.16
Total:	1.425	9884.7	100.00

\* 7400 cu-ft./ton (30 m.30°F.)

Characteristics of a Typical Spilgas-Process Tar

Carbonization Temperature (Gas entering carbonization zone)	Distillation			
	Excluding Light Oil	Including Light Oil.	Excluding Light Oil	Including Light Oil.
	Up to 180°C.	Up to 320°C.	Up to 225°C.	Up to 325°C.
650°C	1.4% vol.	43.8%	47.4%	52.0%
590°C	1.4% vol.	44.5%	46.5%	46.5%
495°C	1.6% vol.	45.3%	48.1%	48.1%

the expert there being Dr. Ipfelkopfer. Lurgi has had no experience with the use of charcoal or other absorbents for selective fractionation of different types of hydrocarbons.

#### VI. CARBONIZATION.

Lurgi Gesellschaft für Wärmetechnik has developed improvements in the carbonization of fuels employing the "Spülgas" or direct heating principle and, in association with Krupp of Essen, a low-temperature carbonization effected by indirect heating of the fuel in closed metal-walled cells or ovens having each an average width of between 70 to 100 mm. According to Dr. Heine's expressed opinion, the former process is the most satisfactory for briquettes and the latter method and apparatus for the processing of weakly-coking coals to provide coke of rugged structure having a residual content of about 10% volatile matter, or less.

##### A. Spülgas Process.

This process of Lurgi was developed primarily for the low-temperature carbonization of lignite, brown coal, xylite and lump fuels having a water content of 25-30%. Early German brown coals having a water content of 50-55% have been also treated but, since they are crumbly and disintegrate rapidly on heating, they are first briquetted (without a binder) and then carbonized. Oil shales, torbanite and cannel coal can be carbonized with very satisfactory results. Long-flame younger coals can be processed ranging from those that disintegrate to those which cake slightly on heating.

Dr. Heine also explained that the Spülgas process had been applied successfully to bituminous coal. At Blechhammer, the 4,200 tons plant (300 tons per generator per day) had 5 generators operating on slightly-coking coal making hard coke for transport producers and the production of hydrogen. The Maurice index of the coal was 12 and the fines were briquetted with 4% sulphite lye and pitch before carbonizing. The tar (10% yield) was divided into asphalts, acids and "Navy oil" over 230°C. (6/7 per cent). The economics of the process were termed "doubtful", when hard coke sold for 22 RM, Lurgi coke brought 25 RM and producer fuel 28 RM; the cost of the coal was 15 RM. Obsolescence was taken at 2 to 3 years.

In the Lurgi Spülgas process, the employed coal is treated successively to drying, carbonization and cooling steps in a retort of special design, the latter step being so arranged that the heat removed from the cooled coke is returned to the carbonization zone. The above three process steps are carried out in zones that are vertically disposed in respect of each other in the retort which

is open at the top (the hopper) and closed at the bottom by the coke-discharging mechanism. In both the drying zone and the carbonizing zone, the descending flow of fuel is treated successively in direct contact to separate circulation of gases that are appropriately preheated usually by admixture with hot combustion products formed extraneously and thereafter introduced into the circulated gases. Manifestly, there is considerable difference in temperature between the two circulated gas-streams; the circulated gases of the drying zone are usually preheated to about 200-250°C. whereas the chosen temperature of those gases circulated into the carbonization zone depend upon the characteristics of the fuel being carbonized and the temperature at which it is best treated, but the usually employed temperature is within the range of about 495° to 650°C. although in special instances, where it is desired to produce a coke having a volatile-matter of from 3-5%, it is feasible to employ preheating temperature of about 800°C. which increases the hardness of the coke; the tar-yield however is not appreciably affected by the temperature of carbonization. Gases leaving the carbonization zone are passed through condensers, tar-extractor and light-oil scrubbers before their return to the carbonization zone. The greater portion of that recirculated gas recycled into the carbonization zone is first, by special construction of the automatic coke-withdrawing pocket, admitted into contact with the hot coke leaving the carbonization zone for cooling the latter; a small portion of said circulation gas supplies the two combustion chambers whereby those gases, admitted to the carbonization zone and the drying zone, are heated.

In consequence of the above method of heating the circulated gas-streams, the formed carbonization gases are of course diluted with combustion products, but they comprise a relatively minor proportion of the total, and the mixed product, in the case of brown coals, burns quite easily as it has a calorific value of 140-200 BTU per cu-ft. In those special instances where there exists a good market for the distillation gases in undiluted form, heating of the recycled gases is effected by combustion-products in indirect contact in tubular heaters. This modification of the process is of value only for those fuels yielding gases containing little carbon dioxide.

The raw coal being thoroughly dried and preheated before it enters the carbonization zone and, in addition, the recycled gas itself being preheated by the hot coke before it also enters the carbonization zone, the carbonization-heat requirement is thus reduced to a minimum. In result, the amount of combustion-products required adding to the recycled gas for heating purposes is relatively small and the volumes of gas that are required to be handled in the condensing, scrubbing and light-oil recovery apparatus are not substantially greater than those encountered in externally-heated retorts.

The product is dried in a centrifuge sufficiently to permit its extrusion and is further dried on a conveyor belt by a blast of hot air to facilitate cutting up the spaghetti into desired lengths. Final drying is carried out at 100°C.

This catalyst is preferably reduced in the synthesis unit with hydrogen at 250 to 350°C for 1 to 4 hours. The rate of hydrogen flow is one cubic meter per kilogram of catalyst per hour. (The lower the rate of hydrogen the higher the reduction temperature required). Reduction with synthesis gas is possible. Synthesis is started at about 180°C and the temperature is raised to 220°C in two days. Over an operating period of three months the temperature should be raised from 220°C to 230°C. Such a catalyst has not been run to exhaustion by Lurgi but it is believed that its life would be about one year.

The catalyst used for high benzine production as illustrated by Fig. 3 is made by impregnating Luxmasse (as used for rough purification of synthesis gas) with 3% metallic copper. The Luxmasse is wet with a solution of copper ammonium nitrate in suitable proportions to give this final copper content. The resulting catalyst is not highly active and requires a high operating temperature, but at this temperature gives a relatively low boiling product with less catalyst deterioration due to CO cracking than would occur with a more active catalyst. In a somewhat similar manner a more volatile product can be obtained by high temperature operation with a cobalt catalyst containing more than the normal proportion of kieselguhr.

It was stated that the most favorable financial position was achieved by working for the maximum yield of hard wax, which was valued at 45 pf. per kilo. The capital cost for a new plant was stated to be 800-900 Marks per ton of liquid product per year, although a figure of 600 Marks per ton per year had been quoted recently for one new plant. The following typical operating costs were cited for a plant using coke for making synthesis gas:

	Pf. per Kg.		Product
Coke per ton	24 Mk.	Gas	14.
Steam " "	2½ Mk.	Cap. & repairs	6
Power per Kw.Hr.	2 pf.	Labor	2
Labor per hr.	1 Mk.	Steam	2
			<hr/>
			24

Lurgi had the contract for the decentralization of Fischer-Tropsch units under the Geilenberg plan, which called for the operation of a few synthesis ovens in conjunction with each of several scattered city gas plants. It was stated that Lurgi officials protested against this plan but could not dissuade Geilenberg. The plan called for 12 ovens at Leipzig, and 2 or 3 each at Dresden (Heidenau), Goerlitz,

Gottbus, Plauen, Wurtz burg, Fuerth, Erfurt and Chemnitz. All of these plants were to be built for atmospheric pressure operation with a cobalt catalyst and most of the ovens were to be moved from the Rheinpreussen or Wintershall plants. The Leipzig, Dresden, and Wurtzburg installations were 80-90% complete and the others were less than 50% complete at the end of the war.

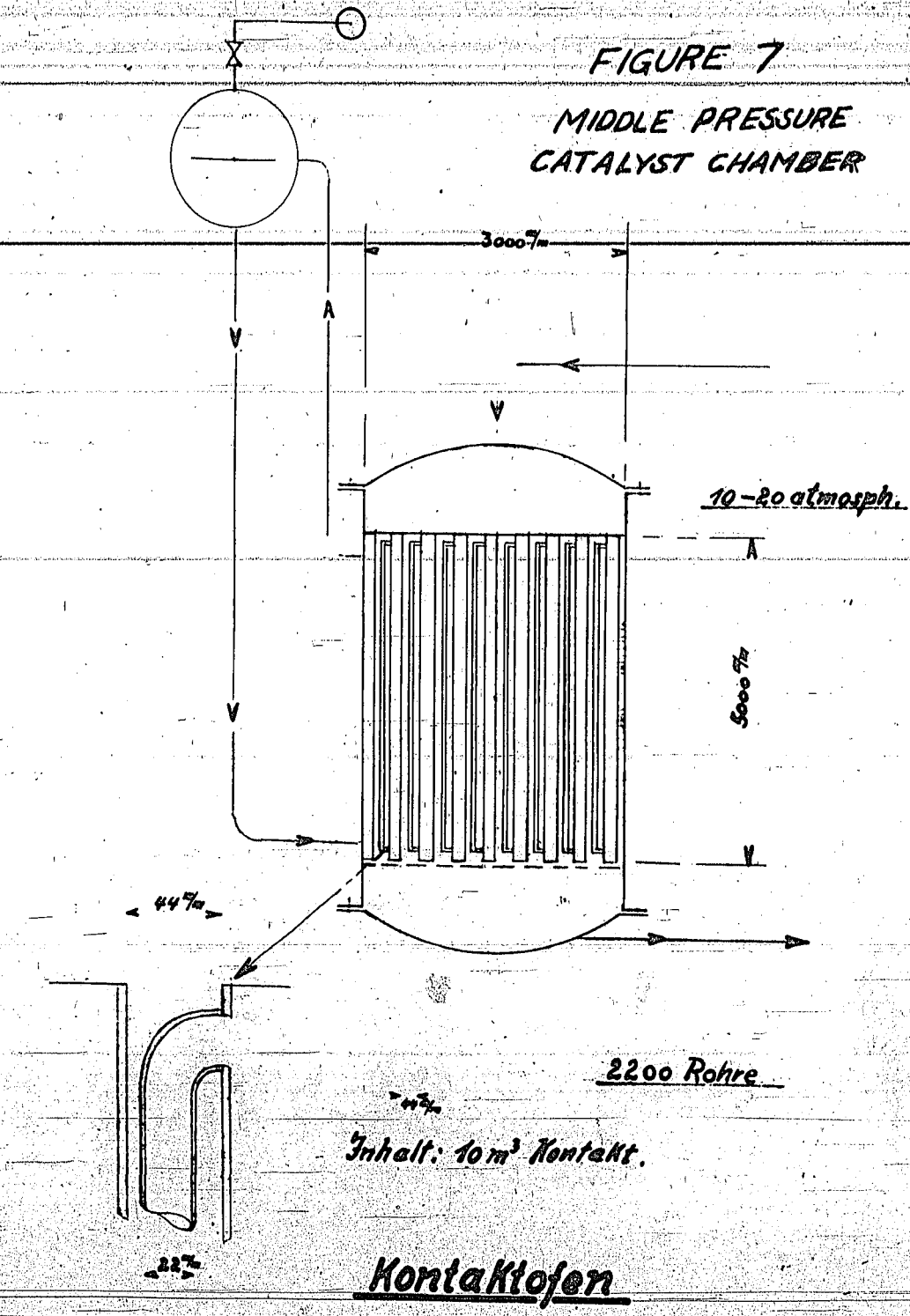
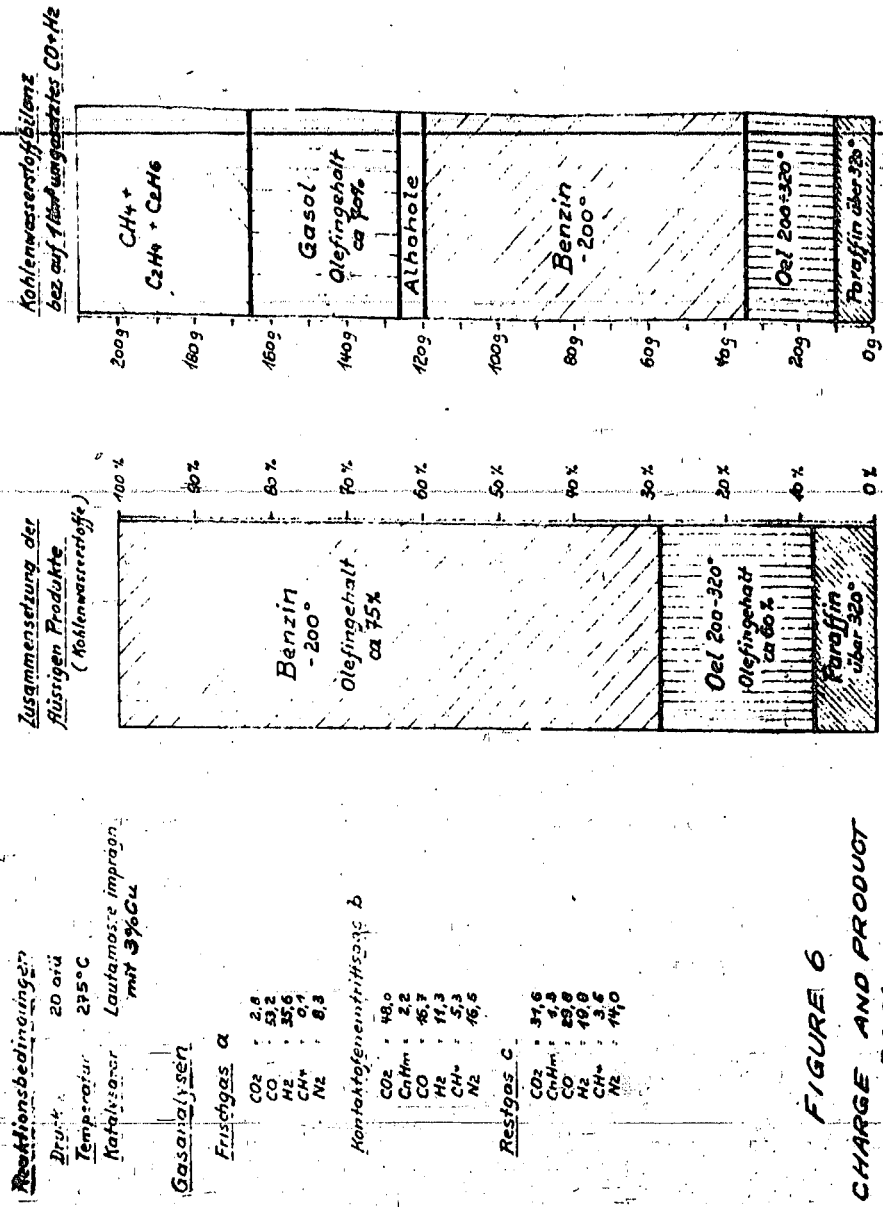
Lurgi has developed a process for de-poisoning city gas, comprising removing the bulk of the carbon monoxide by passage over an iron catalyst to make chiefly methane with some incidental hydrocarbons. This process would normally be used with Lurgi high pressure coal gasification units. The catalyst for this de-poisoning treatment would have the following composition:

100 Fe, 5-10 Cu, 2Al<sub>2</sub>O<sub>3</sub>, 120 Kieselguhr.

Single stage treatment with this catalyst at 20 atmospheres pressure and 250-280°C. is stated to leave only 5% of CO in the gas. A second stage would reduce the CO content to 1% but would probably not be economical. The liquid production amounts to 30-40 grams per cubic meter, of which about half is high melting paraffin. The diesel fuel fraction has a cetane number of 65 which could be raised to 90-100 by hydrogenation. The gasoline fraction contains 50-60% olefins and has an octane number of about 70 which can be raised to about 80 by the Ruhrchemie isomerization process using a non-metallic catalyst at 320°C. An experimental plant of this type located between Boehlen and Pulgar and treating between 1000 and 1500 cubic meters of gas per day from an adjoining Lurgi high pressure generator was inspected by CAFT No.3 on 28 April, 1945.

Lurgi has built the charcoal absorption units for nearly all of the Fischer-Tropsch plants in Germany. They believe that charcoal absorption is definitely more economical than oil absorption up to 10 atmos. pressure and that oil absorption is preferable above 15 atmos. pressure, with the choice between 10 and 15 atmospheres depending on local conditions. The Lurgi built plants used I.G.charcoal which was made from peat at Leverkusen, and is believed to be a little more active than American charcoal. It was stated that there have been no new developments in charcoal manufacture in Germany during the war. The I.G.experts in this field are Drs. Neimann and Doeptke.

A recent advance in charcoal absorption technique has been the design of a plant to work at -50°C, the objective being the separation of methane. Reference was made to similar work by the British Magnesium Corp. at Swansea designed to keep the CO percentage in the gas below 5 per cent. Carbon for the purpose should be highly reactive (S-Kohle or nut shell charcoal). Studies made in conjunction with the Linds Co., indicated costs to be slightly less than for refrigeration. Reference was made to a plant in the gas works at Nuremberg to produce 3000 cubic meters of methane per day (purity 85%),



**Reaktionsbedingungen:**

Druck: 20 atm  
 Temperatur: 230°  
 Katalysator: Au:Fe:250:91:8:0:5  
 2%O 30SiO<sub>2</sub>

**Gasanalyse:**

**Fruchtgas a**

CO<sub>2</sub> = 5.8  
 CO = 37.6  
 H<sub>2</sub> = 48.1  
 CH<sub>4</sub> = 0.1  
 N<sub>2</sub> = 8.4

**Olefinhaltiges Gas b**

CO<sub>2</sub> = 22.9  
 C<sub>2</sub>H<sub>4</sub> = 1.4  
 CO = 26.9  
 H<sub>2</sub> = 34.8  
 CH<sub>4</sub> = 2.0  
 N<sub>2</sub> = 15.0

**Restgas I c**

CO<sub>2</sub> = 28.2  
 C<sub>2</sub>H<sub>4</sub> = 1.9  
 CO = 22.6  
 H<sub>2</sub> = 27.0  
 CH<sub>4</sub> = 2.7  
 N<sub>2</sub> = 17.6

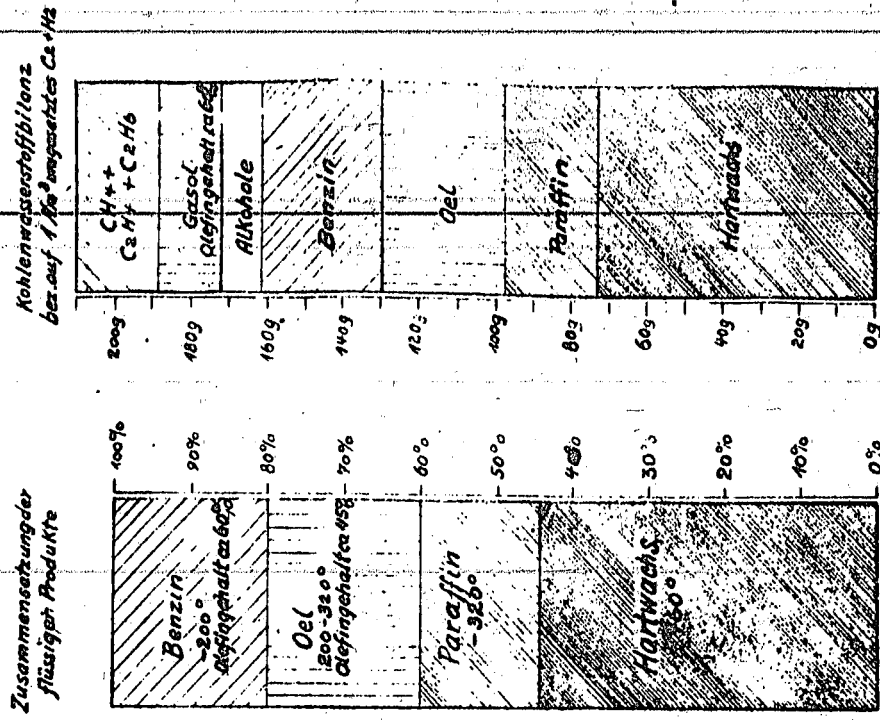
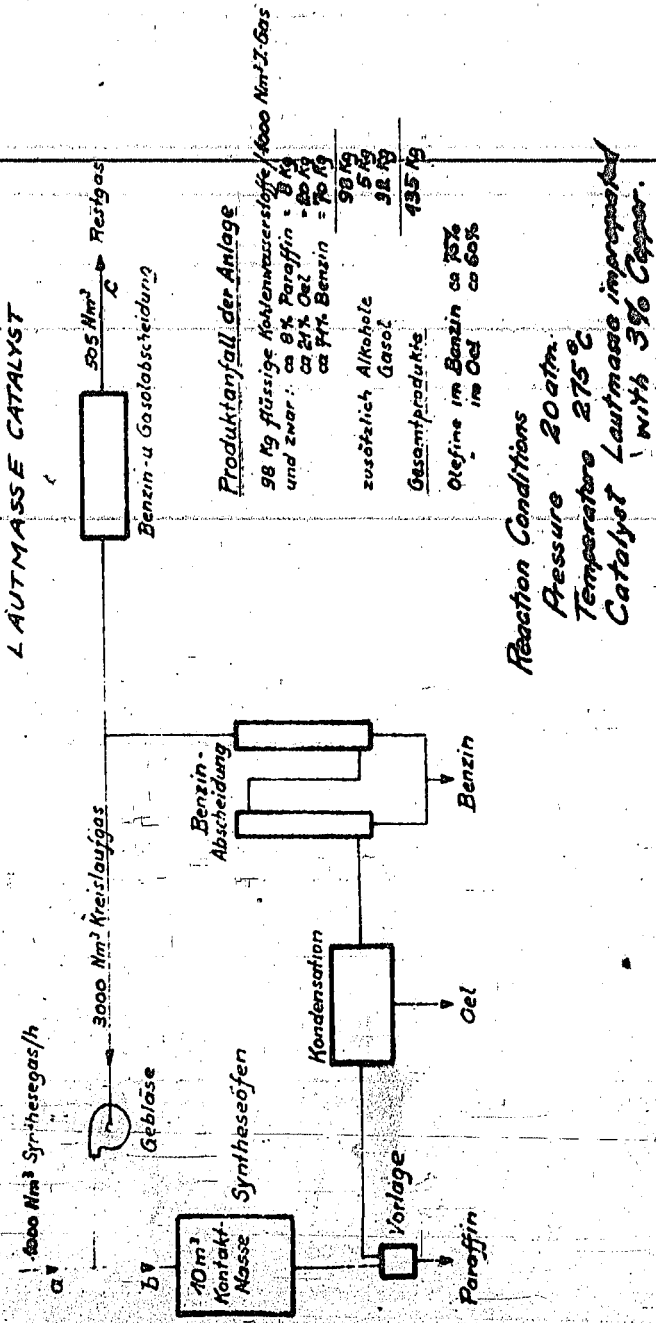


FIGURE 4  
 CHARGE AND PRODUCT  
 DATA  
 PERTAINING TO FIG 3

FIGURE 5  
 RECYCLE SYNTHESIS  
 WITH  
 LAUTMASSE CATALYST



**Reaktionsbedingungen:**

Druck 14-10 atü  
 Temperatur 190-225°C  
 Katalysator 100 Co : 570 Os : 8 Mg O : 200 Mg

**Gasanalyse**

**Frischgas a.**

CO<sub>2</sub> : 10,5  
 CO : 51,4  
 CH<sub>4</sub> : 0,4  
 N<sub>2</sub> : 5,9

**Kontaktgasenrichthtesgas b.**

CO<sub>2</sub> : 19,7  
 CO : 56,1  
 CH<sub>4</sub> : 5,2  
 N<sub>2</sub> : 10,8

**Energies hinter I. Stufe c.**

CO<sub>2</sub> : 23,5  
 CO : 4,0  
 CH<sub>4</sub> : 29,9  
 N<sub>2</sub> : 9,0

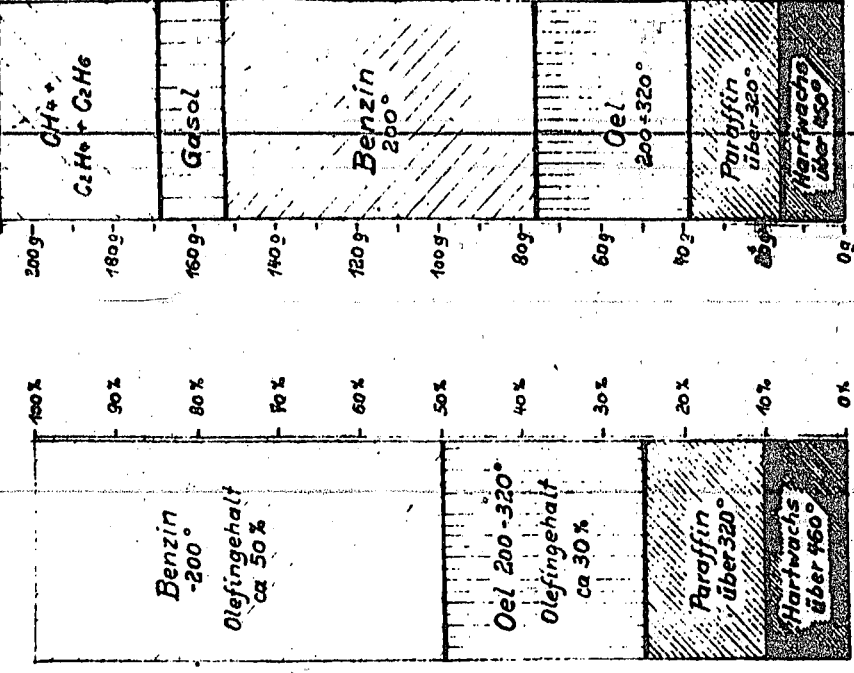
**Energies hinter II. Stufe d.**

CO<sub>2</sub> : 37,4  
 CO : 2,0  
 CH<sub>4</sub> : 22,0  
 N<sub>2</sub> : 13,7

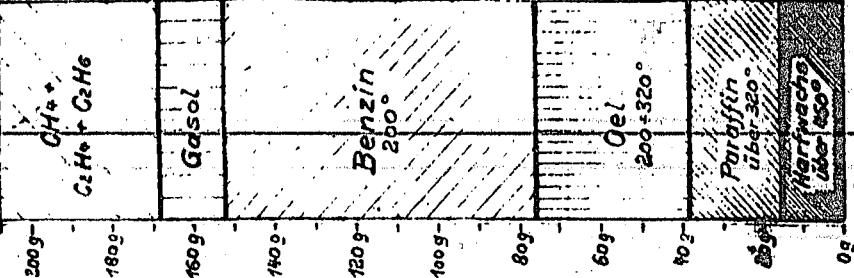
**Restgas hinter II. Stufe e.**

CO<sub>2</sub> : 41,8  
 CO : 0,5  
 CH<sub>4</sub> : 6,3  
 N<sub>2</sub> : 23,5

**Zusammensetzung der flüssigen Produkte**

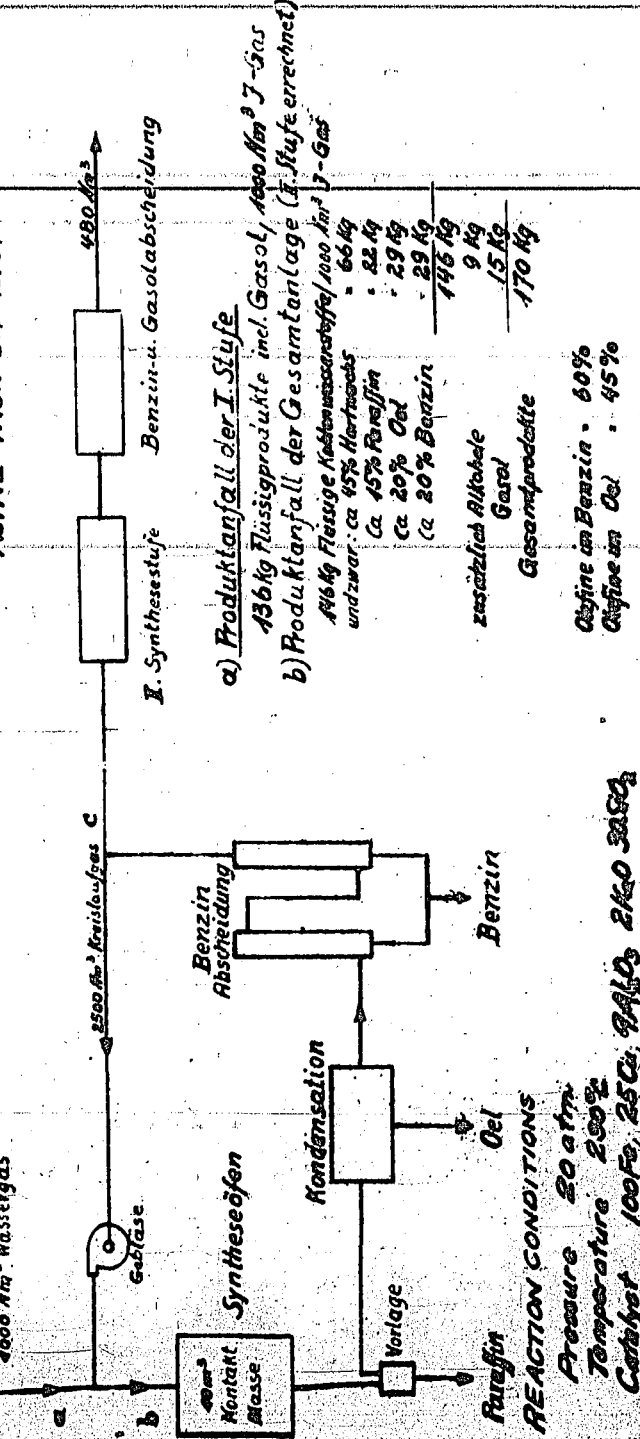


**Kohlenstoffbilanz bez. auf flüchtigstes CO<sub>2</sub>**



**FIGURE 2  
 CHARGE AND PRODUCT DATA  
 PERTAINING TO FIG. 1**

**FIGURE 3  
 RECYCLE SYNTHESIS  
 WITH  
 ACTIVE IRON CATALYST**



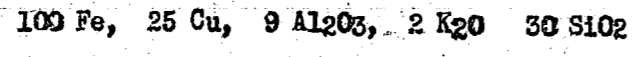
pressure could be controlled to give the desired synthesis temperature. The unit was operated under varied conditions with a Ruhchemie cobalt catalyst for about two years. Single pass operation with this catalyst gave a yield of 155-162 grams of liquid plus "gasol" per cubic meter and the liquid product had the following composition:-

Benzin boiling below 200°C	...	45%
Oil boiling 200/300°C	...	25%
Wax boiling above 320°C	...	30%
(Wax boiling above 460°C)	...	12%

The octane number of the benzin was 40 and the cetane number of the reaction boiling from 120° to 280°C was above 100. With a recycle ratio of about 3:1 it was possible to increase the throughput by 30% without sacrifice of yield in grams per cubic meter or to obtain a yield of 170 grams per cubic meter at the same throughput. With recycle operation charging water gas instead of ideal gas the olefin content of the liquid was 50-80% instead of 20% as normally. The general characteristics of the preferred recycle operation with a cobalt catalyst are shown by Figures 1 and 2, pages 9 and 10. Figures 3, 4, 5, and 6, pages 11, 12, 13 and 14 show the characteristics of recycle operation with two different iron catalysts.

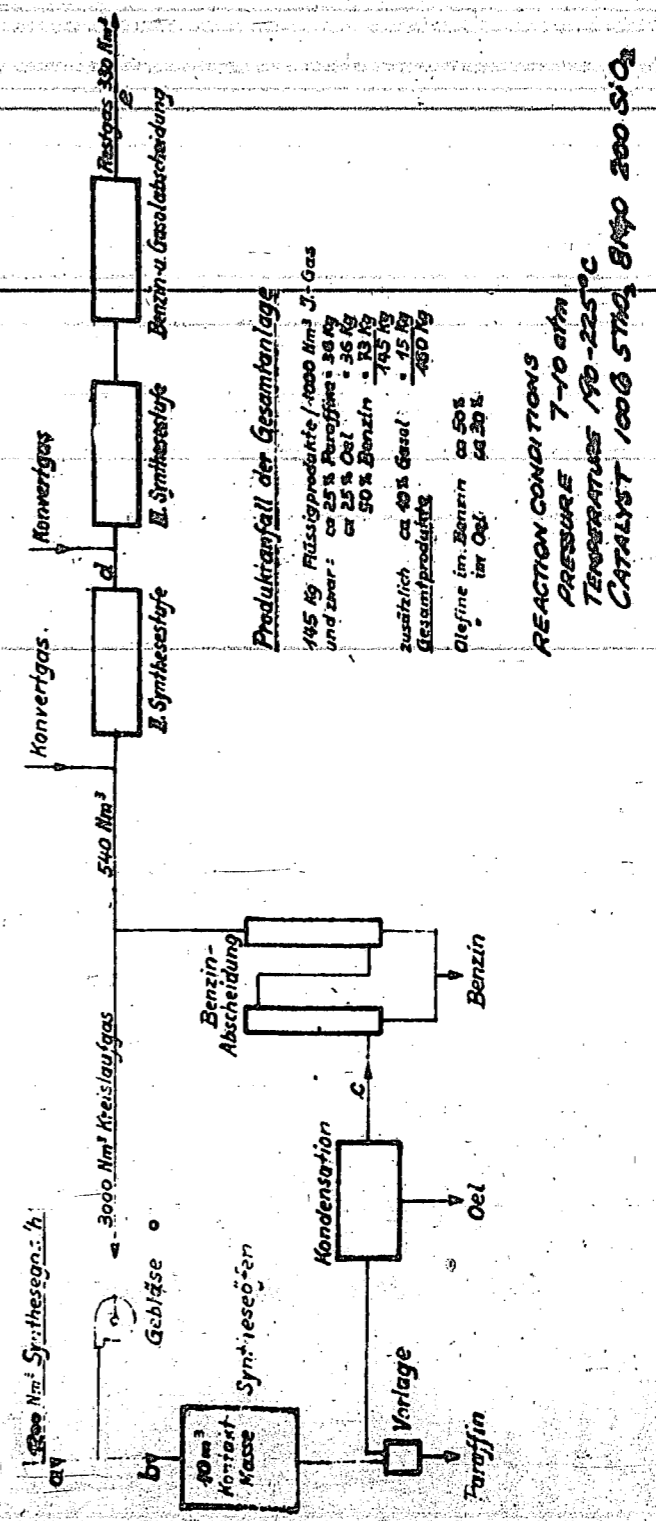
Figure 7, page 15, illustrates the conventional middle pressure reactor preferred for such operations. It was stated that six different engineering firms had collaborated in developing this reactor. Its upper limit of operating temperature is 230 C. and it is doubtful if it can stand much more than the commonly used synthesis pressure of 20 atmos.

Dr. Herbert stated that the best iron catalyst developed by his laboratory has the following composition:



To make this Catalyst, copper nitrate and aluminum nitrate are dissolved in a 10% solution of ferric nitrate in such quantities as will give the specified ratio of metals. The solution is heated nearly to boiling and a 1% solution of sodium carbonate is added rapidly at about 70°C. in the quantity required to precipitate the metals as hydroxides. The kieselguhr is then added rapidly and stirred for about one minute. Lurgi has used kieselguhr No.120 from the Deutsche Kieselguhr Co. The exact nature of this kieselguhr is unknown and no information is available about the suitability of other grades. The kieselguhr-hydroxide mixture is washed with hot water to a p H of 8.0 after which it is washed with a potassium carbonate solution to incorporate the specified quantity of K<sub>2</sub>O. Herbert told Capt. Chaffee that it is important to remove nitrates completely in order to make subsequent reduction temperature control easy and positive.

FIGURE 1  
RECYCLE SYNTHESIS  
WITH  
COBALT CATALYST



#### IV. GERMAN OIL DEVELOPMENTS

Hubmann expressed some views on the German oil position during the war. Since 1942 all efforts have been made to increase the output of low temperature tar from brown coal. The biggest effort had been made in the Sudeten area where the brown coal had a high tar content.

The Brüx plant, which was a Herman Goering project, was capable of producing a million tons of low temperature tar a year. He believed that the actual output prior to the damage from bombing was at the rate of about 600,000 tons per year. The carbonisation ovens were designed for a through-put of 250 tons per day, but actually operated at 350 tons.

The production programme for finished products had to be changed to meet altered circumstances. When the plant started operating the primary product was gasoline. The programme was then altered to provide for a maximum output of diesel oil and then a further change was made to convert the plant to the output of high octane aviation fuel. Just before the war ended, plans were being made to make Brüx the principal producer of jet fuel.

No attempt was made to bring in any feedstocks, either tar or crude oil, from other areas. The low temperature tar from the Leipzig area was unsuitable for Brüx as the tar contained a proportion of arsenic which poisoned the catalyst.

Hubmann expressed the opinion that the petroleum crude oil produced in the latter stages of the war had been extremely expensive, the producing operations being very uneconomical in comparison with pre-war standards.

#### V. FISCHER-TROPSCH

Small scale research on the Fischer-Tropsch process has been conducted at the Lurgi laboratory east of Frankfurt, and pilot plant research has been carried out in the plants of customers, particularly Hoesch Benzin at Dortmund. This research was concentrated on the development of middle pressure recycle operation, particularly with iron catalysts. This type of operation was claimed to have the following attractive characteristics:

1. Use of charge-gas having a ratio of hydrogen to carbon monoxide little if any above 1:1, which is essentially water gas.
2. Easier temperature control due to high gas velocities through catalyst bed.
3. More olefinic product.
4. Formation of less carbon on the catalyst and less methane.

In working along these lines Lurgi appears to have been ahead of Ruhrchemie or more aggressive (or both) and developed a patent position which led to a contract with Ruhrchemie giving Lurgi the exclusive right to build Fischer-Tropsch plants under Ruhrchemie patent licenses in all countries except the United States and Japan. Lurgi's favorable position in the field of high pressure gasification was also a factor in reaching this agreement. Up to the end of the war Lurgi had exercised the rights thus acquired only in the building of city gas conversion units under the Geilenberg plan as discussed later in this report. The pilot plant work at Hoesch Benzin had demonstrated the merits of middle pressure recycle operation so that both Hoesch and Schaffgotsch had given orders for the conversion of their commercial plants to this type of operation. However these conversions had not been accomplished because of bombings of the plants in question and scarcity of necessary materials and equipment.

The Lurgi laboratory had done considerable work on the development of iron catalysts. It was stated that Ruhrchemie, Brabag, Rheinpreussen, and I.G. were also known to have worked on iron catalysts. At the request of some government bureau comparative tests on iron catalysts from different sources were conducted by Ruhland, but the results were inconclusive since different criteria of catalyst quality were used by different companies depending on the products which they desired to make. Details of these Ruhland tests were not available at Frankfurt. The catalyst submitted by I.G. for these tests was believed to be their fused ammonia synthesis catalyst containing some alkali and uranium and was inferior to some others tested. No commercial synthesis plants using an iron catalyst have been built in Germany, but one such plant was designed by Ruhrchemie and Lurgi for erection in Italy. It was stated that Lurgi could produce with the iron catalyst liquid products containing at least 60% wax.

Lurgi has been particularly interested in recycle operation of the Fischer-Tropsch process using a conventional externally cooled middle pressure reactor and a relatively low recycle ratio. This is to be distinguished from the I.G. process developed by Michael, which involves a very high recycle ratio, so that the heat of reaction is carried away by the circulating gas. Dr. Herbert stated that this I.G. process has been operated only on a pilot plant scale and in his opinion the cost of such recycling on a commercial scale would be prohibitive.

The Lurgi low recycle operation was developed in a pilot unit installed at Hoesch Benzin at Dortmund in 1938. This unit consisted of a single tube of the standard middle pressure reactor as built by Gutehoffnungshütte. (See Fig. 7, page 15). The annular layer of catalyst had an inside diam. of 24 mm. and an outside diam. of 44 mm. with an overall length of 5 meters. The tube was water-jacketed with a vapour chamber connected to the top by means of which the steam



Manshu Gosei Nenryo, Chinchow (Kinshu). This plant is also known as Fushin. Hubmann believed that this Fischer-Tropsch plant probably started producing in 1940. It was a standard size Ruhrchemie unit with a capacity of about 30,000 tons per annum. (See Note 4).

Rumoi, Hokkaido. Lurgi had been interested in a high pressure gasification prospect in the vicinity of Rumoi, the intended plant being a high pressure gasification unit employing oxygen. Some information on this subject would be in some files that had been taken by a previous CIOS investigator. Hubmann was doubtful whether there was an oil plant actually in Rumoi and he thought that it was more probable that if any synthetic oil was being produced in this area, the plant or plants would be located more inland and in the vicinity of Fukagawa, Takigawa, and Sunakawa at which three places coal deposits were in the course of being exploited. The Japanese were intending to produce gas for commercial purposes in this area and it was possible that they were also intending to develop steel production.

Chosen Sekitan K.K., Eian. In 1930 there was a small low temperature carbonisation plant at Eian which was processing about 600 tons of coal per day. This plant yielded from 15,000 to 20,000 tons per annum of paraffinous brown coal tar. Hubmann thought it was probable that this tar was hydrogenated elsewhere, possibly at Kirin or Fushun, but he had no information on this subject.

Chosen Chiasso Hiryo K.K., Agochi. Informant knew of no hydrogenation plant at Agochi.

South Sakhalin Mining and Railway Company, Naihoro. The plant here was originally a low temperature carbonization plant, having four ovens. It was probable that the Japanese intended to increase production from this area and Hubmann suspected it was for this purpose that the Japanese government took over the Lurgi license rights. The coal at the south end of the Karafuto Peninsula is an older brown coal with a high content of paraffinic tar (about 15%), the water content being only 15%. It is probable that the Japanese have been making plans for the hydrogenation of this tar.

Note:

4. Air cover shows no extension to the original plant, capacity of which is provisionally estimated at 35,000 tons. Serial J/007 para (6) (a) iii gives estimated production 100,000 tons.

Nissan Ekital Nenryo K.K., Wakamatsu. Bruggemann was responsible for starting one LTO oven at Wakamatsu in 1934 at the site of a coal mine that had been started at this place. Hubmann did not believe that any representative of the company had been to Wakamatsu since that date. The bituminous coal which yields a coke that is valuable for domestic purposes, has a content of 8-9% of low temperature tar. Nothing was known about a hydrogenation plant in this vicinity.

Ube Yuka Kogyo K.K. (No. 2), Ube. The Koppers company had been responsible for a low temperature carbonisation plant at Ube which operated in conjunction with a synthetic ammonia plant. Tar production was believed to be small and Hubmann, who knew of no synthetic oil plant at Ube, considered that tar production from this place was not likely to be important.

On the other hand Hubmann believed that the Japanese were planning to build a hydrogenation plant to treat the whole of the tar production of South Honshu and Kyushu. He considered that the most probable site for such a plant would be at Tokuyama or, failing which, at Ube. Hubmann doubted whether this project had been completed although he could not give any specific reasons for this doubt. (See Note 5).

Manshu Yuka Kogyo K.K., Sawingsai. Hubmann knew nothing about a plant in this vicinity. Likewise, he knew of no other plants.

Hubmann considered that it was probable that the Japanese would have taken steps to develop low temperature carbonisation plants in the Tientsin and Peking areas where there were deposits of bituminous coking coal. It was to be expected that the Japanese would at least attempt to develop the bituminous coal available at Kalgari, West of Peking.

Although Hubmann's information upon Japanese synthetic oil operations was scanty, he was strongly of the opinion that the extent of Japanese achievement was likely to be very much less than published figures had indicated. He stated that it was customary for the Japanese to talk very big about their oil developments but in most cases these statements could be discounted by at least 50%.

Note:

5. Serial J/007 6 (c) reports the existence of a hydrogenation plant with a capacity of 100,000 tons p.a. Air cover indicates a possible hydrogenation plant which, however, appears to be still in the course of construction.

other technical Commissions had visited the Lurgi officials as follows:

2 and 3 April, 1945	A party of about 12, headed by Commander Abbott.
7 April	A party of unidentified investigators who discussed synthetic fuels and carbonization in general.
13 and 14 April	A British Commission under Lt. Col. Hollings, who discussed synthetic fuels and town gas.
14 and 15 April	A party of 3 American Officers, all stated to be affiliated with Standard Oil in Chicago, and 1 working in Oklahoma.

#### II. OWNERSHIP OF METALLGESELLSCHAFT-LURGI

The Lurgi Companies are entirely owned by the Metallgesellschaft A.G. of Frankfurt am Main, and the latter is a stock company with ownership of shares distributed about as follows:-

50% by private individuals.  
15% by De Gussa of Frankfurt/Main and Henkel of Düsseldorf.  
20% by Amalgamated Metal Co., of Great Britain.  
6 to 7% by I.G. Farbenindustrie.

#### III. JAPANESE RELATIONSHIPS.

Dr. Otto Hubmann, the director in charge of fuel research, provided information upon business dealings with Japan. Dr. Hubmann stated that the documentary material in Frankfurt on these dealings had been handed over to previous CIOS investigators.\*

Dr. Hubmann was in Japan from May to July, 1939. He stated that little information was obtained by him during this trip outside the projects on which he was engaged, the Japanese being extremely secretive about their plans. He had no information on developments after Pearl Harbor and their representative in Tokio, Dr. C. Kraye, had not been at all informative; he was of the opinion that Dr. Kraye was now in semi-confinement in Japan. Hubmann was not clear as to the present status of Nippon Lurgi Goshi K.K., which was the Lurgi office in Tokio.

\* In this connection refer to Japanese Document Report No. CD/037 relating to SHAEF G-2 (Japanese Section) Report Serial J/007 of 24th April 1945.

#### PATENT RIGHTS.

At the beginning of 1942 the Japanese acquired all the low temperature carbonization patents of Lurgi for Japan, Manchuria and North China. The agreement gave the Japanese the right to construct plants and an exclusive utilization of patents. A flat payment of approximately 800,000 RM, was received from the Japanese, this sum being cleared through the German government.

#### JAPANESE PLANTS.

Hubmann provided the following information upon plants in the Far East. His knowledge of the subject was fragmentary and better information should be obtainable from Bräggemann who was engaged in the erection of low temperature carbonization plants in Japan until 1942. Bräggemann was not in Frankfurt and he was believed to be somewhere in Central Germany, possibly in the vicinity of Böhlen.

Shale Plant at Fushun, Manchuria. Hubmann did not know what progress had been made with this plant since 1939. He believed that the Shale plant was probably capable at that time of producing about 200,000 tons of raw oil. He thought it probable that the Navy had a small trial hydrogenation plant at Fushun for the production of diesel oil and gasoline and with a capacity of not more than 10,000 tons per annum. He did not know of any aluminium plant in the Fushun area but he believed that the Japanese were intending to put up such a plant somewhere between Mukden and Tientsin on the Chinese side of the border.

Mitsui Kozan K.K., Mike (Omuta). Hubmann believed that this plant got off to a satisfactory start in 1939. (See Note 1.) He knew that coke production and the manufacture of water gas was proceeding satisfactorily in the summer of 1939. The coke ovens were built by Koppers and possibly also the water gas producers. (See Note 2.) Lurgi had installed an activated carbon plant to operate with the Fischer-Tropsch plant, the latter having, he thought, a capacity of 30,000 tons. He saw no reason why the Japanese should encounter any particular difficulties in the production of Fischer-Tropsch fuels, especially as the plant had a good gas purification system. He considered it probable that the Mike plant had been extended since 1939. (See Note 3.)

#### Notes:

1. Reports generally agree that this plant was put into operation late 1939 or early 1940.
2. 5 Koppers Water Gas Generators are known to have been ordered in February, 1939. Capacity of these was 37,500 cbm/hr. synthesis gas, representing about 35,000 t.p.a. product.
3. Air cover reveals that no extensions have been made. The information in SHAEF G-2 (Japanese Section) Report Serial J/007 para 6 (b) giving production at 100,000 tons would, therefore, appear to be inaccurate.

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Personnel of Teams.

Capt. C.C.Chaffee, U.S.Army, Ordnance.	
Lt. Col. Oliver F.Thompson	} British Ministry of Fuel and Power
Maj. J.G.King	
" H.Bardgett	
" E.Spivey	
Mr.Harold V.Atwell, U.S.Pet. Adm. for War	
Irvin H.Jones, U.S.Bureau of Mines.	

1. INVESTIGATION PROCEDURE.

On 5 April 1945, Capt.C.C.Chaffee of CAFT No.3, 12th Army Group Headquarters interrogated Messrs. Lumme, Oetkin, Hubmann, Herbert, Meyer, Danulat, and Kohrt of Metallgesellschaft and Lurgi in Frankfurt. In addition to obtaining considerable general information personally, Capt. Chaffee arranged for the preparation of flow diagrams and memoranda descriptive of Lurgi's war-time activities of interest to the oil industry.

On 1 May, Lt.Col. Oliver Thompson and Mr.H.V.Atwell interviewed H.G.Heine, head of the Metallgesellschaft patent department and arranged with him for a meeting with other officials on 3 May. With Mr.Heine an inspection of the patent files at Kronberg Castle and Kronberg Hof was also made on 1 May. On 3 May at Lurgi Haus, Gervinusstrasse 17, Frankfurt, Lt.Col. Thompson, Mr.I.H.Jones and Mr. H.V.Atwell interrogated Messrs.Oetkin, Danulat, Herbert, Hubmann, and Ruepping. On 9 May, Mr.Atwell again interrogated Dr.Herbert at Lurgi Haus.

On 5 and 6 May, Major J.G.King, H.Bardgett and E.Spivey interrogated Messrs. Oetkin, Danulat, Herbert, Ruepping, Heine and Siebert at Lurgi Haus. They also visited Dr.Herbert's research laboratories at Mouson Strasse and the evacuation premises at the Technische Hochschule at Friedburg where Dr.Siebert was conducting research on distillation problems and where Dr.Danulat's section had been working before being bombed out.

The information obtained at all of these Conferences is consolidated in this report.

For the meeting on 3 May, Mr.Oetkin had prepared at our request statements of the organization and activities of Metallgesellschaft and its Lurgi subsidiaries, copies of which are attached as Appendix A.

In the conference of 3rd May, Dr.Oetkin gave a general picture of their Fischer-Tropsch and coal gasification activities. Afterward Lt.Col. Thompson discussed Japanese relationships with Dr.Hubmann. Mr. Jones discussed carbonization and gasification with Danulat, and Mr.Atwell discussed Fischer-Tropsch activities with Herbert. It was stated that

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METALLGESELLSCHAFT-LURGI FRANKFURT AM MAIN  
GERMANY

Reported by

Captain C. C. CHAFFEE, U.S. Army Ordnance  
Lt. Col. OLIVER F. THOMPSON, Min of Fuel & Power  
Major J. G. KING, Min. of Fuel & Power  
Mr. HAROLD V. ATWELL, U.S. Pet. Adm. for War  
IRVIN H. JONES, U.S. Bureau of Mines

on behalf of

British Ministry of Fuel and Power

and

U.S. Technical Industrial Intelligence Committee

September 14, 1945

Date of Trip: April & May 1945

CIOS Target Nos. 30/6.06 & 30/6.08

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

RESTRICTED

54p. diagrs.

ITEM NO. 30.

COPY NO. ~~1~~

FILE NO. XXXI-23.

PB-12624

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

RESTRICTED

**METALLGESELLSCHAFT-LURGI  
FRANKFURT AM MAIN**

*Chaffee*

RESTRICTED

**COMBINED INTELLIGENCE OBJECTIVES  
SUB-COMMITTEE**

f. All-Purpose Grease

Composition	70% Oil, 50% Calcium Stearate
Water Content	0.5 - 1.0%
Consistency at 50°C	0.5 - 0.8 kg.
Ash	0.8 - 1.5%
pH Value	Must be slightly alkaline
*Water Resistance	Satisfactory
**Bleeding Test, Wt. % Loss	0.5 Max.

Water Resistance Test:

\*Grease is placed in cylindrical sieve which is rotated in a beaker of water at 50°C for 24 hours. After the end of the test the water and the grease are examined for signs of decomposition of the grease.

\*\*Bleeding Test:

The grease is placed in a sintered bottom glass crucible and kept in oven for 24 hours at 75°C. The crucible is weighed to determine the loss of oil. The loss must not exceed 0.5% by weight.

SPECIFICATIONS ON GERMAN WEHRMACHT FUELS AND LUBRICANTS

It was stated that the German Army did not have numbered specifications similar to those used by the Air Force. Herr Schmidt supplied the following specification points, which were in effect at the WIFO, from memory; however, it is considered that they are substantially accurate.

a. Army Delivery Motor Fuel

Density at 20°C.	0.6 Max.
Vapor Pressure, Reid	0.6 atm., Max.
Calorific Value	9000.0 Kilo Cal/gr., Min.
Octane Number	72.0 Min.
Gum	10.0 Mg/100 cc Max. Glass Dish

Distillation:

Init. B.P.	20°C. Min.
at 75°C.	25% evap., Max.
at 100°C.	30% evap., Min.
E. P.	220°C. Max.

Benzol content	20% Min.
Sulphur	.02% Max.
Freezing point (Mar. to Oct.)	-40°C. Max.

b. Benzol Delivery Specification

Sulphur	0.03% Max.
Gum content	10.00 mg/100 cc. Max.
Flash Point	0°C. Max.
Distillation (Kraemer-Spilker) Init B.P.	77°C.
End Point.	200°C.
% Distilled up to 135°C.	95% Max.

c. Diesel fuel (Automotive grade)

Density at 20°C	0.92 Max.
Flash Point (Pensky-Martin)	21°C. Min.
Octane No.	9500 Kilo Cal/gr., Min.
Sulphur	0.2% Max.
*Corrosion Test Loss	10. mg. Max.

\*Corrosion test run on copper and Zinc specimens, 10 cm. long, 1 mm. thick and 1 cm. wide. When exposed to 25 cc. of oil at 75°C. for 3 hours, the maximum loss must not exceed 10 mg.

d. Automotive Lubricating Oil Summer Winter

Density at 20°C.	0.95 Max.	-
vis. at 50°C.	8.0 - 9.0° E.	4.0° E.
vis. at 80°C.	2.5 - 2.8° E.	ca. 1.8-1.9° E.
Pole Height	2.1 max.	2.1 Max.
Flash	250°C.	-
Pour Point	-36 to -40°C.	-40°C.
Carbon Residue	0.2 - 0.3%	-
Noack Test (1 hr., 20 mm. Vac. at 120°C)	12 - 14% Loss	12 - 14% Loss

e. Gear Oils Summer Winter

Density at 20°C.	0.88- .93	Same
Vis. at 50°C.	8 - 9° E.	5.0 - 6.5° E.
Vis. at 80°C.	2.7 - 2.9° E.	-
Pole Height	1.9 - 2	Same
Flash Point	225 - 250°C.	190 - 200°C.
Pour Point	18 to 20°C.	-45°C.
Noack Test (1 hr., 20 mm. Vac. at 200°C)	9 - 13% Loss/Max.	Same
Sap. No.	2.8 - 3.1	Same
Neut. No.	0.8 - 1.1	Same
Carbon Residue	0.25 - 0.4%	Same
*Corrosion Test	Perfect	Same
Fatty Oil Content	-	5% Min.

\*Corrosion Test run on copper and Zinc specimens of dimensions as in Diesel Fuel Test, for 24 hours at 75°C. The visual test inspection of specimens must show perfect.

c. Products or Materials in Storage.

There was a large stock of empty Jerry cans and drums on the premises. The gasoline storage and blending tanks were destroyed or empty. There was a fair supply of gear oil (Betriebsöl Wehrmacht SE), of motor oils, mainly Nachlaufschrnierstoff T 42 in drums (reported removed from Nordhausen when the V Factory was located there) Motorenöl in summer and winter grades, Mekanol, Glistine (Glycerol substitute), and all purpose grease. There was no diesel fuel, aviation gasoline, or special oils, and a careful search failed to reveal the presence of any additive materials.

Samples of all or the above materials were obtained for examination and full-scale tests, if desired, by agencies in the United States and Great Britain.

INVESTIGATION OF THE TARGET

Although the administrative building had been stripped of all documents and the Manager's office had been partially destroyed, interrogation of Herr Johannes Friede, Manager (party member) and Herr K. Schmidt, Head of the laboratory, provided the following useful information:

GENERAL INFORMATION

Herr Friede, Dipl. Eng., previously a design engineer with an aero company (Arada), came with the WIFO by answering an ad, not because of party membership, in 1936. His first assignment was to help choose the layout, design the tanks and RR siding, then instal the canning equipment. He said that this was the first of the smaller WIFO's and that the first oil was received in July 1938.

Herr Friede stated that the WIFO was definitely first organized as a construction company and that Barben was the first plant built, which was in either 1934 or 1935.

The products turn-over in the WIFO was always large and hence they had experienced little or no trouble with products going off test in storage. The WIFO had never been called upon to utilize alcohol in any fuel blends. Benzol of good quality, low gum and sulphur content was used in 25% blends early in the war; this was later dropped to 20% minimum due to a benzol shortage.

The Motor fuel base handled was mainly from Fischer-Tropsch synthesis and rarely from natural sources, in which cases the source was Rumanian. The base gasoline octane varied from 53 - 62 and with the benzol used required the use of .02 - .04% TEL to make the required octane, which was 78 at first, then 74 and finally 72 octane Number.

The WIFO rarely made any Diesel-oil blends and handled no Jet-Plane Fuels. Until 1941 they got lubricating oil direct from Ruhr Chemie and not from Main Depots.

It was stated that no records of personnel had been evacuated to this area and it was confirmed that the tech records are in the underground WIFO at Stassfurt and that Herr Wehling (General Manager of WIFO's) is at Oberhausen, near Neuberg, Bavaria.

Although motor-fuel stability had never been a serious problem, equipment had recently been received to make such tests. Herr Schmidt stated that the test was to require a 10 mg./100 cc max. test on gasoline after storage at 100°C. for 10 hours in a bomb under 15 atm. air pressure. This was stated to be the method adopted at Derben for testing aviation gasolines in 1939. The report on this oxidation stability and gum test is subject to confirmation.

They had no difficulty from Sulphur, even from benzol, since the Fischer-Tropsch material handled are practically sulphur-free.



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Personnel of Team

Maj. D. A. HOWES, British Ministry of Fuel  
and Power  
Dr. H. SCHINDLER, U.S. Petroleum Administration  
for War  
Capt. C. C. CHAFFEE, U.S. Ord. Dept.

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WIRTSCHAFTLICHE FORSCHUNGSGESELLSCHAFT m.b.H. (WIFO)  
Eferbachtel (3 km. S. of Heiligenstadt, Germany)

INTRODUCTION

a.) This is one of the so-called smaller ARMY COMMAND DEPOTS, "WIFO" installations, blending, testing, and packaging Fuels and Lubricants for the German Army, and was visited because the larger MAIN DEPOT at STASSFURT, reported to contain a larger variety of special materials and the evacuated Technical Records of the Central WIFO office, was not available for investigation at the time.

DESCRIPTION OF THE TARGET

a.) The WIFO which was located in a heavily wooded area with the equipment well dispersed, operated until Easter, 1945, and had not been noticeably damaged until the last three months. However, four of the 12 storage tanks and some facilities had been damaged by aerial bombing and the laboratory had been wrecked by an explosive charge met by the Germans before leaving.

b.) The installation contained the following equipment and products:

- One office building (approximately 60 in office staff).
- One laboratory equipped with gasoline and Diesel test engines and normal petroleum-testing apparatus.
- Gasoline blending plant (with TEL blending).
- Lubricating Oil blending plant.
- Can-reconditioning, washing and handling plant.
- Packing plant for oils, greases and Blitz-can filling.
- Barrel reconditioning plant.
- Underground power plant.
- Four storage buildings for finished packaged products.
- Miscellaneous storage for filled and empty drums.
- Miscellaneous buildings for the 300 workmen, workshops, etc.
- Train and truck loading racks.

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WIRTSCHAFTLICHE FORSCHUNGSGESELLSCHAFT m.b.H. (WIFO)  
FUEL BLENDING STATION

Heiligenstadt, Germany

1 July 1945

REPORTED BY

Capt. C.C. CHAFFEE, U.S.

H. SCHINDLER, U.S.

Maj. D.A. HOWES, Br.

on behalf of

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

CIOS Target No. 30/78

Fuels and Lubricants

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

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IVES

ITEM No. 30

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Classification Cancelled,  
by authority of  
The Joint Chiefs of Staff,  
by Col. E. V. Grinn.

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WIRTSCHAFTLICHE FORSCHUNGS

G.M.B.H. FUEL BLENDING

STATION,

HEILIGENSTADT.

*Chaffee, C.E. (2)*

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COMBINED INTELLIGENCE OBJECTIVES

SUB-COMMITTEE

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 Alkalisierung des Esters wird die Bisulfitlösung zugegeben. 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 Zähler (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 ausgefüllt. Von jeder Partie wird sofort die Oberflächenspannung, der pH-Wert und der Gehalt an flüchtigen Bestandteilen bestimmt (Probe 1). Der obige Sammelansatz Ester ergibt fünf Sulfittierungsansätze zu je 1100 Liter dazu noch einen sechsten Ansatz zu etwa 800 Liter (immer gemeinsam im Emailliekessel). 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 Sulfittierung 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 Dismulgen.

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 Dismulgen lieferten.

Proben:

1.) Von jeder Sendung Athylhexanol 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 Uberschuss von 5% über Theorie genommen.

LIST OF COMPLETED PROJECTS

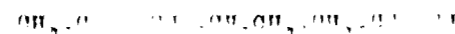
INSTRUCTIONS FOR MAKING AND TESTING PRODUCTS

Process for preparation of Huxetol (V)	4 Feb. 1944
Kaurit MKF	Sep. 1945
Instructions for making Kaurit MKF	11 Jan. 1944
Process for making pure Hydroflavin	18 July 1938
Process for making Thiodiphenylamine C	4 Oct. 1944
Process for making Dismulgen IV	24 Apr. 1941
Instructions for making Dismulgen 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.	

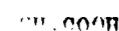
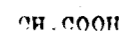
Mainkur, den 25. Oktober 1944

Vorschrift zur Fabrikation von Dismulgan VII

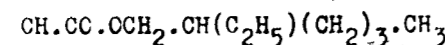
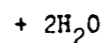
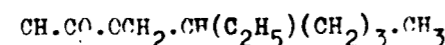
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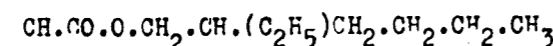
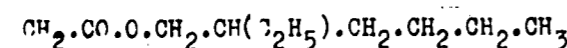
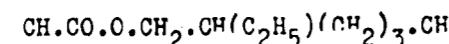
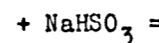
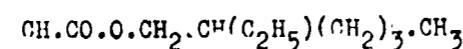
Mischung



Mol. 116



Mol. 340



$\text{SO}_3\text{Na}$  Mol. 444

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

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Einsatz:

4307 kg Äthylhexanol 100%

1830 " Maleinsäure 100%

75 " p-Toluolsulfosäure 50%ige Paste

4330 " Spiritus 95%ig

2194 " Bisulfit Pulver 100% - 2250 kg affektiv

190 " Natronlauge 33° B $\bar{e}$

Ausbeute:

Die Ausbeute

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 Maleinsäure 100% und 75 kg p-Toluol-sulfosäure Paste (etwa 50%ige Paste) 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 mit überdestilliertes 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 Schaugläsern der Kühl-schlangen 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 Emailekessel). Dazu laufen aus einem Maßgefäß (Nr. 5) 750 kg Spiritus 95%ig. Unter Rühren wird der Ester durch vorsichtige Zugabe von Lauge

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-3I-

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 1-2 Stunden bei dieser Temperatur stehen gelassen, die untere Schicht in den Kanal abgezogen. Unter Rühren gibt man dann nochmal 200 l 50%ige Schwefelsäure zu, rührt kurz und lässt 6 Stunden stehen. Nachdem nun die untere Brühe wieder abgelassen ist, läuft der klare Äthylanilid in den Sulfierkessel (Pos. 6) ein. 50 g Natriumhydroxyd werden zugegeben.

#### Sulfierung:

Der Inhalt des Sulfierkessels wird unter Rühren abgekühlt und nach 1-2 Stunden bei 0° mit 50 kg Natriumhydroxyd (Pos. 5b) zugegeben. Die Temperatur darf nicht unter 0° sinken, die Temperatur mindestens 15°.

Nach Beendigung der Sulfierung wird dieselbe sofort langsam in den homogen verbleiten Spitzkessel von 700 l gedrückt (Pos. 6) in den 3000 l Wasser und ca. 30-400 kg Eis vorgelegt sind. Die Temperatur des vorgelegten Wassers muss 0° betragen, während des Eindrückens der Sulfierung wird noch mit Kälteauge 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 Lösung des Äthylanilids wird dann über die Vorlage Pos. 7 in den Destillationskessel (Pos. 8) gedrückt.

#### Neutralisation und Wiedergewinnung des Trichloräthylens:

In dem ausgesteineten 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 phenolphthaleinalkalisch reagieren. Dann wird langsam angeheizt und das Trichloräthyl abdestilliert. Bei 78° Innentemperatur beginnt das Gemisch von Wasser und Trichloräthyl zu übergehen, bei 85° geht die Hauptmenge Trichloräthyl über und bei 98-100° ist die Destillation beendet. Während der Destillation werden Proben genommen, die dennoch 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

(phenolphthaleinrot) 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 Trichloräthyl wiedergewonnen, die zur Entfernung des Wassers 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, Feuchtigkeitsgehalt 35%, siehe Probe 5 von Humitol OX). Die Farbe ist hellbraun. Netzwert 1,0-1,20 Sek. Netzleitfähigkeit 2,0-2,5.

Anzahl: 1000 kg

Ein gusseiserner Doppelkessel mit Rührer, Dampfbad, isolierte Doppelrohrdruckleitung nach Kessel 1). In die Doppelrohrleitung ist eine Lauge zur Beobachtung des Schwefelsäure- und des Sättigungs mit dickerem Öl.

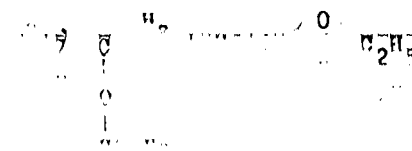
Früher war es so.

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Mainkur, den 24. April 1941.

Verfahren zur Herstellung von  
Dismulgan IV

1. In den Sulfonator des Ölwerkstoff



Verfahren:

1.) Olsäurechlorid:

In den zuvor ausgeleiteten Doppelkessel von 1000 l Inhalt (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 verbleibenden 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. 65 kg). Im Scheider befindet sich das Olsäurechlorid.

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

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

2.) Olsäureäthylanilid:

In dem gusseisernen Doppelkessel von 2000 l (Pos. 3) werden 300 kg Lauge, 200 l Wasser und 214 kg Monoäthylanilin vorgelegt. Bei 20° läuft das Olsäurechlorid aus dem Scheider (Pos. 2) unter Rühren während 8-10 Stunden zu, wobei dauernd mit Kälte-lauge gekühlt wird. Nachdem alles eingelaufen ist, wird auf 50° angewärmt, 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

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Mainkur, den 4. Oktober 1934.

Vorgang zur Herstellung  
Thiodiphenylamin S.

Chemischer Vorgang:

Diphenylamin und Schwefel werden unter Zusatz des katalytischen  $AlCl_3$  bei ca.  $170-180^\circ$  zur Reaktion gebracht. Der frei-

III. Einsatz:

1700 kg Diphenylamin  
646 kg Schwefel  
20-25 " Aluminiumchlorid

IV. Ausbeute: 2000 kg Diphenylamin

V. Arbeitsweise:

1700 kg Diphenylamin werden aus dem 20 obm Lagerkessel über den Wiegekessel in Kessel 1 eingedrückt. Dazu kommen 20 kg Aluminiumchlorid. Es wird auf  $160-165^\circ$  angeheizt. In Kessel 2 werden 646 kg Schwefel bei ca.  $130^\circ$  geschmolzen. Im Laufe von 2-3 Stunden wird nun der geschmolzene Schwefel in dünnem Strahl langsam in das Diphenylamin im Kessel 1 bei von  $165-180^\circ$  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  $180-200^\circ$  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.

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Laboratoriumsproben:

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

Probe 2: Das Endprodukt muss einen Schmpkt. von  $173-175^\circ$  haben.

Erfahrungen:

Beim Zusammenschmelzen von Diphenylamin und Schwefel in Gegenwart von  $AlCl_3$  beginnt die Reaktion bei ca.  $155^\circ$  und steigert sich in wenigen Minuten zu grosser Heftigkeit, wobei dabei freierwerdendes  $H_2S$  - 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 vorlegt und die andere bei Reaktionstemperatur langsam zulaufen lässt. Ausser der in diesem Verfahren beschriebenen Arbeitsweise kann man auch das Aluminiumchlorid vorlegen und das zusammengesetzte Gemisch von Diphenylamin und Schwefel bei  $165-180^\circ$  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 Leitungstücke 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 saurem  $AlCl_3$  gefüllt ist. Von der letzten Vorlage führt eine 150 mm  $\emptyset$  gusseiserne Leitung zur Absorptionsanlage für  $H_2S$ .

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zu der neutralisierten Trypsinlösung: (Der Tothahn der Leitung nach Kessel 7) ist ganz offen). Damit das fertige Produkt die richtige Kristallform erhält, die den normalen Kochsalzgehalt von 2,5 - 3,5 % gewährleistet, ist es nötig, sowohl das Salzwasser, als auch die Trypsinlösung KONSTANT 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 erst fällt das getrocknete Trypsin etwa 2,5% aus. Nach der Fällung rührt man noch solange (3-4 Minuten) bis das gesamte Niederschlagene Trypsin abgetrennt ist.

Das Wasserbad von Kessel 5) darf während der ersten Stunden nicht gekühlt werden. Erst nachdem die Temperatur am nächsten Tag auf 29 - 24° gesunken ist, ist es angebracht, das Bad unter gleichzeitigen 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 abfließt, was je nach der Form des Produktes 3-5 Stunden dauert.

#### Trocknen

Je 3 Partien verteilt man partieweise, gleichmäßig und gut zerkleinert auf je 2 Hängeherden 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

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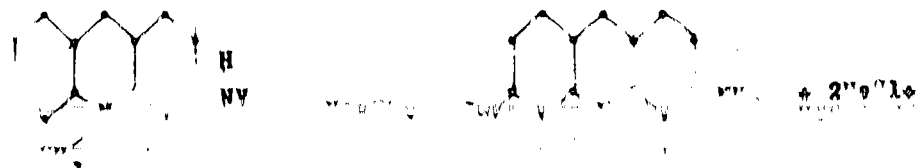
des Trockners sollte der Wassergehalt 6,3% betragen (Probe 1). Da derselbe erfahrungsgemäß beim Mahlen und Umfüllen durchschnittlich um 1,3% sinkt, enthält die Probe 1) nur die gewünschte Menge Wasser.

Ausbeute je Partie: etwa 12 kg (ca. 100%)

#### Mahlen

Je 3 Partien (ca. 97 kg), die man nach den Analysenwerten zu wägen hat, werden etwa 24 St. in der Mahlmaschine 7)

24152-2



Einzelst:

100 g Tryptophan  
 100 g Carboraffin  
 100 g Soda  
 100 g Wasser

Ausbeute:

Arbeitsweise:

Der Kessel 1), der ca. 500 Ltr. Carboraffin-Waschwasser der vorangegangenen Partie enthält, wird bei einer Temperatur von 35-40° mit dem Tryptophan 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 Lackmuspapier soll nach einigen Minuten nur einen sehr geringen Überschuss an Soda anzeigen. Nunmehr bringt man das Volumen mit Trinkwasser auf 580 Ltr. und rührt 3 Stunden bei 35-40°.

Nach Vorlauf dieser Zelle drückt man den ganzen Kesselinhalt auf den Sauger 2) und saugt solange in den Kessel 1) zu rück, bis das Filtrat völlig klar abläuft (Saugglas am Steofilter). Dann wird umgestellt (vorheriges Evakuieren von Kessel 5), und das Filtrat nunmehr in den Kessel 5) geleitet. Das in den Kessel 1) zu Beginn anhängen gebliebene trübe Filtrat wird wieder auf den Sauger gedrückt. Der Carboraffinrückstand auf dem Sauger wird zunächst mit etwa 40 l Trinkwasser von 30° gewaschen, das Filtrat läuft in Kessel 5). Inzwischen hat man in Kessel 1) 400 l Trinkwasser auf 70° erwärmt und wärmt 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) zu rück. Die Carboraffinrückstände enthalten kein Tryptophan mehr und werden fortgeworfen.

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

Inzwischen hat man im Kessel 7) Salzwasser von 24° B<sub>e</sub> 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)

Mainkur, den 18. Juli 1938

Vorfahren zur Herstellung von  
Tryptaflavin

A.) Chemischer Teil.

Konstitution und Zusammensetzung:

Tryptaflavin ist eine Mischung von 3.6-Diamino-10-Methylacridiniumchlorid und 3.6-Diamino-10-Methylacridinhydrat;

Normale Zusammensetzung:

69	67	% Diamino-10-Methylacridiniumchlorid	Mol. 259,5
21,4	25,7	% Diamino-10-Methylacridinhydrat	Mol. 245,7
7	8	% Wasser	
2,5	3,5	% Steinsalz.	

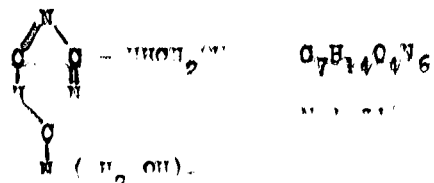
II. Chemischer Vorgang:

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

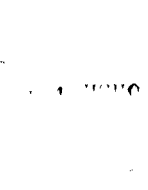
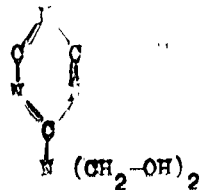
Schmiedeeiserner, innen hölzernen verbleiteter  
Doppelpresse mit Rührwerk n = 14 und n = 67,  
Inhalt 1250 l. Druckleitung aus Eisen nach 2)

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

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

Vorschrift zur Herstellung von Keurit MKF.V. mischer Teil.Konstitution:

Chemischer Vorgang der Polymerisation

Melamin  
Mol. 126Formaldehyd  
Mol. 30Tetramethylolmelamin  
Mol. 216,2II. 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 Triäthanolamin  
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 Sodälösung 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 Manteldampf 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 Sodälösung 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 (in Winter 24 Std., in Sommer 48 - 50 Std.) so fest wird, dass sie mit Spachteln in faustgrossen Stücken ausgestochen werden kann. Die Stücke werden in einer Raspelmaschine zu einer brümeligen Masse verkleinert (Probe 2). Der Keurit MKF in dieser Form ist...

Proben:Materialproben:Azamin rein

126 g Azamin rein 100% sollen sich in 243 g Formaldehyd (37 Gewichtsprozentig mit Soda neutralisiert) bei 10 - 15° vollständig klar und farblos lösen. Die Lösung soll bei 40° mindestens 93% abgeben.

Betriebsproben:

- 1.) Formaldehyd wird mit ca 20%iger Sodälösung auf einen pH-Wert von 7,6 - 8 eingestellt.
- 2.) 170 g Keurit 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 Indikatorpapier "Bayer" Nr. 8 benutzt. Die Lösungen I und II sollen nach 20 - 24 stündigem Stehen noch klar sein.

Nach beendeter Sulfierung wird noch 1/2 Stunde nachgerührt zum besseren Drucke noch 1/6 kg Trichloraethylen aus dem Massgefäß (Pos. 46) zugegeben, und dann die Sulfierung in den ausgeleiteten Schellier (Pos. 6) von 7000 l. in dem 2800 l Wasser vorgelegt und auf 20° abgekühlt waren, unter Rühren geht durch Zugabe von Eis (ca. 1600 kg) sorgt man dafür, dass die Temperatur nicht über 35° geht. Man wartet um 4-6 Stunden.

#### Destillation:

Über eine ausgeleitete Vorlage (Pos. 7) kommt jetzt die angeworbene Sulfierung in den ausgeleiteten Destillationskessel von 4000 l (Pos. 8). In dem Destillationskessel sind 300 l Wasser und 250 l = 325 kg Natronlauge 33° Bé (aus Pos. 9) vorgelegt; unter Rühren wird die Sulfierung zugeedrückt, 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 Zedrückens wird die Phenolphtaleinreaktion schwächer, sodass nach und nach noch ca. 40 l = ca. 52 kg Natronlauge 33° Bé mit zugeedrückt 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 100% zugesetzt. Dann kommen noch ca. 10 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 Nitwert bestimmt (siehe Nr. 4) und auf 2678 kg eingestellt. Spez. Grav. 1,02 - 1,03. Ferner wird durch Verfügen eine Fettbestimmung angefertigt, siehe Nr. 5.

Langzeitbeständigkeit des Typs:

Anforderung: 2000 kg

Die Langzeitbeständigkeit des Typs entspricht den Anforderungen Humectol CX.

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

dem Wassergefäß (Pos. 1a) 100 kg Phosphortrichlorid = 63,5 l, wobei die Temperatur auf 45° zurückgeht. Das Rührwerk läßt wa dann 3 Umdrehungen machen, stellt wieder ab, erhitzt auf 50° und läßt 3 Stunden bei dieser Temperatur. Nach dieser Zeit ist die Reaktion beendet. Die entstandene phosphorige Säure geht als gelbes Öl an Boden ab. Der Rest einbalt wird dann in den Scheider (Pos. 2) gegeben. Nach 3 Stunden ist die Reaktion beendet. Die entstandene phosphorige Säure geht als gelbes Öl an Boden ab. Der Rest einbalt wird dann in den Scheider (Pos. 2) gegeben. Nach 3 Stunden ist die Reaktion beendet. Die entstandene phosphorige Säure geht als gelbes Öl an Boden ab. Der Rest einbalt wird dann in den Scheider (Pos. 2) gegeben.

Isobutyramid

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 Olsäurechloridscheider (Pos. 2) bei einer Temperatur von bis zu + 20° unter Kühlung mit Sole und fortwährendem Rühren das Olsäurechlorid zu. Nachdem alles Olsäurechlorid eingelaufen ist, wird die Suspension auf 50° angeheizt und eine Stunde gehalten. Nach dieser Zeit ist die Amidierung beendet und muss phenolphthalein-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 beträgt 45 - 50°. Das Diisobutylamid der Olsäure 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 Mischung nochmals mit 125 l 50%iger Schwefelsäure gewaschen. Der Scheider bleibt das Amid als klares Öl zurück.

Ausbeute: 835 - 840 l 710 kg Isobutyramid  
Bestimmung der Gehalte an freier Olsäure und Amid  
Der Gehalt an freier Olsäure beträgt 58 - 60%

Sulfurierung:

Die Sulfurierung erfolgt in dem Scheider (Pos. 4) bei einer Temperatur von - 3 bis - 5°.

Bei dem Amid (10 kg freie Olsäure) sind 100 kg Amid vorhanden.

Beispiel: Nach Sulfurierung enthalten 135 kg Amid 100 kg Amid und 35 kg freie Olsäure. Um das Verhältnis Amid-Olsäure zu freier Olsäure zu erhalten, braucht man bei 442 kg Amid-Olsäure 238 kg freie Olsäure. Da im Amid aber bereits 58 kg freie Olsäure enthalten sind, müssen noch 180 kg Olsäure nachgesetzt werden. Es sind dann in der Sulfurierung insgesamt 680 kg Olsäure vorhanden.

Aus dem Scheider (Pos. 4) wird das Amid in den Sulfurierungskessel (Pos. 5) abgelassen, aus dem Massgefäß (Pos. 5a) 180 kg Olsäure und aus dem Massgefäß (Pos. 4b) 730 kg Trichloräthylol zugufügt. 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 beträgt 10% mehr als die eingesetzte Gesamt-Olsäure bei 680 kg, also 748 kg Monohydrat. Die angegebene Sulfurzeit ist nur einzuhalten bei einer Kaltesole von

Mainaur der 4. Klasse

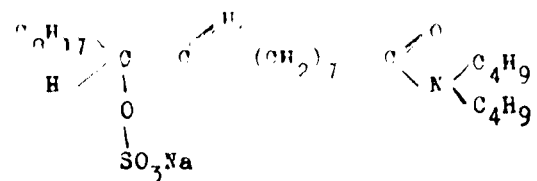
zur Herstellung

Humectol CX

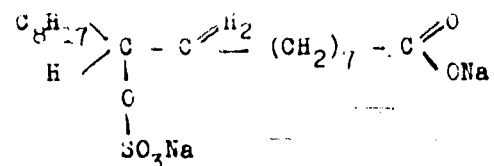
dient als...

### Chemischer Vorgang:

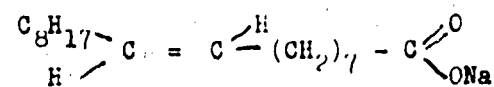
Ölsäure wird in Ölsäurechlorid übergeführt, dieses mit Diisobutylamin zu Ölsäure-di-isobutylamid übergeführt und mit Natriumhydroxyd in die Sulfocenter übergeführt. In der Sulfocenter zugemessene Ölsäure wird ebenfalls in den Sulfocenter übergeführt, so dass das Humectol CX in der Hauptsache aus einem Gemisch des Sulfocenter des Ölsäure-di-isobutylamids und des Sulfocenter des Natrium-Ölsäures besteht.



Sulfocenter des Ölsäure-di-isobutylamids Mol. 518.



Sulfocenter des Ölsäuren Na. Mol. 424.



Ölsäures Natrium Mol. 304.

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### 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 i Schwefelsäure 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 Ofenbach und werden auf 50° angewärmt. Unter die Oberfläche kommen dann aus

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TABLE II

RESEARCH PROJECT ASSIGNMENTS

- 1.) Dr. Brunner Synthetische selbsttrocknende Lackharze der aromatischen Reihe.
- 2.) Dr. Fleischmann Derivate der Cellulose, insbesondere der Amino-Cellulose
- 3.) Dr. Goffert a) Formaldehyd-Kondensationsprodukte von Kalkstickstoff, Cyanamid, Dicyandiamid etc. als Anilinisierungsmittel und zur Verbesserung der Echtheiten substantieller Färbungen.  
b) Prüfung von Stabilisierungsmitteln
- 4.) Dr. Hechter Schwefelfarbstoffe.
- 5.) Dr. Henold a) Hochmolekulare Sulfonamide (entsprechend Superpolyamide)  
b) Egalisierungsmittel für Käpen und Schwefelfarbstoffe.
- 6.) Dr. Keller Derivate des Melamins und ähnlicher Substanzen für Lackharze und Kunststoffe.
- 7.) Dr. Kortzen 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 polymerisierbaren Verbindungen.
- 9.) Dr. Müller, Rud. Herstellung von Stabilisierungsmitteln für Poly-vinyl-chlorid und anderen chlorhaltigen Kunststoffen.
- 10.) Dr. Riedmair a) Weichmacher für Kunststoffe (Sulfosäure-ester).  
b) Derivate des Thio-diphenylamins als Schädlingsbekämpfungsmittel und Wurmmittel.  
c) Sulfonamide ("Prontosil").
- 11.) Dr. Ritter 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 Textilindustrie.
- 13.) Dr. Schubert Kondensations-Produkte von heterocyclischen Aminen mit Formaldehyd.
- 14.) Dr. Schütz Papierleimungs- und Hydrophobierungsmittel (Uniformen).
- 15.) Dr. Schultze Derivate des Kalkstickstoffes, die sich als Lösungsmittel eignen, insbesondere die Dialkyl-cyanamide.
- 16.) Dr. Schwemmer Papierveredlungs-Mittel, insbesondere Grundlegende Melamine.
- 17.) Dr. Tröckner Kapillareaktive Substanzen zur



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

9. Insecticides: Thiamine S, crude thiodiphenylamine, is used for spraying swarms to kill malaria-bearing mosquitoes. Production amounted to 40 tons per month. Production for export is reported on Page 55.

Dr. Giesler stated that Malinkur had done no work on vaccines or sera, nor on any chemical warfare agents. He said that he had no contact with, or knowledge of, Japanese activities. From conversation and observation at the plant and from inspection of captured documents he had obtained the following information:

Keurit MKF  
Ocenol Sulfonate (Laboratory)  
Trypoflavin, pure  
Thiodiphenylamine  
Thiodiphenylamine  
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. Childers 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, Germany

#### List of Chemiker

##### Werksleitung:

Direktor Dr. Giesler

Leiter Schwefelf

Dr. Schiek

Dr. Frohlich

Dr. Kohl

Dr. Murauchi

Dr. Will

##### Abteilung Kfzfarben:

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. Vogt

Dr. Heintz Dr. Müller

Dr. Menzer ) Im

Dr. Ziegler ) Heeresdienst

Wissenschaftliches Laboratorium

Dr. Zerweck Dr. Kortan

Dr. Ritter Dr. Müller

Dr. Blodmeier Dr. Fleischhauer

Dr. Schütz Dr. Frister

Dr. Brunner Dr. Sondag

Dr. Keller Dr. Löwe  
im Heeresdienst

Dr. Selkowski

Dr. Schultis

Dr. Schwamberger

Dr. Kunze

Dr. Schubert

Dr. Honold

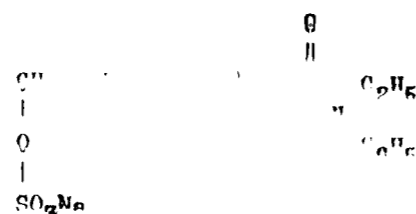
Dr. Hechtenberg

Dr. Gofferje

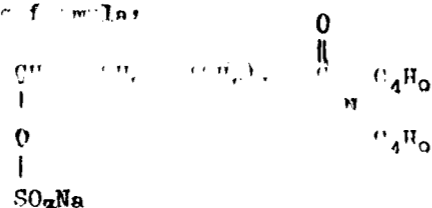
Dr. Trösken

Dr. Kleinhans

**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 correct, and correcting typographical errors, the formula would be:

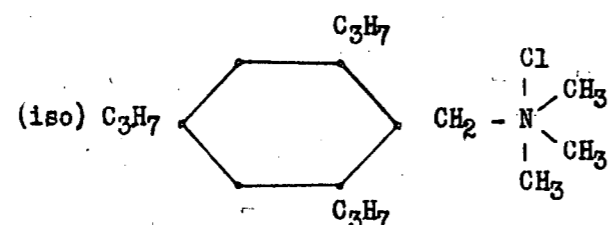


**Dismulgan V:** This was stated to be the sulfate of oleylethylanilide but the written record, which is presumably more dependable, indicates that it is the sulfate of ethylaniline with the following formula:



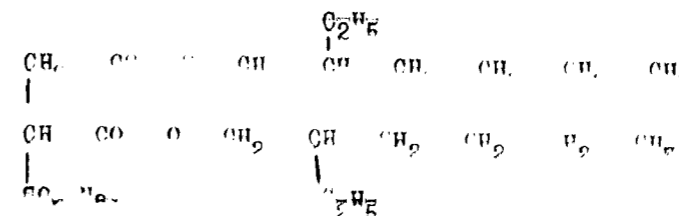
It is made like Dismulgan IV but using Diisobutylamine in place of N-ethyl aniline, as described in Page 8. Apparently in conversation the two compounds were inter-changed, and the final neutralization with caustic soda was not mentioned.

**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 formula:



Instructions for the preparation of this material are reproduced in part on Page 17.

Page 17

#### Miscellaneous Chemical Products

- 1. Dyes and Colors:** Production amounted to '00-500 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 13 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 melamine 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 acryloyl 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.

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

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**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 CPT assessors Capt. C.C. Chaffee and Capt. G.A.A. Moir 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 21st 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." Information obtained on both visits is summarized below.

**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 structure of the plant is summarized and discussed below.

**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 Petriet 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.

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DEPARTMENT OF COMMERCE

WASHINGTON, D. C.

G. FARBENINDUSTRIE

REPORT NO. 100

Reported by

Gen. C. C. Chaffee, U. S. Army  
W. V. Atwell, U. S. Army

[1945]

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

**NOTE:** The Publication Board, in approving and disseminating this report, hopes that it will be of direct benefit to U. S. science and industry. Interested parties should realize that some products and processes described may also be the subject of U. S. patents. Accordingly, it is recommended that the usual patent study be made before pursuing practical applications.

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c. Diesel Fuel (Automotive Grade)

Density at 20°C	.92 Max.
Flash Point (Penky Martin)	21° C. Min.
Cetane No.	9500 Kilo-Cal./gr., Min.
Sulphur	0.2 % Max.
*Corrosion Test Loss	10 mg. Max.

\*Corrosion test run on Copper and Zinc specimens, 10 cm. long, 1 mm. thick and 1 cm. wide. When exposed to 25 cc. of oil at 75°C. for 3 hours, the maximum loss must not exceed 10 mg.

d. Automotive Lubricating Oil Summer Winter

Density at 20°C	0.95 Max.	-
Vis at 50°C	8.0 - 9.0°E.	4.0°E.
Vis at 80°C	2.5 - 2.8°E.	ca. 1.8 - 1.9°E.
Pole Height	2.1 Max.	2.1 Max.
Flash	250°C	-
Pour Point	-30 - -40°C	-40°C
Carbon Residue	0.2 - 0.3%	-
Noack Test (1 hr., 20 mm Vac at 120°C)	12 - 14% Loss	12 - 14% Loss

e. Gear Oils Summer Winter

Density at 20°C	0.88 - .93	Same
Vis at 50°C	8 - 9°E	5.0 - 6.5°E
Vis at 80°C	2.7 - 2.9°E	-
Pole Height	1.9 - 2	Same
Flash Point	215 - 230°C	190 - 200°C
Pour Point	18 to 20°C	-45°C
Noack Test (1 hr, 20 mg. Vac, at 200°C)	9 - 13% Loss, Max.	Same
Sap. No.	2.8 - 3.1	Same
Neut. No.	0.8 - 1.1	Same
Carbon Res.	0.25 - 0.4%	Same
*Corrosion Test	Perfect	Same
Fatty Oil Content	-	3% Min.

\*Corrosion Test run on copper and Zinc specimens of dimensions as in Diesel Fuel Test, for 24 hours at 75°C. The visual test inspection of specimens must show perfect.

F. All Purpose Grease

Composition	70% Oil, 30% Calcium Stearate
Water Content	0.5 - 1.0%
Consistency at 50°C	0.5 - 0.8 kg.
Ash	0.8 - 1.5%
P. H. Value	Must be slightly alkaline
*Water Resistance	Satisfactory
**Bleeding Test, Wt.% Loss	0.5 Max.

Water Resistance Test:

\*Grease is placed in cylindrical sieve which is rotated in a beaker of water at 50°C for 24 hours. After the end of the test the water and the grease are examined for signs of decomposition of the grease.

\*\*Bleeding Test: The grease is placed in a sintered bottom glass crucible and kept in oven for 24 hours at 75°C. The crucible is weighed to determine the loss of oil. The loss must not exceed 0.5% by weight.

gasoline, or special oils, and a careful search failed to reveal the presence of any additive materials.

Samples of all of the above materials were obtained for examination and full scale tests if desired by agencies in the United States and Great Britain.

### III. INVESTIGATION OF THE TARGET

Although the administrative building had been stripped of all documents and the manager's office had been partially destroyed, interrogation of Herr Johannes Friede, Manager (party member) and Herr R. Schmidt, Head of the Laboratory, provided the following useful information:

### IV. GENERAL INFORMATION

Herr Friede, Dipl. Engr., previously a design engineer with an aero company (Arada), came with the WIFO by answering an ad, not because of party membership, in 1936. His first assignment was to help choose the layout, design the tanks and RR siding, then install the canning equipment. He said that this was the first of the smaller WIFO's and that the first oil was received July 1938.

Herr Friede stated that the WIFO was definitely first organized as a construction company and that Derben was the first plant built, which was in either 1934 or 1935.

The products turn-over in the WIFO was always large and hence they had experienced little or no trouble with products going off test in storage. The WIFO had never been called upon to utilize alcohol in any fuel blends. Benzol of good quality, low gum and sulphur content was used in 25% blends early in the war; this was later dropped to 20% minimum due to a benzol shortage.

The Motor Fuel base handled was mainly from Fischer Tropesch synthesis and rarely from natural sources, in which cases the source was Rumanian. The base gasoline octane varied from 55 - 62 and with the benzol used required the use of .02 - .04% TEL to make the required octane, which was 78 at first, then 74 and finally 72 Octane Number.

The WIFO rarely made any Diesel oil blends and handled no Jet Plane Fuels. Until 1941, they got lube oil direct from Ruhr Chemie and not from Main Depots.

It was stated that no records or personnel had been evacuated to this area and it was confirmed that the Tech Records are in the underground WIFO at Stassfurt and that Herr Wehling (General Manager of WIFO's) is at Oberhausen, near Neuberg, Bavaria.

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Although motor fuel stability had never been a serious problem, equipment had recently been received to make such tests. Herr Schmidt stated that the test was to require a 10 mg./100 cc. max. test on gasoline after storage at 100°C. for 10 hours in a bomb under 15 atm. air pressure. This was stated to be the method adopted at Derben for testing aviation gasolines in 1939. The report on this oxidation stability and gum test is subject to confirmation.

They had no difficulty from Sulphur, even from benzol, since the Fischer Tropesch materials handled are practically sulphur-free.

### V. SPECIFICATIONS ON GERMAN WEHRMACHT FUELS AND LUBRICANTS

It was stated that the German Army did not have numbered specifications similar to those used by the Air Force. Herr Schmidt supplied the following specification points, which were in effect at the WIFO, from memory, however, it is considered that they are substantially accurate.

#### a. Army Delivery Motor Fuel

Density at 20°C.	0.8 Max.
Vapor Pressure Reid	0.6 atm., Max.
Calorific Value	9000.0 Kilo Cal/Gr., Min.
Octane	72.0 Min.
Gum	10.0 Mg/100 cc Max. Glass Dish.

#### Distillation:

Init. B. P.	20°C. Min.
at 75°C.	25% evap., Max.
at 100°C.	30% evap., Min.
E. P.	220°C. Max.

Benzol content	20% Min.
Sulphur	.02% Max.
Freezing point (Mar. to Oct.)	-40°C. Max.

#### b. Benzol Delivery Specification

Sulphur	0.05% Max.
Gum content	10.00 mg/100 cc. Max.
Flash Point	0°C. Max.
Distillation (Kraemer-Spilker)	
Init B.P.	77°C.
End Point	200°C.
% Distilled up to 135°C.	95% Max.

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Personnel of Team  
(Part of CIGS Party 551)

Maj. D. A. HOWES, Ministry of Fuel & Power  
Mr. E. SCHINDLER, TIC Ord. Team  
Capt. C. C. CHAFFEE, U. S. Ord. Dept.

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WIRTSCHAFTLICHE FORSCHUNGSGESELLSCHAFT m.b.H. (WIFO)  
EWEERBACHTEL (3 km. S. of Heiligenstadt, Germany)

I. INTRODUCTION

a. This is one of the so-called smaller ARMY COMMAND DEPOT, "WIFO" installations, blending, testing, and packaging Fuels and Lubricants for the German Army, and was visited because the larger MAIN DEPOT at STASSFURT, reported to contain a larger variety of special materials and the evacuated Technical Records of the Central WIFO office, was not available for investigation at the time.

II. DESCRIPTION OF THE TARGET

a. The WIFO which was located in a heavily wooded area with the equipment well dispersed, operated until Easter and had not been noticeably damaged until the last three months. However, four of the 12 storage tanks and some facilities had been damaged by aerial bombing and the laboratory had been wrecked by an explosive charge set by the Germans before leaving.

b. The installation contained the following equipment and/or products:

- One office building (approximately 50 in Office staff)
- One laboratory equipped with gasoline and Diesel test engines and normal petroleum testing apparatus.
- Gasoline blending plant (with TEL blending).
- Lubricating Oil blending plant.
- Can reconditioning, washing and handling plant.
- Packing plant for oils, greases and Blitz can filling.
- Barrel reconditioning plant.
- Underground power plant.
- Four storage buildings for finished packaged products.
- Miscellaneous storage for filled and empty drums.
- Miscellaneous buildings for the 300 workmen, workshops, etc.
- Train and truck loading racks.

c. Products or Materials in Storage.

There was a large stock of empty Jerry cans and drums on the premises. The gasoline storage and blending tanks were destroyed or empty. There was a fair supply of gear oil (Betriebsöl Wehrmacht 88), of motor oils, mainly Nachlaufschmierstoff T 42 in drums (reported removed from Nordhausen when the V Factory was located there) Motor-eneel in summer and winter grades, Methanol, Glistina (Glycerol substitute), and all-purpose grease. There was no diesel fuel, aviation

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WIRTSCHAFTLICHE FORSCHUNGSGESELLSCHAFT m.b.H. (WIFO)  
FUEL BLENDING STATION

1 July 1945

Reported by

Capt. C. C. CHAFFEE, U. S. Ord.

CIOS Target Number 30/78

Fuels And Lubricants

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

7 p.  
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ITEM No. 30  
FILE No. XXVI-68

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

~~RESTRICTED~~

WIRTSCHAFTLICHE FORSCHUNGS G.M.B.H.,

EFERBACHTEL FUEL BLENDING STATION.

*Chaffee, cc*

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COMBINED INTELLIGENCE OBJECTIVES  
SUB-COMMITTEE

Statement No- 4.

Showing stocks of finished goods

<u>Material</u>	<u>Tons</u>
Buna S	600
Buna 85	50
Igetex (rubber lat)	100
Buna 32	30
Polyvinyl chloride	370
Polystyrene	190
Ethyl alcohol	400
Butanol	130
Acetic acid	20
Acetone	30
Phthalic acid	200
Formaldehyde (100%)	60
Diglycol	250
SS oil (lubricating oil)	80
Tetrahydro furane	20
Trichloroethylene	60
Ethyl chloride	20
Caustic soda lye (NaOH)	500
Aluminum chloride	300

Product	Quantity	From where obtained
Iron	2 (used)	
Iron (30)	61	From where obtained
Dipropylamine	30	I.G. at Wolfen Elektrochemische Werke at Hüllriegelstraße near Mühlheim
Calcium chloride	6	I.G. at Griesheim
Acetic acid	40	I.G. at Griesheim
...	10	Schkoppe
...	2	I.G. at Ludwigshafen
...		Hauptstadt Wien

Statement No. 3.

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

Product	Tons	Will last for
Brown coal	-	-
Coke	-	-
Anthracite	15,000	1 1/2 months
Burnt chalk	-	-
Hydrogen	-	-
Benzene refined	130	4 days
Special electrodes	70	4 to 5 months
Tar	70	2 months
Pitch	90	3 months
Blankets for electrodes	100	25 days
Chlorine	-	-
Caustic soda lye (NaOH)	500	4 months
Mercury	60	1 year
Nitric acid	-	-
Sulphuric acid (SO <sub>3</sub> )	50	10 days
Ferrous sulphate	70	1 3/4 months
Phosphoric acid (P <sub>2</sub> O <sub>5</sub> )	100	1 month
Caustic potash lye (KOH)	25	1/2 month
Caustic potash	10	1 month
Butyl amine	10	1 month
Silica gel	70	6 months
A-coal	15	3 months
Calcium chloride fused	5	1 month
Aluminum chloride	300	10 months
Naphthaline	250	3 months
Oleum (SO <sub>3</sub> )	20	10 days
Potassium persulphate	10	10 days
Diperoxide	25	4 months
Calcium chloride	50	6 days
Acetic acid	20	1/2 month
Phenyl-naphthyl amine	700	6 months
Talcum powder	80	2 months
Synthetic fatty acids	50	1 month

APPENDIX I

STATEMENT NO. 1

Statement No. 1

Product	Quantity	Value	Value
Buna S	1,000	1,800	-
Buna 72 and 85	270	270	-
Vinyl chloride	500	300	-
Polyvinyl chloride	200	200	-
Polystyrene	50	50	-
Ethyl alcohol	800	800	500
Butanol	500	500	350
Acetic acid	700	700	-
Acetone	200	200	-
Phthalic acid	540	270	-
Formaldehyde (100%)	800	800	-
Diglycol	700	700	-
SS oil (lubricating oil)	500	500	-
Tetrahydro furane	300	300	-
Trichloroethylene	400	400	-
Ethyl chloride	120	120	-
Waste lime (refuse used as manure)	14,000	14,000	8,000
Caustic soda lye (NaOH)	4,500	2,500	-
Aluminum chloride	700	700	-

Statement No. 2.

showing the products and the quantities which are required per month in case (c) and naming also the factories and places where they were manufactured: fact.

Unless otherwise specified the quantities are in tons per month.

Product	Quantity	From where obtained
Brown coal	10,000	Weisel district (near Merseburg)
Coke	10,000	Waldenburg (Saxony)
Anthracite	10,000	Ruhr district
Burnt chalk	5,000	Harz district
Hydrogen	5,000	Leuna
Benzene refined	1,000	hour
Tar (or coal)	60	Arbeitslehre
Special electrodes	15	Leuna or I.G. Werke
the	35	Siemens-Elenia at Ratibor
Iron	30	Raschig at Ludwigshafen
Phosphate	120	Verkaufsgesellschaft für Teererzeugnisse at Essen
Chlorine	25	Teererzeugnisse at Essen
Caustic soda lye (NaOH)	130	Arnsdorf (Place) or I.G. at Bitterfeld
Mercury	4	Schkopau
Nitric acid	40	Italy
Sulphuric acid (SO <sub>3</sub> )	140	Piesteritz (Place)
Ferrous sulphate	40	I.G. at Wolfen or at Döberitz
Phosphoric acid (P <sub>2</sub> O <sub>5</sub> )	100	I.G. at Leverkusen
Caustic potash lye (KOH)	50	Piesteritz (Place)
Caustic potash	10	I.G. at Bitterfeld
Butyl amine	10	I.G. at Bitterfeld
Silica gel	12	I.G. at Ludwigshafen
A-coal	5	I.G. at Oppau
Calcium chloride fused	5	I.G. at Leverkusen
Aluminum chloride	30	I.G. at Bitterfeld
Naphthaline	85	Schkopau
		Verkaufsgesellschaft für Teererzeugnisse at Essen

asymmetrical compound polymerizes very readily and exothermally, and must be stabilized for safe handling. Distilled at 0.2% concentration, is used for this purpose.

#### Other Chloro-compounds

A 100% product is available for use in the production of butadiene.

#### Aluminum Chloride

This plant adds the own supply of aluminum chloride to the plant.

#### Butenediol and Tetrahydrofuran

A process developed at Ludwigshafen under Dr. R. W. Schkopau which gave an alternative route to butadiene, using less acetylene than the acetylene-acetaldehyde aldol-butylene glycol synthesis, was operated in part at Schkopau. This synthesis consists of forming butenediol by the addition of two moles of formaldehyde to one mole of acetylene. The butenediol is then hydrogenated to butanediol, and dehydrated to tetrahydrofuran which in turn was shipped to Ludwigshafen for conversion to butadiene. The Schkopau plant was merely a pilot plant for Ludwigshafen.

Butenediol was prepared with the aid of a cuprous acetylide catalyst, made by precipitating copper hydroxide on silica gel. The reaction was carried out at 3 atm pressure and 90 - 130°C by passing both acetylene and 30% formaldehyde solution downward through a multitubular reactor packed with the catalyst. The acetylene was used in excess and recirculated. The rate of feed of the formaldehyde solution was adjusted to obtain complete consumption of the HCHO in a single passage. Some explosions were experienced, due to the instability of the cuprous acetylide catalyst, till steps were taken to prevent its drying out by injecting steam into the incoming

stream of dry acetylene. These explosions also occurred in the pipe lines carrying the recirculating acetylene until they adopted the expedient of filling these large pipes with small pipes, thereby increasing the total surface and the heat transfer and reducing the chance of a local temperature rise and of the propagation of decomposition. The butenediol itself gave no trouble, especially as under their operating procedure it was not isolated, but was processed in solution as obtained. The hydrogenation was done at atmospheric pressure, the catalyst being a paste of a mixture of nickel and manganese oxides.

The butenediol solution resulting from the hydrogenation of butenediol could be and usually was, further processed without removal of the water. The dehydration of the diol was carried out at 90 atm pressure and 270°C, using o-phosphoric acid as a catalyst. The tetrahydrofuran, boiling at 64°C, could then be distilled out readily. Most of this was shipped to Ludwigshafen for butadiene production, but little was used on site for the production of butadiene.

#### Naphthalene Derivatives

Naphthalene was used for the production of Nektal 100 (dibutyl-naphthalene sulphonic acid sodium salt) and for phthalic anhydride. Some of the latter was esterified with 1,3-butylene glycol to obtain a plasticizer for resins.

W. S. CALCOTT,  
CWS, HQ  
ETOUSA

atmosphere pressure. Steel equipment is used throughout, as no corrosion problem is presented. The heating medium is steam at 60 atm pressure, obtained from the power plant. The cooling water is at 170 atm pressure. The reaction is carried out at 170 atm pressure.

h. Buna

Besides Buna S for motor and aircraft tires, the plant also produces Buna 85 for tires.

i. Chlorine and Caustic

Salt was electrolyzed in mercury cells to produce chlorine and sodium hydroxide solution. The cells were of the type at Electrochem, which were used for the production of chlorine and caustic.

j. Glycerol

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

k. Hydrochloric Acid

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

l. Vinyl Chloride and Polyvinyl chloride (PCU).

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

differential of 40 - 50°C. Some mercuric chloride sublimes out of the catalyst chamber and is lost, but has never caused any trouble. The output was 2 kg of vinyl chloride per liter of catalyst per day, and the catalyst life about 200 hours per liter. The vinyl chloride is recovered by liquefying in a condenser cooled with brine at -30 to -40°C. The condensate is then fractionated, the products obtained being acetylene, vinyl chloride and 1,1-dichloroethane. The vinyl chloride is stored at -20°C, but is considered stable enough to ship in uninsulated tank cars at 7-8 atm pressure to Walfen, a firm which was usually made in 24 hrs. or less. The stabilizer of any sort is used in the vinyl chloride.

About 800 tons/yr of PCU (unchlorinated vinyl chloride polymer) were made at the Schkopau plant. Potassium persulphate or hydrogen peroxide were used as catalysts, the latter only when the PCU was to be used as an insulator. Mers' lab, from Leuna, was used as the polymerizing agent. This polymer is used for the production of PVC.

m. Lubricating Oil

SS 903 and SS 916, having Encler viscosities at 30 of 3 and 6 respectively, were produced at the plant. The catalyst used was iron chloride.

n. Tetrachloroethane

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

o. Trichloroethylene

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

p. Dichloroethylene,  $CH_2 = CCl_2$

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

The acetylene produced contained no oxygen, but did contain hydrogen sulphide and phosphine derivatives, all of which were removed by treatment with chlorine water. The acetylene was purified by treatment in a ...

#### Ethylene

Ethylene was made from acetylene by hydrogenation using hydrogen from Leuna. The temperature used in the hydrogenation was 100-150°C, being cooled as the reaction proceeded.

#### Styrene

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

#### e. Polystyrene

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

#### f. Acetone and Acetaldehyde.

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

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

#### g. Acetic Acid

This was made by air oxidation of acetaldehyde, using a copper-manganese catalyst.

#### h. Al'ol

By condensing acetaldehyde there was obtained a crude al'ol containing crotonaldehyde, acetaldehyde, al' and "polymers" (tri and tetra acetaldehyde condensation products). The entire mixture was distilled and the separation of the various components was effected.

crotonaldehyde	Butyl alcohol
acetaldehyde	Butylene glycol
al'ol	Hexane triol
"polymers"	

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

#### i. Nokal BX

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

#### j. Butadiene

Butylene glycol is dehydrated catalytically, the catalyst consisting of a mixture of sodium dihydrogen phosphate ( $\text{NaH}_2\text{PO}_4$ ), orthophosphoric acid, and dibutyl ammonium phosphate ( $(\text{C}_4\text{H}_9)_2\text{NH}_2 \cdot \text{H}_2\text{PO}_4$ ), supported on graphite. The reaction is carried out at 280°C, and one

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INDUSTRIAL PUNA

1. INTRODUCTION.

This plant was visited on 14 May, 1945. It was somewhat damaged, chiefly by bombs, but was still capable of producing 4000 tons/mo of Buna S as compared with its original capacity of 7000 tons/mo. A complete statement of the plant's current capabilities is given in Appendix I.

Information concerning the plant was obtained from Mr. A. S. Selkirk, Manager of the plant.

2. RAW MATERIALS

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

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

3. PRODUCTS AND PROCESSES.

a. Formaldehyde

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

b. Acetylene

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

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DEPARTMENT OF COMMERCE

WASHINGTON, D. C.

INDUSTRIAL PAPER

Reported By

W. S. Calcott  
CWS, HQ. ETUSA

L19457

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

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14 p.  
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RESEARCH PROGRAMME - ORGANIC

The field of investigation was the province of Dr. Harold  
er... was responsible for the following general  
or

1. Development of new uses for high molecular weight alcohols.
2. Finding uses for phenols, cyclohexanol, and cyclohexanone.
3. Development of new catalysts for the synthesis of i-butylene.
4. Improving processes for the extraction of phenols. Improved methods for the manufacture of maleic anhydride, adipic acid, and the laboratory synthesis of plasticizers.

W. S. CALCOTT  
CWS, Hq ETUSA

The lining of the towers had at first been done by using thin sheets of resin, cemented in place, but this had not been found satisfactory so that they had switched to the use of a solution applied by spraying while the equipment was heated to a temperature of 100-200°. This latter procedure had been found quite satisfactory. At Leuna they had made 4500 tons (metric) per month of Mersol prior to the damaging of the plant by bombs. This would give a capacity of 225 tons per month per tower. The product as made contained 30% unreacted oil and about 70% of sulphonyl esters. If the 70% 5-6% was more sulphonyl, the remaining being a mixture of 41 and 42 per cent of the product.

h. Aldehydes

i. Fatty acids were made at this plant from the highest fractions of the alcohols remaining from iso-butyl alcohol manufacture by caustic fusion and precipitation with sulphuric acid. Some of these fatty acids were esterified with trimethylolethane to form a castor oil substitute for use as a plasticizer. The remainder was shipped to the IG plant at Wiesheim, where the calcium magnesium salt was prepared. This salt was known by the trade name of "Solignum".

j. Contrary to one report received, there had been no work done on the synthesis of benzene at Leuna. The work referred to was due to a misunderstanding; it had been directed toward the production of benzene, or motor gasoline.

k. At the Leuna plant alanine (1 aminopropionic acid) which they termed "alkazidlaug" is used for the removal of carbon dioxide from process gases. This solution absorbs CO<sub>2</sub> when cool and under pressure, and releases it when heated without pressure.

l. The aliphatic amines were made by the usual procedure of reacting the alcohols with ammonia in the presence of a catalyst, and were shipped to other IG plants, chiefly to Ludwigshafen, for further processing.

m. Adipic acid was made by the route phenol-cyclohexanol-cyclohexanone - cyclohexanone oxime - caprolactam-adipic acid. This route was preferred to a simpler one as it gave more by-products and intermediate products which could be used as the raw materials for other syntheses.

n. The only resin or plastic made at this plant was of the phenol-formaldehyde type (Mepasin).

o. The production figures for January, 1944 are an approximate measure of the capacity of this plant before it was bombed.

Product	Production Metric Tons	Product	Production Metric Tons
Crude methanol	15850	SS oil	128.1
Crude i-butanol	14690	Spindle (Ac)	17.8
Refined Methanol	11287	Cutting oil	29.1
Anhydrous "	487	Machine oil	77.1
Dimethyl ether	75.8	Hydraulic oil	32.5
Propanol	302.9	Mesomoll	
Dipropyl ether	4	Weichmacher	
Crude i-butanol	137.6	Thiocompound	7
Refined "	848.5	Methylamines	
Pentanol	216.2	i-Butylamine	1.6
Ketoalcohols	37.5	Other amines	12.6
Other alcohols	66.7	Catalysts	277.1
Intrasolzone (Butanol + anil)	638.3	Mepasin	300.0
Formaldehyde	114.9	Mersol, D.H. 30	2068
Propionaldehyde	65.2	Mersolat & Mersapen	80
Other aldehydes	.6++	Hydrochloric acid	140
Trimethylolethane	60.0	Ethyl chloride	159
Ketone oil	179.8	Dichloroethane	12
Carbon dioxide	111.3	Carbolic oil	6.1
i-Butylene	32.9	Phenol-pure & crude	342
i-Octane	488.7	Cresols	498
Alkylate (ET 120)	1693.7	Xylenols	287
x1 Arabin	55.1	Crude catechol	250
HS olefin & polymers	51.8	Tech "	5
x2 Noral	11.6++	Oxime (cyclohexanone)	240
		Lactam ( " )	155
		Adipic acid	95

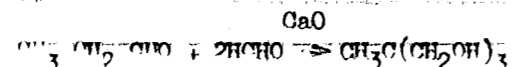
++February figures. No January production.

x1-The last fraction of the catalytic cracking process, hydrogenated.

x2-A special fraction used as a solvent for insecticides.

x3-A polymerized ethylene lubricating oil. Two grades SS 903 and SS 906 were made, with Engler viscosities at 100°C of 3 & 6 respectively.

d. A glycerine substitute was made here, by condensing formaldehyde with propionaldehyde in alcoholic solution in the presence of calcium oxide.



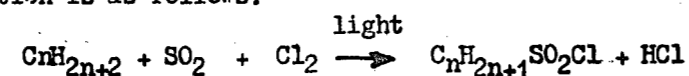
The reaction was carried out at 30-40°C by merely mixing the reagents. It is very rapid and strongly exothermic, so that cooling is necessary. The product was filtered off, washed with 1-butyl ketone and crystallized from water. It was used chiefly as a glycerine substitute in making the other types of soap.

e. The same method was used to produce hydrogenated oil.

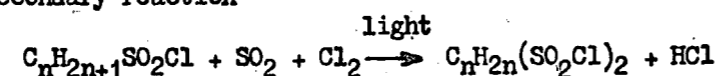
f. The same method was used to produce the other types of oil.

g. One of the important products made at Leuna was a soap substitute, or soap extender. This was made from a special fraction of Fischer-Tropsch oil known as Kogasin, which was the fraction having a distillation range of 230-330°C, and an average chain length of 15 carbon atoms. It was a straight chain aliphatic compound, with no branching. As received, it contained both oxygen derivatives and unsaturated compounds, which were eliminated by catalytic hydrogenation, using a nickel tungstate catalyst, a pressure of 200 at. and a temperature of 300-350°C. By this procedure was obtained a mixture of straight chain hydrocarbons, containing the compounds from C<sub>12</sub>H<sub>26</sub> to C<sub>18</sub>H<sub>38</sub>, but chiefly C<sub>15</sub>H<sub>32</sub>. This mixture is then treated with sulphur dioxide and chlorine, while being irradiated with "ultra violet" light. The temperature is held at 30-40°C by means of an external pipe-type heat exchanger.

The reaction is as follows:



and as secondary reaction



-4-

The reaction is carried to only 70% of completion, as too much disulphonyl chloride is formed if it is attempted to react more than this percentage of the oil. The product was, for the most part, shipped to soap makers (who saponified it together with their other soap making materials):

A cresol ester was also prepared from this sulphonyl chloride and used to plasticize polyvinyl chloride. Also, a small amount of the sodium salt (Mersolat) was made by saponifying the Mersol at 70°C with 10% NaOH solution. The resulting solution of sodium alkyl sulphonate was allowed to stand until most of the oil separated, the clear aqueous layer being then drawn off and dried on a drum drier. About 5% of the oil remained in solution in the aqueous layer, and was steam distilled off during the drum drier operation. The recovered oil contained chlorine derivatives, and was further chlorinated and sold for use as a solvent.

The finished sodium alkylsulphonate was sent to Schkopau for use in the preparation of polyvinyl chloride emulsions. According to Dr. Giesen, the Mersolat should not be considered as a soap but a solvent, but which it is believed would be more accurately referred to as a dispersing agent. The equipment in which the production of the sulphonyl chloride was carried out consisted of 20 vertical steel towers, each approximately 20 feet high by 8 feet inside diameter; the bottom or reacting section of each of these containing 16 glass tubes 7 inches inside diameter running across the steel cylinder in a horizontal position. In each of these there were placed six 40-60W daylight lamps; that is, the type which start on a tungsten filament and then operate as a mercury vapor light. In order to transmit as much ultra violet light as practicable, the glass tubes were made of "Normal" glass. The reacting section was 8 feet high. The entire steel cylinder and all pipe lines were lined with an acid resistant resin of the "Haveg" type which lasts from six months to one year. The upper part of the reactor was filled with rings of this same resin. In operating, the Kogasin was circulated from the bottom of the reactor through a heat exchanger consisting of water cooled six inch pipe also lined with "Haveg" and was returned to the top of the tower. Chlorine and sulphur dioxide were passed in at the bottom of the reactor and the hydrochloric acid, mixed with some excess sulphur dioxide, passed off at the top. Circulation was brought about by means of a centrifugal pump with a porcelain impeller, and the valves were steel, lined with "Haveg."

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