

ITEM NO. 30.

COPY NO. 1

FILE NO. XXXI-23.

PB-12624

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RESTRICTED

METALLGESELLSCHAFT-LURGI
FRANKFURT AM MAIN

Chaffee

RESTRICTED

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

RESTRICTED

METALLGESELLSCHAFT-LURGI FRANKFURT AM MAIN
GERMANY

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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, SHAEP (Rear), APO 413

RESTRICTED

54p. diagrs.

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1. INVESTIGATION PROCEDURE.

On 5 April 1945, Capt. C. C. Chaffee of CACT No. 5, 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

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. Krayer, had not been at all informative; he was of the opinion that Dr. Krayer 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 Brüggenmann who was engaged in the erection of low temperature carbonization plants in Japan until 1942. Brüggenmann 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 KK., 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.

Manshu Gosei Nenryo, Chinchow (Kingsu). This plant is also known as Fushin. Hubmann believed that this Fischer-Tropsch plant probably started producing in 1940. It was a standard size Buhchemie 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 CLOS 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 carbonization 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 Chisso 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., Ssuningkai. 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%.

Notes:

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.

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 Bräx 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 Bräx 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 Bräx 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 Gutehoffnungshutte. (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

Pressure could be controlled to give the desired synthesis temperature. This unit was operated under varied conditions with a Ruhrchemie 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 fraction 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 50-60% 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.

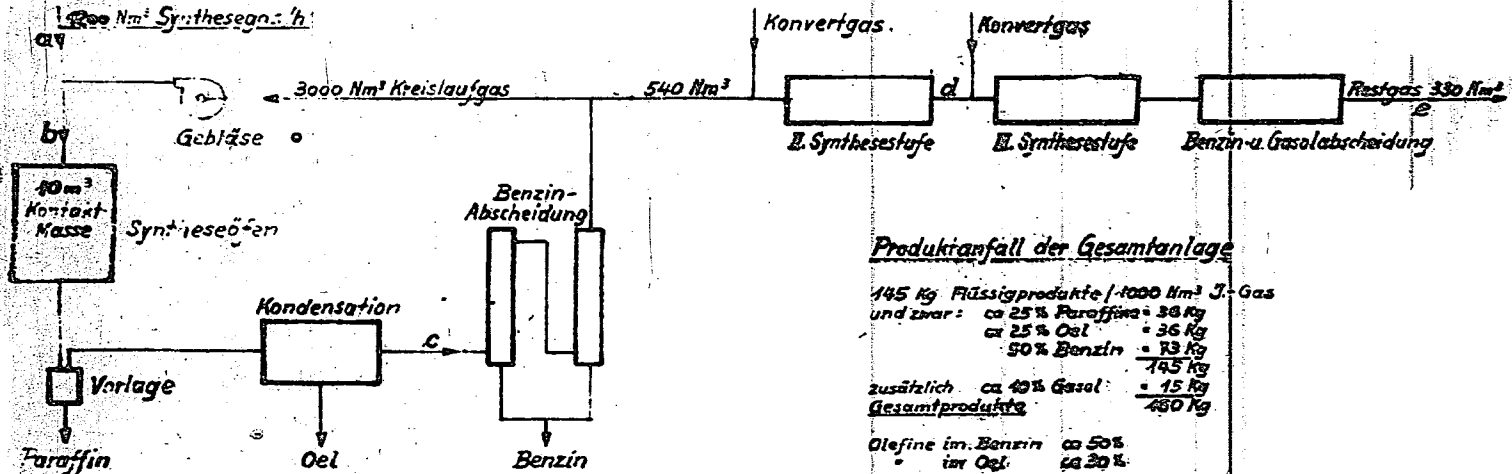
Figure 7, page 15, illustrates the conventional middle pressure reactor preferred for such operations. It was stated that six different engineering firms had collaborated in developing this reactor. Its upper limit of operating temperature is 230 C. and it is doubtful if it can stand much more than the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

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

100 Fe, 25 Cu, 9 Al₂O₃, 2 K₂O 30 SiO₂

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 10% 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 Deutsch 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 K₂O. 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



Produktanfall der Gesamtanlage

145 Kg Flüssigprodukte / 1000 Nm³ J.-Gas
 und zwar: ca 25% Paraffine = 36 Kg
 ca 25% Oel = 36 Kg
 50% Benzin = 73 Kg
 145 Kg
 zusätzlich ca 40% Gasol = 15 Kg
 Gesamtprodukte 160 Kg

Olefine im Benzin ca 50%
 im Oel ca 30%

REACTION CONDITIONS

PRESSURE 7-10 atm

TEMPERATURE 190-225°C

CATALYST 100g 5% Co, 8% Ni, 200 g SiO₂

Reaktionsbedingungen:

Druck 7+10 atü
 Temperatur 190-225°
 Katalysator 100 Co : 5 ThO₂ : 8 MgO : 200 Kgr

Gasanalysen

Frischgas a

CO₂ 10,5
 CO 31,8
 H₂ 51,4
 CH₄ 0,4
 N₂ 5,9

Kontakteneintrittsgas b

CO₂ 19,7
 C₂H₄ 0,7
 CO 28,3
 H₂ 35,4
 CH₄ 5,2
 N₂ 10,7

Endgas hinter I. Stufe c

CO₂ 23,5
 C₂H₄ 1,0
 CO 26,9
 H₂ 28,9
 CH₄ 7,0
 N₂ 12,7

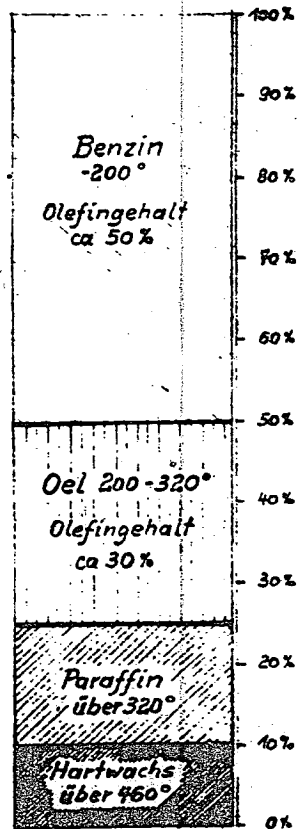
Endgas hinter II. Stufe d

CO₂ 37,4
 C₂H₄ 1,1
 CO 16,9
 H₂ 12,1
 CH₄ 13,1
 N₂ 19,7

Restgas hinter II. Stufe e

CO₂ 44,8
 C₂H₄ 0,8
 CO 9,5
 H₂ 6,1
 CH₄ 16,3
 N₂ 22,5

Zusammensetzung der flüssigen Produkte



Kohlenwasserstoffbilanz bez. auf 1 Liter ungasartiges CO+H₂

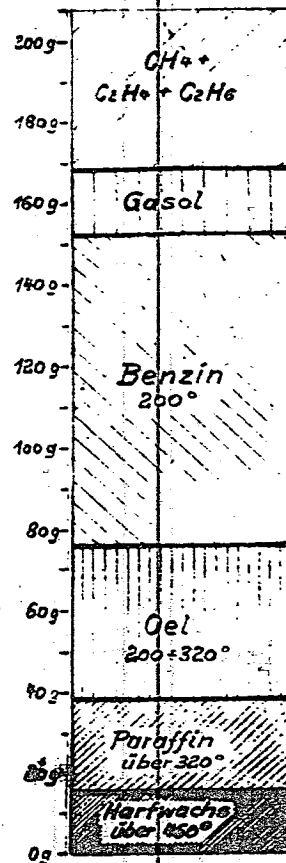
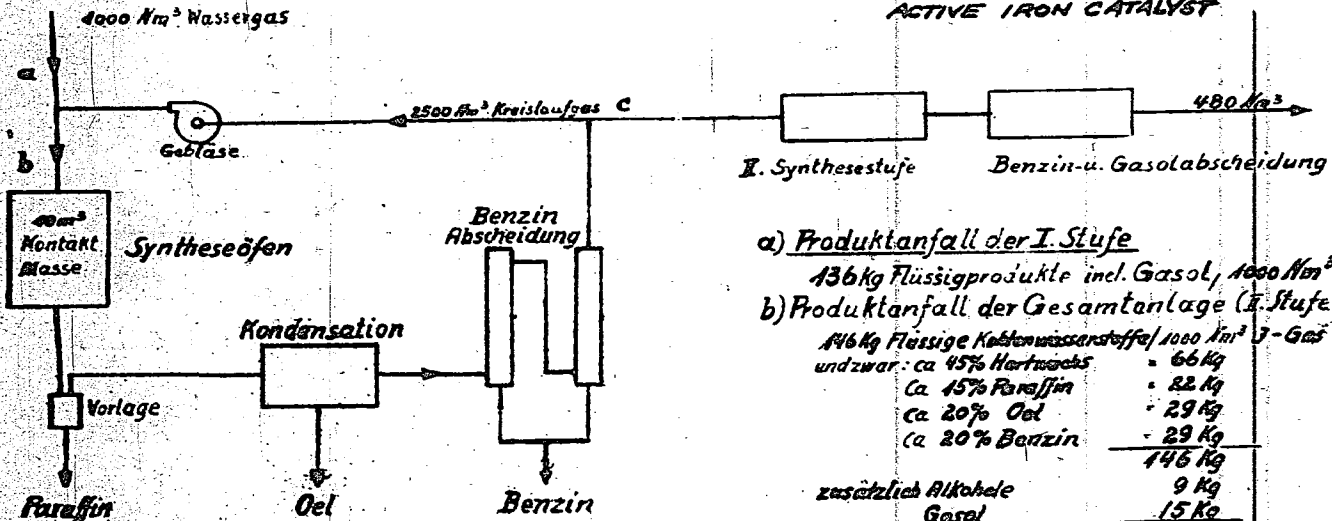


FIGURE 2
 CHARGE AND PRODUCT
 DATA
 PERTAINING TO FIG. 1

FIGURE 3
 RECYCLE SYNTHESIS
 WITH
 ACTIVE IRON CATALYST



REACTION CONDITIONS

Pressure 20 atm

Temperature 250°C

Catalyst 100Fe, 25Co, 9Al₂O₃, 2K₂O, 3As₂O₃

a) Produktanfall der I. Stufe

136 Kg Flüssigprodukte incl. Gasol, 1000 Nm³ J-Gas

b) Produktanfall der Gesamtanlage (I. Stufe errechnet)

146 Kg Flüssige Kohlenwasserstoffe/1000 Nm³ J-Gas

und zwar: ca 45% Hartwachs = 66 Kg

ca 15% Paraffin = 22 Kg

ca 20% Öl = 29 Kg

ca 20% Benzin = 29 Kg

146 Kg

zusätzlich Alkohole

9 Kg

Gasol

15 Kg

Gesamtprodukte

170 Kg

Ölfine im Benzin = 60%

Ölfine im Öl = 45%

Reaktionsbedingungen:

Druck: 20 atü

Temperatur: 230°

Katalysator: 100Fe:25Cu:9Al:2Os

2K₂O 30SiO₂

Gasanalysen:

Frischgas a

CO₂ = 5.8

CO = 37.6

H₂ = 48.1

CH₄ = 0.1

N₂ = 8.4

Ofeneintrittsgas b

CO₂ = 22.9

C₂H₄ = 1.4

CO = 26.9

H₂ = 34.8

CH₄ = 2.0

N₂ = 15.0

Restgas I c

CO₂ = 28.2

C₂H₄ = 1.9

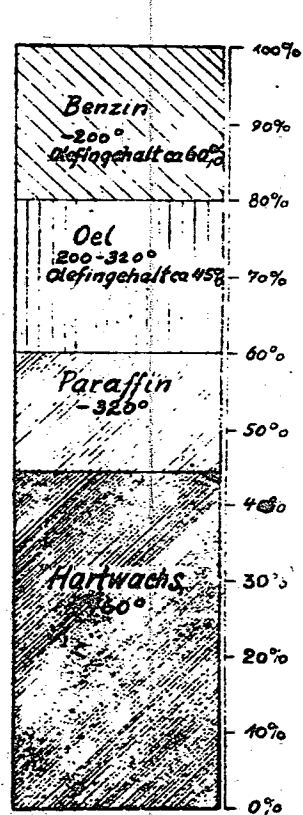
CO = 22.6

H₂ = 27.0

CH₄ = 2.7

N₂ = 17.6

Zusammensetzung der flüssigen Produkte



Kohlenwasserstoffbilanz bez. auf 1 Mol eingesetztes C₂H₂

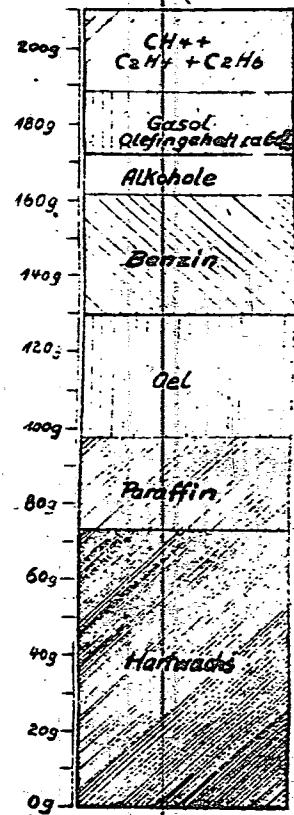
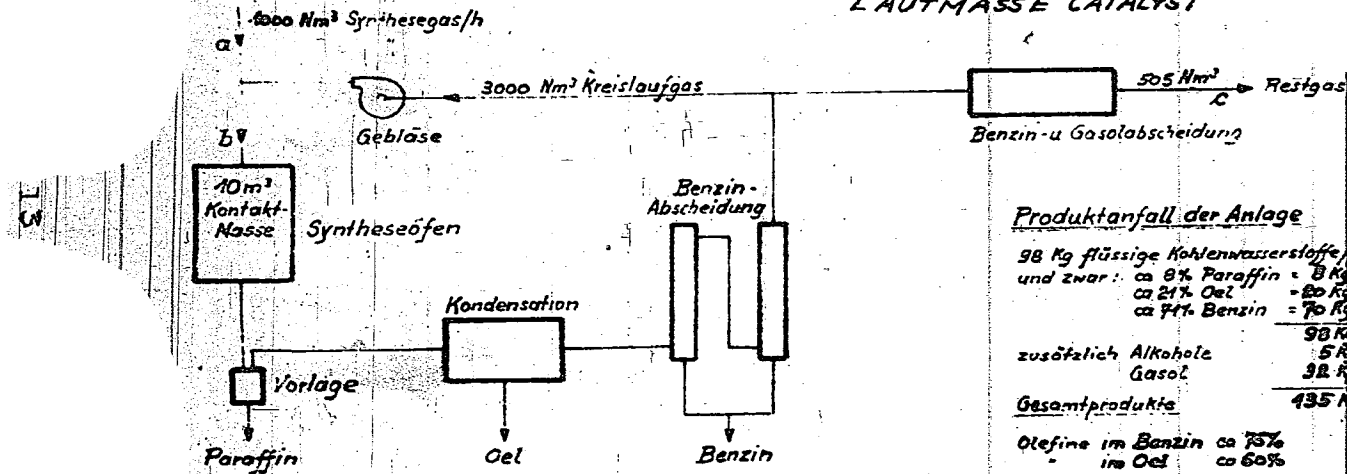


FIGURE 4
CHARGE AND PRODUCT
DATA
PERTAINING TO FIG 3

FIGURE 5
 RECYCLE SYNTHESIS
 WITH
 LAUTMASSE CATALYST



Produktanfall der Anlage

98 kg flüssige Kohlenwasserstoffe / 4000 Nm³ I.-Gas
 und zwar: ca 8% Paraffin = 8 kg
 ca 21% Öl = 21 kg
 ca 71% Benzin = 70 kg

zusätzlich Alkohole 98 kg
 Gasöl 5 kg
 92 kg

Gesamtprodukte 435 kg

Olefine im Benzin ca 75%
 im Öl ca 60%

Reaction Conditions

Pressure 20 atm.

Temperature 275°C

Catalyst Lautmasse impregnated
 with 3% Copper.

Reaktionsbedingungen

Druck : 20 atü
 Temperatur : 275°C
 Katalysator : Laufmasse imprägn.
 mit 3% Cu

Gasanalysen

Frischgas a

CO₂ : 2,8
 CO : 53,2
 H₂ : 35,6
 CH₄ : 0,1
 N₂ : 8,3

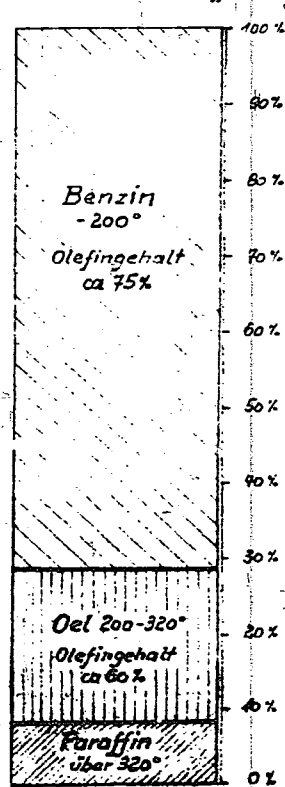
Kontaktofeneintrittsgas b

CO₂ : 48,0
 C_nH_m : 2,2
 CO : 46,7
 H₂ : 11,3
 CH₄ : 5,3
 N₂ : 16,5

Restgas c

CO₂ : 31,6
 C_nH_m : 1,3
 CO : 29,8
 H₂ : 19,9
 CH₄ : 3,5
 N₂ : 14,0

Zusammensetzung der flüssigen Produkte (Kohlenwasserstoffe)



Kohlenwasserstoffbilanz bez auf 100% umgesetztes CO+H₂

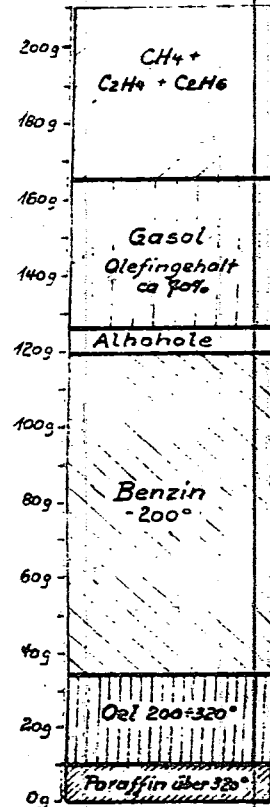
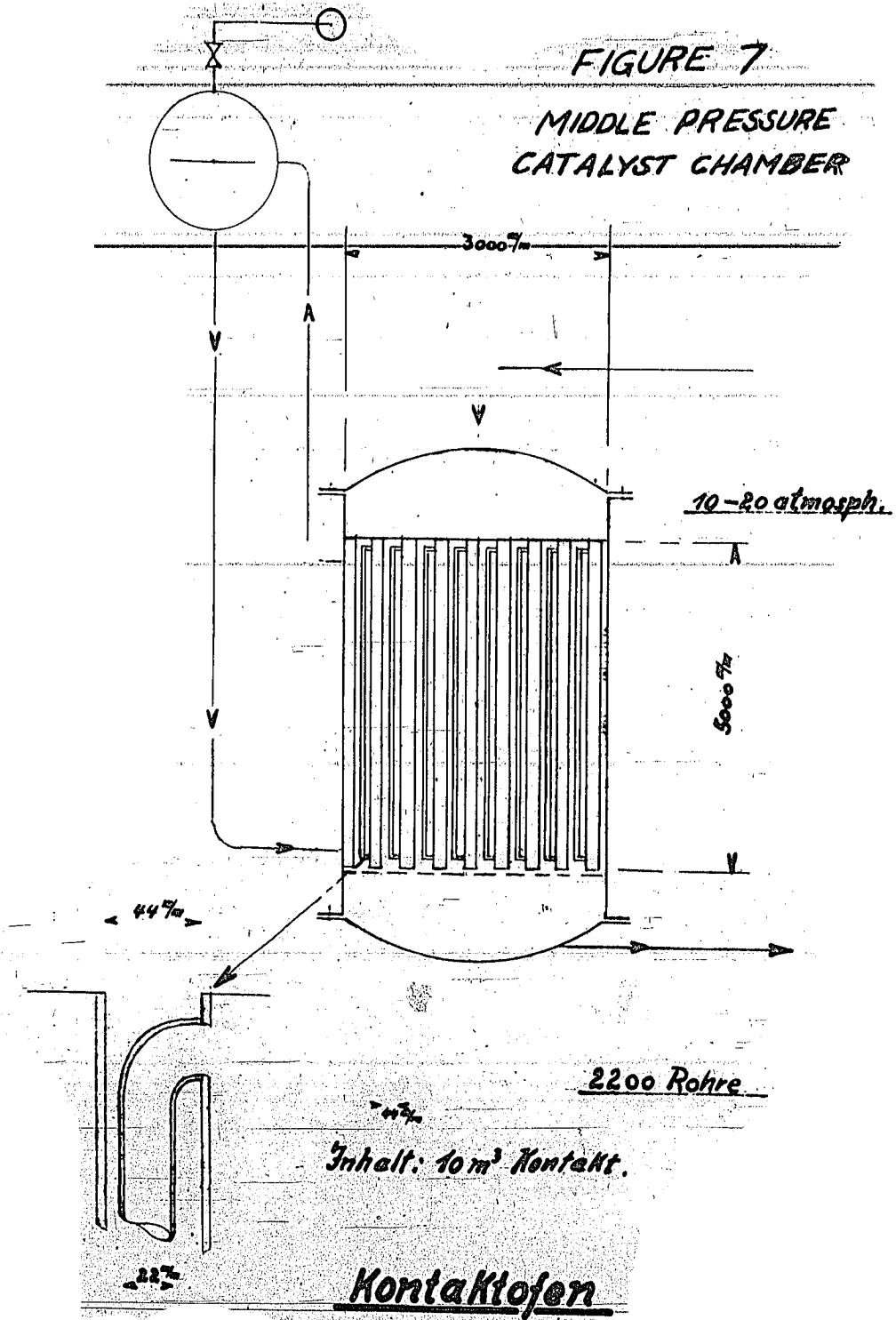


FIGURE 6
 CHARGE AND PRODUCT
 DATA
 PERTAINING TO FIG. 5

FIGURE 7
MIDDLE PRESSURE
CATALYST CHAMBER



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 benzol 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:

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

Lurgi had the contract for the decentralization of Fischer-Tropsch units under the Gellenberg 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 Gellenberg. The plan called for 12 ovens at Leipzig, and 2 or 3 each at Dresden (Heidenau), Goerlitz,

Gottbus, Pilsen, 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, $2Al_2O_3$, 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 Linde 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%),

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%. Earthy 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 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 Spülgas 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 "Spülgas" 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 Spülgas 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 Spülgas plant operating on brown coal briquettes containing 16% water and 14.3% tar, as determined in the Fischer-Hempel assay.

| <u>Input</u> | <u>Lb.</u> | <u>B.T.U.</u> | <u>% Heat</u> |
|-----------------------|--------------|---------------|---------------|
| 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 | 0.081 | 2.9 | 0.03 |
| Residue | 0.009 | 122.2 | 1.23 |
| Gas loss | 0.001 | 12.4 | 0.13 |
| Heat in cooling H ₂ 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.60°F.)

Characteristics of a Typical Spülgas-Process Tar

| Carbonization Temperature (Gas entering carboni- zation zone) | Distillation | | | |
|--|---------------------|-----------------|-------------------------|-----------------|
| | Excluding Light Oil | | Including Light Oil. | |
| | Up to 180°C. | Up to 320°C. | Up to 325°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% |

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

| <u>Carbonization Temperature</u> (Gas entering carbonization zone) | <u>Creosote & Asphalt Contents of Tar.</u> (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

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 those 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. | 30 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/m³ and a relatively low hydrogen content. Depending upon the coal, the gas-yield is 70-120 Nm³/ton.

The recovered benzin 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 ³ Gas | 320 Nm ³ Gas |
| 8000 Kcal/Nm ³ (gross) | 4600 Kcal/Nm ³ (gross) |
| 11 kg. Crude Gasoline | 11 kg. ammonium sulphate. |
| 60 kg. Tar usable as heating oil. | 9 kg. Crude light oil |
| | 34 kg. tar. |

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 Moist Coal 0.846 to 1 m³

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. - 9.8%~~

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% |
| | | <hr/> |
| | | 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 H₂ 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.

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

| | | |
|--------------------------------|---|--------------------------|
| Carbon dioxide | - | 4.5% |
| C _n H _{2n} | - | 2.5% |
| C _x H _y | - | 0.8% |
| Oxygen | - | 0.2% |
| Carbon Monoxide | - | 3.5% |
| Hydrogen | - | 23.8% |
| Ethane | - | 13.6% |
| Methane | - | 46.1% |
| Nitrogen | - | 5.0% |
| Density | - | 0.805 |
| Gross Heating Value | - | 8137 Kcal/m ³ |

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 m³ per ton of said coke:-

| | | |
|-------------------------------|---|--------------------------|
| C _x H _y | - | 0.3% |
| Carbon Monoxide | - | 7.4% |
| Hydrogen | - | 77.2% |
| Methane | - | 12.5% |
| Nitrogen | - | 2.0% |
| Gross Heating Value | - | 3840 Kcal/m ³ |

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 ash 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 (4000-4800 Kcal/Nm³) that is, substantially equivalent to coke-oven gas. A typical crude gas and its purified

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

| | <u>Crude Gas</u> | <u>Purified Gas.</u> |
|-------------------------------|------------------|----------------------|
| CO ₂ | 30.6% by vol. | 3.0% by vol. |
| C _n H _m | 0.6 | 0.5 |
| O ₂ | 0.1 | 0.1 |
| CO | 16.5 | 22.8 |
| H ₂ | 34.0 | 48.7 |
| CH ₄ | 16.3 | 22.6 |
| N ₂ , 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 m² 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/Nm³ 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 H₂ or CH₄ are always accompanied by the production of the inerts CO₂ and H₂O; and the high conversion of chemical energy is thus not influenced by large CO₂ formation.

The pressure-generator gas has the following characteristics:-

- a) Relative Density (Air = 1) less than 0.5;
- b) Oxygen content " " 0.3% by vol.;
- c) It is substantially free of H₂S, NH₃, Naphthalene and tar;
- d) Its content of organic sulphur when employing high-sulphur coals is less than 20g/100 Nm³ and by strong aeration of the water employed for removing the CO₂ it can be further reduced.

In consequence of this scrubbing of the crude gas with water under pressure, the effluent gas contains not more than 5-10 grams $H_2S/100 Nm^3$; 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:

1. Hirschfelde near Zittau: This was the first commercial plant and had a capacity of 5 million m^3 per year of City gas.
2. Böhlen near Leipzig: Two plants having a total capacity of 150 million m^3 per year.
3. Brax in Czechoslovakia: This plant was designed for a capacity of 100 million m^3 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 Brax 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.S.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-

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 V₂A 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 m² cross-section when operating at 20-30 atm. can gasify:-

100 to 130 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 Nm³ city gas per hour, or 20,000,000 Nm³
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 Nm³ gas having a gross
heating value of 4300-4500 Kcal/m³.

1 ton lean coal yields 1570-1670 Nm³ having a
gross heating value of 4100-4300 Kcal/m³.

~~1 ton lean coal yields 1570-1670 Nm³ having a
gross heating value of 4100-4300 Kcal/m³.~~

Results of Pressure-Generator Operations on Various Fuels.

| | <u>Brown Coal</u> (Lausitz) | <u>Brown Coal</u> (Middle Germany) | <u>Lean Coal</u> Fines (Ruhr) |
|--|--------------------------------|---------------------------------------|-------------------------------------|
| 1. Composition of the Fuel | | | |
| 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 |
| 2. Operating Pressure at. | | | |
| | 20 | 20 | 20 |
| 3. Throughput Kg/m²h | | | |
| | 750 | 770 | 310 |
| 4. Composition of Purified Gas. | | | |
| CO ₂ % | 3.0 | 2.3 | 1.0 |
| C _n H _m % | 0.5 | 0.9 | 0.3 |
| O ₂ % | 0.1 | 0.2 | - |
| CO % | 22.8 | 22.0 | 27.9 |
| H ₂ % | 48.7 | 50.7 | 52.4 |
| CH ₄ % | 22.6 | 21.8 | 16.9 |
| N ₂ % | 2.3 | 2.1 | 1.5 |
| Density Gas (air = 1) | 0.448 | 0.435 | 0.432 |
| Gross heating value Kcal/Nm ³ | 4280 | 4500 | 4100 |
| 5. Gas Yield Nm³/to coal | | | |
| | 760 | 680 | 1690 |

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 ³ /Nm ³ Gas | 0.15 | 0.145 | 0.198 * |
| 7. Steam requirement Kg/M ³ gas | 0.01 | 1.06 | 1.40 |

* For comparative purpose, it is noted that the Winkler Generator employs slightly more than twice as much oxygen per m³ 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/M³

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 8-9 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 m³ of water per hour per 6000 m³ 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 m³ 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 Nm³ 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 |
| | <hr/> |
| | 100% |

B. Heat Output

| | |
|--|-------|
| 1. Chemically combined heat in the town gas. | 62.2% |
| 2. In tar and benzine | 14.3% |
| 3. Phenols in gas liquor | 0.9% |
| 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% |
| | <hr/> |
| | 100% |

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 m³ 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.

Synthesis Gas.

In the Fischer-Tropsch synthesis of hydrocarbons from CO and H₂ as usually practised with the cobalt-thoria catalyst, the employed Synthesis Gas preferably has a H₂/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 H₂/CO₂ 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/CO₂ 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 ₂ | - | 29.3% |
| CO | - | 21.8% |
| H ₂ | - | 44.0% |
| CH ₄ | - | 3.3% |
| N ₂ | - | 1.5% |

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

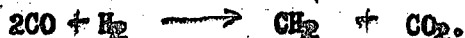
following composition:-

| | | |
|-----------------|---|-------|
| CO ₂ | - | 1.0% |
| CO | - | 30.7% |
| H ₂ | - | 61.8% |
| CH ₄ | - | 4.6% |
| H ₂ | - | 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 H₂/CO ratio differing from the requirement of the cobalt-thoria catalyst; the preferred synthesis pressure with the iron catalyst is 22 atm. and the H₂/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 H₂/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 (Bamag);

2. Pressure Gasification with Cracking of the Crude Gas (without Regenerators);
3. Atmospheric Gasification with addition of CO₂;
4. Atmospheric Gasification with addition of CO₂;
5. Pressure Gasification with Cracking of Crude Gas (with Regenerators);
6. Pressure Gasification with Cracking of Residue Gas (Koppers);
7. Pressure Gasification with CO₂ 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 CO₂ and some of the H₂S 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 H₂. 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 CO₂, the formed crude Synthesis Gas has then the following composition:-

| | | |
|-------------------------------|---|------|
| CO ₂ | - | 2.0% |
| C ₂ H ₆ | - | 0.2 |
| CO | - | 42.2 |
| H ₂ | - | 50.7 |
| CH ₄ | - | 2.4 |
| N ₂ | - | 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 m³ thereof, 0.317 m³ 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 H₂/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, Boshlen 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

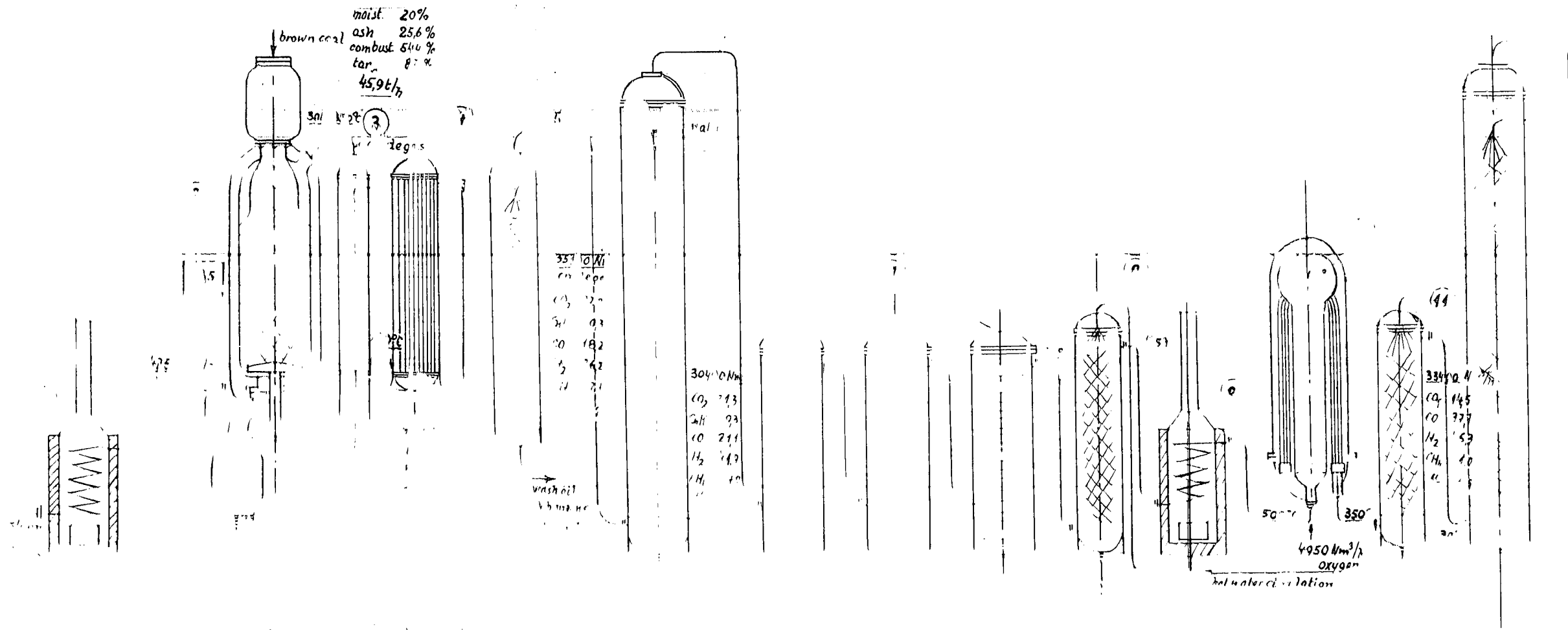
the Societa Italiana Carburanti Sintetici project, which was to use the Lurgi Gasification process, was placed with Messer, not because their estimate of power consumption was lower than that of Linde (0.536 as compared with 0.56 Kw. per Nm³ of O₂), but because Linde was unable to undertake the contract.

For the purpose of estimating the cost of oxygen production, Lurgi use a figure of 0.65-0.68 KwH per Nm³ 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 Nm³ per hour capacity was estimated as 2.2 to 2.5 pfg. per Nm³ 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 Nm³ 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 Nm³ 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:-



- ① steam superheater
- ② gas producer
- ③ spray cooler
- ④ tube cooler
- ⑤ benzine scrubber
- ⑥ water wash tower I
- ⑦ extraction of H₂S
- 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

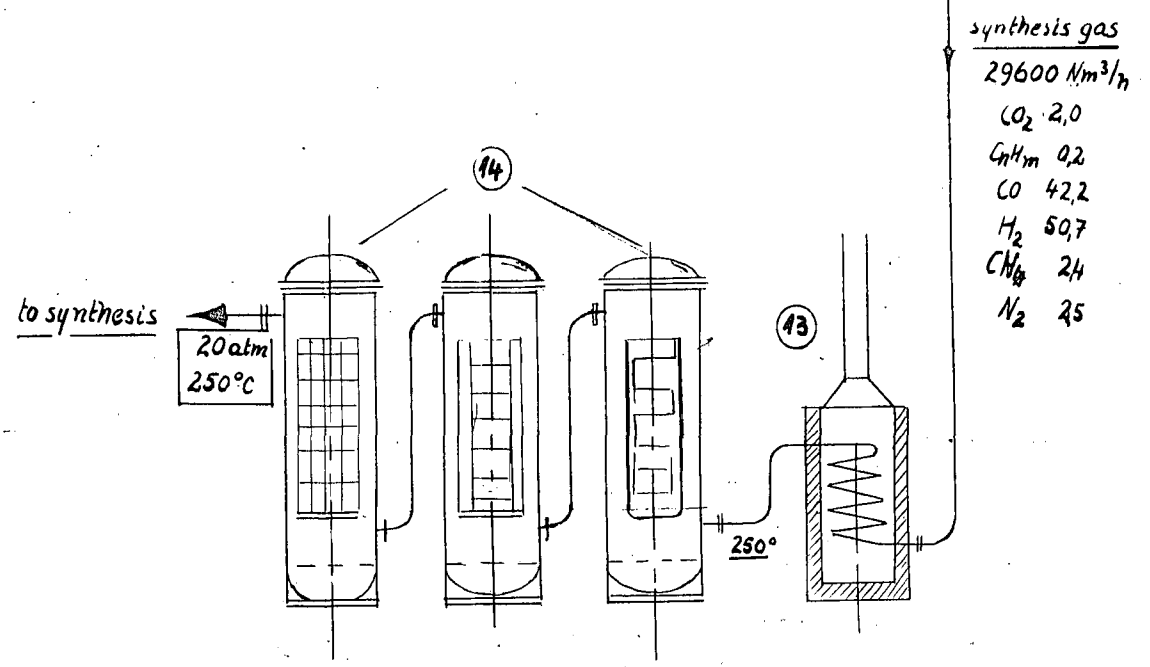


Fig. No. 8.

Lurgi Gasification Process.

Ffm. 4.12.45 Dec

(1) Compressors and Blowers:-

Gutthoffnungshütte, Oberhausen,
Maschinenfabrik SAAG, Essen

(2) Evaporating Apparatus

Linde Werke, Hiltrup, Dortmund

(3) Injection Machines

DeVandell, Berlin-Tempelhof

(4) Aluminum Packing for the Regener

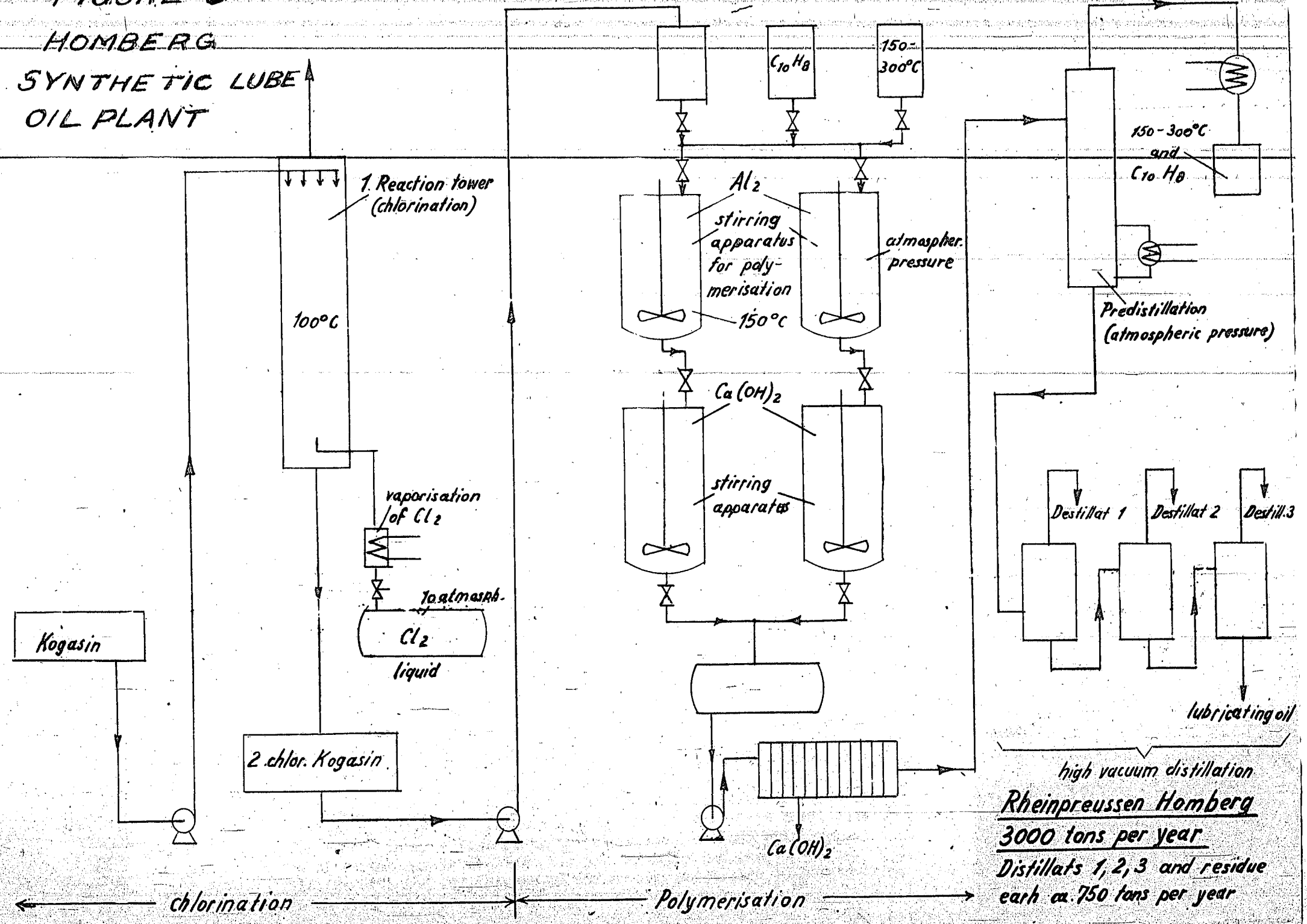
Kolbel, Augsburg

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 Hedderneim, 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.

FIGURE 9
HOMBERG
SYNTHETIC LUBE
OIL PLANT



high vacuum distillation
Rheinpreussen Homberg
3000 tons per year
Distillats 1, 2, 3 and residue
each ca. 750 tons per year

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 DuPont 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 Ossag 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.G. 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.

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 Boehlen. 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 Hubda 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.Göhre
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.Herbe
Power Plants: Dipl.Ing.Müller
Dr.Rueping.

Lurgi Werkstätten G.m.b.H.

Dr.Räder
Dir.Wätcher
Dr.Oetken.

The activities of the Lurgi Companies are as follows:-

Lurgi-Gesellschaft für Chemie und Hüttenwesen:

Construction of Sintering Plants (Dwight-Lloyd)

" " Roasters

" " Sulphuric Acid Plants.

Apparatus for the Cellulose Industry.

Various Process for Lead and Zinc Smelters.

Lurgi-Apparatebau G.m.b.H.

Cottrell Electrical Precipitators for Gas Purification

Mechanical De-dusters.

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.

b) Steam Engineering.

Evaporating Plants.

Drying Plants.

Crystallization Plants.

Plants for Treatment of Vegetable Oils and Fats.

Distillation Plants.

Extraction Plants.

c) Recovery of Solvents (Active Carbon).

APPENDIX B.

LABORATORIES OF METALLGESELLSCHAFT A.G.

MAIN RESEARCH (Z.F.)

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

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

- 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)

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)

Zinc Alloys.

- a. Castings
- b. Forging Material.

Bearing Metals
Lead Alloys.

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

Principal Office:- Erbstadt.

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

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.

b. Department of Surface Phenomena.

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

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 C.

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

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 H₂S and O₂
- 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.

- 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.

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 benzine 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-Benzine.
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-Benzin.
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 4182 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.