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ITEM No. 30

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Lurgi Gesellschaft Fur
Warmetechnik
Frankfurt-am-Main

Hollings & King

RESTRICTED

COMBINED INTELLIGENCE OBJECTIVES

SUB-COMMITTEE

RESTRICTED

LURGI GESELLSCHAFT FÜR WÄRMETECHNIK

LURGI HOUSE, FRANKFURT-AM-MAIN

Reported by
DR. H. HOLLINGS

and

DR. J. G. KING

on behalf of

BRITISH MINISTRY OF FUEL AND POWER

and

U.S. TECHNICAL AND INDUSTRIAL INTELLIGENCE COMMITTEE

[1945]

CIOS Target No. 30/6.08
Fuels and Lubricants

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

RESTRICTED

12 p.

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PERSONNEL OF TEAMS

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This report deals with the activities of the Lurgi Company in the fields of low temperature carbonisation, active carbon, distillation of light hydrocarbons, high pressure gasification and Fischer Tropsch synthesis. It contains also some information concerning the cost of oxygen production. The information was collected by Dr. H. Hollings and Messrs. J.H.G. Plant and R.N. Quirk on April 12th-13th and by Dr. J.G. King, Mr. H. Bardgett and Dr. E. Spivey on May 5th and 6th, 1945.

The personnel interviewed were:-

Dir. Ötken	- head of Wärmetechnik
Dr. Danulat	- high pressure gasification
Dr. Herbert	- Chief Chemist
Dr. Heine	- low temperature carbonisation
Dr. Röpzig	- active carbon
Dr. Siebert	- distillation

Visits of inspection were paid to Dr. Herbert's research laboratories at Mouson Strasse and evacuation premises at the Technische Hochschule at Friedburg where Dr. Siebert was researching on distillation problems and Dr. Danulat's section had been working before being bombed out.

1. THE ORGANISATION

The whole group of Lurgi companies is a subsidiary of the Metallgesellschaft, a Company with very wide interests in metals such as copper, zinc, etc., in finance, insurance, etc.

Director Dr. Ötken explained that there were four subsidiaries of the Lurgi organisation.

- (i) Gesellschaft für Chemie und Hüttenwesen, dealing with the metallurgical industries, paper, pulp, sulphuric acid, fertilisers, etc.
- (ii) Apparatebau Gesellschaft, covering for instance electrical precipitation plant for gas purification.
- (iii) Gesellschaft für Wärmetechnik - consisting of a number of divisions of which the principal were concerned with
 - (a) Drying and concentrating processes; refining of fats and vegetable oils; distillation, etc.
 - (b) Distillation of mineral oils including propane and butane (see later - there were large plants at Brück and Gelsenberg); Recovery of phenols from effluent liquors.

- (c) Producer gas, high pressure gasification for towns gas.
 - (d) Low temperature carbonisation of hard coal, brown coal and oil shale.
 - (e) Active Carbon.
- (iv) Lurgi Werkstätten Gesellschaft - Manufacture of electro-filters - workshops and laboratories.

2. LOW TEMPERATURE CARBONISATION

Dr. Ötken gave the following information on Low Temperature Carbonisation:-

Under the four year plan "Spülgas" plants, similar to those erected before the war in Canada and New Zealand, had been built;

- (i) for soft brown coal at Offleben
Znachterstedt
Deuben
Profen
Regis (this plant makes briquettes by the Krupp ring roller press)
Deutzen
Böhlen
Hirschfelde - also pressure gasification
Rositz
Webau
Koeppen
Leuna
Magdeburg
Zeitz
Lützkendorf
Espenhain - see later
- (ii) for hard brown coal (glanz coal) at Brück
- (iii) for non-coking or slightly coking bituminous coal at Blechhammer, Karsten-Zentrum Grube, Auschwitz (I.C. Chem. works).

There are none of these plants in the Ruhr, except that at Hoesch Benzin there was an experimental plant.

Krupp-Lurgi L.T.C. plants with as one of the main objects naval fuel oil, were erected at:

Wanne-Eickel - the only one in the Ruhr
Velsen in the Saar

and Heinitz -- a pilot plant replacing the earlier Salerno plant. It is not certain if it is working now.

Here it may be noted that the Wanne Eickel Krupp Lurgi L.T.C. plant was inspected on April 16th. In 1939 there were 56 "old" ovens obtained from Lurgi. Some of these were destroyed and then 24 "new" ovens were put to work. These incorporated an entirely different bottom door. There was one door (instead of two) for each 6 chambers and this was removed on a truck. Immediately above the door was a large iron grid supporting the coke in each chamber. After taking away the door the grid was moved horizontally a few inches so as to leave the coke free to fall. Then the coke was pushed down from the top. There were some alterations to the heating flues, but details were not obtained. The heating gas temperatures were given as 610/620°C inlet and 560/580° outlet.

Only a part of the L.T. coke went to the water gas generators (the rest sold for domestic use) and only a part of the synthesis gas was made from L.T. coke. In reply to questions as to the advantage of L.T. coke, it was stated that it gave a H₂/CO ratio of 1.35 (c.f. 1.25/1.20 with H.T. coke). The generator operates at a lower temperature. There is less trouble with clinker. On a coal basis credit is taken for the L.T. tar and spirit. The gas output from the generator is, however, reduced.

In the field of L.T.C. it was learnt from Dr. Otken that a Borsig-Geissen process was started in 1937 at Kilkwitz but it has been discontinued. This was not a Lurgi plant.

There is also a Brennstoffe-Technik design which is similar to Krupp Lurgi, but in which coke is discharged by moving the retort wall instead of by a vertical pusher. This plant is installed at:

Marienau (Saar)
Mizluwitz Grube (Silesia)

The Lurgi Co. took over a tunnel furnace design for Estonian shale. There were several under construction when the Russians occupied Estonia. In Germany there is one plant at Heide in Holstein.

In the Lurgi-Schweitzer system for carbonising shale there is sufficient internal heating of the retort to distil off the oil - a mixture of air and steam is admitted. The only plant is at Schömberg (Württemberg).

Near Schömberg "Government engineers" have built piles similar to charcoal Meilers.

Dr. Heine explained that the Lurgi-Spülgas process had been applied successfully to bituminous coal. At Blechhammer the 4,200 tons plant (300 tons per generator per day) had 5 of the generators working on slightly-caking coal making hard coke for transport producers and hydrogen production. The Meuric index of the coal was less than 12 and the smalls below 6 m.m. were briquetted with 4% sulphite lye and 2% pitch before carbonising. The tar (10% yield) was divided into asphalt, acids and "navy oil" over 230°C (6/7 per cent). The economics of the process were termed "doubtful", when hard coke fetched 22 Mk., Lurgi coke fetched 25 Mk. and producer fuel 28 Mk., the cost of the coal being 15 Mk. Obsolescence was taken at 2/3 years.

Dr. Heine's opinion was that the Krupp-Lurgi process was the most satisfactory for weakly caking coal and the Spülgas process best for briquettes.

Experimental work had been done on tar treatment using cold extraction methods. Plants had been under erection at Hirschfelde and Altenberg. The process had been applied only to brown coal tar and involved.

- (i) extraction of tar with amyl and butyl acetates to recover phenols.
- (ii) 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 suitable for blending with Fischer-Tropsch oil.

Dr. Herbert explained that the research work had been done in his laboratory at Mouson Strasse.

Dr. Otken when outlining his general views on the future of liquid fuel technology, suggested that more attention should be paid to low temperature carbonisation. He included bituminous coal in his remarks. The coke would go for domestic use and he thought that the tar would repay more research. This led him to draw our attention to the application of the Edeleanau process of SO₂ solvent extraction to low temperature tar derived from brown coal at Espenhain, where there is a Lurgi Spülgas L.T.C. plant, regarded as pre-treating brown coal used for power production. Half the tar produced goes to a hydrogenation plant and the other half to the extraction plant which started in 1942.

This extraction process has not been applied to bituminous coal, but on the basis of their experience, Lurgi would be prepared to submit a scheme.

3. ACTIVE CARBON

Dr. Ötken, Dr. Rüping and Dr. Herbert gave the following information about active carbon.

The carbon was mainly brands manufactured by the I.G. at Leverkusen, the experts being Drs. Neimann and Döptke. There had been no outstanding improvements in quality recently.

The plant working under pressure at Nordstern had been moved to Alma Gelsenkirchen and, in reply to direct questions, Dr. Rüping said he was quite sure there was no other similar plant. Further, he was not aware of any special plants at coke ovens for the extraction of hydrocarbons, such as butane. The use of carbon under 10 ats. pressure had really been worked out for the Fischer plants in which gas recirculation was employed.

There had been an intensive laboratory study by Lurgi of the extraction of ethylene, etc., by oil and by carbon at pressures from normal to 20 ats. Up to 10 ats. the use of carbon was considered more economical than oil. "S-Kohle" was used.

At 20 ats. pressure, the Lurgi oil washing process is preferred to the carbon process. In this compressed gas is washed with benzole to remove the light fractions, including ethylene, and then with oil to remove benzole.

Dr. Herbert and Rüping were emphatic that no commercial plant had been erected (several were projected but steel could not be released) except that at Nordstern there had been some adaptation of an existing oil washing plant - hence the removal of the carbon plant to Alma.

A recent advance had been the design of plant to work at -50°C , the objective being the separation of methane. Reference was made to similar work by the Brit. Magnesium Corp. at Swansea designed to keep the CO percentage in gas below 5 per cent. Carbon for the purpose should be highly reactive (S. Kohle or nut shell charcoal). Studies made in conjunction with Linde Co. indicated costs to be slightly less than those of refrigeration.

Reference was made to a plant in the Gas Works at Nuremberg to produce 3000m^3 of methane per day (purity 85%), the expert there being Dr. Ipfelkopfer.

The main development in the active carbon field during the war had been the application of automatic control, particularly in Fischer plants.

Dr. Herbert expressed the view that, where Fischer synthesis gas was derived from the carbonisation of coal, it still contained gum forming substances, which should be removed from the gas before the final purification for sulphur removal. Further information was stated to be available at Kamen (Essenersteinkohle) and Schaffgotsch.

4. DISTILLATION OF LIGHT HYDROCARBONS

Dr. Otken was asked what had been the activities of Lurgi in the field of distillation of light hydrocarbons. He said they had their own process - briefly the special feature is the removal of permanent gas before fractionation. This had been installed to deal with the by-products from every Fischer plant (except Schaffgotsch), and from some hydrogenation plants. Also the process had been installed to deal with natural gas. Iso-butane and iso-octane were prepared from the products, or mixtures were made.

~~There were no such plants installed in connection with carbonisation plants except possibly where coke oven gas is used as a source of synthesis gas.~~

Dr. Siebert was interviewed at Friedburg and his laboratory inspected. The methods of separation were not novel, even the claim to separate permanent gas before fractionation. The distillation curves showed very sharp separation of certain hydrocarbons. Dr. Siebert's ideas had not yet been developed for full-scale use but were intended mainly for gasol separations.

5. HIGH PRESSURE GASIFICATION

Dr. Otken and Dr. Danulat gave the following information on High Pressure Gasification:-

The first plant started at Hirschfelde (Zittau) before the war has continued to work satisfactorily. The only new plants were at Böhlen (1939) and Brück (1942), capacity 150 and 100 million cu.m. per year respectively. In these plants the internal diameter of the generator has been increased from 1.24 to 2.5 m. Full details of the plant at Böhlen appear in the report of target 30/4.05 and some drawings of the Brück plant were among the official documents seized at Böhlen. The Brück generators, which have not been seen, have an internal hydraulic ram in the coal pouch for opening the upper valve. Dr. Otken considers the process is now foolproof, but only suitable for larger central works. The plants are operated with a uniform output summer and winter and supply gas into a grid.

These plants are gasifying brown coal and Dr. Otken evidently considered the financial results depended on the yields of by-products. He indicated the results would be very different

~~with an anthracite coal. Bituminous coal had been gasified successfully (Karsten - Zentrum area) but optimum conditions had not been established.~~

The use of high pressure gasification for synthesis gas has not been applied on the large scale although the Italian order had been secured.

The process uses from 0.5 to 0.2 cu.m. or less oxygen per cu.m. of gas made. Under the conditions for manufacture of town gas there is more exothermic production of methane and, therefore, a lower oxygen consumption. In any case it is claimed the oxygen consumption is lower than in the Winkler process, which Dr. Otken does not like.

Dr. Danulat referred to fundamental work published in 1942 and 1943 in Gas u. Wasserfach in which reaction equilibria had been studied and to a paper in which he had suggested an alternative to the Dent process of hydrogenation. On the question of oxygen purity he stated that the higher the reactivity of the fuel the lower could be the oxygen purity. At Zittau the first plant had used oxygen of 72-75 per cent O₂. Dr. Danulat said the best way of making synthesis gas in this process would be to make normal gas at 20 atms. and then crack the CH₄ at about 1200°C.

Referring to the limiting size of a Lurgi generator Dr. Danulat said the only limit to diameter was imposed by considerations of travel on the railway. The max. at present was 2.5m. (3.0m.o.d.). He suggested a pilot plant to work at 40 atmos.

It was stated that Ruhrchemie had considered this process for production of synthesis gas from bituminous coal, but no plant had been installed; for reasons explained elsewhere there have been no new Fischer plants recently.

In a general outline of his views on future developments, Dr. Otken said Lurgi had a pilot plant at Böhlen working under Dr. Herbert, which was designed:

- (i) To increase the calorific value of gas, particularly that from high pressure gasification. (The increase was only 200/300 cal. per cu.m.)
- (ii) As a means of gas detoxification.
- (iii) To produce 40 gms. of liquid fuel per cu.m. of gas.

An iron catalyst made by Lurgi is used (Fe 100, Cu 10, Al₂O₃ 9, Kieselguhr 120). It was pointed out that the proportions of the activators might be changed. The catalyst is used at a temperature 70° higher than in the Fischer process.

This pilot plant is described in the report on target 30/4.05.

6. LURGI AND FISCHER-TROPSCH

Dr. Otken and Dr. Herbert gave the following information:

(i) Position vis a vis Ruhrchemie

For some years Lurgi have been doing experimental work on various aspects of Fischer-Tropsch:-

- (a) recirculation of gases
- (b) 20 atms. operation
- (c) the iron catalyst

Lurgi (under the name of Metallgesellschaft) have been responsible for a number of patents. We were told that these patents had so frightened Ruhrchemie that they were induced to enter into a comprehensive Agreement with the Lurgi Company. Under this Agreement the Lurgi people are given rights in connection with the construction of future Fischer-Tropsch plants and there is to be some interchange of technical information. We secured the firm's original copy of this Agreement.

Ruhrchemie had not patented either recirculation nor had they made so much headway with pressure gasification and the use of iron catalysts. The advantages were stated to be:-

- (a) higher gas velocity and easier heat control
- (b) the hydrogenation of olefines to paraffins is stopped
- (c) cracking is suppressed by high CO/H₂ ratio giving both less CH₄ and less carbon.

The experimental work had been done by Dr. Herbert using single tubes of full size, which we saw at the laboratory at Mouson Strasse, and at the Hoesch-Benzin Plant.

The Lurgi company had definitely the full rights of building plants in the gas industry and nine had been completed, or started, in the Geilenberg programme.

(ii) Hydrogenation and Fischer-Tropsch

The Company admitted that the Fischer-Tropsch process had been subject to a good deal of criticism (a) because of the poor yields from the process at the start (b) because the petrol is low grade and (c) because Dr. Krauch and the I.G. staff at the Ministry were more favourable to hydrogenation.

The Lurgi people admit that hydrogenation is far more suitable for the production of high octane fuel. (Dr. Otken suggested that the trend towards high octane fuel, with engines specially designed for the use of these fuels, had perhaps been overdone.) But they appear to consider the Fischer Tropsch process of very considerable potential importance. They mentioned that the Fischer-Tropsch process was better than hydrogenation for the treatment of high ash coals and also that the wax and other products made it potentially of great interest from the chemical point of view.

(iii) The working of the Fischer-Tropsch process

Dr. Herbert said that operation under medium pressure enabled the quality of the product to be rather delicately controlled, particularly when combined (a) with recirculation of gases and (b) with the iron catalyst. On the other hand, under medium pressure operation with cobalt catalyst it was not very satisfactory.

He prepared certain diagrams, of which copies were taken, of the yields of products obtained (a) at 7-10 atms. with a cobalt catalyst and (b) and (c) at 20 atms. with two iron catalysts. The compositions of the products were quoted. For gas of high CO/H₂ ratio, water gas was made. The data are abstracted in the Table below which refers to processes employing recirculation in the first stage in every case.

<u>Catalyst</u>	<u>Co/Th₂</u>	<u>Fe/Cu</u>	<u>Lautemassee Cu.</u>
<u>Pressure Atm.</u>	7	20	20
<u>Temp. °C.</u>	190-225	230	275
<u>Stages</u>	3	2	1
<u>Oil Yield gm/m³ of CO + H₂</u>	145		165
<u>Product gm.</u>			
Hard Wax (over 460°)	15	66	-
Soft Wax (over 320°)	31	22	8
Oil	36	29	20
"Benzin"	73	29	70
Gasöl	15	15	32
Alcohol		9	5
	<u>160</u>	<u>170</u>	<u>135</u>
<u>Olefines in benzin %</u>	50	60	75
" " oil %	30	45	60

With the recirculation process under 10 ats. pressure, it was possible to restore the olefine content and to obtain the same products as under atmospheric pressure but with higher yield.

The iron catalyst was stated to work at 218-230°C. at 10 atmos. (H_2/CO ratio = 2). Ruhrchemie did not like it because of the high H_2/CO ratio and the difficulty of removing the liquid product rich in wax. Given recirculation the Fe catalyst was stated to work satisfactorily with water gas. Since it was less active than Co, however, the inerts in the gas had to be below 10 per cent. A second stage of conversion was necessary but no more; additional hydrogen was not necessary since the CO and H_2 were consumed at equal rates.

The catalyst has the composition:-

Fe, 100; Cu, 25; Al_2O_3 , 9; K_2O , 2; SiO_2 , 30

and is made by precipitating Fe, Cu, and Al nitrates from boiling solution with sodium carbonate in the presence of Silica. The precipitate is washed, potassium carbonate added to adjust the proportion of K_2O and the catalyst prepared as extruded cylinders. It was stated that the addition of some K_2O was not injurious to the catalyst but promoted the formation of hard wax. A life of 1 year was assured.

A further catalyst in which Lautamasse was activated with 3% copper had been tried at 20 atm. using a gas of 1.6 ratio. This required no second stage, giving 85% conversion in Stage 1 and 135 gm. per cu.m. The product contained 75% olefines.

(iv) Design of Catalyst Ovens

Operation at 20 ats. pressure required a rather high steam pressure at high steam temperature. There were those that argued that for these conditions, the double tube medium pressure oven was not very satisfactory. Dr. Herbert said that the atmospheric pressure Lamellen oven enclosed under 20 ats. pressure might have advantages, though Dr. Otken doubted this.

It was stated that 6 engineering firms had collaborated in producing the double-tube pressure chamber (24 mm. external with 44 mm. internal outer tube) but it was not known if it would stand 30 atmos. It was considered that its temperature limit was 230°C.

(v) Economics: It was stated that the most favourable financial position was achieved by working for the max. yield of hard wax; the average price of all products was stated to be 45 pf. per Kg.

Capital for new plant was put at 800-900 Marks per year tonne of liquid product (a recent plant was quoted at 600 Mk.).

The following costs were typical when using coke, steam, etc., at the figures shown.

		Pf. per Kg. product
Coke per tonne	24 Mk.	Gas 14
Steam " "	2 $\frac{1}{2}$ "	Cap. & Repairs 6
Power per kW.hr.	2 Pf.	Labour 2
Labour per hr.	1 Mk.	Steam 2
		<u>24</u>

7. OXYGEN PRODUCTION

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

The Messer Co. of Frankfurt 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 0.65 kWh. per Nm³ of O₂) but because Linde were 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 of oxygen 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 Böhlen of 1.2 kWh., including the power for compression to 23 ats.) 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 Pf. 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 Pf. 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 has operated on "rich air" containing 70-75% of oxygen.

8. DOCUMENTS AND PAPERS

During the period of the visit a search was made for technical reports and detailed drawings of plant, particularly of the high-pressure generator.

- (i) Basement of Lurgi Haus. - The files there proved to be correspondence only with some technical matters arising in correspondence. The drawing offices had been cleared and the drawings evacuated but copies of diagrams of interest to the Fischer-Tropsch group were obtained from Dr. Herbert.
- (ii) The Hochschule at Friedburg. More correspondence files were found and some technical files relating to new projects. Certain of the latter were taken, namely:
- File: Carbonisation of bituminous coal - Krupp-Lurgi
Reports 1937 to Sept. 1941
- File: Ringwaltzen Press:- Technical Reports to 1942.
- File: E.T.4. Projected Plant at Hoesch using air circulation 1939-1944.
- File: Fischer-Tropsch - Miscellaneous projects at different works including Japan.
- Prints: (a) Recovery Systems of Fischer-Tropsch plants
4 different arrangements.
- (b) Flow diagrams of Fischer-Tropsch yields by 3 recirculation systems.
- (c) Miscellaneous drawings of experimental plant.
- (d) Drawing No. 200.612 of the Brux plant.
- (iii) During the visit to Frankfurt information was received which led to the liberation during a subsequent visit of the team to Bohlen and Grossdeuben, nr. Leipzig of Drawings and working data relating to the Lurgi High Pressure Gasification process.