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KRUPP TREIBSTOFFE WERKE G.M.B.H.

WANNE-EICKEL

FUELS AND LUBRICANTS

Capt. C. C. Hall, Brit.

Dr. A. R. Powell, U.S.

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Report I - Inspection of Fischer-Tropsch Plant

Personnel making inspection

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Capt. C. C. Hall, Br.

Date of Inspection

April 15, 1945

Personnel Interrogated

Dr. E. Combles, Manager for the whole works
Dr. H. Fischer, Manager of the Fischer-Tropsch Plant

General

The works are divided by a railway line into two distinct sections. On the one side is the Krupp-Lurgi low-temperature carbonization plant and on the other, the Fischer-Tropsch plant. The two sections are quite independent (or can be made so), i.e., the Fischer plant does not depend on low-temperature coke for water-gas production and the Krupp-Lurgi plant does not rely on the Fischer residual gas for heating. The investigation of the Krupp-Lurgi section is dealt with in a separate report. The Fischer-Tropsch plant was badly damaged.

The Fischer-Tropsch Plant and Process

Production and purification of synthesis gas. Water gas is made in 10 standard Humphreys and Glasgow generators, two being fitted for dry removal of ash. Low-temperature coke from the Krupp-Lurgi installation is used in two of the generators and normal high-temperature coke in the remainder. The output of water-gas is 58,000 m³/hr. and its composition:

CO₂ 6%, CO 39%, H₂ 50%, N₂ 5%.

The H₂S is reduced from 3 gm./m³ to 0.08 gm./m³ in a system of 4 towers (3 in operation) each containing 1000 tonnes luxmasse. The organic sulphur is then reduced from 0.14 gm./m³ to 0.002 gm./100m³ in the usual feinreiniging system of 3 streams of 2 towers, each tower containing 70 tonnes of luxmasse-soda contact obtained from Ruhrchemie. At the inlet to the feinreiniging the gas contains 0.2 gm. oxygen/m³, but the out-going gas is oxygen-free. The mass is discarded when the sulphur content reaches 8 per cent.

One-third of the purified water gas is converted at 450°C. in the presence of an iron catalyst obtained from Ruhrchemie and the product blended with the remainder to give 63,000 m³ of syngas/hour. The syngas composition was given as

CO₂ 14%, CO 27%, H₂ 54%, N₂ 5%.

The Synthesis

The process is operated in two stages, one at atmospheric pressure and one at medium pressure (10 atmos.). The first stage comprised 72 normal tube and plate ovens (64-66 on streams at any one time) arranged in the usual manner in an enclosed oven-house. The medium-pressure stage had obviously been added later and was in a separate building, located at a considerable distance from the normal pressure stage. The M.P. oven-house was of the 'open' type, i.e. the double rows of six ovens each on each side of the house were open to the atmosphere and only the central portion where the steam drums and control instruments were located was roofed-in. Of the 24 medium-pressure ovens, 8 were the double-tube type seen at Sterkrade, but the other 16 were of a modified type. They were, externally, identical with the normal type, but contained wider tubes (7.2 cm. i.d.) fitted with a fin-assembly made of sheet metal which was apparently driven into the tubes after the latter had been made hot. They were known as the "Taschen-rohren Ofen". Their only apparent advantage is an increased catalyst capacity (95-96 per cent of that of the normal-pressure oven compared with 90 per cent for the double-tube type). As all the ovens were on one stage, it was impossible to tell which type functioned the better, but it was found that the Taschen-rohren type were difficult to discharge; the spent catalyst had to be rammed-out with the aid of steel rods.

All the medium-pressure ovens were constructed by Krupps and had an improved design of bottom fitting, which had adequate strength with the use of less metal and permitted discharge of the catalyst without interference with the product outlet connections.

A drawing of the Krupp Taschen-rohren oven was obtained.

The same cobalt catalyst, obtained from Ruhrchemie, was employed in both normal and medium-pressure stages. It was purchased on the basis of cobalt content and the proportions of thoria and magnesia were not known. The data for the 1st stage were given as follows:

Temp.: 180-190°C., pressure: 2 m. water-gauge, pressure drop across oven: 25 cm. (w.g.), gas contraction: 58-60 per cent., yield: 100-110 gm. liquid hydrocarbons + 10 gm. C₃ + C₄/m³ ideal gas, catalyst life 3 x 10⁶ m³ gas/oven or ca. 4 months. After about 600,000 m³ of gas has passed through an oven (ca. 25 days) the catalyst is reactivated by extraction with 20 m³ benzine, and after draining off the solvent, by passing hydrogen for 6-8 hours at a rate of 1000 m³/hr. at the maximum oven temperature (ca. 205°C.). A total of 4 or 5 such reactivation treatments are carried out during the catalyst's life. As 4 ovens are operated as a unit with a common steam drum, a bank of 4 ovens is reactivated at one time.

The gases leaving the 1st stage pass through a direct condenser and the usual active carbon plant to a gas holder. The residual gas, 26,000 m³/hr., of composition: CO₂ 33%, CO 17%, H₂ 32%, CH₄ 8%, N₂ 9-10% passes to a bank of 5

compressors where it is compressed to 9-10 atmos. and then enters the 2nd stage of 24 medium-pressure ovens (20-24 in operation). The conditions in this stage were given as: Temp.: 180-202°C., gas contraction: ca. 32%, catalyst life 6×10^6 m³ gas/oven i.e. about 8 months. No reactivation is carried out in this stage. Recycling of residual gas had been employed occasionally due to shortage of inlet gas from the 1st stage. The outflow from the ovens passes through the usual condensers and active carbon plant.

The over-all yield from the two stages was given as 130 gm. liquid and solid hydrocarbons + 14-16 gm. C₃ + C₄/m³ ideal gas. The final residual gas had the composition: CO₂ 52%, CO 6-8%, H₂ 8-10%, CH₄ 14%, N₂ 16%, and was used as fuel for the feinreinigung, the Krupp-Lurgi plant and the boilers.

Products

With the exception of the wax flowing directly from the medium-pressure ovens, the condensed oil and active carbon benzin from both stages were combined for distillation, stabilization, etc. It was not possible, therefore, to obtain separate compositions for normal and medium-pressure products. The composition of the combined product was given as:

1. C ₃ + C ₄	11 - 13%
2. Benzin 39 - 176°C.	42 - 45% (Olefines 25%)
3. Diesel oil 176 - 230°C.)	
4. Rohrol 230 - 320°C.)	41 - 43% (Olefines 15%)
5. Gatsch 320 - 450°C.)	
6. Hard wax, m.p. 80 - 91°C.	4 - 6%

The gasol (1) was sold as Treibgas for automobiles. The benzin (2) was blended with 8 - 10% of Krupp-Lurgi spirit (O.N. 80) and was then stated to have an octane No. of 56. (This would suggest that the unblended Fischer benzin had the abnormally high octane No. of 54.) The blended spirit was despatched to the central distributing station in Dortmund, (3) was sold as diesel fuel, (4) was despatched to I.G. for conversion to 'Mersol' and (5) to Witten for oxidation to fatty acids. The hard wax (6) was steamed to remove traces of soft wax and sold.

The total output of Fischer-Tropsch products was 196 tonnes per day, i.e. ca. 65,000 tonnes/annum.

Cracking Plant

The works included a German-designed cracking plant which had not been used during the war. It was formerly used to crack the over 260° portion of the primary product in the vapour phase at 450-530°C. and 16-18 atmos. It had also been "borrowed" by Ruhrchemie to crack their products.

Polymer Gasoline Unit

This was a U.O.P. phosphoric-acid unit built to process 14-16 tonnes of C₃ + C₄ per day at 42 atmos. It had not been in operation since the U.S.A. entered the War.

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Report II - Krupp-Lurgi Low Temperature
Carbonization Plant

Personnel Making Inspection

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Date of Inspection

April 15 - 16, 1945

Purpose of Inspection

This works of Krupp at Wanne-Eichel was primarily a Fischer-Tropsch plant for production of synthetic oil. Located here, however, was also a commercial plant for carbonizing coal at low temperature, and it was understood that part of the coke at least from this plant was used in connection with the making of synthesis gas for the Fischer-Tropsch process. A rather brief and undetailed inspection was therefore made of this special type of carbonization plant.

Description of Plant

The coking chambers of this plant were vertical slots with the heating flues alternating between slots. With respect to this general arrangement of alternating coking chambers and heating flues arranged in a long line, there was some resemblance to a coke-oven battery, but there the resemblance stopped.

The coking chambers were made of cast metal (composition not determined). The inside width at the top was 76 mm., and at the bottom 100 mm., this taper being provided to facilitate discharge of coke through the bottom when coking was completed. The chambers were 3.1 m. long and 2.1 m. high.

Each six chambers composed an "oven" and this oven was the operating unit, not each individual chamber. This distinction between "chamber" and "oven" must be kept in mind throughout this report to avoid confusion. Over the top of each oven was the top door which was simply an insulated metal lid with the turned-down edges dipping into a water seal, and at the bottom was a similar door also provided with a water seal around the four edges. Thus, each oven was effectively sealed against gas leakage, but there was free gas communication between all six chambers of a single oven unit.

In the older ovens at Wanne-Eichel, the coal discharge had been supported directly by the bottom door, but this had been found unsatisfactory due to the dropping out of hot coke while the door was being removed. In the newer improved ovens, this trouble has been eliminated by providing a coal supporting grate (Rost) which presses directly against the chamber bottoms and which stays in place when the bottom door is removed. This grate (Rost) is a kind of a heavy metal grid with the bars normally covering the bottoms of the six chambers. After the bottom door has been entirely removed, this grid is slid sideways several inches and the coke may be discharged through the grid openings. After coke discharge, the grate or grid is slid back in place to again provide a support for the coal. Thus, the bottom door serves simply as a gas seal and does not support the coal in any way.

Along the top of the battery of ovens there is a combination charging and discharging machine which runs on rails much after the fashion of a coke-oven larry car. When an oven is to be discharged, both the top and bottom doors are removed, a small hot-coke car is run under the oven, the grid at the bottom is slid open, and the top machine lowers six rams simultaneously to assist in the discharge of coke. After discharge, the hot coke is taken away to be quenched, the grid slides back in place, and the bottom door is replaced. The top machine then fills the six chambers simultaneously, after which the top door is dropped into place and another coking period is started.

Each oven (six chambers) has a volume of 3.5 m^3 and has a coal capacity of 2.75 metric tons per charge. The gross coking time is 6 hours, thereby giving each oven a coking capacity of 11 metric tons of coal per 24 hours. The actual or net coking time is somewhat less than 6 hours. The plant contains 56 ovens, thereby making the plant coking capacity 20,000 metric tons of coal per month.

The oven heating system used in this plant is definitely a low-temperature scheme since there is no combustion whatsoever of gas in the oven heating flues. Hot combustion gas recirculates through the flues of the battery and this recirculating gas is continuously heated by injection of high-temperature gas from central combustion chambers. The oven heating gas used is Fischer-Tropsch tail gas (Restgas), in some cases supplemented by a part of the distillation gas (Schwelgas) from the low-temperature ovens.

Two recirculating heating gas mains extend the length of the battery, one on each side, and these passages are more or less an integral part of the battery. These passages are of rectangular cross section, about the same height as the oven chambers and connect directly with all of the oven flues which lie between each coking chamber. By means of recirculating gas blowers the hot gas flows along one side of the battery, then through the battery along the flue passages between each coking chamber, thereby heating the coal charge from both sides. The heating gas then returns through the main on the other side of the battery, is reheated by the injection of high temperature gas from the central combustion chambers, and then resumes its circuit through the battery. Periodically, the direction of flow of the heating gas is reversed from one side of the battery to the other side in order to ensure uniform heating conditions in the coking chambers. Several recirculating heating gas systems are provided, each system handling several ovens.

The temperature of the recirculating heating gas entering the battery is 610-620°C. and the temperature of the gas leaving the battery is 560-580°C., so that there is relatively little temperature drop. The underfiring or fuel gas requirement is 500 k. cal. per kg. of coal (dry basis). This is equivalent to 900 BTU per lb. of coal.

The by-product recovery equipment of this plant was not inspected in detail, but it was understood that it consisted of the usual coolers and condensers for recovery of the low-temperature tar and washers for recovery of light oil from the gas.

Coal Used and Products Recovered

It was stated that the coal carbonization in this plant was a blend of high-volatile coking coals from the Ruhr district. It was claimed that non-coking coals could also be carbonized in this type of equipment giving, of course, a char instead of coke. As stated before, an average of about 20,000 metric tons of coal per month were carbonized.

Low-temperature coke was produced at an average rate of 17,000 tons per month, indicating a total coke yield of 85%. This coke contained 7-8% volatile matter. After screening, the large sized coke was charged to two of the ten water-gas generators producing synthesis gas for the Fischer-Tropsch plant. It was claimed that this low-temperature coke gave water-gas with a 1.35 ratio of hydrogen to carbon monoxide, which is considerably higher than obtained from high-temperature coke. Such gas would require, of course, less conversion by the shift catalyst to make synthesis gas. The small sized coke has been sold for house heating and it is claimed that it has proven to be an excellent fuel for this purpose.

The total yield of tar was not given, but it was stated that there was a production of 1,000 tons per month of Heizol (heating oil), which is presumably a tar distillate. There was also a production of 160 tons per month of Schwebbenzin, which represented the light oil recovered from the gas. This light oil was quite different in character from the usual high-temperature coke plant light oil, being largely parafinic and containing very little aromatic hydrocarbons.

Evaluation of Process by Plant Personnel

The plant people interrogated about this process were quite enthusiastic about its commercial possibilities. In conjunction with a Fischer-Tropsch plant, they claimed that this process gave 30-35% more total oil (from coal carbonization and Fischer-Tropsch plant combined) than would be obtained from a high-temperature coke plant plus a Fischer-Tropsch plant. They claim that, in view of their experience, they would again combine a Krupp-Lurgi low-temperature carbonization plant with a Fischer-Tropsch plant in any possible future project for which their opinion would be asked. Even aside from Fischer-Tropsch operation, they claim that the process would be economically feasible for production of smokeless house-heating fuel, but time was not available to enlarge on this point.

A. R. POWELL