

Interviews In London.

April 25th., 1945 Interrogation conducted by:

Major A.J.V. Underwood, British, Ministry of Fuel and Power

May 1st., 1945 Interrogation conducted by:

Mr. G. S. Bays, Jr.,	
Dr. W.A. Horne,)
Mr. J. P. Jones,) U.S., Petroleum Administration
Mr. B. L. MacKusick,) for War
Col. J. A. Oriel,)
Lt. Col. R. N. Quirk,)
Major. A. J. V. Underwood,) British Ministry of Fuel
Mr. A. E. M. Murray,) and Power

(Note. The following information, which is additional to that given in previous sections of this Report, has been compiled from notes supplied by Major Underwood and from a report prepared by Messrs. Bays, Horne, Jones and MacKusick.)

Production of Fischer-Tropsch Plants.

Figures given by Martin and Hagemann for capacity of F.-T. plants are shown below, together with figures given in M.E.W. report on "The German Oil Industry" of October 1944.

<u>Plant</u>		<u>Martin & Hagemann's</u>	<u>M.E.W.</u>
		<u>Figure</u> <u>Tons per year</u>	<u>Estimate.</u> <u>Tons per year.</u>
Deschowitz	30/5.09	60,000	110,000
Dortmund	30/5.04	60,000	90,000
Sterkrade-Holten	30/5.01	70,000	125,000
Homburg	30/5.05	75,000	190,000
Kamen-Dortmund	30/5.06	75,000	100,000
Lützkendorf-Michel	30/5.08	*80,000	150,000
Ostrop-Rauxel	30/5.03	50,000	100,000
Ruhland-Schwarzeiche	30/5.07	210,000	350,000
Wanne-Eickel	30/5.02	60,000	130,000
		<u>740,000</u>	<u>1,345,000</u>

* Nominal capacity - actually production was only about 30,000 t/yr., owing to difficulties with Schmalfeldt gasification process.

Production and Purification of Synthesis Gas.

Martin said that there had been no new developments in synthesis gas production during the war; there was no direct gasification of coal except of brown coal at Lützkendorf and Ruhland. At Ruhland, the Didier plant had been found to be heavy on repair costs and the Koppers plant was the main source of gas.

At Lützkendorf the Schmalfeldt process was not very successful. There was much trouble with cracked products and sulphur compounds and arrangements had to be made with I.G. to borrow an expert. In order to make the process workable and to achieve the desired output, oxygen (0.25m^3 per m^3 synthesis gas) had to be employed. The gas still required oil washing followed by active charcoal to remove compounds which upset the Feinreinigung process.

The Lurgi pressure gasification process using oxygen operates best (from the point of view of synthesis gas production) with low-temperature, brown-coal coke. When using coal difficulties arise due to incompletely cracked tar.

Martin stated that Krupp had tried out the Thyssen-Galocsy gasification process on the large scale. He did not know what results had been obtained but they were believed to be good.

In the medium-pressure synthesis, removal of H_2S is always carried out before compression.

If coal or coke oven gas is used for synthesis gas production, oil washing or active carbon seems to be necessary to remove resin-forming compounds which affect adversely the life of the sulphur purification catalyst and synthesis catalyst. Essener Steinkohle had an active carbon plant before the organic sulphur purification and extended the life of the cobalt catalyst to 8 months from 5-6 months. Use of active carbon reduces the size of sulphur purification plant and generally seems desirable. Synthesis gas at Essener Steinkohle was bad, although made from coke. Possibly this was due to the use of lower temperatures in the water gas generator with coke of low melting point ash. No change had been made in the catalyst for sulphur purification.

Synthesis with Cobalt Catalysts.

Martin gave the composition of the cobalt catalyst used for both normal and medium pressure in all plants as: 100Co, 1 ThO₂, 10MgO, with the keiselguhr proportion adjusted to maintain 800kg. cobalt per oven charge of 10m³ of catalyst. (This ratio of thorium to magnesia conflicts with that given in Germany).

To ensure receipt of keiselguhr of the quality required for catalyst preparation, Ruhrchemie had installed a chemist at the mine (near Münster) who supervised the selection of the material and checked each consignment.

Pure hydrogen is considered preferable to the hydrogen-nitrogen mixtures used by Ruhrchemie for catalyst reduction. The reduction is more rapid and takes place at lower temperatures.

Martin gave the life of the cobalt catalyst as 5-6 months in the normal-pressure and 7-8 months in the medium-pressure process. The life depends greatly on the skill of the operator, particularly when starting-up a fresh catalyst. Starting conditions have to be varied according to the time the catalyst has been in storage or transit since its reduction.

Martin thought it was possible to increase the overall life of the catalyst by operating the first stage at a low conversion (e.g. 50% gas contraction) and a low temperature.

Various patented processes for the continuous extraction of the catalyst by reflux washing have not been adopted as they appear to cause some cracking.

There is usually no difficulty in removing spent catalyst from the ovens if solvent extraction and hydrogenation has been carried out. If the temperature has been too high, particularly at the start, carbon may have been formed and this makes the cleaning of the catalyst spaces in the oven difficult.

Experiments had been carried out at Sterkrade and at Castrop-Rauxel on the use of catalyst suspended

in oil. During the process, the lighter products distill off and the proportion of heavy products in the oil increases. The throughput obtained was not high, the conversion was low and the methane formation was not reduced.

Difficulties had been encountered in the complete removal of catalyst from the oil. This was essential if the oil was subsequently to be cracked, as traces of cobalt cause excessive production of methane in the cracking process. The power cost of the process also proved to be high.

Use of Iron Catalysts.

Martin considered that the iron catalyst was mainly of interest for hard wax production. It was this aspect of the process which had attracted the Italian concern (S.I.C.S.). There was little to choose between cobalt and iron in sensitivity to poisoning by sulphur.

Martin stated that the I.G. sintered iron catalyst was very heavy and would cause structural difficulties on the large scale.

Condensation and Product Recovery.

Direct condensation was used for the atmospheric pressure process and indirect condensation for the medium-pressure process. Indirect condensation permits the recovery of alcohols and fatty acids formed in the synthesis. Schaffgotsch Benzin recovered 8-10 tonnes /month of C₂- C₄ alcohols (about 0.02% of the primary products) by distillation of the condensed water.

In the medium-pressure process it is usual to carry out the active carbon scrubbing after expansion, i.e., at atmospheric pressure. Oil scrubbing is not considered so satisfactory owing to the incomplete recovery of gasol.

A pre-war Linde project for the recovery of all products including gasol, by cooling under pressure had not been tried out.

Carbon dioxide was extracted from the residual gas after the active carbon plant by the Alkazid process and used for catalyst protection. The residual gas was used as fuel.

Product Utilisation.

The petrol fraction was blended with benzole for motor fuel and certain light fractions also found use as solvents, paint thinners, etc.

The fraction 180-230°C. was employed as a high-grade diesel fuel blending agent.

The I.G. had taken 8000 tonnes per month of the heavy diesel oil for conversion to Mersol detergents.

The hard wax of m.p. 90/95°C. had a good market for use in polishes, candles and explosives. The Ruhrchemie production of this hard wax from the medium-pressure process was about 500 tonnes/ month.

Martin stated that I.G. had a lubricating oil plant which utilised Fischer-Tropsch products in conjunction with the low-temperature hydrogenation of brown coal.

Special Products.

Fatty Acids. The addition of 0.2- 0.5% of acetic acid (based on the liquid products) to the synthesis gas in the medium-pressure, cobalt-catalyst process increased the yield of directly synthesised acids.

The OXO aldehydes could be readily converted into acids by the addition of sodium carbonate and air blowing.

Soaps prepared from the C₁₃ and C₁₄ acids were better detergents than those prepared from natural oils.

Alcohols. In this interview Martin stated that an iron catalyst containing up to 10% copper was employed in their laboratory synthesis of alcohols at 180°C. and 200 atmos. pressure.

Catalytic Cracking.

Martin said that Ruhrchemie were building a catalytic cracking plant to produce C₃, C₄, and C₅ olefines from the heavy fractions (above C₇). The catalyst to be used was "Granosil", an acid-treated clay.

The plant was about 60% complete when construction was abandoned in the Summer of 1944. The

pilot plant tests indicated that the catalyst would have a life of about 6-8 weeks after which it would be discarded. The process involved the removal of carbon deposits by oxidation at intervals of 8 - 10 hours. The olefines were to be used for the production of polymer gasoline.

Ruhrchemie had not carried out alkylation, and, had made no high-octane gasoline or components.

Yield and costs of Fischer-Tropsch Operations.

Martin stated that from an economic standpoint, the principal war-time improvements in Fischer-Tropsch operations were increased yields of primary products, and better utilization of the by-products. Whereas, before the war an average yield of primary products of 140 gms. per cubic meter of synthesis gas was realised, a yield of 150 gms./cubic meter is now obtained from most plants. One plant, Essener Steinkohle, gets yields as high as 160 gms. This particular plant operated at atmospheric pressure, used three stage conversion with 50% of the total conversion taking place in the first stage and 25% (approximately) in each of the remaining two stages. This plant also had a rigid schedule for catalyst changing every six months regardless of activity, and especially good (according to Martin) technique for removing and replacing catalyst; they had constant coke quality and exceptionally good gas purification (including the use of activated carbon). All these factors contributed to making the yields and efficiency of the Essener Steinkohle plant outstanding. The complete utilization of by-products such as of tail gas in cracking and lubricating oil manufacture and the use of CO₂ recovered by Alkazid to blanket the catalyst during the preparation, improves the economics.

The records of the Essener Steinkohle plant should be found at Muessner Allee in Essen (Home Office) and those for the Hoesch Benzin plant at the Hoesch Steel Works in Dortmund. Copies of records of all plants should be at Reelkirchen and there is practically nothing at Ringelheim.

Martin stated that one kilogram of primary product per 5 kilograms of coke was considered the normal

yield for his low-pressure plants although in winter it was necessary to bring in process steam not accounted for in the above coke consumption. As a capital investment cost of the plants, the atmospheric or low pressure units were generally figured at 800 marks per tonne annual production capacity (primary products), which is exclusive of land and utilities. The medium-pressure plant cost was somewhat higher. This figure, according to Martin, is difficult to translate to a foreign basis because it is a construction company figure, and the construction firms were steel producers who put their own price on the steel, and may have used unnecessarily large amounts of it.

Martin stated that Ruhrchemie were to deliver olefines to the OXO plant at 45pfg./kg. of feed material, the unreacted portion being returned to them. The estimated cost of production of the alcohols was given as 95pfg./kg. including 10% amortization. (Note: These costs are higher than those given at Sterkrade, C.I. Report 4, p.40).

Japanese Contacts

Ruhrchemie made a contract several years ago with Mitsui giving them a general license; I.G. Farbenindustrie had made similar agreements with Mitsubischi; Martin professed to know nothing about the details. Koppers built a Fischer-Tropsch plant for Mitsui about five years ago at Mu'ke which is about 30 kilometers from Tokyo. This had an initial capacity of 20,000 tons per year but may have been enlarged since. Then Ruhrchemie periodically gave information on developments to the Japanese in accordance with their agreement, but the Japanese always had 'no information' to exchange. The Japs were quite anxious to have cobalt shipped to them. The last information was passed to the Japs about September or October 1944, and included the latest information on the Ruhrchemie iron catalyst. Records of such information given to the Japs are in Ruhrchemie files at Oberhausen-Holten. Mitsui also was reported to have built a plant in Manchukuo near a steel mill.

The Mu'ke plant had been built with the help of a Dr. Schenk who went to Japan for that purpose and whom the Japs have allegedly never allowed to return nor to send back reports.

Lurgi also had agreements with Mitsui and gave them information on oil processing and on coal gasification. Records of such information should be in Lurgi files in Frankfurt/M. Dr. Hubmann from Lurgi went to Japan in this connection and has since returned.