

Restricted

Summary and Conclusions.

General.

There had been no increase in the number or capacity of the Fischer-Tropsch plants in Germany during the War, and the total, average annual production of primary products from the nine plants amounted to rather less than 700,000 tonnes, i.e., only about half that estimated in the Item 30 briefing documents.

The processes used in these plants were substantially those known or believed to be in use before the War. The cobalt catalyst (Co:100, ThO<sub>2</sub>:5, MgO:8, kieselguhr: ca.200) had remained unchanged in composition since 1938 and was the only one employed on the full scale for both normal and medium-pressure (9-11 atmos.) operation. The methods employed for catalyst preparation, reduction and regeneration were substantially those known before the War.

Some increase in yield and catalyst life had been achieved in the atmospheric-pressure process by more rigorous purification of the synthesis gas and by improved operation and control in the catalyst chamber house - in particular, the starting-up of fresh catalysts, the methods and time schedules used for regeneration 'in situ' and the schedule for switching catalyst chambers from one stage to another. The average yield claimed for both normal and medium-pressure operation was 150 gm. C<sub>3</sub> and higher hydrocarbons per Nm<sup>3</sup> ideal gas; the catalyst life was given as 5-6 months for normal pressure and 8-11 months for medium-pressure operation.

No new design of catalyst chamber had been introduced. The rectangular, multi-plate vessel was still standard for normal-pressure synthesis and a vertical, cylindrical, double-tube chamber was used for operation at 9-11 atmospheres.

The Fischer-Tropsch process was regarded by Ruhrchemie primarily as a means of producing chemical products and was not thought to have much future as a fuel-producing process in a free world economy.

Even during the war, only the spirit fraction (e.p. 160°C.) and the light diesel oil fraction (160-230°C.) were used as fuel. The heavy diesel oil was sent to

I.G. for conversion to Mersol detergents and the soft wax to Witten (Deutsche Fettsäure Werke) for conversion to soap and edible fat. There had been no reforming of the motor spirit fraction or cracking of the heavy fractions (except to make raw-material for the manufacture of lubricating oils) during the war, although partly completed large-scale plants for catalytic cracking, polymer petrol production and conversion of the heptane cut to toluene, existed.

The main trend of the Ruhrchemie war-time research work was in the direction of producing chemical and special products which were in short supply.

### The Sterkrade-Holten Plant

Water gas was produced from coke in standard generators and purified from hydrogen sulphide and organic sulphur compounds by the normal processes. A portion of the water gas was converted to hydrogen by the usual shift catalyst and the product blended with unconverted water gas to give the required ratio of carbon monoxide to hydrogen.

The synthesis step was carried out in two independent sections :-

1. An atmospheric section comprising 52 catalyst chambers in two stages supplied with gas of  $H_2: CO = 2 : 1$ .
2. A medium-pressure section of 72 chambers arranged in three stages which received gas of  $H_2: CO$  ratios 1.4:1, 1.6:1 and 1.8:1 respectively.

Direct condensers were employed on the atmospheric-pressure stream, and indirect condensers on the medium-pressure stream. Active carbon was used in both cases for recovery of light spirit and 'gasol', and the products were refined in conventional stabilising and distillation equipment.

The annual output from the whole plant was approx. 70,000 tonnes of primary products.

### Synthetic Lubricating Oil.

At Sterkrade-Holten, 1400 tonnes per month of high-grade synthetic lubricating oil was produced by

aluminium chloride polymerisation of the olefines derived from soft cracking of the over 170°C. portion of the primary products. The oxidation stability of these oils had been markedly improved by the incorporation of phenathiazine or, more recently, elemental sulphur in the polymerisation process.

The oils were blended with petroleum oils and used in army vehicles.

### Research and Development Work.

The OXO Process. The Sterkrade-Holten works included an almost completed plant for the production of 10,000 tonnes /annum of C<sub>12</sub> - C<sub>18</sub> alcohols by the reaction of water gas with C<sub>11</sub> - C<sub>17</sub> Fischer-Tropsch olefines, using a powdered cobalt catalyst in the liquid phase at 135°C. and 150 atmos. pressure to form the C<sub>12</sub> - C<sub>18</sub> aldehydes, which were then hydrogenated to the corresponding alcohols at 180°C. and 150 atmos. The process was to be operated by the OXO Gesellschaft (Ruh Chemie + I.G. + Henkel et Cie.) with the object of producing detergents from the alcohols. The OXO reaction appeared to be generally applicable to olefinic compounds.

### The Cobalt - Water gas - Recycle Process.

Arrangements were almost complete for the operation of the Sterkrade-Holten synthesis plant with all the 72 medium-pressure ovens in one stage at 11 atmos. using water gas as the feed material and recycling 3 parts residual gas to 1 part fresh gas. After adjusting the H<sub>2</sub>: CO ratio to 2 : 1 the tail gas was passed through two atmospheric-pressure stages.

The products from the medium-pressure stage were said to have a high olefine content and would have provided suitable raw-material for the OXO process.

Iron Catalysts. Research work which had started before the War had resulted in the development of an iron catalyst of the general type:- Fe:100, Cu:2-5, CaO: 10, kieselguhr: 30-150, which gave good performance with water gas at 215°-230°C. and 20 atmos. pressure. The process was preferably operated in two stages with a 2:1 recycle-gas : fresh gas ratio in the first stage. The products were rich in olefines and contained a high proportion of hard wax; the yields and life of catalyst were said to be similar to those obtained with cobalt.

This process had only been operated on the pilot-plant scale ( $100\text{m}^3$  gas /hr. = 1/10th. full scale).

Apart from the question of War-time availability of materials, there was no suggestion that this process was superior to that based on cobalt.

The iron catalyst was said to be more expensive to prepare, it required higher operating pressures, and tended to give products contaminated with oxygen-containing bodies. Its use was not essential for the production of olefine-rich products; these could equally well be obtained by the cobalt - water gas - recycle process. The production of hard wax was the only virtue specifically claimed for the iron catalyst.

Chemical Products. A considerable amount of research work had been carried out along the following lines :-

1. The extraction of the small amounts of alcohols and fatty acids present in the normal products and attempts to modify the process in such a way as to increase the proportion of these compounds.
2. The synthesis of n-primary alcohols at  $200^{\circ}\text{C}$ . and 200 atmos. pressure using cobalt and iron catalysts.
3. The oxidation of the hard wax with nitrosyl sulphuric acid to produce fatty acids of greater chain length and freedom from byproducts than are obtained by the Witten (aerial oxidation) process.
4. Chlorination and dechlorination of the waxes to produce olefines which yield special lubricants on polymerisation and detergents by sulphation.
5. The production of new products by the addition of acetylene to the synthesis gas.

#### Japanese Intelligence.

The Directors of Ruhrchemie appeared to have little knowledge of value concerning Japanese developments in the Fischer-Tropsch field during recent years. The exchange of information appears to have been 'one-way'.

Such information as was obtained has been passed to the appropriate authorities.