CHAPTER II.

VISIT TO THE RUHRCHEMIE PLANT AT HOLTEN,
on Monday, 31st Oct. 1938.

Present: the parties from Standard, Kellogg, I.G. and Shell, as mentioned before.

Furthermore from Ruhrchemie:

Director Alberts
Drs Feiszt
Wilke
Bahr
Von Asboth
Schaller

The works at Holten (Ruhrchemie and Ruhrbenzin) consist of the synthesis plant proper, a large ammonia plant, a separate plant for manufacturing catalysts, which also serves some 4 other synthesis plants in Germany, laboratories, pilot plants and auxiliaries.

The synthesis plant combined with blue water-gas plant has an extensive lay-out, separated from the other plants. The production of the synthesis plant at the time of the visit was given at 90 tons primary product per day, or 34,000 tons per year, with a gas throughput of about 32,000 m3/hour. It was expected that within a few weeks the first units of the pressure synthesis plant would be put into operation. After completion of this pressure plant the production will be increased to 85,000 - 90,000 tons of primary products per year.

The manufacturing of blue watergas.

Eleven Demag generators, system Humphries, Glasgow, were installed, 6 of which have a rated capacity of 6800 m3/hour each, the 5 newer units having 7800 m3/hour. In actual operation these units produce 7400 and 8500 m3/hour each respectively. Four units were in operation with one as stand-by. The generators are operated automatically, which gave full satisfaction. Coke is charged to the generators every 3 minutes at the rate of 200 kg, i.e. about 100 tons per 24 hours. The cycle is:

1 minute blowing with air
1 " blowing steam from top
1 " blowing steam from bottom.

The composition of the gas produced was given as follows:

6 %	CO_2
40 %	CO
50 %	H_2
0.6%	CH_4
3.4%	N_2

The pressure in the generator was about 900 m water, with a water seal at the bottom of the furnace.

Each furnace is equipped with one 18 atm. steam boiler for waste heat, while at the same time the furnace jacket produces low pressure steam ($2\frac{1}{2}$ atm.), used for blowing the furnaces.

Extensive equipment was available for the transportation of the coke in special railway containers (4 containers of 10 tons each upon 1 railway carriage) to bunkers on top of the generators.

After the generator plant comes the CO conversion plant, where about 1/3rd of the total gas output, after sulphur purification, is passed over a catalyst together with steam, to convert the CO with steam into CO_2 and hydrogen, thus producing a gas with a higher H_2 content, which, after mixing with the untreated 2/3rds, yields the synthesis gas of about the following composition:

13.5%	CO_2
28 %	CO
55 %	H_2
0.4%	CH_4
3.1%	N_2

The conversion is effected catalytically at a temperature of about 450°C. This plant was originally placed before the final purification; afterwards this was reversed on account of difficulties encountered with the converter catalyst (see figs 9 and 10, Annex A).

Next to the CO conversion plant a turbo compressor station was in course of erection. In a building of about 15 x 65 m, equipped with a 15 ton crane, four turbo compressor units, each unit consisting of one l.p. and one h.p. compressor, are placed. The l.p. compressors have a working pressure of 5 atm. and a capacity of 20,000 m3/hour at 3,000 revs/min. The electric motor of 2000 kW at 1500 revs/min. is connected to the turbo by a gear-box, manufacturer Deutsche Werke; the turbos are from Gutehoffnungshütte; the motors from Siemens.

The high-pressure compressors have the same capacity and are of the same type and make, the delivery pressure being 10 - 14 atm. The motor is rated 1000 kW. One unit of two compressors is driven by steam, produced by the above-mentioned 18 atm. boilers. The lining-up is

such that two units are used for water gas and one for converted gas, whereas the fourth unit is stand-by for both purposes.

H_2S and organic sulphur removal plant.

Inorganic and organic or final purifiers are set up in one line, the whole space being covered by a 30 ton travelling crane, with an extension on one side over a free space and a railway track for emptying and refilling the screen-boxes. At one end of the purifiers a blower station is built (at the other end a filling station for bottled gas).

In this blower station 3 blowers of 40,000, 40,000 and 80,000 m3/hour capacity respectively are installed. Only one of the first-mentioned blowers was running, passing the gas from the gasometer (capacity 20,000 m3) through purification, converter and subsequently synthesis (see fig. 10, Annex A).

The delivery pressure is about 3.4 m water. Each blower has 2 stages at 3000 revs/min., coupled by a gear-box (in the same way as the compressors mentioned before) to an electrical motor of 640 kW (one blower of 40,000 m3/hr) or to steam turbines (one blower of 40,000 and one of 80,000 m3/hr).

Difficulties were encountered on account of sulphur depositing on the vanes of the blowers, which necessitated a shut-down every 8 weeks.

For this reason a change of the flow was intended, i.e., passing the gas through the H_2S purifiers before it enters the blowers. In Annex A. fig. 9 shows the gas flow when starting the plant, fig. 10 as it is now and fig. 11 as it is intended to be in the future.

In the blower station (dimensions about 24 x 20 m) also a steam-reducing station was installed.

The gas leaving the blowers at a temperature of about 60°C passes through a set of coolers with direct water sprays and then enters the H_2S removal towers. These towers are of the common type, as used in coke gas oven plants.

Two sets of 4 towers are available at Holten, the gas passing 3 towers in series, one tower of the set being refilled. Each tower has 16 screens with purifying mass in layers of 400 - 500 mm.

The Lux mass in these towers should not contain more than 50 % water; when preparing this mass, about 20 % spent mass is mixed with fresh mass, so as to obtain a better porosity. The average H_2S content of the inlet gas is about 3 g per m3; the purified gas contains 0.2 g H_2S per 100 m3 and, in addition, 150 - 200 mg organic sulphur per m3.

The organic sulphur removal plant, consisting of 5 sets of 2 towers each, of which one set is spare, is equipped with one heater per set, using tail gas and air, both from a main line.

A circulation blower serves the purpose of increasing the fuel gas velocity through the gas heaters.

These gas heaters are equipped with vertical tubes and as the gas enters from one side, thus heating the tubes direct exposed to the incoming gas, more than the others, it was necessary to provide for a separate expansion for the first half of the tube bank. Thus the outlet-cover on the top consists of two halves, each having a separate flexible outlet connection with separate balancing weights.

Two types of towers are in use, described fully in the Ruhrchemie report. The price of the flat screen towers is somewhat higher than the price of the cylindrical screen towers; moreover, the first type needs a hoisting equipment, but the flat screen towers can be recharged in 4 - 6 hours, whereas this takes 36 hours for the cylindrical towers.

Oxygen is introduced before the H_2S removal boxes, in such a quantity that the gas contains 0.3 - 0.5 % total oxygen as a maximum.

As inlet and outlet for each layer can be closed independently, it is possible to operate with two or more layers in series.

The synthesis plant.

The synthesis plant is housed in a large building about 170 m long and 35 m wide. A platform at a height of about 5 m crosses the building in the centre from one end to the other, the converters being situated left and right of this long corridor.

In the older half length of the building the l.p. synthesis ovens are placed.

In the centre an ample space has been reserved for operating control (central operating room).

In the newer half length of the building the h.p. synthesis ovens were partly ready and partly in course of construction.

As the overall length of the converters is only about 6 m, an ample space is left alongside the walls (open from the base to about 2½ m height) for a railway track on which the catalyst-containers are transported.

The weight of the large containers (dia.

3100 mm) is 5600 kg, taking one oven filling of catalyst (10 m3 is 3250 kg, or, if heavily loaded with paraffin wax, 5000 kg). The capacity of the crane travelling over the ovens and railway track is 15 - 17 tons.

A special ring equipment is available for tipping the container above the oven.

When an oven has to be emptied, a special scraper conveyor is attached to the bottom, from which, with a bucket conveyor at the back of the oven, the mass is transported to the container. Vide Annex A, fig. 12.

At Holten 52 l.p. ovens are available, of which 48 - 50 are in operation, whilst 2 to 6 are out of use on account of changing catalysts and at the same time overhaul of auxiliaries, etc., also for re-activating the catalyst with pure hydrogen.

At Holten sets of 2, 4 and 6 converters are connected to one steam drum. The temperature of each oven is regulated from the central operating room by adjusting the pressure at which the boilers deliver their steam to the central steam line.

The boilers are designed for a working pressure of 30 atm. The normal pressure in the central steam line is 9 - 10 atm. When an oven with fresh catalyst is started, the temperature corresponds to about 5 - 7 atm.; during this short period of a few hours the steam is blown off in the low pressure line.

Feed of boilers is automatically controlled by Hanomag feed-water regulators, which work quite satisfactorily. The feed-water is softened in a permutite plant and the water is afterwards heated to 70°C, where under a vacuum all carbon dioxide and air are removed.

Quantities of inlet and outlet gas to and from each separate converter are measured.

Of the 48 - 50 ovens in use, 36 are connected in first stage and 12 - 14 in the second stage. These figures, however, may be altered to 32 in the first stage and 16 in the second stage, depending on the "newness" of the catalyst.

The gas enters the synthesis converters with a temperature of about 200°C, which, however, varies with the required temperature of the organic sulphur removal, the latter depending on the catalyst age.

The outlet gas of the first stage, with a temperature of 180 - 200°C, enters the intermittent coolers with direct water spray, of the same construction as the second-stage coolers mentioned further on, where about 50 % of the total liquid products formed in the first stage are condensed.

At Holten, where the capacity and the line-up of the charcoal plant do not allow of treating the total quantity of gas after the second stage, the cooled gas of the first stage is then passed through a set of 4 charcoal adsorption vessels and from there, via a water-cooler, to the second stage . This water-cooler had to be installed afterwards to equalize the outlet temperature of the charcoal plant, which changes during the adsorption period; the temperature of the gas entering the second stage is about 25°C.

After leaving the second stage, the gas is cooled again in a water spray tower and passed on to another set of charcoal vessels to be stripped, up to about 80 % of the C_3-C_4, thus 20 % being lost in the tail gas. Further details about

the condensation and the charcoal plant will be given below.

It was stated that the complicated construction of the l.p. ovens, which does not allow of much tube expansion, was nevertheless very satisfactory. In the course of 2 years' running, only very minor repairs to the oven-tube systems had been necessary. For these tubes normal seamless boiler tubes with tensile strength of 45 kg/mm2 are used.

To avoid leakages it was said that the oven was brougth up to temperature slowly (24 hours) and taken out of use also gradually. On a later occasion this was contradicted, when it was stated that the whole process of taking the oven out of use, dropping the catalyst, filling it up again and bringing the oven into production, required no longer than 48 hours.

As already stated, an oven with fresh catalyst is started at a temperature corresponding to a steam pressure of 5 - 7 atm., which in the course of about 4 months is raised to 15 atm.

The steam pressure controls were said to be exact within limits of 1/10 atm.

When filling a new oven with catalyst, special care has to be taken to leave only a minimum of free space.

A surplus filling of, say, 1/2" on top of the tubes and cooling fins would entail excessive formation of methane and excessive heating of the top layer, with the resultant danger of burning of the catalyst and clogging up of the oven.

The catalyst building has room for 72 h.p. ovens.

The operation of the converters is controlled by registering the CO_2 percentage of inlet and outlet gas. As average figures the following CO_2 percentages were given:

inlet first stage	13	%
outlet " "	32 - 34	%
" second "	45 - 47	%

On entering the first stage the gas has a pressure of about 1500 mm and on leaving: 1100 mm; on entering the second stage: 1000 mm and on leaving this stage: 650 mm.

Condensation.

The condensation after first and second stages of the low-pressure synthesis is effected in vertical cylindrical towers, of which 2 with a diameter of about 4 m and 1 with a diameter of 5 m is available, filled with Raschig rings 90 x 90 mm, with direct water sprays.

The condensate, together with the cooling water, is piped to settlers, where the product is separated and from where the water is re-circulated via cooling towers. The acid content of the circulating water is kept at an equivalent of 650 mg KOH per litre, by discarding 15 - 20 m3 water per hour per stage; make-up water is added at the upper inlet of the coolers.

All parts coming into contact with these liquids are made of acid-resisting material, as the products contain CO_2 and small amounts of fatty acids.

In the newly built part of the cooling plant product and water are separated in acid-proof brick-lined trenches.

At the Holten plant all valves in the gas lines (partly riveted, partly welded) are hand-operated, even to diameters of 1500 mm.

The charcoal adsorption plant, consisting of 5 adsorbers for first stage and 6 for second stage, (for the low-pressure synthesis plant). For the H.P. 4 new adsorbers were installed, built by Bamag (Lurgi system) and fully automatic (the valves operated by hydraulic pressure and controlled by electrically-driven camshaft). This system was said to be the first of its kind and gives complete satisfaction.

The adsorption vessels are placed on a structure of about 5 m height. The small vessels contain about 20 m3 = 8.7 tons of activated charcoal; the large ones have a capacity of 40 m3 = about 18 tons. Valves and connecting lines are arranged under the vessels.

The charcoal plant is running with the following cycle:

	1st stage	2nd stage
adsorbing	40 min.	20-60 min.
steaming	20 "	20-20 "
drying	20 "	40-20 "
cooling	20 "	40-20 "
	100 min.	120 min.

When steaming, the first part of the gas expelled, with a high CO_2 content, is blown off. Then the vapours are cooled while the uncondensed C_3-C_4 go to a gasometer and are afterwards compressed at 35 atm. and cooled, the condensate being stabilized.

The charcoal takes up about 4.5 % of its own weight when removing gasoline and (80 %) C_3-C_4.

Up to now 500 kg product per kg charcoal have been produced, whereas a figure of 800 kg has been guaranteed. The opinion was expressed that charcoal is very sensitive to resin-forming substances and paraffin mist, because these products cannot be removed by steam.

In the charcoal plant at Holten the coal rests on aluminium screens. No difficulties were encountered with clogging up of same; on opening the vessels only very slight pulverization of the charcoal was found.

The effect of the charcoal after the first stage on the CO_2 content of the gas entering the second stage is rather small. When the gas entering the charcoal has 30 % of CO_2, it enters the second stage with not less than 27 %. This proves that the greater part of the carbon dioxide is not retained by the charcoal.

Drying and cooling of the charcoal is done with tail gas, the temperature during drying being 130 - 140° and during cooling as low as possible. When cooling the tail gas in circulation with water, the amount of cooling gas is equal to the amount of treated gas.