FIRST EXPERIENCES AND RESULTS OF THE 200 T/D PILOT PLANT BOTTROP

Dr. J. Langhoff, Dr. E. Wolowski, H. F. Tamm
RUHRKOHLE OEL UND GAS GMBH

Dr. G. Escher, H. Hosang, B. Schmedeshagen
VEBA OEL ENTWICKLUNGSGESELLSCHAFT MBH

FIRST EXPERIENCE AND RESULTS GAINED DURING OPERATION OF THE 200 T/D COAL OIL PLANT IN BOTTROP

Dr. J. Langhoff, Dr. E. Wolowski, H. F. Tamm
RUHRKOHLE OEL UND GAS GMBH

Dr. G. Escher, H. Hosang, B. Schmedeshagen
VEBA OEL ENTWICKLUNGSGESELLSCHAFT MBH

Contents

1. Introduction

In a joint project with a 60 % participation of RUHRKOHLE AG and a 40 % participation of VEBA OEL AG, the largest European pilot plant for coal hydrogenation with a capacity of 200 t/d was erected in Bottrop.

On 21st May, 1979, the foundation stone for this large-scale experimental plant was layed. The first coal was hydrogenated on 25th November, 1981.

The aim of the trial operation is the optimization of GERMAN TECHNOLOGY (modified Bergius/Pier process) and its further development towards commercial feasibility.

2. Project Description

2.1 Plant Description

The Bottrop COAL OIL plant is designed for a throughput of 200 t (waf) hard coal per day, which are converted into hydrogen gases (C_1 to C_4), approx. 30 tons of light oil (C_5 to 200 °C), and approx. 70 tons of medium oil (200 to 325 °C) (Table 1).

The reason for Bottrop to be chosen as plant site was that an economy-priced system for supply and disposal could be built up in connection with the near-by Prosper cokery. The new parts of the plant to be constructed could be limited for the most part to the process plants, as there are:

- coal/catalyst preparation
- hydrogenation
- atmospheric distillation
- vacuum distillation
- flash evaporation
- light oil stabilization
- residue disposal.

The supply for the COAL OIL plant with coal, heating gas, process water and vapour, as well as the disposal of flash gas, polluted process waste water and hydrogenation residues are realized by the cokery. The hydrogen required for hydrogenation is taken from a hydrogen pipeline running along the plant boundary.

The space required for the COAL OIL plant with a length of approx. 267 m and a width of approx. 215 m amounts to a total of approx. 57,400 m². The main process plants, including the measuring and the control stations, are arranged in four process fields. The corresponding space required with a length of 130 m and a width of 100 m amounts to 13.000 m², that makes about a quarter of the total space required.

The following slides show some views of the plant. Picture 1 shows a general view of the plant. In the foreground you can distinguish from the left to the right: the flare, the blow-down system, the coal preparation building with the coal conveyor belt of the Prosper cokery, the high-pressure hydrogenation chamber and the compressor building.

On the next Picture 2 there is the central element of the plant, the high-pressure chamber with the hydrogenation reactors and the heat separators in the background, and the feed/product heat exchangers in the foreground. On the right hand side there are the oil scrubbing units and the flash vessels. In the upper part of the high-pressure chamber a portal crane is provided for mounting and service.

The atmospheric and the vacuum distillations are illustrated on the next Picture 3.

The steel structure shown on Picture 4 contains the residue solidification with the cooling belt. The final Picture 5 shows the measuring station. Above the indicating and recording instruments there are the alarm tableaus and an overall flow chart of the plant.

Parallel to this project a testing program for the further processing of COAL OIL is carried out. Due to the different composition of oils which are derived from mineral oil and coal, special processing methods have to be developed and/or well-tried mineral oil methods have to be adapted. Characteristic for the COAL OIL are its low sulphur content, its relatively high nitrogen content, and its oxygen content - which practically does not exist in mineral oil. Also the high density and the low hydrogen content are distinctive of COAL OIL.

For these development works a laboratory was installed at the VEBA OEL AG in Gelsenkirchen-Scholven, where the suitability of COAL OIL as raw product for fuel and heating oil production as well as for chemical feedstock is investigated. The COAL OIL is at first separated by distillation in low and in medium boiling fractions, the individual fractions are separately processed. Light and medium oil are subjected to a refining process. After the two-stage refining process the light coal oil is then converted in a reforming process into carburettor fuel. The refined medium oil can be used as admixed component for EL-heating oil (equivalent fuel oil No. 2). There are some other possibilities such as the high-pressure hydrogenation of medium oil to diesel fuel and/or the hydrocracking of medium oil into gasoline.

The total expenditure for this project amounts to approx. 420 million DM, of which approx. 170 million DM are investment, approx. 220 million DM are required for the operation of the COAL OIL plant up to 1983, and approx. 30 million DM for the operation of the further processing laboratory. The project is largely financed by the Minister of Economic Affairs of the Land North-Rhine Westfalia.

2.2 Process Description

Picture 6 shows a simplified process flow diagram of the main process stages of the Bottrop COAL OIL plant. The hydrogenation, central element of the process, is completed by the coal/catalyst preparation, the distillation plants, the gas treatment and the residue disposal.

In the following the basic process stages shall be described in detail.

The feed coal is dried down in the milling and drying installation - which is not shown here - to a moisture content of minus 1 weight per cent, and ground to a grain size of minus 1 mm. Under the addition of an iron catalyst, the prepared coal is then slurried with an oil mixture recycled from the process itself - medium/heavy oil in a 40:60 proportion - and further ground in a wet grinding mill to minus 0.2 mm. The resulting slurry contains approx. 40 weight per cent coal.

The coal-oil suspension is brought up to a pressure of 300 bar and after the addition of recycle gas and fresh hydrogen it is heated up to the reactor inlet temperature of approx. 425 °C in a feed/product heat exchanger and a downstream pre-heater. In the feed/product heat exchanger a part of the exothermal heat of reaction from the hydrogenation is recovered. Downstream of the pre-heater the heated reaction mixture passes, and ascends, through three reactors connected in series. The exothermal hydrogenation reaction is controlled by feeding-in quench hydrogen in some defined reactor levels in such a way that the maximum temperature of 475 °C is not exceeded.

Downstream of the reactors the products obtained are separated within the hot separator . Gases and vapours are drawn off at the top, liquids and solids from the bottom. The top products are further cooled down in the before mentioned feed/product heat exchanger and by additional cooling. The condensates accumulated in separators - a water and COAL OIL-mixture - are led off or pumped into the atmospheric distillation. The remaining hydrogenation gas is led under process pressure into an oilscrubbing unit, where part of the hydrocarbons C_1 to C_5 produced during hydrogenation is recovered and returned to the cokery, where it can be used as heating gas after its cleaning.

The gas recovered from the oilscrubbing unit is recycled as recycle gas after the addition of hydrogen.

The atmospheric distillation basically consists of a heater to supply the required heat, and of a top column to separate the COAL OIL into light, medium and heavy oil fractions.

Downstream of a two-stage flash, in the vacuum distillation the bottom product of the hot separator is separated into distillate oil and residue. After its cooling down, this residue which contains mineral particles, unconverted coal and catalyst is granulated on a steel conveyor belt, the resulting product is returned to the cokery. In an industrial plant the residue would be gasified into hydrogen.

In the vacuum distillation we try to concentrate the solids up to approx. 50 weight per cent. If this cannot be achieved in the way described the hot separator bottom products can be concentrated by means of a combined flash evaporation and vacuum distillation. The five possible routes are illustrated in Picture 7.

The number of flash stages as well as the corresponding pressures can be varied. In two of these alternative processes the bottom product from the hot separator is stripped with hydrogen and then subjected to flash evaporation. Moreover it is possible to increase the pressure before stripping. Between the different flash stages the temperature may be increased if necessary.

The oil collected from flash evaporation and vacuum distillation is led to a solvent vessel, where it is mixed with the required quantity of medium and heavy oil obtained during the atmospheric distillation, and is then recycled into the coal slurrying process.

3. Planning and Construction

After the conclusion of a preliminary project at the end of 1977, the decision was taken to construct the Bottrop COAL OIL plant. Picture 8 shows the time schedule including the basic planning and construction activities. The basic engineering was carried out in 1978/79 by the RURHKOHLE AG and the VEBA OEL AG. The first partial approval (buildings, opening-up of the building ground, etc.) was obtained in the IV. quarter of 1978, the second partial approval (main process plants) during the III. quarter of 1979. The detail engineering was jointly prepared in 1979/80 by RUHRKOHLE OEL UND GAS GMBH, the VEBA OEL AG in cooperation with the Chemische Werke Hüls AG and some other German engineering offices.

On 21st May,1979 the foundation stone for the COAL OIL plant was layed and the opening-up and construction works started. The mechanical completion and the setting into operation of the individual partial plants followed in 1981. On 25th November, 1981 the first coal was hydrogenated.

4. Setting into Operation and First Operational Results

During the preparation of the overall plant for the 'coal-in', in some of its parts mechanical failures and difficulties with the equipment were observed. Important problems arose with regard to conventional components such as mills, furnaces, H_2-compressors, high-pressure valves etc., but these could be considered as difficulties which usually arise when such plants are set into operation. They have been for the most part eliminated in the meantime by constructional and material modifications, such as for example:

- modification of the milling equipment, increase of the revolutions of the coal mills
- modification and different materials of the H_2-compressor's piston rings
- constructive and material modifications of the seat/cone sets of the high-pressure valves.

To start-up the plant, at first an anthracen oil was used, which was substituted in the subsequent periods by a solvent produced in the process itself.

The start-up conditions, design parameters and the values obtained up to now during operation (as of 06.06.82) in the Bottrop COAL OIL plant are compiled in Table 2.

The first 'coal-in' run was started on 25.11.1981 with a coal throughput of 750 kg/h, that is 8.5 % of the design parameter. In the course of that first run the coal throughput was increased to 2.5 t/h. Products such as light and medium oil were obtained. During this first run the vacuum residue disposal system was not yet operated. After 7 days of uninterrupted hydrogenation the plant was turned off on purpose after both hydrogen compressors had failed.

Following the repair of the compressors several runs were carried out - all of them of different duration due to different mecnanical failures - which permitted to increase the throughput and the coal/oil ratio in the slurry as far as to the design output. The largest run lasted for 25 days.

As soon as during the 6th run we succeeded in producing hard, granulated vacuum residue. The material produced is at present used for gasifying tests in the gasifying plant operated by RUHRKOHLE/RUHRCHEMIE according to the Texaco-process in Oberhausen/Holten.

Up to 6th June,1982 approx. 2000 h of 'coal-in' operation have been performed in the COAL OIL plant, with a total coal throughput of approx. 9000 t. The overall operating time - oil cycle and coal-in operation - adds up to approx. 4200 h.

To sum up it can be said that up to now only satisfying results have been obtained. We have not met any important difficulties arising from the GERMAN TECHNOLOGY-process. The problems occured at first with regard to the equipment could be eliminated to a large extent, so that an improved availability of the COAL OIL plant can be expected in the future.

5. Selected Main Aspects of Equipment Development

The operation of the Bottrop COAL OIL plant permits the testing of different equipment components with the object of optimizing the different stages of this method and of further developing the units with an adequate enlargement factor for large-scale industrial plants. In the following we should like to consider some selected aspects of equipment development.

High-Pressure Slurry Pumps

The high-pressure slurry pumps had to be designed extremely resistant to wear due to the high compression and temperature stresses as well as to the solids contained in the coal-oil-suspension.

In the Bottrop plant three high-pressure plunger pumps were installed, two of which of a different construction. The pumps were equipped with three parallel plungers each. For reasons of testing in two pumps the plungers were arranged vertically, in the third one horizontally.

The valves are installed separately from the plunger via a hydraulic column of clean oil. Part of this oil is lost during each plunger stroke and filled up with fresh oil. The column permits the plungers to operate without abrasion and avoids the wear which would otherwise inevitably arise.

Different valves and materials were used in order to gain experience with regard to wear resistance.

Pre-Heater

While the coal is heated up it swells considerably within the solvent. During this so-called gel-phase the viscosity of the coal-oil-suspension increases, although it should in fact decrease due to the temperature rising. It reaches its maximum value at 300-350 °C and does not decrease until then, its abrupt drop being furdered by the chemical decomposition of the coal. In the high viscosity region the heat transfer deteriorates considerably due to the laminar flow, and the coking tendency increases. It is for this reason that the pre-heater has to be designed with particular care. In Bottrop two different pre-heaters were installed - a convective-type heater and a radiation heater.

In the convective-type heater the heat is transferred convectively by flowing hot flue gases against some hairpin-shaped pipes, through which the coal-oil-suspension is passing. Its construction is similar to that one used during the thirties and fourties already in IG-plants. The heat transfer output is relatively low, thereby reducing to a minimum the coking tendency of the coal-oil-suspension in the hairpin pipes. The disadvantages of this type of heater consist in its largeness required and the high costs involved.

When using the radiation heater which is normally employed in mineral oil refineries, there is an increased danger of coking due to the higher temperatures. The purpose of the operational tests carried out in the Bottrop plant is to reduce this risk, at the same time utilizing the higher heat transfer value, the smaller dimensions and the lower investment cost level.

Hydrogenation reactors

Downstream of the pre-heater the reaction mixture passes, and ascends, through three reactors, which are connected in series. These reactors are welded multilayer vessels, which have been provided with an internal heat insulation for cost-saving reasons (Picture 9). This internal insulation is protected against abrasion by a thin metal shirt.

During the operation, gas - particularly hydrogen - penetrates from the reactor inside behind the metal shirt into the insulating layer, filling up the existing pores under operating pressure. In order to ensure that the gas returns into the reactor during the pressure release, the metal shirt was provided with pressure equalization outlets.

Whereas a gradual pressure release in the system ensures a presure compensation relative to the insulating layer, in case of a quick pressure release an overpressure develops in the insulating layer and may cause a mechanic deformation of the metal shirt towards the reactor inside. According to the degree of deformation the internal insulation loosens and the pressure-bearing reactor wall is no longer completely heat insulated. Under such conditions it is absolutely necessary to put the reactors out of operation and to replace the internal heat insulation together with the metal shirt in order to ensure that local overheatings of the pressure-bearing reactor wall do not occur any more once it has been put into operation again.

We are just now developing a new reactor type which in case of pressure changes, in particular of pressure drops, does not imply these technical disadvantages and ensures a maximum availability and service life. The basic element of this development is a pressure bearing metal shirt between the internal insulation and the reactor inside. It is planned to use and try-out this reactor type in Bottrop in 1983.

Encs.

Table 1: FEED AND PRODUCTS (STANDARD CASE)

Feed (t/d)	
Coal (m.a.f.)	200
Catalyst (Fe_2O_3)	8
Hydrogen	12
Products (t/d)	
Gases (C_1, C_2)	22
Propane / Butane	18
Naphtha (< 200 °C)	26
Middle Oil (200 - 325 °C)	66

Table 2: DESIGN CONDITIONS, START-UP CONDITIONS AND OPERATING RESULTS OF THE COAL OIL PLANT BOTTROP

		DESIGN CONDITIONS	START-UP CONDITIONS	MAX. OPERATING RESULTS up to 06-06-1982)
Coal Feed Rate	t(m.a.f.)/h	8,3	0,75	8,3
Coal/Oil-Ratio in Slurry	Wt.-%	40 : 60	15 : 85	40 : 60
Slurry-Rate	t/h	20,8	5,0	20,8
Temperature	°C	475	440	478
Pressure	bar	300	280	280

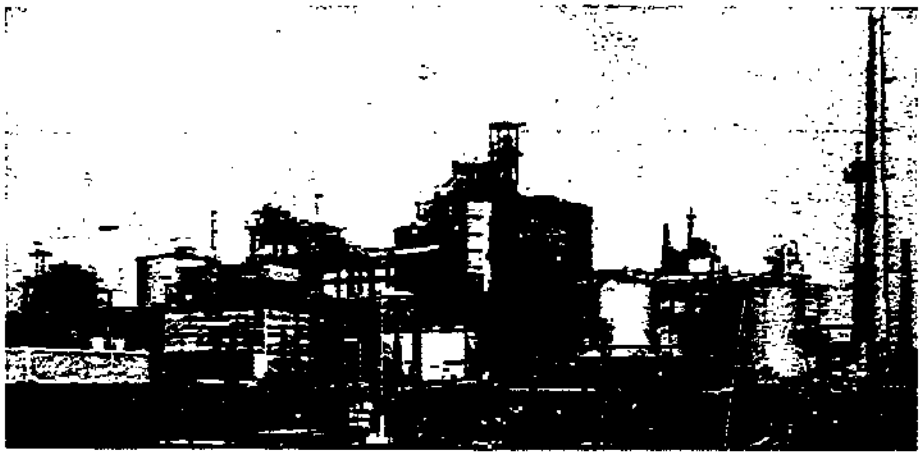


Figure 1: Coal-Oil Plant Bottrop

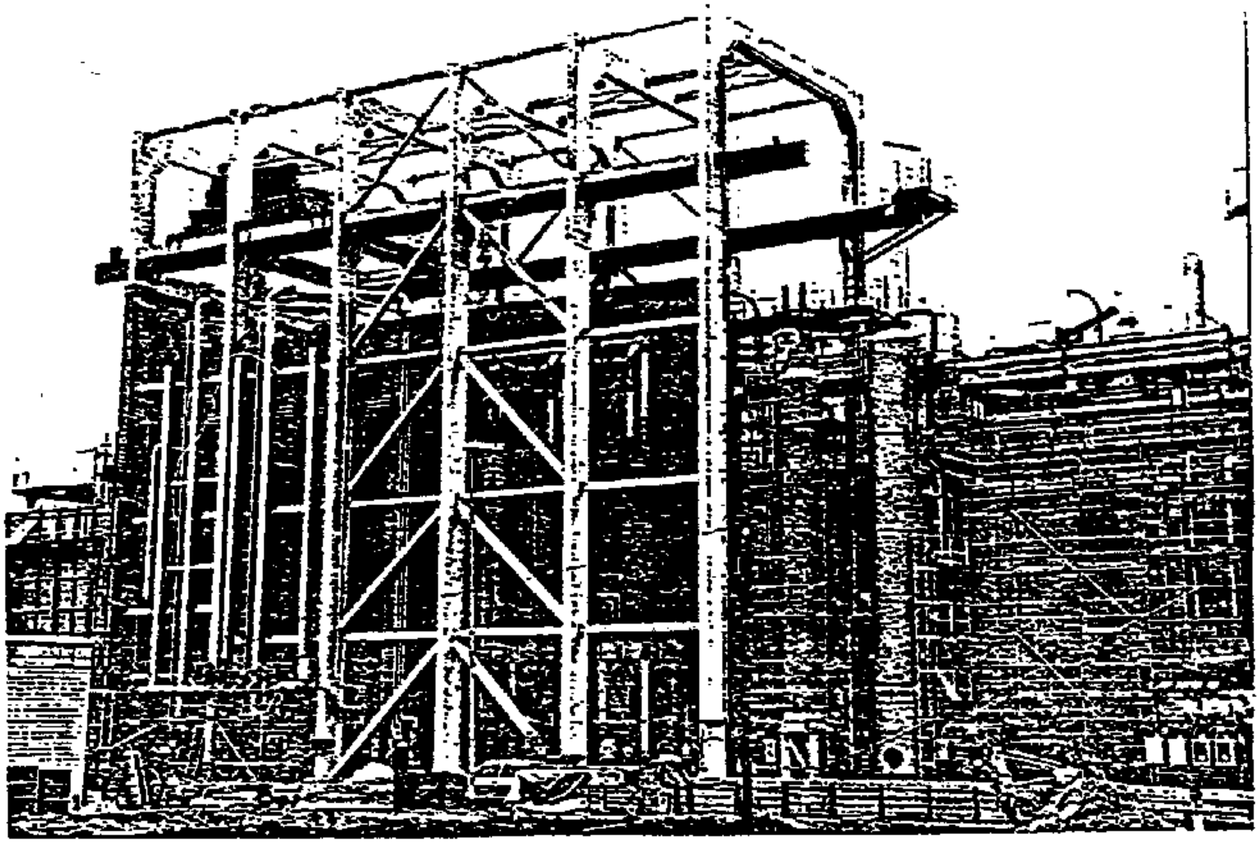


Figure 2: Hydrogenation

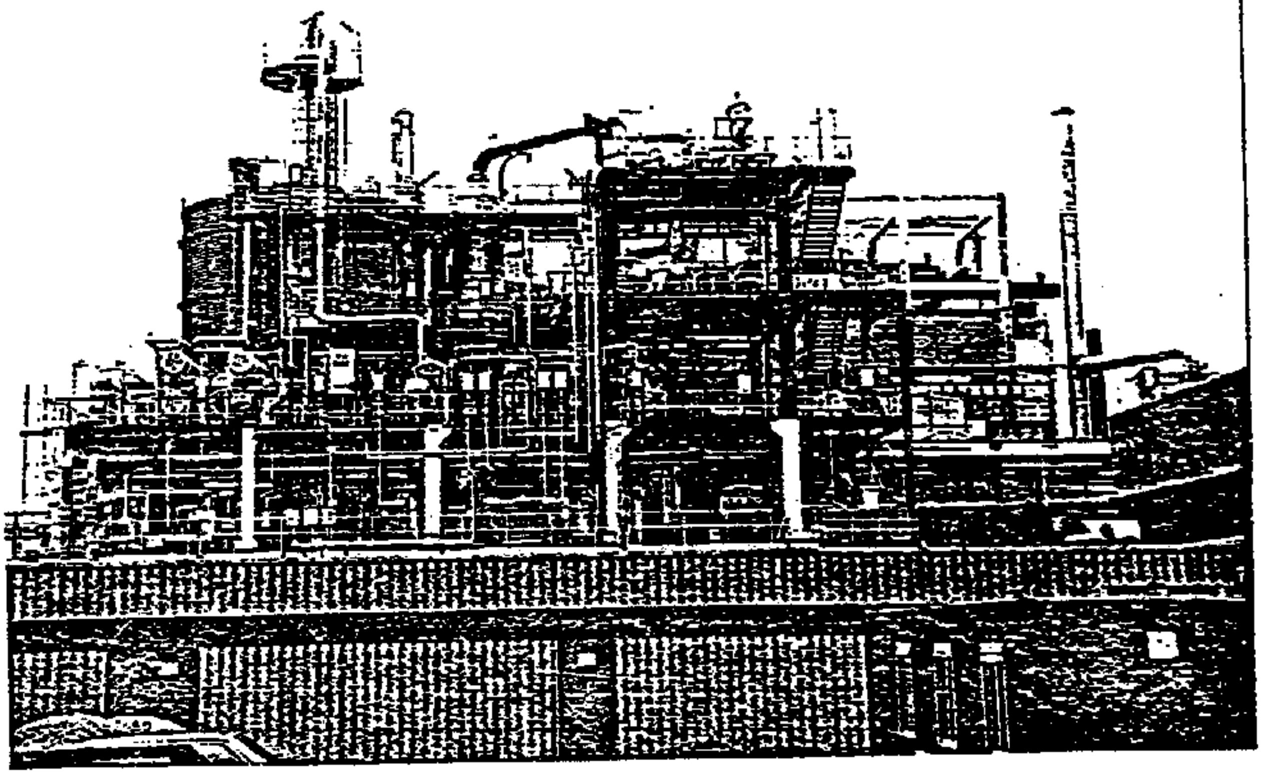


Figure 3: Distillation Units

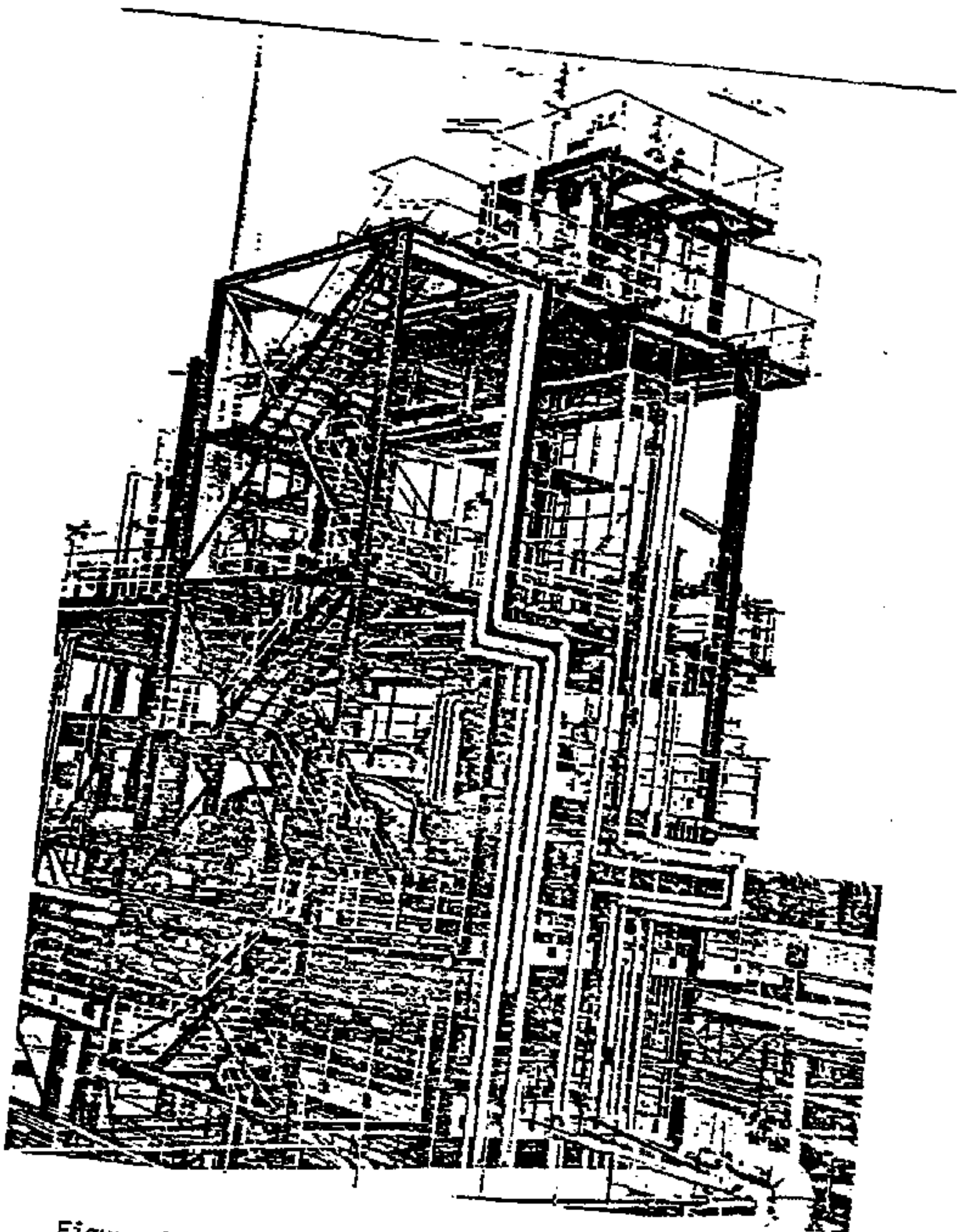
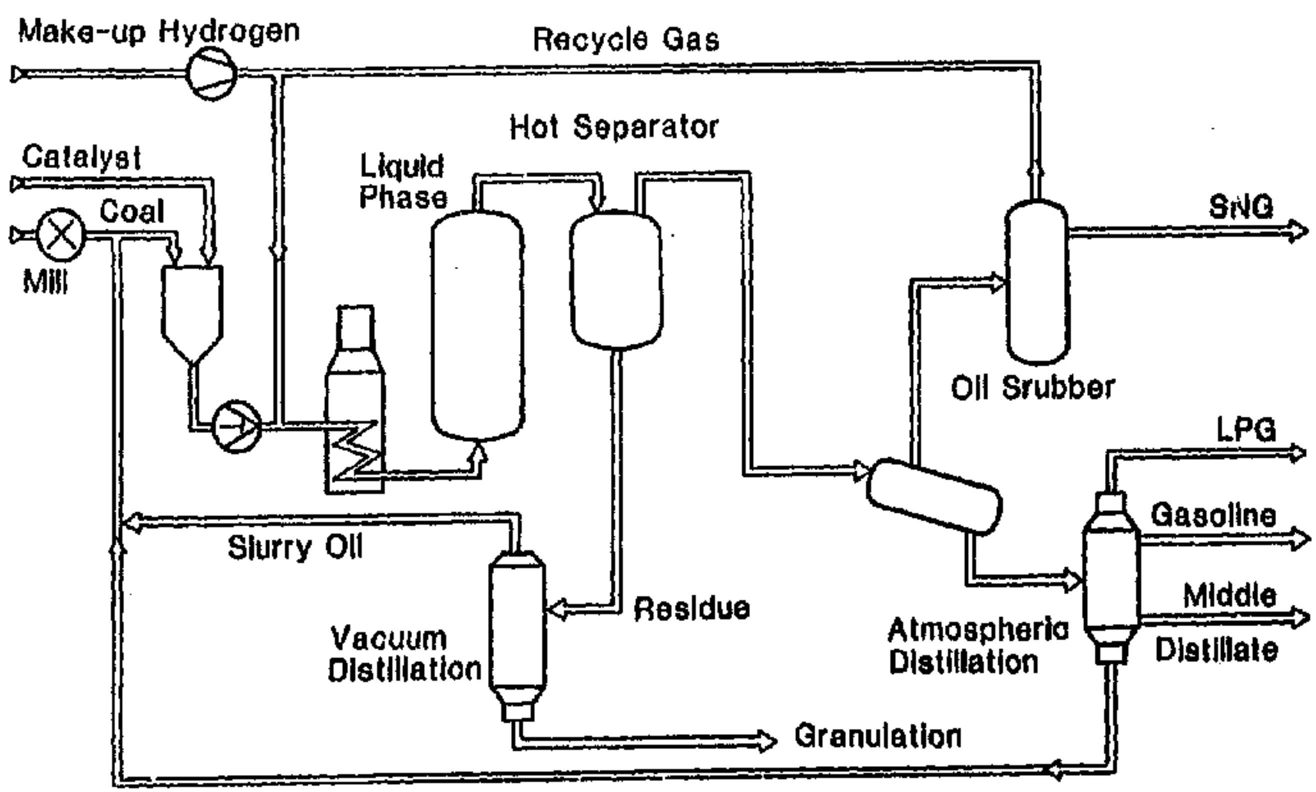


Figure 4: Residue solidification

Figure 5: Control Room

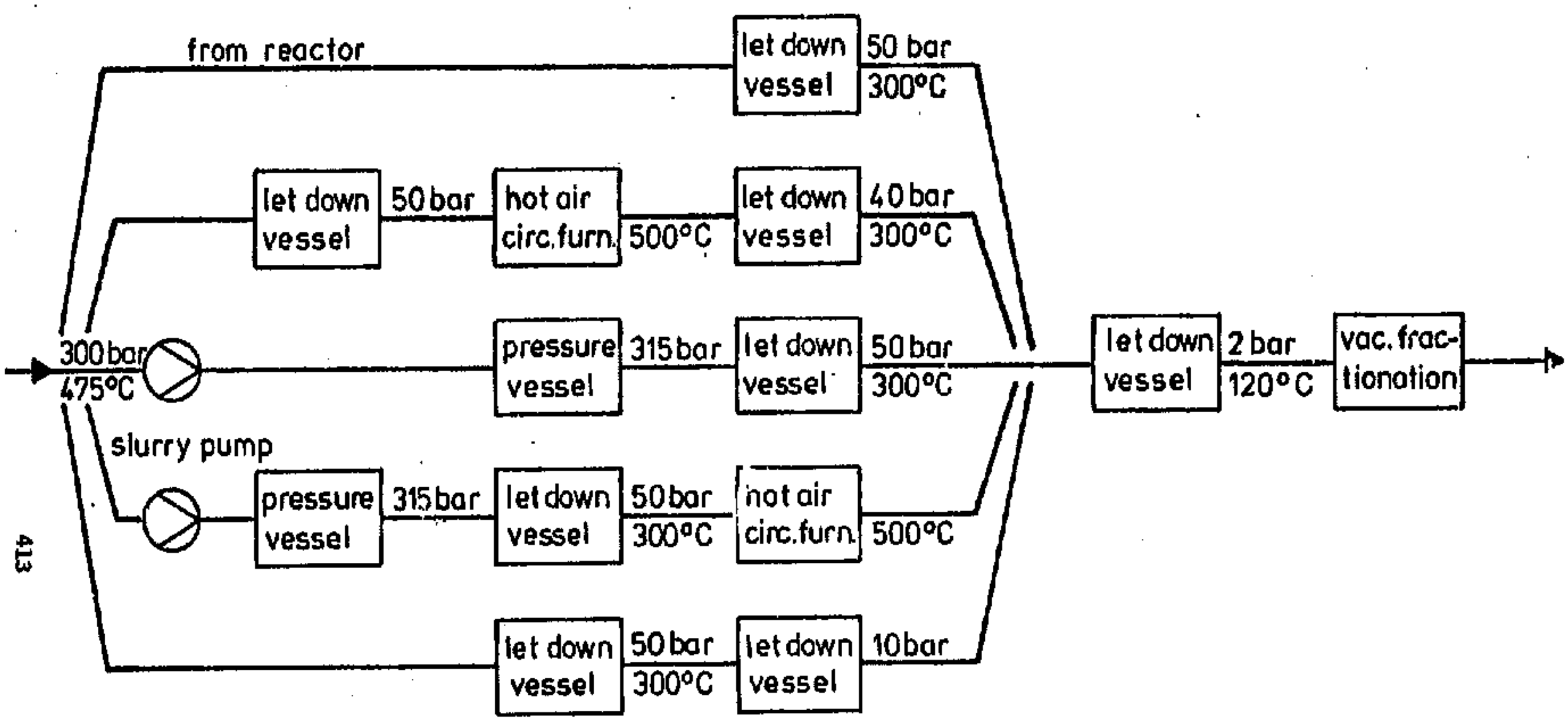


RUHRKOHLE AG
VE A OEL AG

Pilot Plant Bottrop – Flowsheet

Fig. 6

555/82 ~09-4



VEBA OEL AG RUHRKOHLE AG	PILOT PLANT BOTTROP - RESIDUE TREATMENT	Fig. 7

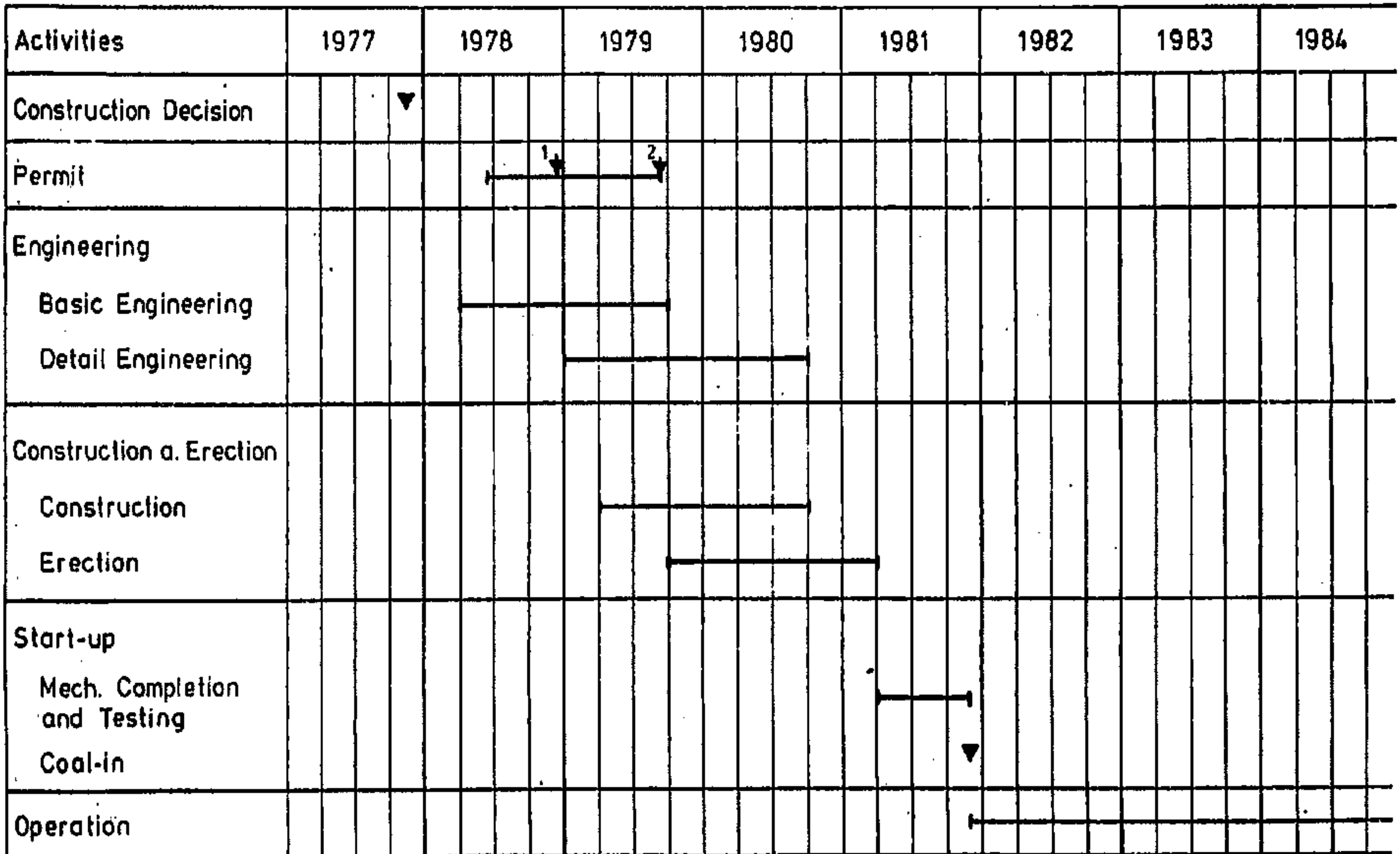
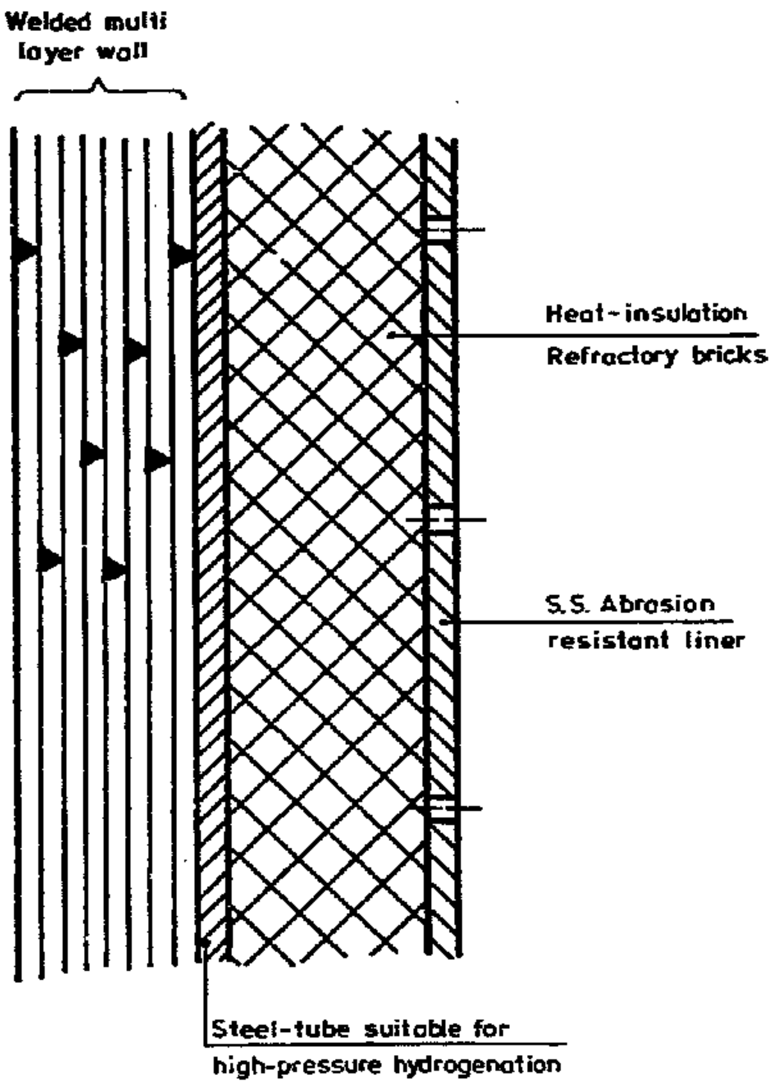


Fig. 8 Time Schedule Coal-Oil Plant Bottrop 1982



RUHRKOHLE AG VEBA OEL AG	DESIGN HYDROGENATION REACTOR	Fig. 9