Section 8

THE COAL HYDROGENATION PLANT AT BOTTROP

by

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1. INTRODUCTION

Ruhrkohle AG/Veba Oel AG have a 200 t/d-pilot plant for coal liquefaction under construction in Bottrop, Northrhine-Westphalia. Construction work was started in May 1979. Completion and commissioning of the plant is scheduled during the first quarter of 1981.

The coal oil obtained from the Bottrop pilot plant will be processed in a semi-commercial plant at Veba Oel AG, Scholven. Investigations on coal oil from a laboratory plant at Bergbau-Forschung GmbH, Essen, are already being carried out.

The whole project is mainly sponsored by the Department of Economics of the Land Northrhine-Westphalia.

2. TARGETS

The following targets have been set for the pilot plant of Bottrop:

- Plant operation in order to demonstrate the expected improvements (already confirmed on a laboratory scale) achievable by the GERMAN TECHNOLOGY versus the coal hydrogenation plants operated up to 1945.
- Optimization of the entire process.
- Testing and improving of plant components.
- Logging of basic data permitting the lay-out of commercial plants.

3. DESCRIPTION OF THE PROCESS

3.1. Background

The GERMAN TECHNOLOGY is based on the process developed by Bergius and Pier (IG-process) which was modified by introducing process steps and equipment to the most recent state of technology.

Compared to the previous IG-technology, the operational pressure was brought down from 700 to 300 bar utilizing the same cheap iron catalyst, i.e. red mud. For preparing the slurry, medium/heavy oil produced by distillation during the process is used in a ratio of 40 : 60 instead of the former thin centrifuge liquid. This prevents asphaltene enrichment in the coal slurry. The residue is to be fed at commercial plants for hydrogen production by gasification.

3.2. Process concept

Figure 1 shows a block flow diagram of the Bottrop plant. As can be seen, the hydrogenation step, properly speaking, is complemented by coal/catalyst preparation, distillation plants, gas treatment and disposal of residue.

The plant is being erected in the immediate neighbourhood of the RAG coking plant "Prosper". Crude gas, wastewater and residual granule are fed to the coking plant for reuse or treatment. The fresh hydrogen will be tapped from a hydrogen pipeline of Chemische Werke Hüls AG passing by the premises.

A more detailed description of the major process steps will be given in the following.

3.3. Coal preparation

Figure 2 shows how the coal is dried/ground and the slurry prepared.

By grinding/drying, the residual humidity of the feed coal is reduced to below 1 % by wt. and the grain size brought down to minus 1 mm. The coal is then stored in a dry bunker. After preparation of a coal slurry by admixture of medium/heavy oil in a 40 : 60 ratio, the coal is wet ground to less than 0.2 mm and the catalyst is added at the same time. The final slurry contains approx. 40 % by wt. of solids.

3.4. Hydrogenation

The hydrogenation step consisting grosso modo of high pressure pumps, heaters, three reactors in series, separators, and oil srubber, is shown in Figure 3.

The slurry is pumped from the slurry preparation tank, via high pressure plunger pumps, into the pressurized section of the hydrogenation.

High wear-resistance demands are put on the pumps because of the high pressure and temperature loads and the solids contained in the slurry.

Three pumps were installed in the Bottrop plant, one of which is meant as a spare unit. Each of them is equipped with three parallel pistons which, for testing, were arranged vertically in two pumps and horizontally for the third pump. Furthermore, inlet and outlet valves of widely varying designs and materials have been used in order to test their suitability.

Upon admixture of hydrogen and recycle gas, the slurry is heated to reaction temperature in a feed/product heat exchanger with subsequent secondary heater. At the same time, part of the exothermal hydrogenation heat is recovered in the feed/product heat exchanger.

When heated, the coal will swell considerably in the solvent. During this so-called gel phase, the viscosity of the slurry goes up although it should be decreasing with rising temperatures. Viscosity reaches a maximum between 300 and 350° C and will then decrease rather steeply, assisted by the beginning chemical decomposition of the coal. Within the high viscosity range, heat transfer is considerably deteriorated due to the laminar flow, while the coking tendency increases at the same time. This is the reason why special attention was given to the lay-out of the secondary heater; two secondary heaters of different design - one convection heater and one furnacer - were installed.

As to the convection heater, heat transfer is achieved by directing a flow of waste gases to the hairpin-shaped tubes arranged in series and which are flown through by the slurry. In this case, the heat transfer capacity is relatively low which minimizes the coking tendency of the slurry in the hairpin tubes. Disadvantages of this type of heater are its dimensions and high cost.

When using a furnace, coking tendency is higher due to the higher temperature level. One of the objectives pursued by the Bottrop plant is to reduce this tendency by utilizing the higher heat transfer potentials and reduced dimensions at lower investment cost.

After the secondary heater, the heated reaction mix flows upwards through three reactors put in series. They are multi-layer reactors which for cost-saving were provided with an internal heat-insulation coating. The internal insulation is retained towards the reactor cavity by a thin metal wall.

During operation, the gases, specifically hydrogen, will penetrate from the interior of the reactor behind the metal retaining wall into the insulation layer and fill any empty spaces at operational pressure. In order to ensure unhindered refluxing of the gas into the reactor cavity in cases of dropping pressure, pressure relief apertures were provided in the metal wall.

While with gradual destressing of the system, pressure balance towards the insulation layer is maintained, in cases of abrupt destressing, however, there may occur a pressure buildup in the insulation layer, leading to a mechanical deformation viz. bulging of the metal wall into the reactor cavity. Depending on the degree of deformation, the internal insulation may come off so that the reactor wall exposed to operational pressure is no longer heat-insulated. To avoid local overheating of the reactor wall exposed to operational pressure upon restarting, in a similar situation the reactor has to be stopped for renewal of the internal heat insulation including the metal retaining wall.

A novel reactor type is under development; it will no longer have the present drawbacks implied with pressure operations, specifically pressure drops, thus promising high availability and service life. The essential component of this development is a pressure-resisting metal retaining wall in between of the internal insulation and the reactor cavity. The new reactor type is scheduled for installation and testing also at Bottrop.

The exothermal hydrogenation reaction will be controlled by feeding quenching hydrogen in a way as not to exceed 475° C.

Subsequent to the reactors, a phase separation takes place in the hot separator, where gases and vapours are removed from the top, while liquid products and solids are discharged from the bottom. The top outlet product is then cooled in the feed/product heat exchanger mentioned earlier, by water quenching and indirect cooling. The condensates obtained in the separator sections, i.e. reaction water, quenching water and coal oil, are withdrawn or pumped to the atmospheric distillation. The remaining hydrogenation gas is then led (at operational pressure) to an oil scrubber where part of the hydrocarbons C_1 through C_5 yielded during hydrogenation will be leached, with simultaneous hydrogen enrichment. Part of the scrubbed gas will be used as recycling gas, whereas the excess is discharged as purge gas.

3.5. Atmospheric distillation

The coal oil obtained in the separator sections of the hydrogenation are subsequently treated by atmospheric distillation as shown in Figure 4. The essential components of the distillation plant are letdown vessels where the coal oil, after having been fed at hydrogenation pressure, are expanded to almost atmospheric pressure, along with removal of the light hydrocarbons, furthermore, a heater providing the heat quantities required for distillation, and a top column for the distillation separation of the coal oil into gasoline, medium and heavy oil fractions. A gasoline stabilizer column is also provided.

3.6. Vacuum distillation

Upon expanding by vacuum distillation, as shown in Figure 5, the bottom product of the hot separator will be separated into distillate oil and residue. The residue containing minerals, undissolved coal and catalyst as solids, will be cooled on a steel belt, further processed to granule which will be supplied to the coking plant.

The target is solids' enrichment up to approx. 50 % by wt.. In case such concentration is not obtainable, the bottom product from the vacuum column can be subjected to a secondary enrichment by high pressure flash. To the effect, the residue is pressurized to approx. 50 to 100 bar and mixed with hydrogen-containing excess gas from hydrogenation. The slurry/gas mixture is then heated up to approx. 460° C and expanded. The oil percentages evaporating during expanding are removed along with the excess gas. As mentioned earlier, the remaining residue will be transferred to a steel belt for cooling.

4. DESCRIPTION OF THE PLANT

Figure 6 is a plot plan of the 200 t/d-pilot plant.

Given a length of 273 m and a width of 215 m, the total space requirement is 58 700 m^2. The main process facilities, including measuring and control station, are located in four sections. With 130 m length and 100 m width, every unit has a space requirement of 13 000 m^3 or one fourth of the total requirement.

The main process facilities are connected to each other by pipe bridges. The four sections are accessible by bulk transport vehicles.

The connection between pilot plant and coking plant is provided by a belt conveying system which serves for supplying the feed coal and evacuating the residue. A flare and blow-down system is installed on the premises. Tanks are provided for intermediate storage of the coal oil fractions. Furthermore, are provided office buildings and staff facilities, workshop, store rooms and an open-air space which could be used for an eventual expansion of the pilot plant.

5. WORK PROGRESS

Ruhrkohle AG and Veba Oel AG were in charge of the basic engineering. Detail engineering was provided by Ruhrkohle Oel und Gas GmbH, by the engineering department of CWH within the Veba AG trust, and by the German engineering group Still/Didier. Authority approval was obtained.

The construction site was formally opened on May 21, 1979, by the Minister of Economy of the Land Northrhine-Westphalia.

Meanwhile, the steel structures for the coal preparation, compressor building, and distillation were completed, the hydrogenation chamber is erected and reactors as well as hot separators were installed. As to the distillation part, most of the equipment is installed. The foundations of the ancillary facilities are partly completed. The fire extinction tank is installed. Construction work of the measuring station, control station, workshop, store house and tank facilities is completed. Work at the measuring/control/adjusting and electric installations has started. The office building and staff facilities were completed so that the staff could move into the office wing in April 1980.

At the end of August 1980, approx. 60 % of the construction and installation work was completed. Completion of the mechanical work is scheduled within the first quarter of 1981.

Figure 7 gives a general impression on the progression at the construction site.

The total expenditure for the project amounts to some 300 million DM, approx. 150 million DM of which account for investment and 150 million DM for the three years' operational period of the plant (cost situation of 1976/1977).

The project is mainly sponsored by the Minister of Economy of the Land Northrhine-Westphalia.

6. FEED MATERIALS - PRODUCTS

Table 1 gives the specification of the standard coal to be fed to the Bottrop pilot plant as well as the flexibility range of this plant. As shown, a wide variety of coals may be used for hydrogenation in the Bottrop pilot plant.

Table 2 summarizes the feed materials as well as the products to be normally yielded by the Bottrop plant. These figures are based on tests ran by Bergbau-Forschung GmbH in Essen on standard coal.

As to medium oil and residues, the yield will vary, depending both on the solids' enrichment in the residue as well as on the composition of the oil for preparing the slurry.

7. UPGRADING OF COAL OIL

A market study was established for determining the present and future demand for chemical raw materials to be derived from coal oil. Permanent investigations are under way as to the processing potentials for coal oil obtained according the the German technology developed by Bergbau-Forschung GmbH.

The results of these investigations form a basis for optimization studies on a semi-commercial scale concerning the upgrading of coal oil.

Table 3 shows the properties of crude and refined coal oil fractions. The crude fractions contain high percentages of S, N, and O compounds which have to be as far as possible removed by refining if they are going to be used as gasoline, diesel fuel, heating fuel or chemical feedstock.

In a first step, the crude coal oil is separated by distillation into three fractions:

Fraction	Boiling range
Light oil	C_5 - 200° C
Medium oil	200 - 325° C
Heavy oil	above 325° C.

The following characteristics of the different coal oil fractions are determined (inter alia):

Density; contents of hydrogen, sulphur, nitrogen and oxygen; constitution analysis; phenol content and basicity; asphaltene content in the heavy fraction (gum formation).

Then the three fractions are processed in the semi-commercial plant as shown in Figures 8, 9 and 10.

Figure 8 shows the processing principals for light oil from coal. The main approach is represented by coal hydrogenation with subsequent refining of the light crude oil. With this approach, refining is followed by a reforming step for obtaining gasoline, aromatics extraction for recovery of the BTX aromatics, and steam cracking.

The light oil to be fed to the reformer has to have a combined sulphur and nitrogen content of less than 1 ppm; with a two-step refining this target is obtainable using commercially available catalysts. Furthermore, continuous investigation work on phenol extraction from light oil is being carried out.

Figure 9 is the schematic process flow for obtaining medium oil from coal. The main approach consists in cold hydrogenation and refining of the crude medium oil in order to obtain light fuel oil.

Apart from density, the specifications for medium oil from coal are comparable to those for EL fuel oil. It is not yet sure, whether it will be necessary to operate at lower densities. Refining, however, appears absolutely necessary in order to enhance storage stability and reduce corrosive properties. The target is achievable with a single-step refining and commercially available catalysts.

Figure 10 is a schematic representation of obtaining heavy oil from coal. Here, the main approach is refining and hydrocracking in order to obtain lighter hydrocarbons.

Aside from the aforementioned tests, investigations on application potentials are being carried out.

These latter may be devided in the two categories:

- Tests on gasoline/diesel fuel and heating fuel complying with given specifications, without requiring any modifications to motors or burners.
- Test considering design modifications of motors or burners to comply with the properties of untreated and weakly pretreated coal-oil fractions.

Motor characteristics are determined using different pretreated light oil. Furthermore, it is planned to use a test and simulation program for determining the relevant traffic behaviour.

The medium oil is tested in view of its application potentials as diesel fuel and EL heating fuel.

As regards its suitability as a diesel fuel, the diesel fuel characteristics (inter alia cetane number) is determined; furthermore, refined medium oil from coal is investigated in a test diesel engine operating according to the ignition system. The light fuel oil will be determined by burner tests for which crude medium oil, weakly hydrogenated medium oil and medium oil meeting the specifications are to be used. The tests were already started.

The results form this semi-commercial plant are to form the basis of planning documents for the erection of coal-oil refineries.

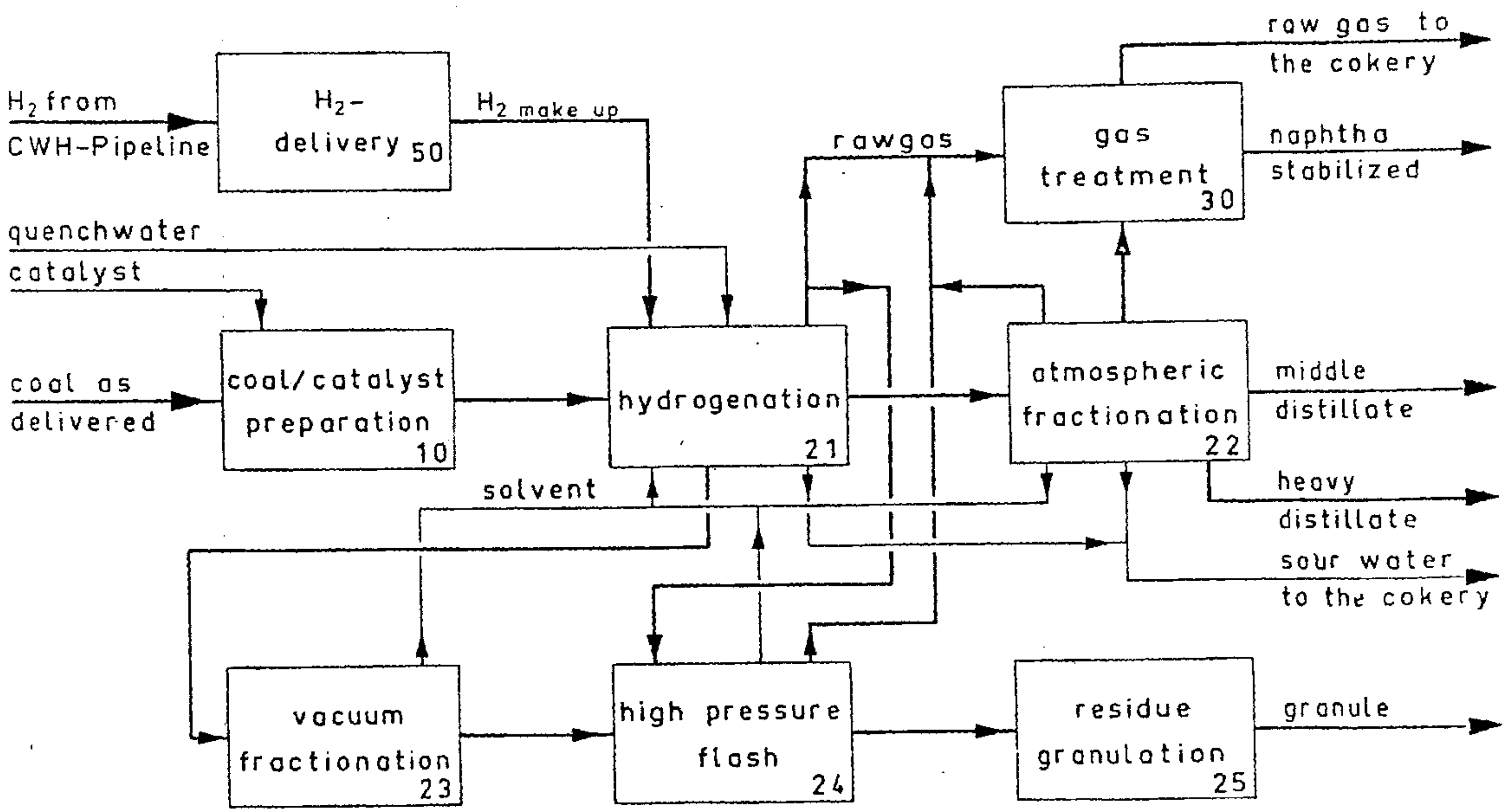

Figure 1: BLOCK FLOW DIAGRAM OF THE 200 T/D PILOT PLANT "BOTTROP"

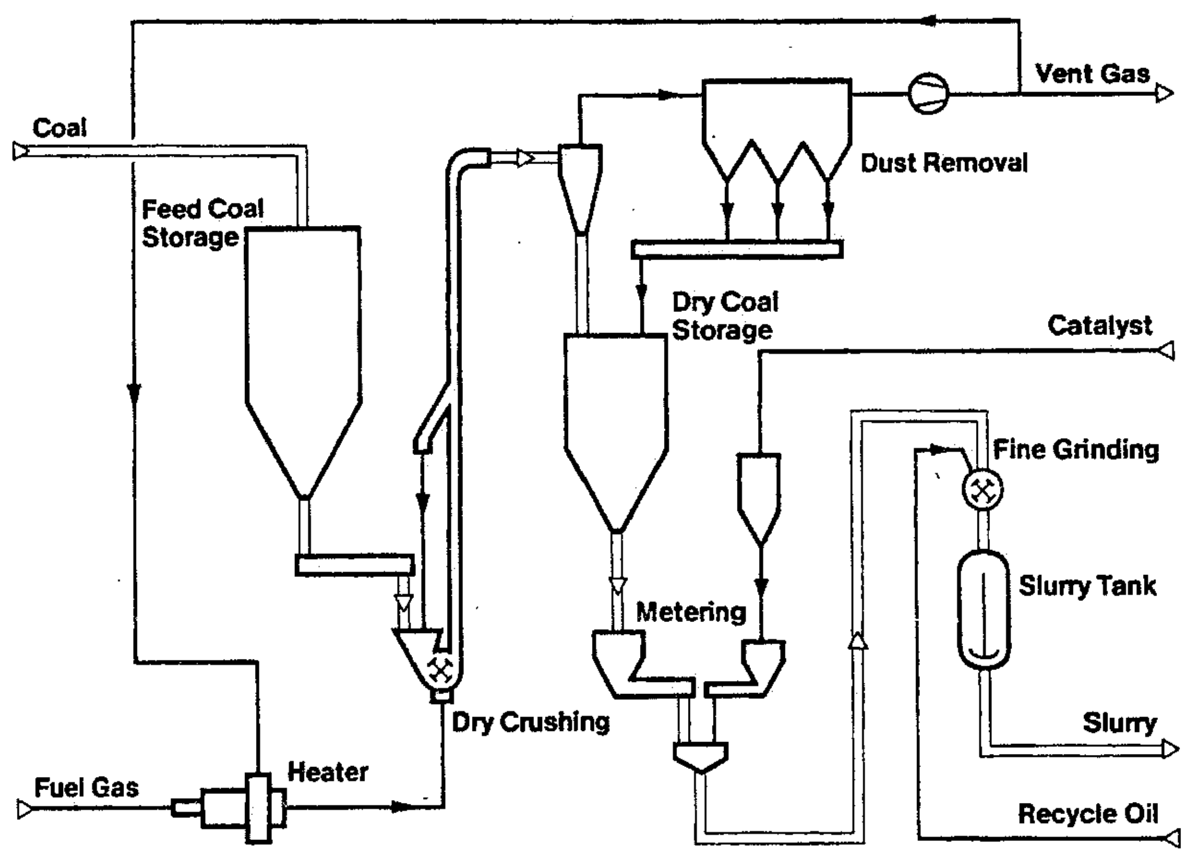

Figure 2: COAL PREPARATION

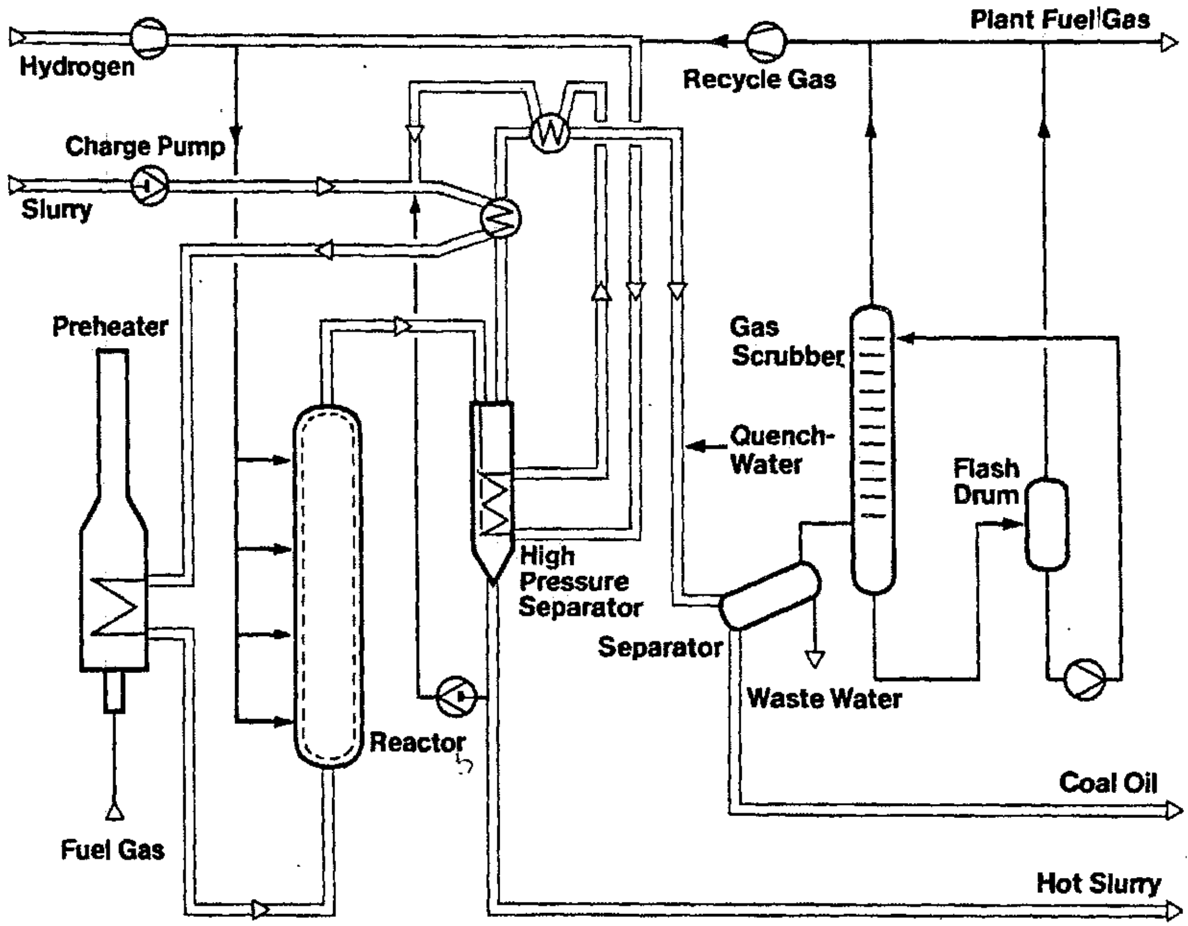

Figure 3: HYDROGENATION

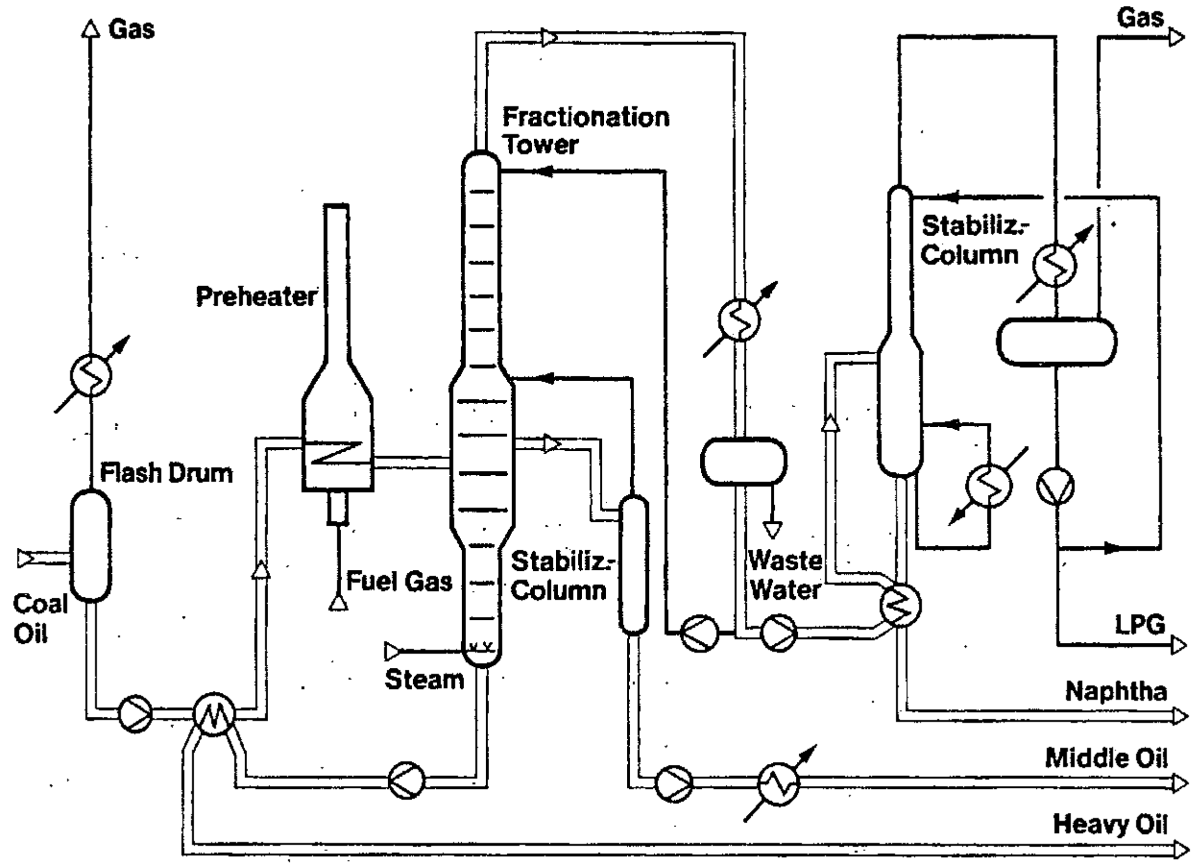

Figure 4: ATM.-DISTILLATION AND NAPHTHA-STABILIZATION

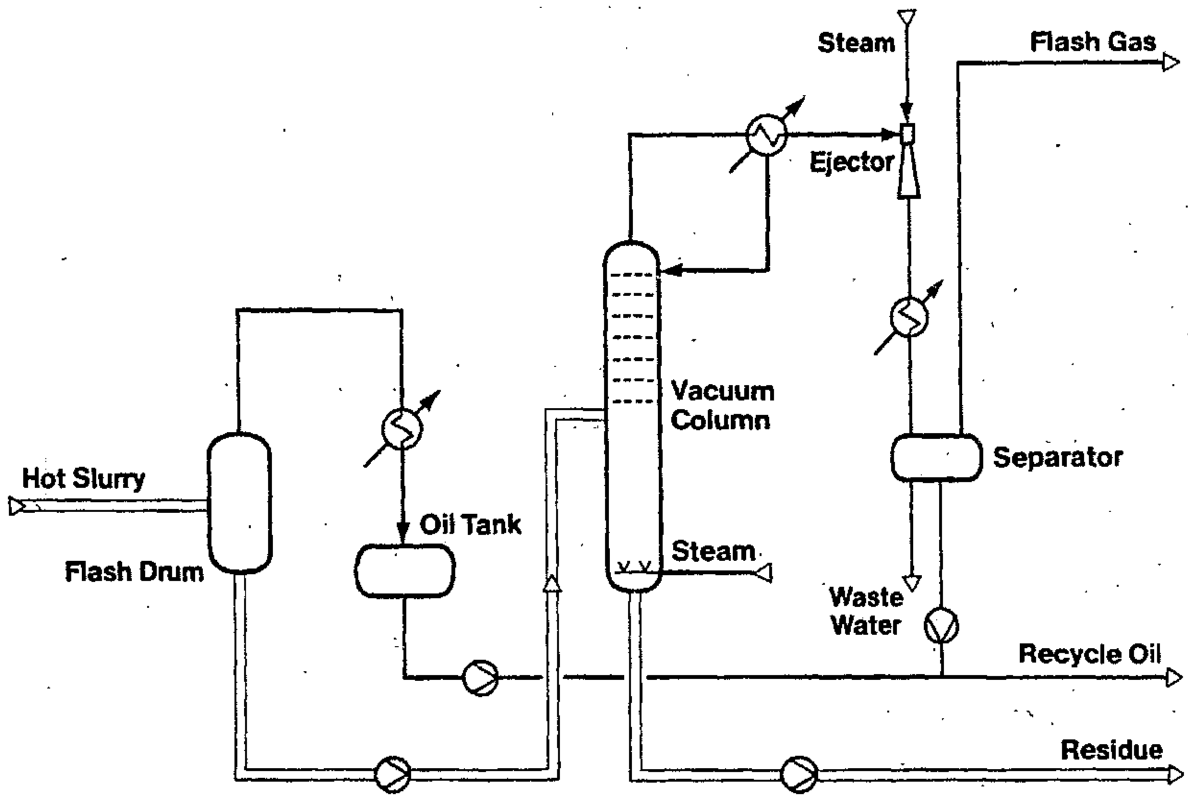

Figure 5: VACUUM-DISTILLATION

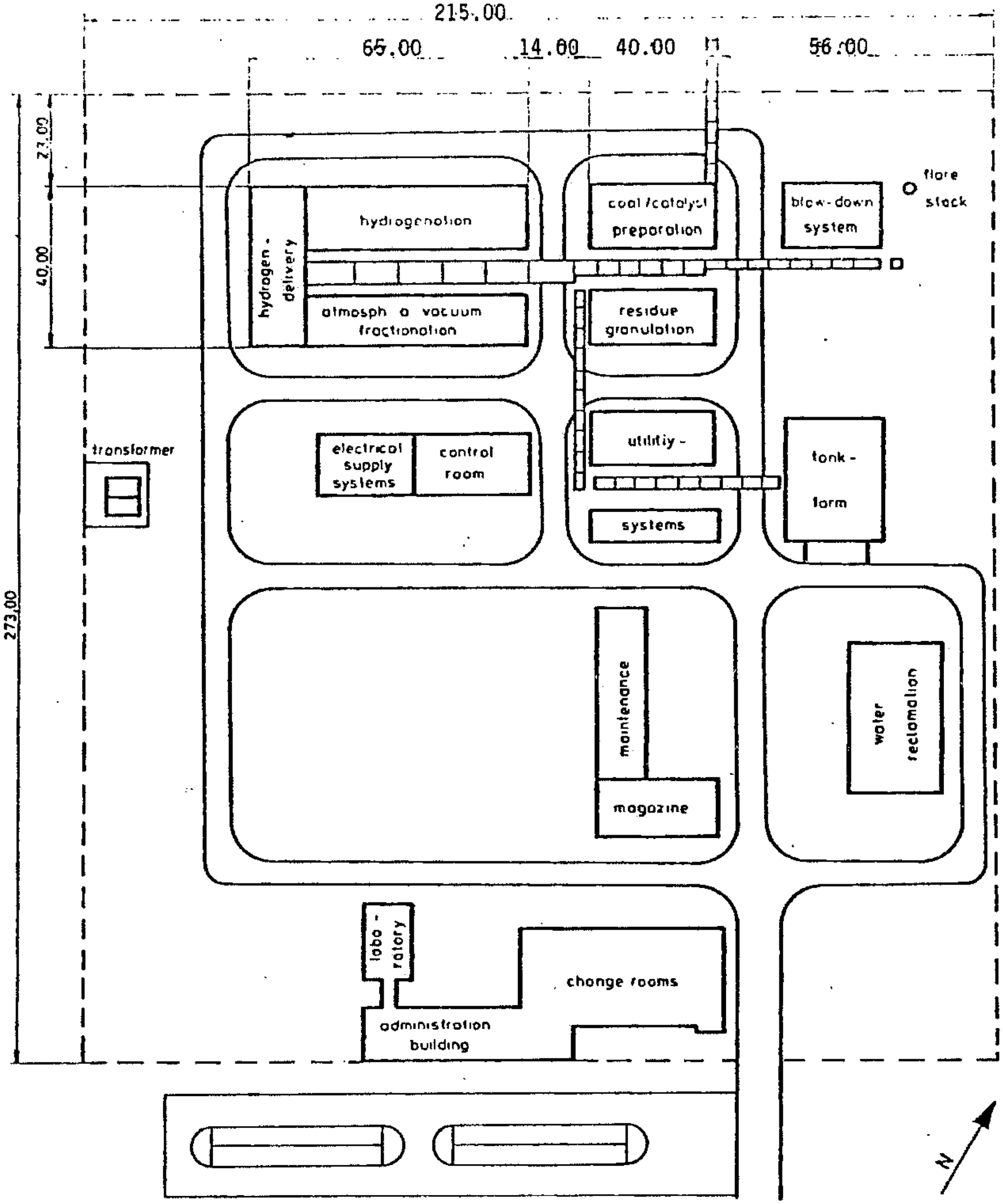

Figure 6: PLOT PLAN OF THE 200 T/D PILOT PLANT BOTTROP

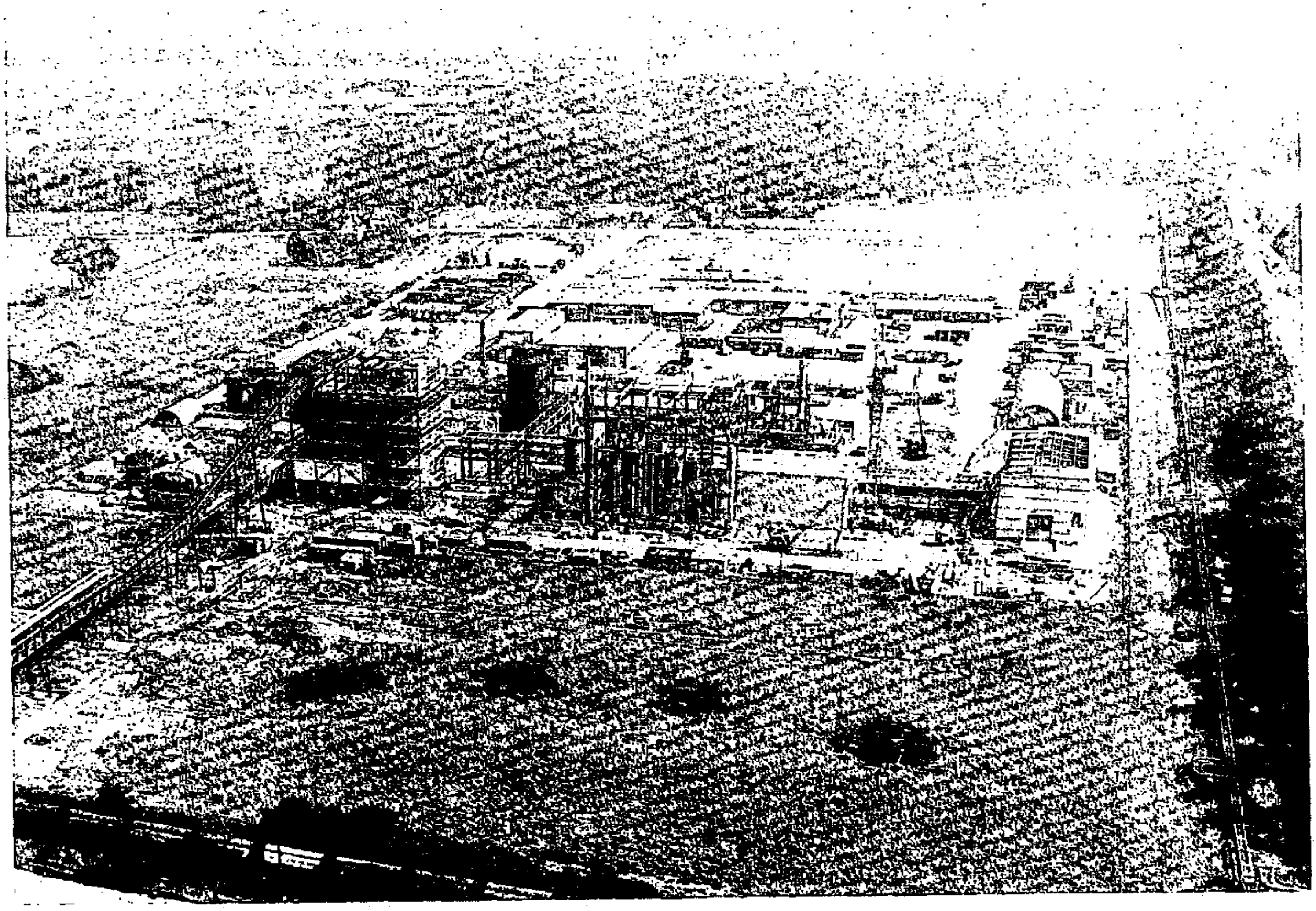

Figure 7: VIEW OF THE CONSTRUCTION SITE OF THE HYDROGENATION PLANT "BOTTROP"

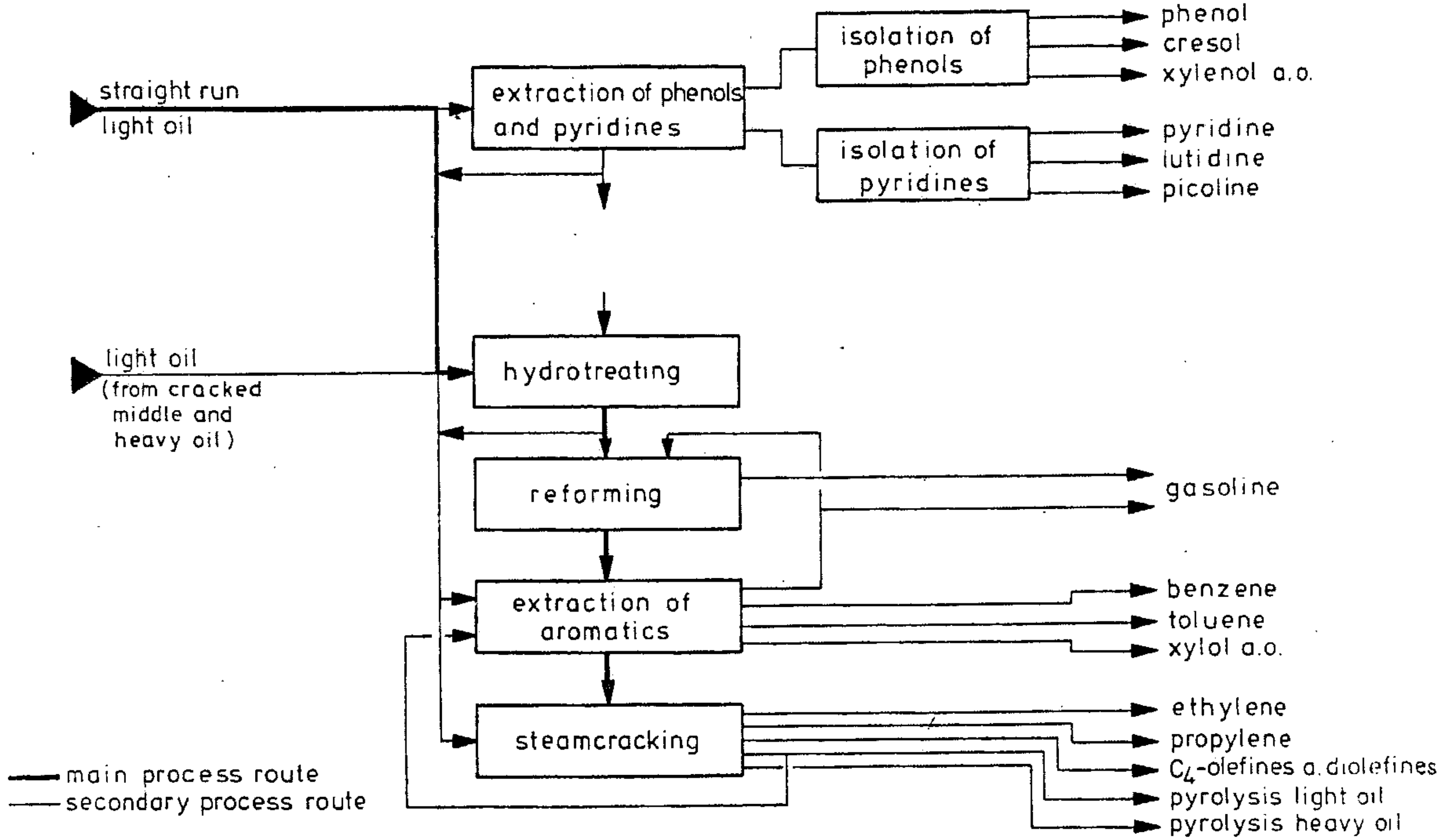

Figure 8: FLOW SCHEME OF UPGRADING LIGHT OIL

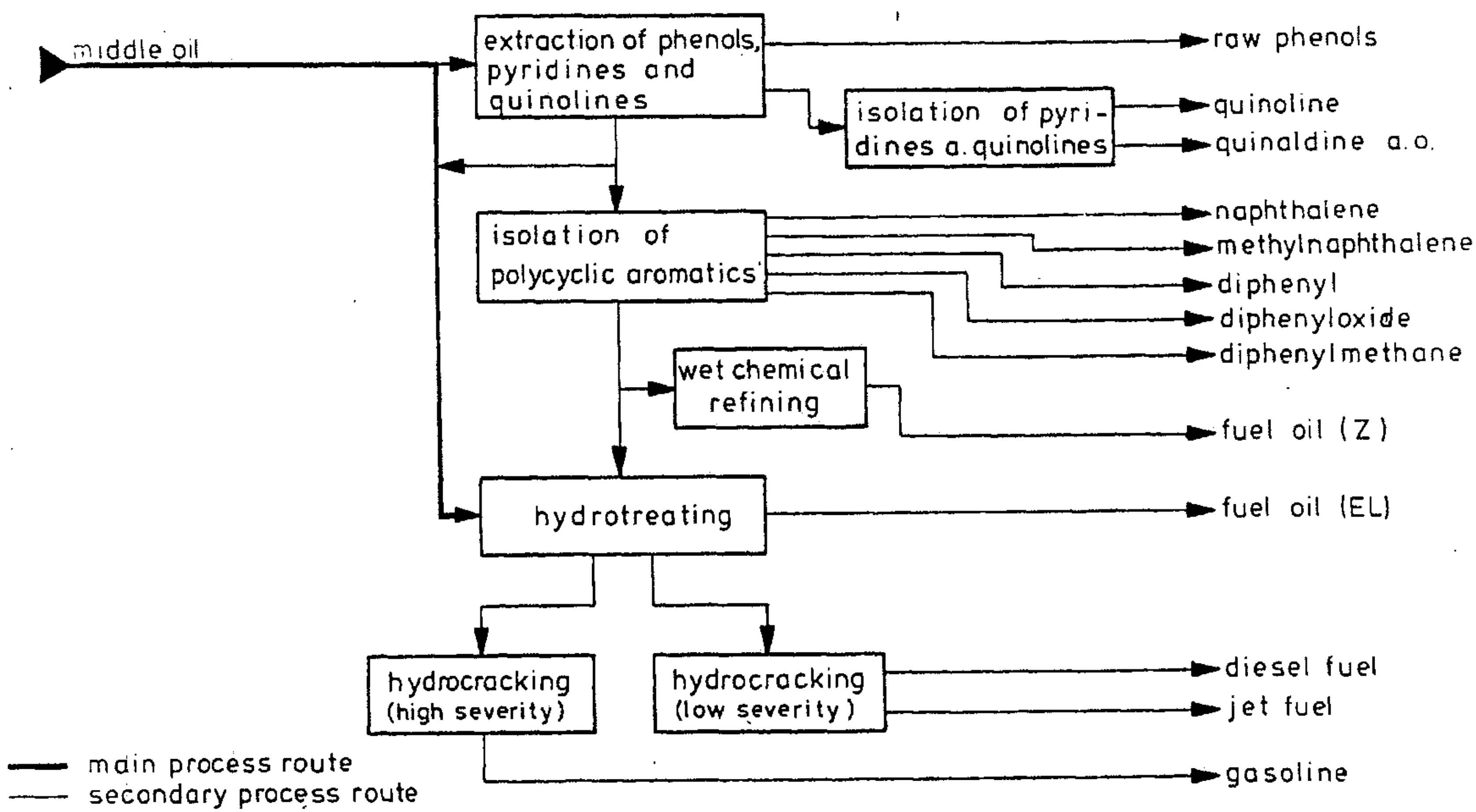

Figure 9: FLOW SCHEME OF UPGRADING MIDDLE OIL

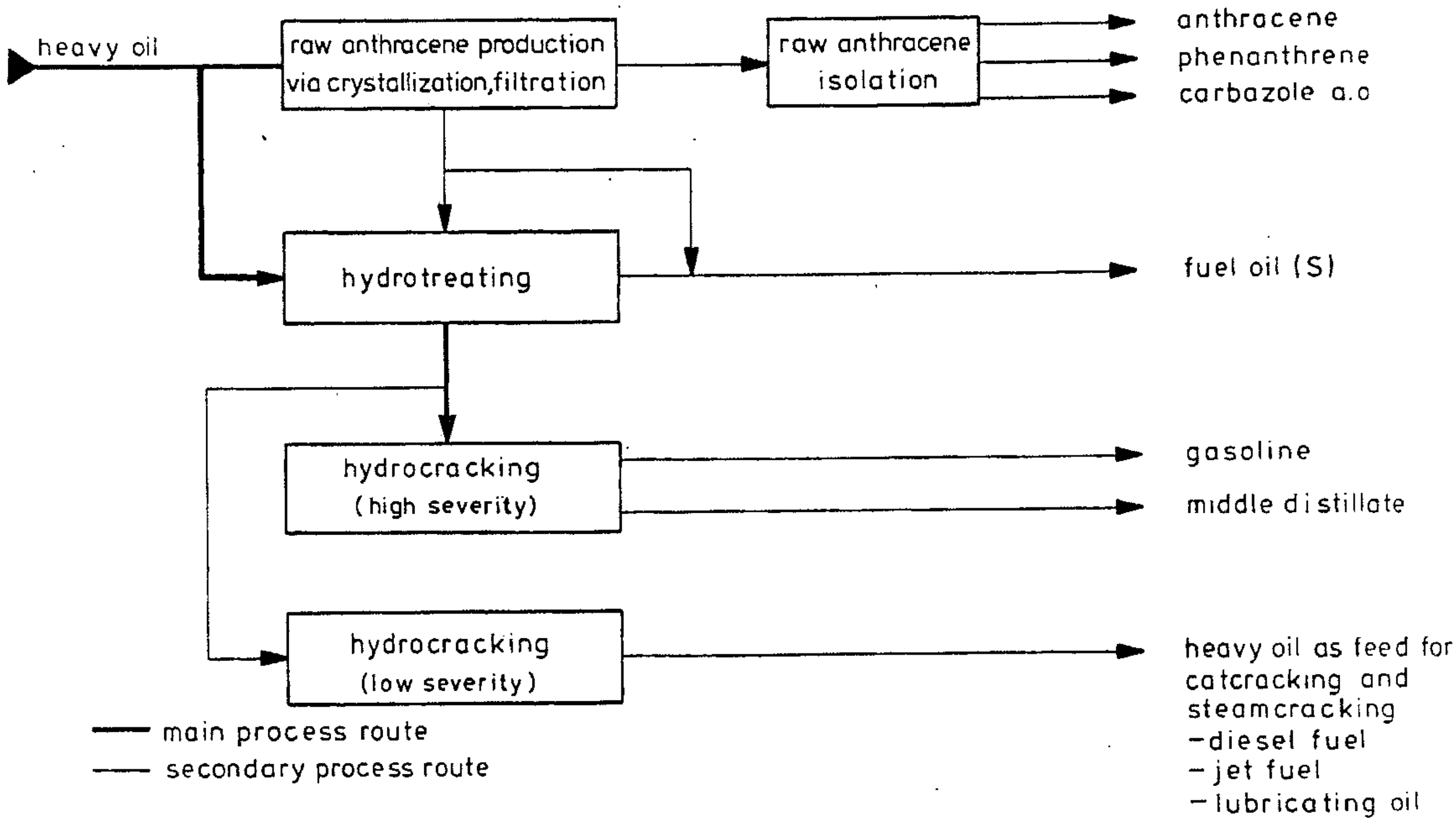

Figure 10: FLOW SCHEME OF UPGRADING HEAVY OIL

		Standard-Case	Flexibility
Moisture	wt.%(raw)	4.30	3.8 ... 14.4
Ash	wt.%(m.f)	4.52	3.0 ... 17.0
Volatile	wt.%(m.a.f.)	37.9	10.0 ... 53.8
Carbon	wt.%(m.a.f.)	84.30	75.0 ... 93.0
Hydrogen	wt.%(m.a.f.)	4.96	3.5 ... 7.0
Oxygen	wt.%(m.a.f.)	7.23	1,5 ... 17.8
Nitrogen	wt.%(m.a.f.)	1.68	1.0 ... 1.9
Sulphur	wt.%(m.a.f.)	1.67	0.5 ... 5.4
Chlorine	wt.%(m.a.f.)	0.16	0.0 ... 0.4
Vitrinite	vol.%	64	61 ... 77
Exinite	vol.%	17	5 ... 20
Inertinite	vol.%	14	1 ... 14
Minerals	vol.%	5	1 ... 9
SiO_2	wt.%	32.92	32.11 ... 64.8
Fe_2O_3	wt.%	32.20	3.71 ... 36.00
Al_2O_3	wt.%	27.63	18.48 ... 34.60
Grindability	^oH	52	> 30
High Heating Value*	kJ/kg(m.a.f.)	34,600	31,500 ... 36,800

Table 1: SPECIFICATION OF FEED COALS FOR THE PILOT PLANT BOTTROP

Feed:

Coal,m.a.f	200	t/d
Hydrogen H_2make up	220,000	m^3/d
Process Water	41.2	t/d
Catalyst(Fe_2O_3)	4.0	t/d
Power	108,000	kWh/d

Products:

Gas	61.3	t/d
Naphtha(stab.) $<$ 200°C	24.4	t/d
Middle Oil 200 ...325°C	69.0...74.7	t/d
Heavy Oil $>$ 325°C	-	t/d
Residue	68.6...74.3	t/d

Table 2: FEEDS AND PRODUCTS OF THE PILOT PLANT BOTTROP (STANDARD CASE)

specifications		light oil		middle oil	
		crude	refined	crude	refined
gravity at 15 °C g/cm³		0.865	0.827	0.990	0.993
aromat. C	wt. %	46	31.6	64	50.0
naphth. C	wt. %	10	27	28	31.3
paraff. C	wt. %	43	41.4	28	18.7
carbon	wt. %	85.25	87.80	87.40	88.5
hydrogen	wt. %	11.15	12.25	9.10	10.75
sulfur	wt. %	146 ppm	2 ppm	0.60	0.23
nitrogen	wt. %	0.24	2 ppm	0.60	0.23
oxygen	wt. %	3.5	0.1	3.0	0.4
base value	mg NH_3/l	1,100	3	8,800	2,990
CFPP	°C	-	-	-26	-47
flash point	°C	-	-	93	67
viscosity	mm²/S	-	-	3,1	2,1 (50 °C)
heating value	kJ/kg	41,000	43,000	38,500	40,900
fuel value	kJ/kg	43,000	46,000	40,500	43,250
phenols	wt. %	15.4	-	16.2	0.29
bases	wt. %	0.25	-	6.5	2.32
benzene	wt. %	-	3.9	-	-
toluene	wt. %	-	5.3	-	-
ethyl benzene	wt. %	-	2.0	-	-
xylene	wt. %	-	4.4	-	-
ROZ (clear)		-	79.2	-	-
boiling range					
IBP	°C	76	43	212	169
10 Vol.-%	°C	102	81	225	105
50 Vol.-%	°C	158	149	253	244
EP	°C	206	211	324	303

Table 3: SPECIFICATION OF CRUDE AND REFINED COALOIL FRACTIONS