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OBERHAUSEN-HOLTEN

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Chem.Fact.Investigation

Fischer-Tropsch-Ruhrchemie Synthesis Plant

Products: Gasoles, Petrol, Diesel Oil, Paraffin

(g) Process with regard to flow sheet annexed.

Watergas which is produced from coke in common manner is stored in the gasholder (2). Hydrogen sulphide is removed from the gas by means of lux mass (3).

Organic sulphur (CS, COS) is taken out in the desulphurizing equipment (4) by alkalised lux mass at temperature of 200 - 300°C. The gas is moved by means of blowers (5). For the synthesis the ratio of hydrogen to carbon monoxyd in the gas must be 2 : 1. To get this ratio a part of the watergas together with steam, is passed through tower (6) filled with catalyst. By catalytic reaction between steam and carbon monoxyde, hydrogen and carbondioxide are produced (convertgas) for the atmospheric pressure synthesis plant.

The convert gas is added in necessary amount directly to the water gas after passing heat exchanger (8). For medium pressure synthesis plant the gas has to be compressed to 10 atü (11)(12). The convert gas passes after compression the pressure scrubber (13) in which the carbon dioxide is taken out with water. The water gas passes the cooler (10) with direct water cooling before compression. The synthesis gas passes then the ovens in the atmospheric pressure plant and the medium pressure plant containing cobalt catalyst. There the synthesis of hydrocarbone takes place by catalytic reaction.

The products of both, the atmospheric pressure and the medium pressure plant, are taken out from residue gas by condensation (14 and 15) and absorption (16 and 17) with active carbon. By steaming the active carbon propane, butane and light hydrocarbons are driven off and stored separately in gasholder (19) and tank (20) After liquefying of propane and butane by compression (20) the mixture is run through the stabilizer (21). The finished products are propane/butane which got to the bottling station (22) and petrol which goes to storage tank (23).
The products from the condensation towers pass separating pit (24) and are stored in tank (25).

The paraffin gatch from the medium pressure synthesis is stored in tank (27) and goes to topping column (28). The top product is added to products from condensation (25). The residue of topping column goes to paraffin plant.

The mixed material from tank (25) is fractionated by destillation at atmospheric pressure.

The products are:
petrol which passes redistillation (34) and bleaching (35) and goes to storage tank 23.
diesel oil (160 - 230°C) after bleaching (32) to storage tank (33)
gogasine (230 - 328°C) which goes to lubricating oil plant
Residue (320 - 460°C) going to paraffin plant

PARAFFINE WAX PLANT

In the storage tanks (36) are stored the stocks of topping and fractioning plant. These are separately carried over by pumps (37, each to a group of the vacuum distillation (38), where they are divided up to 2 distillates and 1 residue each taken over into receivers (39). The paraffines of low melting point from the vacuum distillation (38) and from the manufacture of paraffin wax 5o/52 - the scouring chambers (4o) are conveyed to the cracking plant (41) of the lubricating oil factory or to shipment (42) respectively.

The raw product for making paraffin wax 5o/52 is conveyed over the caustic soda treatment device (43) to the scouring chambers (4o). The paraffines of medium melting point 5o/52° obtained here pass through bleaching device (45) and filter press (46) and are pressed to the cooling press (47).

The residues of vacuum distillation (38) pass through bleaching (48) and filtering (49) devices of the same kind as above and are then cooled and formed in scales on cooling drums (5o). The final products thus received are high melting plastic wax (51), super hard wax (52) and hard paraffines (53). The fuller earth loaden with paraffines obtained after the bleaching (54 and 55) are likewise utilized.

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(h) Description of apparatus.

14 units for production of water gas (construction Humphreys-Glasgow) with an average capacity of 6.000 m^3/h each.
Each unit consists of

producer, initial chamber, off-heat boiler, receiver and scrubber.

1 gasholder 20.000 m^3

8 desulphurizing towers (capacity 40.000 m^3 gas/h)

5 units for the removal of organic sulphur
Each unit consists of 2 towers for the mass with heat exchanges and gas heater.

3 blowers of turbine construction

2 units for production of hydrogen by conversion of CO.
Each unit containe 1 saturator, 2 contact ovens and 1 cooler

4 sets of turbo compressors for compressing synthesis gas to 10 atm. (capacitiy 20.000 Nm^3/h each.)

1 set of turbo compressors as above, with a capacity of 35.000 Nm^3/h acting all in two steps.

2 scrubbers for the removal of CO_2 combined with

2 sets of turbines and pumps to feed water saturated with CO_2 to CO_2 removing column and water freed from CO_2 from CO_2 removing column to scrubbers.

1 CO_2 removing column

1 electro motor to drive pumps
heat exchanges and injection coolers of normal construction

52 furnaces (reaction chambers) for the normal pressure synthesis process. Furnace space divided up by iron sheets penetrated by tubes. Between the sheets and around the tubes lies the contact mass, through the tubes flows water. In order to carry off the heat from the water-filled parts of the furnace steam assembling units are provided for. 12 of these furnaces are combined to units of three, the rest to such of four oven per set. Content of contact mass = 12 m^3 per oven.

72 contact furnaces (reaction chambers) for a pressure of 15 atm. in the gas part and 25 atm. in the water part. The effective space is penetrated by double tubes. The cylindric space between these double tubes is filled with catalyst amounting to 10 m^3 per oven. Otherwise arrangements are the same as with the ovens of the normal pressure system. 4 ovens are combined formerly one unit with the respective steam assembler.

2 condensers
acting directly for the normal pressure system and indirectly for the medium pressure system both of normal construction.

15 adsorbers, acting at normal pressures. 4 of which contain 18 tons of active carbon, 11 of which contain 9 ton of active carbon equipment as usual with pre-heaters, cooling coils, circulation blowers and condensers. The plant is working absolutely automatically.

4	tanks of	100 cbm	content
1	" "	200 cbm	"
1	" "	500 cbm	"
4	" "	1000 cbm	"
9	" "	2000 cbm	"
1	" "	5000 cbm	"

for storage and mixing up of liquid products obtained.

3o receivers for gasoles, petrol, diesel oil, have a capacity of 25 cbm each.

1 stabilisation unit, normal construction

1 fractionating plant, normal construction

1 redestillation plant, normal construction

1 topping plant, normal construction

Paraffin Wax Plant:

3 tanks for storage of raw material (residues) of 100 cbm content each

1 Vacuum distillation unit of normal construction several receivers and containers, normal construction

4 scouring chambers (normal)

3 desintegrators (normal)

4 filter presses (of normal construction)

3 cooling drums (normal)

L u b r i c a t i n g o i l

(g) Description of process with regard to flow sheet annexed.

The feed for the plant is stored in tank (56). The products of the dubbs olefine cracking equipment (57) are:

cracking gas (58)
petrol condenset by refrigeration (59) and
cracking petrol (60)

In the stabilizer (61) propane and butane (62) are separated and go to the bottling station. The stabolized cracking petrol is stored in tank (63), dried in tower (64) and polymerized by means of aluminium chlorid in the synthesis equipment (65). The product of the synthesis is transferred to the settling tower (66) in which the luboil containing material (upper layer) is separated from contact oil.

The luboil containing product is treated with amounts of zinc oxyd and bleaching powder to eliminate the chlorine (67). The residue of zinc oxyd and bleaching powder is filtered off in kellyfilters (68) and filter presses (69). Light petrol (73) and heavy petrol (74) are taken out from the chlorine free product by distillation at atmospheric pressure. They are stored in tank (23) and (33). The residue of atmospheric distillations (raw oil) is fractionated in vacuum distillation (71). The products of distillation are subdivided in prerun first, second, third distillat and residue. The prerun goes to tank (33) containing dieseloil, the other products are stored in tanks (76). The different distillates and the residue are mixed to lueb-oil. The mixture is treated with bleaching powder (78), which is filtered off in kelly-filters and filter presses)79). The finished lubricating oils for shipping (81) are prepared in the mixture (80).

(h) Discription of apparatus.

The equipment of lubricating oil plant comprises:

1 dubbs olefine cracking outfit of special construction (U.O.P.C., USA.)

1 stabilizer

7 polymerisation units equipped with stirrer, heating and cooling coils

6 dechlorination units equipped with stirrer, heating and cooling coils

1 distillation outfit for atmospheric pressure

1 distillation outfit for vacuum distillation
4 kelly filters
4 filter presses
gasholder, tanks and receivers.

sulphur from synthesis gas of Ruhrchemie-Fischer-Tropsch process. Deliveries destinated chiefly to factories residing in the British zone, besides shipments to Upper Silesia and exports to Swizzerland. There the mass is used for purification of gas which serves to another chemical process.

Gasoles as a motor fuel

We are at a loss to give any exact dates about the destination of our shipments for the pass, this product having been sold by the Benzol-Verband or Zentralbüro für Mineralöl respectively. According to the nature of this business steel cylinders filled here might chiefly have been left in the British zone, where as high pressure tank waggons could have been despatched to more distant regions. Exports never have been made. It may be assumed that the greatest part of our production remained in the region now under British occupation.
Sales were made by the different destributing agencies of BV or ZB directly to motor lorry users.

Petrol, diesel oil

The motor fuels were either sold through BV od ZB, which mixed up our products, as they could not be used as fuels as they were. No exports. Same considerations as above as to destination and categories of customers.

Low melting paraffines

These paraffines with a melting point of about 3o° C were chiefly sold to consuments and merchants residing in the British zone. Categories of customers which got known to us, were e.g. manufacturers of leather oils, leather fats and shoe-polish.

Paraffin wax 5o/52

Sold chiefly to firms residing in the British zone, deliveries to the rest of Germany and exports of no importance.
Categories of customers: Manufacturers of candles, vaseline, shoe-polish, waxed paper, cosmetics.

Hard paraffin wax of highest melting point (9o/95°C)

Sold all over Germany, statistics not available, some exports.
Main categories of customers:
Manufacturers of ceresines, candles, floor-polish, waxed paper, electrical appliances, synthetic lubricants, artificial flowers, emulsors, impregnants, boot-polish, textile auxiliaries.

Lubricating oils

Main part of our lubricating oil production was sold and stocked by Wifo, which distributed it for the use in automobile motors. Regional distribution not known by us. Few deliveries for electric and other purposes. No exports.

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C a t a l y s t m a s s .

(g) Description of process with regard to flow sheet annexed.

The used catalyst, returned by the synthesis plants, is dissolved under heating in concentrated nitric acid (1). The solution is filtered off from undissolved kieselgur in filter presses (2) The clear filtrate, containing the soluble components of the catalyst (cobalt, thorium, magnesium) and the soluble impurities of the kieselgur (iron, aluminium, calcium) is treated to remove the impurities. By the first precipitation, which is aneutralisation with soda ash solution (3) to Ph 5,5 the iron and aluminium and also the thorium is thrown out. The latter is recovered by special process (4). In the second precipitation step the solutions are freed from calcium by means of sodium fluoride (5).

The pure solutions are brought to the composition necessary for the precipitation of catalyst by diluting and adding thorium and magnesium as nitrate solutions.

For the precipitation of catalyst the solution of catalyst components is heated to boiling point and run into boiling solution of soda ash (6). When the precipitation is finished the kieselgur is added (6). After thoroughly mixing the suspension is filtered (7) and the catalyst washed with hot destilled water, which is prepared in a separate equipment. Following the filter cake is charged to the dryer (9) by special arrangement (8) The dry product is calibrated by means of special screens (1o). Over size and fines are separated from the material of 1 - 3 mm size by vibrating screens (11).

The catalyst has to be activated by reducing part of the cobalt content (5o - 6o %) to the metallic state. This is done by treatment with hot hydrogen at a temperature of about 4oo° C (12). The reduced catalyst is cooled by means of circulation nitrogen and saturated with carbon dioxyd. This latter treatment is necessary to prevent quick oxydation during shipping and loading in the synthesis ovens.

(h) Description of apparatus.

The equipment for production of cobalt, catalyst comprises:

18 dissolving tanks (breacklined or of acid resistant material)
34 filter presses (wood-iron)
4 dryers (steam heated)
2 rotary dryers (gas heated)
4 gas heaters
1 gas drying outfit (silica gel)
1 gas cooling outfit (refrigerating)
1 6-stage equipment for destilled water

All equipment is of normal techn. construction.

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d e s u l p h u r i z i n g m a s s .

(g) Description of process with regard to flow sheet annexed.

The mass used by Fischer-Tropsch synthesis plants for removal of organic sulphur from the synthesis gas is prepared from the residue of bouxit treatment (1) which contains about 5o % moisture by mixing it with calc.soda ash (2). The wet mixture is calibrated on special screens (3) and dried (4). Over size and fines are separated on vibrating screens (5). The finished product contains about 33 % sodium carbonate.

(h) Description of apparatus.

The equipment comprises:

6 mixers
3 dryers (steam heated)
6 calibrating screens
2 vibrating screens
2 dust filters

All equipment is of normal techn.construction.

7 (a) (continued)

ITEMS	Requirement for 1932		Requirement for 1938		Requirement for 1943		Requirement for 1944	
	normal capacity	actual production	normal capacity	actual production	normal capacity	actual production	normal capacity	actual production
FISCHER - TROPSCH - RUHRCHEMIE - PRODUCTS								
Products 1) to 8)	tons	tons	tons	tons	tons	tons	tons	tons
Coke	-	-	193 000	162 486	350 000	281 485	350 000	193 956
Products 1) to 3) (Raw Products from outside)								
	tons	tons	tons	tons	tons	tons	tons	tons
Cracking stock	-	-	-	-	10 000	13 940	10 000	8 573
Products 2) and 3), 7) and 8) (Raw Products from outside)								
	tons	tons	tons	tons	tons	tons	tons	tons
Cracking petrol	-	-	-	-	10 000	1 643	10 000	3 274

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7 (f) SOURCES OF EACH RAW MATERIAL:

Coke oven gas	Gutehoffnungshütte Oberhausen and Steinkohlenbergwerk Neumühl
Lime	Rheinische Kalksteinwerke Wülfrath
Sulphuricacid	Deutsche Ammoniak-Verkaufsvereinigung Bochum
Cobalt	Gesellschaft für Elektro Metallurgie Dr. Paul Grünfeld, Berlin-Charlottenburg 2
	Union Miniere du Haut Katanga, Brüssel Montagne du Parc 8
Thorium oxide	Auergesellschaft A.G., Berlin N 65
Soda	Sodasyndikat Bernburg
Infuserial Earth	Vereinigte Deutsche Kieselgurwerke G.m.b.H., Hannover 1
Luxmasse	Luxmasse G.m.b.H., Ludwigshafen /Rhein
Cracking stocks and cracked petrols	Different factories operating the Fischer-Tropsch-Ruhrchemie process
Coke	Bergwerksgesellschaft Hibernia Zeche Scholven, Herne
	Gutehoffnungshütte A.G., Oberhausen, Zeche Jakobi.

II.

F I S C H E R - T R O P S C H P L A N T .

The Fischer-Tropsch plant is a plant for the synthetic production of hydrocarbons. It compdses the following productions:

1) Water gas production
2) Synthesis plant
3) After-treatment
4) Paraffin plant
5) Lubricating oil plant.

II. General Survey.

For the synthetic production of hydrocarbons according to the Fischer-Tropsch process Ruhrchemie uses coke as raw material. From the latter water-gas, i.e. the mixture of carbon monoxide and hydrogen necessary for the synthesis, is produced in known manner by the alternative action of air and steam in a fully automatic generator plant combined with a waste heat utilizing equipment. With regard to the activity and life of the highly sensitive catalyst the gas must be freed to a large extent from sulfur compounds. Therefore, hydrogen sulfide is removed by purification over Lux mass (ferric hydroxide) and organically bound sulfur is eliminated by a hot purification of the gas over shaped soda-containing Lux mass.

The mixture used for the synthesis must be richer in hydrogen than water-gas, it must approximately correspond to the ratio 1 : 2 necessary for the formation of hydrocarbons. For this purpose, part of the water-gas produced is transformed by catalytical reaction with steam into converter gas and mixed again with the rest of the water-gas. In the <u>normal pressure synthesis</u> this is done directly and in the <u>medium pressure synthesis</u> after previous separate compression of the two gas components to 8 - 10 atm. gauge pressure, the pressure being utilized to remove the carbondioxide from the converter gas by washing with water under pressure in order to thus obtain a gas for synthesis poorer in inert components.

Both synthesis processes use the same catalyst which contains oxides of cobalt, magnesium and thorium on kieselguhr as carrier and both practically work in the same temperature range of 180 - 200° C. In the medium pressure synthesis the slightly raised pressure gives rise to a stronger shifting of the boiling range of the products so that instead of the lighter, petrol-like hydrocarbons heavier ones, especially larger quantities of paraffins with higher melting points are obtained. The synthesis gas is worked up by a two- or three-stage process with intermediary condensation of the oil-like products and the reaction water by direct or indirect water-cooling. The petrol vapors as well as the gaseous hydrocarbons which can be used as fuel are removed in a following adsorption plant by means of activated carbon, in the medium pressure synthesis after previous expansion of the final gases containing the said vapors.

By distillation of the crude products thus obtained there are prepared by after-treatment: fuel gas and petrol (stabilization plant), Diesel oil and Kogasin (fractionating plant) gatsch (jelly-paraffin) paraffin in plates and hard wax (paraffin plant).

The highest capacity of both synthesis plants amounts to yearly 70 000 tons of hydrocarbons. About 50 % thereof are produced by "Aufbaustufe I" (reconstruction stage I) which, according to the scope of the damages occurred, provides first of all the repair of the medium pressure synthesis plant, the plant for producing water-gas belonging thereto and the other appliances necessary for operation.

The total quantity of the products obtained according to both synthesis processes may be classified about as follows:

		normal pressure synthesis:	medium pressure synthesis:
		percent by weight	percent by weight
propane, butane	fuel gas	13	9
boiling range 35-160°C.	petrol	45	36
" " 160-230°C.	Diesel oil	18	16
" " 230-320°C.	Kogasin	16	18
" " 320-400°C.	gatsch	5	12
" " 400-460°C.	paraffin in plates	1	2
" range above 460°C.	hard wax	2	7

A plant for the production of synthetic lubricating oils is annexed. The olefinic hydrocarbons required therefor as starting material are obtained by cracking of Kogasin and gatsch in a Dubbs plant. The olefines are polymerized by means of aluminium chloride. The crude polymerization products formed are freed from chlorine, filtered and subsequently worked up by distillation under atmospheric and under reduced pressure. The maximum capacity of the lubricating oil plant amounts to 18 000 tons yearly. The output of the available Dubbs cracking plant, however, suffices only to produce the starting material for a production of 12 000 tons of lubricating oil per year, the simultaneous use of products purchased from other sources being necessary. The condition of the plant is such as to allow again this amount of production. If the total capacity of the lubricating oil synthesis proper shall be

utilized, one of the cracking plants available in other synthesis works must also be operated while using further amounts of products from other sources.

Annexed hereto are the descriptions of the different processes and plants, the schemes and flow-sheets belonging thereto as well as detailed statements as to the condition of the different plants and the work necessary for repairing them.

A synthesis plant was being constructed which by using the existing medium pressure contact furnaces should directly produce hydrocarbons rich in olefines for the preparation of lubricating oils and fatty alcohols. This synthesis is possible with use of water-gas in a cyclic process, the proportion of fresh gas to circulating gas being 1 : 3, and that under the conditions of the medium pressure synthesis at somewhat elevated temperatures and with use of the same catalyst. For after-treatment the resultant final gas should be conducted with addition of converter gas to the succeeding normal pressure synthesis.

II/1) Water-gas production including feed water purifying and re-cooling plant.

(see the drawings: folder II/1)

General:

The water-gas necessary for the synthetic production of hydrocarbons is produced by a generator plant working fully automatically according to the system of Humphreys and Glasgow, London. The plant has been built by the firm "Demag", Duisburg, as licencee.

It comprises 14 generators: 7 thereof having a shaft diameter of 3 450 mm, built in 1936 (construction stage I), 4 having a shaft diameter of 3 550 mm, built in 1938, all with wet ash removal and, furthermore, 3 of the same diameter but with dry ash removal, built in 1943 (construction stage II) arranged at both sides of the engine house. The corresponding gas outputs amount to about 6 200 and 6 800 Nm^3/h (= cu.m.p.h. under normal conditions [0°C. and 760 mm Hg]). Taking into account the necessary reserves for maintenance and interruptions of service the total gas output of the plant amounts to 65 000 Nm^3/h. The necessary coke, having a size of 40 - 100 mm and containing at least 85 % of carbon is supplied from neighboured coking plants; it amounts to 1000 tons per day. The transportation is effected by special trains. The buckets containing 10 tons of coke are lifted by two travelling cranes, each having a lifting power of 16.5 tons, and emptied above the coke bunkers.

A special re-cooling plant with 2 cooling towers supplies the necessary cooling water. The feed water is supplied by a centrally situated water purifying plant which is described separately.

Plant and apparatusses:

Each group of generators comprises a generator and the coke bunker arranged thereover, having a capacity of 240 tons, the necessary devices for charging coke and discharging ashes, the ignition chamber, the gas receiver, the waste heat boiler and the washer.

The lower part of the cylindrical generator forms a jacketed vessel (A) for low pressure steam (2.5 atm. gauge pressure). Its cooling action on the contents of the generator prevents a caking

together and adhering of slags to the wall. The upper part is lined with refractory bricks and carries at its top the charging device (B) which like a sluice is provided with an upper and inner closing device. The grate (C) mounted rotatably for removing the ashes is secured against the atmosphere by a water seal. This device consists of a large tub filled with water. A special dip closure arranged under the jacketed walled vessel dips into the water bath. The tub itself carries the oval grate whose upper part consists of rings arranged in a stair-like manner. Two shovels, one at each side, dive into the tub. By turning the grate the ashes are thus thrown outside over the edge of the tub and removed over slips (D) into suspended-railway buckets (E). The ashes of the 3 generators built in the last construction stage and provided with dry ash removal are pressed by means of shovels into two ash casings and discharged from there at intervals into the suspended-railway buckets. The total amount of ashes is collected in two bunkers at the height of the engine house and of a capacity of 65 tons each. The ashes are transported away by railway cars.

The succeeding ignition chamber (F) is connected with the generator by a lateral brick-lined connecting pipe. It has an outside diameter of about 3.5 m and contains refractory masonry cross-like assembled grate stones.

In the lower part the ignition chamber is connected with the waste heat boiler (G). This serves to produce high pressure steam (18 atm. gauge pressure). Before and behind the bunch of pipes there are chambers with fireproof lining. The chamber which is situated towards the ignition chamber comprises an overheater which is constructed from 18 several pipe coils. The chamber at the exhaust-side of the boiler is connected to the chimney (H) which conveys the waste gases over the roof. In the pass of the chimney a closing device, the chimney flap (I), is provided.

In several groups of generators there is inserted, for the purpose of separating flue dust from the blower gas, a special receiver (K) in which by reversal of the gas flow above a water surface the dust is caught by the water contained in the receiver and washed away.

The gas receiver (L) is a cylindrical apparatus of 3 m diameter which is filled with water up to a certain height. As a securing device against back-flowing of gas from the gas-holder the pipe for conveying the water-gas produced dips into this water with a dip pipe. For cooling and purifying the water-gas produced scrubbers (M) are provided which have a height of 10 m and a diameter of 3.5 m and are protected against corrosion by an acid proof brick-lining. Inside, almost to the full height of the scrubbers, there are wooden hurdles arranged in a crosswise manner. From above reflux cooling water is trickling down on these hurdles. The gas exhaust ports are attached on top and joined to a collector pipe (N) which leads to a water-gas-holder of 20 000 cu.m.

The water, running off from scrubbers and receivers of the individual groups is conveyed through a special system of channels to a clarifying plant (O) and from there by means of pumps to the reflux cooling plant.

The air which is required for blowing hot the coke is produced by in toto 9 blowers (P) which are arranged in the engine house. The capacity of the blowers amounts to 54 000 cu.m. p.h. (0° C., 760 mm Hg) and a pressure of 850 mm water column (construction stage I) and 60 000 cu.m.p.h. (0°C., 760 mm Hg) and a pressure of 1000 mm water column (construction stage II). These blowers are preferably steam-driven and in 2 cases only they are electrically driven. In the engine house air is sucked in through underground sucking shafts and then it is conveyed to the individual generator groups by blast mains arranged overground with a diameter of 1000 - 1400 mm.

Furthermore there is a station (Q) in the engine house for production of compressed water of 35 - 40 atm. required for automatic operation of the plant. For current operation 2 centrifugal pumps are provided, with an output of 375 l p.min. each, 4 plunger pumps with an output of 125 l.p.min. each form the reserve.

Finally pumps (R) necessary for the feedwater supply of the jacketed boilers and the waste heat boilers are mounted herein. 2 steam pumps with a capacity of 90 cu.m.p.h. each and one electric pump with a capacity of 270 cu.m.p.h. are available.

For maintaining most exact switching times at the generators a control pedestal (S) is attached to each generator group. Governed motions of the gas sliding valves, pressure valves blast valves and charging valves which are necessary for blowing and gasifying are performed from the control pedestal by means of hydraulic control valves and electric drives. To each valve in the generator group there is allotted a control valve in the control pedestal. Governing is managed by means of levers and rods in a fixed cycle which is adjustable between certain limits. A mechanically operating supervision apparatus is built in to avoid failures of control and explosions, it exactly controls each valve motion and stops the control pedestal in the case of failures of governing. The levers and rods which operate the control valves are moved by means of a synchronous motor and a gear. By especially assembled contact devices also the pressure of the blower air in the blast pipes is controlled since a failure of the blowers might entrain serious troubles. These contact devices too can stop the control pedestal. The below described mode of operation of the plant has blowing periods of 60 sec. and gasifying periods of 90 sec. The air blowers in the engine house are mounted and fitted in such a way that the capacity of one blower suffices for operating 3 generators. The switching times of the control pedestals are adjusted to start the blowing of the generators in a previously fixed order of succession. By using synchronous motors for the control pedestals shifting of the switching times is avoided. A separate transformer station supplies current to the synchronous motors. Adjustment of the swiching times is centrally effected for all generators by way of this transformer station.

Mode of operation.

The glowing coke stocked in the generator is transferred into water-gas by blowing-in steam (T) (2.5 atm. gauge pressure). This gas enters the ignition chamber (F) through the connection pipe and from there it is introduced through a gas pipe into the reciver (L) bubbles through the water dip closure, and, after passing the scrubber (M), it enters into the gas collecting pipe (N). The coke contents of the generator is cooled by the endothermic water-gas reaction so that after a short period the gas production is considerably reduced. After a fixed gasifying period, therefore, the supply of steam (T) is shut-off, the air valve (U)

is opened and, air is blown into the generator from below. The blowing gas leaving the generator and still containing carbon monoxide is burned in the ignition chamber (F) with a further addition of air. (V). The heat evolved by this process is accumulated in the ignition chamber (F) and serves for the steam production in the following waste-heat boiler (G). Through the chimney (H) and the chimney flap (I) the flue gases which are no more useful can escape into the open.

After a blowing period of about 60 sec. the gasifying is turned on. The air valves (U) as well as the chimney valve (I) are shut and the steam valve (T) is opened. In order to avoid migration of the fire in the generator from bottom, to top by gasifying and blowing from below the gasifying period is subdivided once more; i.e. into a [illegible] period of gasifying from the bottom of approximately 50 sec. and a period of gasifying from the top of about 40 sec. In the latter case the steam is conducted through the ignition chamber (F) and therein it is overheated. Gas passes the grate whence it is also conveyed to the gas receiver (L) through a special conduct (W).

To produce 1 cu.m. of water gas approximately 1 kg of steam is required. One third of this amount is generated in the jacketed vessel, while two thirds are produced in the waste-heat vessel. The high-pressure steam of 18 atm. gauge prefsure which is produced in the waste heat boiler is released to 2.5 atm. gauge pressure in the driving turbine of the air-blowers. Then, in addition to the steam from the jacketed boiler, it serves as gasifying steam for the water-gas process.

Condition of the plant:

The generator groups 1 - 7 of the construction stage I, including the generator house, are badly damaged by bombs. The iron constructions, steam- and water pipings, the crane way and the bunkers of the generators 1, 5 and 6 are severely damaged. The generator groups 8 - 14 of the construction stage II show minor damages except the generators 8, 13 and 14. The jacketed boiler and the ash removal of generator 8, the gas and the blast pipes of generator 13, the scrubber, the blowing gas receiver and the waste-heat boiler of generator 14 are more or less damaged. The air blowers

of the engine house are slightly damaged, the suction shafts and the electric distributors, however, are destroyed. Both ash-bucket lifts are damaged requiring extensive repairs. Particularly great damages occurred in the reflux cooling water system hsince both the cooling towers are burned and the house with the reflux cooling water pumps. The clarifying plant too was hit; also part of the pipe bridges are considerably damaged.
is destroyed
For construction stage I, comprising about 50 % of the former capacity of the plant, repair of the generators 8 - 14 as well as the new erection of a cooling tower with the necessary pumps is provided. The water gas holder is destroyed and must be replaced by a new one. The time of construction required for such a reservoir of 20 000 cu.m. must be estimated at 7 months. Within this space of time the repairs according to construction stage I can be finished supposing, however, that enough workmen are available and the supply of materials is guaranteed.

Feedwater purifying plant.

Description:

The feedwater demand of the hydrocarbon plants and of part of the central power station is met by the feedwater purifying plant. The water purifying plant is operating according to the base exchange process of "Permutitgesellschaft", Berlin. There exist 5 filters (A), with a height of 3.5 m and a diameter of 2.5 m, which are filled with permutite mass. The fresh water continuously flowing through is softened by this mass. After a certain period, about every 8 - 10 hours, depending on the hardness of the water, regeneration of the mass is necessary. This is effected by pressing a solution of common salt through the permutite mass. For removal of oxygen which is dissolved in the water and has a corrosive action and of carbonic acid a degassing plant is inserted behind this filtration plant. The softened water is introduced into the degassing tower (B); here it is warmed up to about 70°C. with steam of 2.5 atm. gauge pressure and subjected to reduced pressure by means of the vacuum pumps (C). The before mentioned gases and the hot damps are sucked off and pressed into the open air by the vacuum pumps (C). Two intermediate pumps (D)-one of them being a spare-pump serve for the continuous removal of the degassed water from the degassing tower (B). In a reservoir (F)

this water is stored with steam-closure. The total output of the filters and of the degassing plant respectively amounts to 300 and 220 cu.m.p.h.; so that it is impossible to soften the total quantity of water required. While the hydrocarbon plants only are supplied with degassed water, the exceeding part of not-degassed water is delivered to the power station. After passing the before mentioned reservoir (E) trisodium phosphate is mixed with the feedwater in the feedwater discharging piping in order to take off the remaining hardness since the permutite filter only soften water down to about 0.1° German hardness.

The feedwater required by the water gas plant flows to this from the reservoir by its own weight and then it is pumped into the boilers. For supply of the synthesis furnace house 3 centrifugal pumps (F) are fitted up in the feedwater purifying plant, 2 of which with a capacity of 60 cu.m.p.h. and one with a capacity of 120 cu.m.p.h. All pumps work against 30 atm. gauge pressure. The big pump and a small one are steam-driven, the remaining small pump has an electric drive. 2 electrically driven pumps with a capacity of about 100 cu.m.p.h. each are available for the water supply of the power station.

Condition of the permutite plant.

One of the 5 available filters has been turned over by a damage of the foundation. At this place the building and part of the pipes lines have been damaged too. So far as it can be seen, the filter itself is allright. From 4 filters the permutite mass has been removed during preparation for displacing and it has been damaged in storage or transportation. But about 70 % of the mass are still available. The degassing plant is undamaged but for some unimportant splinterholes. The small steam-driven pump, one of the 3 feedwater pumps for the synthesis furnace house, has been destroyed, furthermore a water supply pump for the power station. The building as well as part of the steam and water pipings are destroyed or badly damaged.

Reflux-water cooling plant.

Description.

In the building of the feedwater purifying plant the reflux-water cooling plant is fitted which is required for supply of cooling water to the hydrocarbon plants. It consists of the pumping

aggregate, the reflux basin (H) and the two cooling towers (I). The total capacity of the cooling plant amounts to 3000 cu.m.p.h. It comprises 4 electrically driven sets of pumps (G) with a capacity of 750 cu.m.p.h. each. In each of the aggregates a cold water pump and a hot water pump are driven by the same shaft. The hot water pump presses the water returning from the plants to the tops of the cooling towers and the cold water pump brings it back to the plants. In a separate pump house a spare aggregate is fitted up. It is driven by a steam turbine and has a capacity of 1500 cu.m.p.h.

Condition of the plant.

Both cooling towers have been destroyed. The basins for the refluxing and for the cold water are badly damaged. The steam-driven aggregate and 3 of the electric driven pumps are allright, the 4th has been destroyed. The motors have been diamantled and in part been brought to the electric workshop but repair is impossible at the moment as important spare parts are not available. Two other motors, which were intended as spare units, have been seriously damaged in the synthesis furnace house, where they had been stored. A 5th motor only is ready for service but at this date it is mounted at an ice machine in the compressor house.

Works for reoperating the feedwater purifying plant will require about 8 weeks. Reconstruction of the reflux-water cooling plant in the old form probably will be postponed since the new cooling plant at the west side of the factory has but slittle damages and may be started without difficulties.

Reflux cooling plant west.

The reflux cooling plant situated to the west of the Primärstraße (Primary street), which was planned to complete the old reflux cooling plant behind the permutite plant and which had to supply the newly constructed plants, comprises the following units:

1 atmospheric cooling tower for 2 000 cu.m.p.h., erected by Bischoff, Essen.
1 pump house with 3 cold water pumps, 1 000 cu.m.p.h., lifting height 50 m.
1 atmospheric cooling tower for 4 500 cu.m.p.h., 3 000 cu.m.p.h. at intense cooling, erected by Balcke, Bochum.
1 pump house with 3 cold water pumps 2 500 cu.m.p.h., lifting height 50 m.

1 basin for warm water reflux and a warm water pump house with 4 warm water pumps, 2 500 cu.m.p.h., lifting height 22 m.

Condition of the plant and its reconstruction.

Both cooling towers are destroyed. The basin and the steel construction of the Bischoff cooling tower (2 000 cu.m.p.h.) exist in damaged condition. The buildings of the pump houses are damaged. 2 pumps 1 000 cu.m.p.h. and 2 warm water pumps are damaged. For construction stage I the Bischoff cooling tower must be erected, the corresponding pump house and 2 damaged pumps 1 000 cu.m.p.h. must be repaired. In the warm water pump house the windows are missing. 2 pumps are saved.

Reconstruction time: 4 months.

II/2) Synthesis plant.

Drawings in folder II/2.

a) Desulfurising plant and operating method:

From the gas holder the water-gas is first conveyed into the desulfurising plant. In the first part of it, which is also called the coarse-purifying or the dry-purifying plant, hydrogen sulfide is removed. Before purifying the sulfur content amounts to 3 - 4 g/cu.m. and after purifying to 0,05 - 0,1 g/cu.m. The plant has been built by the firm Klönne at Dortmund and consists of 8 purifying towers with a diameter of 11,5 m and a height of 9.8 m. Each tower contains approximately 600 tons of purifying material spread on 16 wooden hurdles in layers of 400 mm height. All layers are passed through in parallel, 3 purifying towers always are connected in series. The separate purifying towers are inserted and shut off by means of water closures. The temperatures lie between 20 and 40°C. The purifying material is pure Lux mass (iron hydroxide, by-product of the alumina production); in former times it was mixed too with 1/3 part of spent mass. The mass is enriched to about 45 % of sulfur, calculated on moist mass, attaining in such a way a period of activity of 5 - 6 months. 4 purifying towers always are provided for a gas through-put of 40 000 cu.m.p.h., 1 purifying tower of each group of four towers serves as a spare unit or is charged anew. The spent mass is sold for being worked up for recovery of the sulfur.

The coarse-purifying plant is followed by the fine-purifying plant for removal of the organically bound sulfur. In the here used water gas this is mainly present in the form of carbon oxysulfide (COS) and carbon disulfide (CS_2); its quantity amounts to about 0.2 g p.cu.m. Among other factors the life-time of the catalyst for the synthesis of hydrocarbons depends on the greatest degree attainable of freedom from sulfur of the synthesis gas. These sulfur compounds are removed by decomposition and simultaneous binding to shaped alkalized Lux mass at temperatures between 200 and 260° C. The fine-purifying plant which is also built by Klönne at Dortmund comprises 5 groups and an after-purifier. Each group consists of one gas heater and 2 towers with a diameter of 4.2 m and a height of 10.5 m. The towers contain set-in tubs or

vertical screens between which the fine-purifying mass lies in layers of 0.90 - 1 m thickness. Both towers are connected in series. Between them a heat exchanger for regulating the gas temperature of the second tower and for pre-heating the arriving gas is arranged. The gas heater consists of a fire box, in which residual gas from the synthesis is burned at 600 - 900° C. and of a nest of tubes wherein the pipes in the lower hot part are made of heat resisting steel, FF 18 or alited, and in the upper part of normal ingot steel St. 37. The gas which is to be purified passes the heat exchanger, the nest of tubes and both purifying towers successively. The mass contents of the towers amounts to 60 tons when using set-in tubs and to 70 tons in the case of the screen towers constructed at last. Normally 17 000 cu.m.p.h. of gas (0° C., 760 mm Hg) are purified by one group. The maximum output is 20 000 cu.m.p.h. (0° C., 760 mm Hg).

The fine-purifying mass contains Lux mass mixed with 33 % of soda and is produced in the mass factory of the Ruhrchemie. The grain size is 10 - 15 mm. Fresh mass is active at 180° C.; with an increasing charge with sulfur the temperature must be raised to 260 - 280° C. The spent mass has a sulfur content of 8 - 10 %, almost entirely in the form of sodium sulfate. Hitherto the spent mass was not further used. The operation time of a group is 30 - 40 days when normally charged; then the contents of the first tower is completely spent while in the next running the second is once more used as first tower.

For the compensation of occasional fluctuations there is an afterpurifier which receives the total gas current without further heating the gas. It consists of 2 towers which are connected in parallel, and similar to the preceding ones. In this way the sulfur content of the synthesis gas is kept below 0.002 g.p.cu.m. (H_2S + org. S).

Before coarse purifying the water gas is adjusted to an oxygen content of 0.4 % by addition of air. 0.2 % thereof are consumed for the process of the sulfur separation in the coarse-purifying mass and the remaining 0.2 - 0.3 % in the fine-purifying mass for binding sulfur in the form of sodium sulfate.

Desulfurizing plant, its condition and reconstruction:

In the coarse-purifying plant there are damaged 4 of the 8 towers, furthermore several pipings and water closures as well as the track of the gantry crane which serves for the charging of the towers. For construction stage I there are provided: 4 towers with the corresponding pipings, 2 of the towers showing considerable damages. Rebuilding time approximately 2 months. In the fine-purifying plant the towers of one group are destroyed, of 2 groups they are damaged and of 2 groups they are allright. Both after-purifying towers are slightly damaged. The spare blower for the combustion air of the gas heaters is destroyed. The pipings are damaged like in all other cases.

For construction stage I there are provided: 3 groups with after-purifier, of which one group with the after-purifier are damaged 1 air blower must be supplied. Rebuilding time approximately 2 months.

b) Hydrogen production plant (CO-conversion), construction and operation.

For the hydrocarbon synthesis a gas is required in which carbon monoxide and hydrogen are in the proportion 1 : 2, whereas water-gas contains these components only in the proportion 1 : 1.3. Therefore, about one third of the water-gas is used in the hydrogen production by which carbon monoxide is converted with steam according to the equation:

$$CO + H_2O = CO_2 + H_2.$$

Various catalysts were used, in general the brown oxide catalyst of I.G. Farbenindustrie which is a mixture of iron oxide and chromium oxide. In a furnace with a diameter of 3.2 m and a capacity of 25 - 28 tons the catalyst is arranged in 4 layers of 750 mm height. Below the furnace there is a heat exchanger with a surface of 1400 sq.m. The height of the furnace and the heat exchanger together is 14.50 m. The arriving water-gas firstly is conveyed through a saturator (a tower with a height of 27 m and a diameter of 2.40 m) in which it is saturated at 70° C., the required water being is taken from the cooler which cools the converted gas leaving the catalystic furnace. By a steam injector with 9 atm. gauge pressure the gas is conveyed from the saturator through the heat exchanger into the catalytic furnace; the required reaction steam is simultaneously added. The heat exchanger transfers the heat of the converted gas to the arriving water-gas. The converted gas finally passes a cooler (a tower with a diameter of 3.10 m, a height of 27 m, filled with Raschig rings in 5 layers) and leaves the plant at 25 - 30° C. In the cooler and in the saturator a formation of sulfur (H_2S) is observed due to the action of certain bacteria on the sulfate ($CaSO_4$) contained in the water. As the converted gas enters directly into the synthesis plant this sulfur formation must be inhibited. Therefore in a separate cycle the cooling water is conveyed through an atmospheric cooling tower, where the water cycle is transformed into a condensate free from sulfate by the condensing excess steam of the conversion process. In that way any sulfur formation is stopped. Occasional irregularities in the water cycle caused by bacteria are counteracted by inoculation with zinc chloride.

The conversion process itself proceeds exothermically at temperatures of 420 - 480° C., the catalyst starts action at 370° C. For lowering the increasing temperature and for saving steam, con-

densate free from residue is injected behind the second catalyst layer. The required steam excess increases pari passu with the temperature. Without previous saturation and condensate injection a steam consumption of 0.8 - 1.0 kg p.cu.m. of used water-gas is attained in practical operation, which is lowered to 0.6 - 0.7 kg p.cu.m. by saturation and injection. If a condensate with a residue on evaporation of 2 - 5 mg p.l. is not available the injection must be given up as otherwise the catalyst is chocked by being coated with the residues. In addition to crusting formation by salts from steam or condensate the catalyst is subjected to a disintegration by the mechanical stress during passage of gases. This limits lifetime to about 2 years and, after screening and repeated use to about 3 - 4 years.

First heating of the catalyst is executed by heating up to 370 - 400°C. with flue gas which is produced by the combustion of heating gas in a small heating furnace. When charging water-gas and steam there is at first a reduction of the contact followed by normal operation after several hours. Before shopping operation for a longer period the catalyst is previously oxidized with a mixture of steam and air or better with a mixture of nitrogen and air.

The present plant, which is erected by Bamag-Meguin A.G., at Berlin, comprises 5 contact furnaces for 6000 cu.m.p.h. of water-gas (0° C., 760 mm Hg) each, 2 saturators and 3 coolers (1 spare cooler). Moreover, the pumps required for circulation of cooling water and the irrigation of the saturator are mounted.

Condition and reconstruction of the plant.

1 catalytic furnace (furnace no. 5) and 1 cooler (cooler no. 2) are destroyed; the other apparatuses are damaged by bomb splinters. 2 pumps of the cooling water cycle are missing. For construction stage I the furnaces no. 1, 2 and 3 will be made ready for service and the missing pumps will be supplied. Furthermore, pipings and measuring instruments are to be substituted or refitted. Rebuilding time: about 3 months.

c) Blower house, attachment and operation.

In the blower house there are the following machines:

1 gas blower, 45 000 cu.m.p.h. (approximately 40 000 cu.m.p.h./ (0° C., 760 mm Hg),
increase of pressure from 1.043 up to 1.343 atm., specific gravity in the suction period 0.937 kg.p.cu.m., number of revolutions 3 700 r.p.m., drive electric by a 640 kW-motor, 5000 volt, 1 480 r.p.min. by means of a gear.

1 gas blower, 45 000 cu.m.p.h. (approximately 40000cu.m.p.h./ (0° C., 760 mm Hg))
as above mentioned, steam turbine driven, live steam of 15 - 20 atm. gauge pressure, back pressure 2.5 atm. gauge pressure, 3 700 r.p. min., 615(700) H.P.

1 gas blower, 91 800 cu.m.p.h. (approximately 80 000 cu.m./ 0° C., 760 mm Hg)
service conditions. As above mentioned, driven by back pressure steam turbine 18/2.5 atm. gauge pressure, 3000 r.p.min., 1 200 H.P.
All gas blowers have been constructed by the Gutehoffnungshütte at Oberhausen in 1935 - 1937.

2 air blowers, 1000 cu.m.p.h., compression 500 mm water column.
The gas blowers suck the total quantity of water through the coarse-purifying plant and press it into the synthesis plant. At normal load, of the synthesis plant 2 blowers 40 000 cu.m. or 1 blower 80 000 cu.m. are operating, thus the reserve capacity amounts to 100%. For separating the dust which is still contained in the water (20 mg.p.cu.m.) and which otherwise would cover the runner of the blower within a short time and thus cause unbalance, there is a coarse-purifying plant preceding the gas blowers. The disadvantage of this mode of operation lies in the reduced pressure which results in the coarse-purifying plant (admissible limit 500 mm water column). Moreover, the gas blowers do not convey the calculated quantities of gas as they are constructed for a suction pressure of 100 mm water column but now suck in with -500 mm water column. Hence, for later times fitting of an electric filter for dust-removal and the insertion of the blowers before the coarse-purifying plant were provided.

The air blowers serve to add air to the water-gas (see desulfurizing plant).

Condition and reconstruction of the plant.

All machines are undamaged and before starting operation only need a thorough cleaning and supervision. The roof and the windows and part of the walls of the machine house are damaged. Rebuilding time: 2 months.

The 2 gas blowers 45 000 cu.m.p.h. are provided for construction stage I. Rebuilding time: about 2 months.

d) Gas-machine house, attachment and operation.

The gas-machine house contains the turbo-compressors and the compressed water purifying plant for the synthesis gas of the medium pressure synthesis.

The subsequent engines are mounted:
4 turbo-compressors, 20 000 cu.m.p.h. (0° C., 760 mm Hg) (22 450 cu.m.p.h. in the suction period), increase of pressure from 1.033 up to 12 atm., spec.gravity 0.7 kg.p.cu.m. (0° C., 760 mm Hg), double-casing type, fitted in parallel. The low-pressure part runs with 5 100 r.p.min., driving motor 2000 kW at 1 450 r.p.min. The motors are directly started. In one of the engines the low-pressure part is driven by a steam turbine, steam pressure 18/2.5 atm. gauge pressure, 2 500 H.P., 5 100 r.p.min., the high-pressure part has 7000 r.p.min., 1000 kW driving motor with 1 450 r.p.min.

Special difficulties with these machines occur at the intermediate gas coolers, 3 of them being fitted in each machine set; for these a final corrosion-proof construction had not yet been designed. At last cells covered with stoving lacquers (Bakelite) were built in. The capacity of the turbo-compressors lay below the calculated, i.e. 17 - 18 000 instead of 20 000 cu.m.p.h. (0° C., 760 mm Hg). All machines have been constructed by the Gutehoffnungshütte at Oberhausen in 1938.

Furthermore:

1 turbo-compressor, 35 000 cu.m.p.h. (0° C., 760 mm Hg), operation conditions as above, 4 200 r.p.min. Driving motor 5 600 kW, 1 450 r.p.min. Starting is operated in 2 stages by means of a starting transformer. Double-casing type mounted on a single shaft.

Hitherto this machine has been operated for a short time only. It is constructed by the Demag at Duisburg in 1943/44.

For normal load of the pressure synthesis approximately 50 000 cu.m.p.h. (0° C., 760 mm Hg) must be compressed. For this purpose 3 compressors, 20 000 cu.m., or 1 compressor, 35 000 cu.m., together with 1 compressor, 20 000 cu.m., are running. One of the compressors, 20 000 cu.m., is operated with converted gas which is conveyed through the carbonic acid absorption tower. Including the compressor, 35 000 cu.m., which is provided for the olefine synthesis, the reserve capacity amounts to 100 %.

In front of the gas-machine house 2 injection coolers of 16 m height and 4 m diameter are erected which cool the hot gas, leaving the fine-purifying plant down to 25° C. In heat exchangers part of the heat to be taken from the gas is transmitted to the converted gas going to the low-pressure synthesis. The water cycle of these coolers is connected to the cooling water cycle of the hydrogen plant, as in the coolers also sulfur formation by bacterial occurs.

Furthermore in the gas-machine house there are 2 aggregates of pumps and turbines for the absorption of carbonic acid from the converted gas, which comes from the hydrogen plant and is conveyed into the medium pressure synthesis. They have the following data:

Pump: 1 500 cu.m.p.h., lifting height 140 m,
power consumption: 870 H.P. 1 470 r.p.min.;
constructed by: Gebr. Sulzer, Ludwigshafen.
Turbine: 1 400 cu.m.p.h., net pressure 100 m;
output: 450 H.P., 1 470 r.p.min.;
constructed by: Voith at Heidenheim (Württemberg).
Motor: 500 kW, 5000 V, 1 470 r.p.min.

The pumps work on 2 towers of 16 m height and 3 m diameter, filled with Raschig rings of 50 mm diameter. From the washer the compressed water flows through the turbine to the degasing tower which has a height of 30 m and a ground of 18 x 18 m. It is ventilated by 4 fans with a capacity of 450 000 cu.m.p.h. Simultaneously it was used for the cooling water cycle of the hydrogen plant. This mode of operation was supposed to be temporary; it was intended to add a separate indirect cooling system to the

hydrogen plant.

Up to this date an increase of the water temperature and consequently a reduced washing out of carbonic acid resulted. The washing towers can be charged with 10 000 cu.m.p.h. of gas (0° C., 760 mm Hg); water supply 1 400 cu.m.p.h.; carbonic acid absorption from 29 to 10 %; loss of withdrawn useful gas ($CO + H_2$) 2,5 % of the introduced gas quantity. The washing towers are followed by a large spraying separator and a float valve which closes the gas discharge pipe when water is carried along.

The absorbed carbonic acid, approximately 3 500 cu.m.p.h. (0° C., 760 mm Hg) was not used hitherto. It had been planned to insert a large available receiver before the degassing tower and to obtain a gas very rich in carbonic acid by preliminary expansion. Carbonic acid is used as a protective gas at many places of the plant.

Condition and reconstruction of the plant.

All machines are undamaged. The suction pipes of the compressors are destroyed over a length of about 10 m; the towers of the carbonic acid absorption plant are damaged by bomb splinters. Overhauling of the compressor, 35 000 cu.m., damaged by an explosion had been finished and the compressor was ready for the test run. The roof and the windows of the building are damaged, the glazing is completely missing.

For construction stage I 3 electrically driven compressors, 20 000 cu.m. and the carbonic acid absorption plant are provided. Damage at pipings and by bomb splinters must be removed, measuring instruments must be replaced, all machines must be cleaned and controlled.

Rebuilding time: 3 months.

e) Contact furnace house for hydrocarbon synthesis, plant and operating method.

The hydrocarbon synthesis proper takes place in the contact furnaces into which the purified gas brought up to the hydrogen content necessary for the synthesis is conveyed by means of the blowers and gas compressors. The synthesis is based on the following equation:

$$CO + 2\,H_2 \longrightarrow (CH_2)_n + H_2O$$

The reaction is an exothermic one. The fundamental idea underlying the construction of the contact furnaces is to remove the heat as completely as possible. The reaction temperature is adjusted by regulation of the steam pressure in the water-containing pipe system of the furnace by means of electric regulators (Siemens). The temperatures vary between 180 and 200° C. and must be observed exactly to ½° C. The catalyst used is a cobalt-magnesium oxide-thorium oxide-catalyst, kieselguhr being the carrier mass. The catalyst preferably has a grain size of 2 - 3 mm. The composition is as follows: 30 % of Co, 2.5 % of MgO, 1.5 % of ThO_2 and 66 % of kieselguhr. A contact furnace contains about 10 cu.m. of catalytic mass with about 900 kg of cobalt. The catalytic mass is delivered by the catalyst plant of the Ruhrchemie in reduced state and is therefore, highly sensible to oxygen; it is contained in closed vats under carbon dioxide as protective gas. Travelling cranes lift and empty the vats by tilting them above the contact furnaces. The cranes have a carrying capacity of 60 tons and can lift an entire contact furnace which for the low-pressure synthesis weighs 46 tons and 49 tons for the medium-pressure synthesis.

The first synthesis according to Fischer-Tropsch carried out on a large scale was the low-pressure synthesis under atmospheric pressure. The contact furnace house contains in its east part 52 low-pressure furnaces, 20 thereof have been furnished by Gutehoffnungshütte at Oberhausen, 20 by Krupp-Germaniawerft at Kiel in the form of tube bend furnaces in 1935/1936 and 12 by Krupp in the form of chamber furnaces in 1937. All furnaces have a contact chamber of 5 m long, 1.50 m broad and 2.50 m high, a sheet iron box through which run 630 tubes of 34/29 mm diameter. On the tubes are mounted 555 iron sheets or fins, 1.5 mm thick, at a distance of 3 mm from middle to middle. The free space for the catalyst thus amounts to 7.4 mm from fin to fin. Hinged screens

prevent the grains of the catalyst from falling downwards. The tubes are connected with each other outside the sheet iron box by bends or chambers, respectively, which in their turn are connected by collecting tubes ending in cylindrical drums. One cylinder drum is mounted above 2, in case of chamber furnaces, above 3 furnaces which, controlled by an electric regulator, are operated under equal pressure, i.e. at equal temperature. The reaction heat absorbed by the water circulating in the tube system corresponds to a steam generation of about 5 kg/kg of liquid product. When starting operation of the furnaces the steam streams into the piping system of 2.5 atm. gauge pressure and later on into the piping system of 9 atm. gauge pressure. The admissible operating pressures amount to 0.3 atm. gauge pressure for the gas and 30 atm. gauge pressure for the steam.

The products formed during the passage of the synthesis gas consist of gaseous, liquid and solid hydrocarbons. Gasol propane + butane, petrol and oil leave the furnace as vapors in the final or residual gas and are recovered in the following plants. The paraffin formed at the surface of the catalyst remains there and enriches the latter up to 50 % by weight. Methane and small quantities of carbon dioxide are formed as undesired by-products (known as gasification) as well as the water of reaction. With the synthesis gas produced at Holten having about 80 % of active components ($CO + H_2$) it is possible to convert up to 92 % of the useful gas whereby the following side products are obtained:

68 % of liquid products (55 % of oil, 45 % of petrol = 130 g/cum. of useful gas)
15 % of Gasol ($C_3 + C_4$-hydrocarbons),
15 % of methane, CH_4,
2 % of carbondioxide CO_2

100 % = converted useful gas ($CO + H_2$).

The gas is conducted in two stages, that is to say, through two groups of furnaces arranged in series. Between the stages the difficultly boiling products and the water of reaction are removed by condensation. The normal charge of a furnace in the first stage amounts to 1000 normal cu.m.p.h., that of a furnace on the plant average 600 - 700 normal cu.m.p.h., calculated upon the charge of the first stage.

The catalyst loses in efficiency by the accumulation of paraffin in the course of 3 - 4 weeks and is then re-activated by trickling it with extraction oil (boiling at 160 - 320° C.) and subsequent treatment with hydrogen (75 % H_2, 25 % N_2 supplied by the nitrogen plant of the Ruhrchemie). This treatment is repeated every three weeks, total lifetime: 3 - 4 months. The discharge of the catalyst is preceded by an extraction by which the paraffin formed at the catalyst is removed. After drying with gas or steam the catalyst falls through the opened screens into a trough below the furnace from which it is carried by a chain conveyer (Redler) into the vats in which it is reconducted to the catalyst plant and worked up again (dissolution and re-precipitation).

In the course of the development of the process in former years disburging difficulties were often encountered by disintegration of the catalyst or by deposition of carbon in the furnace due to insufficient heat elimination. In view of this fact only 80 % of the low pressure furnaces worked with full output even before damages were caused by bonds. Since preference was given to the medium pressure synthesis, the low-pressure synthesis was not entirely utilized.

The development led in 1938 to the <u>medium-pressure synthesis</u> which with better liquefaction yields more valuable products, especially paraffins with higher melting points. With a transformation of about 93 % ($CO + H_2$) there are obtained on the average:

75 % of liquid products (30 % of paraffin, 30 % of oil, 40 % of petrol)
12 % of Gasol ($C_3 + C_4$ hydrocarbons),
11 % of methane, CH_4,
2 % of carbondioxide, CO_2

100 % = from converted useful gas ($CO + H_2$) = 93 % of the charge.

The operation of the synthesis is similar to that of the low-pressure process. The synthesis gas, compressed to 9 - 10 atm. gauge pressure, is introduced into the contact furnaces which are operated in 3 stages. The converter gas is freed from carbondioxide by passing through the washing towers so that its contents in active components amounts in the first stage to about 86 %.

The construction of the contact furnaces for the medium-pressure synthesis differs from that used for the low-pressure synthesis.

About 2000 double-tube elements with a length of 4.5 m are arranged in a vertical vessel with a diameter of 2.7 m. The catalyst lies in the interstices of 10 mm between the tubes of 44/48 mm and 21/24 mm diameter; the water flows through the inner tube and around the outer tube. Hinged screens shut the tube bunches at the bottom. Always 4 furnaces work together with 1 cylindrical drum and form a block. The admissible operating pressures are 15 atm. gauge pressure for the gas and 25 atm. gauge pressure for the water. In the west part of the furnace house were 73 furnaces, 16 thereof having been constructed by Krupp at Essen and 56 by Mannesmann at Witten. 2 of these were trial constructions: one was a head furnace in which the cylindrical drum was replaced by an enlargement at the upper end of the furnace and the other one was a pressure luminae furnace (built by Gutehoffnungshütte), i.e. a low-pressure furnace resistant to pressure. Both types were operated in exactly the same way as the normal double-tube furnace. A regeneration of the catalyst similar to that applied in the low-pressure process can be dispensed with. Only before being discharged, the catalyst is freed from the paraffin by extraction. In the synthesis the paraffin is obtained in the liquid state and flows out through the final gas pipes. Oil and water are condensed at the end of the different stages. In order to avoid epoxy separation within the furnaces, the gas is pre-heated to 160° C. prior to the second and third stage by means of steam in tube heaters. The material used for the plant is ingot steel which is affected by the organic acids (acetic acid and higher organic acids), formed to a large extent, as soon as they condense. Care must, therefore, be taken that the final gas pipes are well insulated so that the temperature will not drop below the thaw point. The life-time of the catalyst which is exactly the same as that used for the low-pressure synthesis is about 6 months. The average load lies at 650 - 700 cu.m. p.h. and furnace, calculated upon the total charge and total number of furnaces.

In the middle of the central furnace house is a central station where all Siemens steam-pressure regulators and the most important instruments for controlling the quantity and composition of the gas are united.

Condition of the plant and reconstruction.

Of the 52 low-pressure furnaces 7 are totally destroyed,
3 are seriously damaged,
18 are slightly damaged and,
24 are intact.

One cylindrical drum is destroyed, 3 are damaged. At two places the synthesis and residual gas pipes between the furnaces are destroyed to a larger extent. Roof and windows of the building as well as the platform between the furnaces are heavily damaged, glazing is missing entirely. Of the 73 medium-pressure furnaces
5 are totally destroyed,
9 heavily damaged, (not certain, whether repair is possible),
11 slightly damaged and
48 intact.

1 cylindrical drum is destroyed, 3 are heavily damaged. Conveying devices (Redler) for the discharge of the catalyst, crane way, lifting device for vats containing catalysts and steam pipings are damaged. 4 main columns of the building are destroyed or damaged roof and glazing are missing. The platforms between the furnaces and rails are damaged.

For construction stage I 52 medium-pressure furnaces are provided; 48 thereof are undamaged but must be overhauled, and 4 must be repaired. Crane way, catalyst conveying plant, platform and tracks must likewise be put into good order.

Roof and building must be repaired to such an extent that freezing is prevented or the whole piping system must be heated and insulated. For the present, the low-pressure plant remains out of work. Reconstruction time: about 6 months.

z) Condensation, plant and operation.

The condensation in the low-pressure synthesis is effected by means of direct cooling; the plant comprises a tower of 3.45 m diameter and 25,2 m height for the 1st stage and a tower of 2.85 m diameter and equal height for the second stage. Both towers are lined with acid-proof stones. In order to avoid corrosion of the equipment by acid, the pipes conducting water and product are made of aluminium, the pumps and armatures are made of V_2A or sicromal. Each tower contains 4 layers of Raschig rings and a trickling in 3 stages. The water is circulated through a special cooling tower. The circulating pumps with a capacity of 130 cu.m.p.h. as well as the oil pumps are mounted in a pumping house. The oil-water-mixture which flows from the towers through dip closures is collected in separators of 4 m diameter and 2.2 m height. From there the separated oil and water overflow into receivers. The oil passes through counters into the supply tank for the fractionation, the water is led to the cooling tower. By addition of a small quantity of fresh water the circulating water is kept at a tolerable acidity. Separators and receivers are mounted in an open pit besides the towers. In this pit there is also a steam-driven piston pump, which pumps the extraction oil to the distillation plant from where the fresh oil is pressed into the furnaces.

A third tower, 3 m diameter, height as above, was originally erected as extension of construction stage I for low-pressure synthesis which was later on carried out for medium-pressure synthesis. The tower is used to-day in connection with the medium-pressure condensation as well as the separating pit made of concrete which belongs thereto and which in this plant replaced the separating vessels. The pumps for this condensation plant, made of V_2A, originally had a capacity of 600 cu.m.p.h. and are now used with silvered gunmers and a capacity of 250 cu.m.p.h. for trickling the low-pressure condensing towers. The pumps stand in a second pump pit besides the separating pit mentioned. The water regulators and measuring instruments are located in a measuring station.

Above this pump pit is a turbo-blower from Jaeger at Leipzig with steam drive for conveying the circulating gas in a cycle during the regeneration of the catalyst by means of nitrogen and hydrogen. It is capable of pressing about 4000 normal cu.m.p.h. from 1 to 1.4 abs. atm., turbine 110 H.P., steam pressure 18/2.5 atm. pressure

number of revolutions 5000 per minute. Before the blower a cooler of 1.60 m diameter and 15 m height is situated which condenses oil and water out of the circulating gas. The off-runnings go to the separators of the condensation plant.

The condensation of the medium-pressure synthesis is fitted with indirect tube coolers. These condensers are preceded at each stage by a neutraliser (tower filled with Raschig rings) and a washer (tower with ring slit bottoms, system Kittel). The washer is trickled with 2 - 3 cu.m. of 1.5 % soda solution, per hour, in order to neutralise the organic acids formed. The originally planned circulation of the soda solution between neutralizer and washer was abandoned later on and operation of the neutralizer was stopped since the washer alone was sufficient. The solution runs off together with the oil and the reaction water. The pH-value of the off-runnings is kept at 5.0 - 6.0. At the first stage the condensers have a cooling surface of 400 sq.m. and at the second stage of 300 sq.m. Behind each stage are pressure vessels as spray separators. The off-runnings are conducted through float pots into a collecting pipe and from there to the pump of the above mentioned condensing-tower (5 m diameter) of the low-pressure condensation, where also all degassing pipings of the pressure condensation end. The expanded residual gases are sent to the adsorption plant working with active carbon, oil with residual soda solution and reaction water to the separating pit. This pit is made of concrete and contains four chambers situated one behind the other. The oil which separates is pumped through counters to the after-treatment plant and the water is pumped to a clarifying tank situated besides the cooling tower for the water-circulation of the low pressure condensation. The overflow of this circulation is likewise led to the clarifying tank where oil is separated once more, above all emulsions which are formed occasionally. The water, leaving the clarifying tank free from oil, is led to the sewer. Oil and emulsion from the clarifying tank as well as other oil and paraffin residues which are obtained at any place are heated in a special chamber and filtered subsequently. The pure oil obtained is used for the production.

The 1.5 %, soda solution is prepared in a special mixing plant which consists of 2 receivers, capacity 20 cu.m., with circulating pumps and a bunker of 30 tons overhead. In order to avoid precipi-

station of lime permutite-treated water is used for preparing the solution. The separation of oil and water is accelerated by adding 3 % of common salt ("salting out") which are carried along with the soda solution into the separating pit.

The pressure condensation plant is made of steel. In practice the neutralisation has proved sufficient, no strong attack of acid could be detected. The tubes of the pressure condensers, however, as of all other coolers of the plant operated by means of water re-cooled by air were heavily attacked by water. Therefore, tubes outside plated with copper were used which, however, corroded also at the rolled joints. Thus, this tube-problem has not been solved as yet. The condensation plants here before described have all been built by Bamag-Meguin A.G. at Berlin.

The third stage of the medium-pressure condensation as a new development has been carried out with direct cooling. Soda solution is circulated through an (existing) washer with bubble cap bottoms and indirect tube coolers which are cooled by means of reflux cooling water. From the washer, through which the synthesis residual gas streams in counter-current, soda solution and condensate flow into a separator. From here the oil flows off as product, whereas the soda solution is sucked off by the circulation pump. By current addition of fresh soda solution the pH-value of the circulating solution is kept at 6.0 - 7.0.

The third stage of the condensation is followed by the expansion of the compressed gas by an automatic regulating valve. The expanded gas streams to the adsorption plant working with active carbon.

The hydrocarbons with higher boiling points are obtained in the liquid state already in the synthesis; they are separated as furnace-paraffin in separators before the condensation plant, and from there expanded into a collecting receiver. From the receiver the furnace paraffin is sent via a counter to the paraffin plant where it is further processed.

Condition and reconstruction of the plant.

The condensation plant principally shows bomb splinter damages and defects of the piping system. The cooling tower for the water circulation of the low-pressure condensation has been destroyed, likewise parts of the clarifying tank and of the separating pit.

For construction stage I the medium-pressure condensation plant in all three stages, including the separating pit, condensing tower, diameter 5 m, and receivers and pumps for the extraction oil, has to be repaired. The pressure condensers must be provided with new tubes, lacquered tube bunches having already been prepared.

Time of reconstruction: 4 months.

a) Adsorption plant working with active carbon.

The adsorption plant was built in two steps by the Bamag-Meguin A.G. at Berlin according to the process of the Lurgi Aktiv-Kohle GmbH. The plant I comprises 11 adsorbers each of which takes up 9 tons of carbon. The adsorbers are cylindric vessels with curved ceiling and curved bottom with a diameter of 3,8 m wherein the height of the carbon amounts to about 1.80 m. The synthesis gas passes through the layer of carbon and leaves the plant after being freed from all products up to the C_3 hydrocarbons. The gas obtained is then used as heating gas in the after-treatment plants and in the steam generator. When the carbon is loaded with 3 - 4 % of petrol and 1 - 2 % of Gasol the stream of synthesis gas is introduced into a fresh adsorber. The loaded adsorber is rinsed with steam, and the steam-petrol-mixture is condensed in pipe condensers and separated into petrol and water in a separator. From the separator the Gasol escapes; it is collected in a moist gasometer of 1000 cu.m. from which it streams to the Gasol compressor for further treatment. Special rectifying columns in the gas stream for separation of the heavy petrol (boiling point above 160° C.) were inactive later on and the working up of the petrol was carried out exclusively in the stabilisation and distillation plants.

The rinsed adsorber is subsequently dried in a special circulation provided with cooler (laminated tubes) and heater (laminated tubes, steam-heated) and cooled in a second circulation provided with cooler. The cooling circulation is, moreover, passed by the gas which has been loaded so that further portions of gasol are adsorbed. The favorable adsorption temperatures lie below 40° C. The blowers in the circulations have a capacity of 22 000 cu.m. p.h. The time change-over periods for the four operations: loading, rinsing, drying and cooling amount to 24 - 60 minutes according to the load of the plant. The gas current is controlled by hydraulically operated disk valves of 700 mm diameter. The hydraulic control is fully automatic and effected by time relays from a central switch desk at the measuring station. When separating the Gasol, the inert gases (CO_2, CO, H_2) are separated and return into the adsorbers in an automatically controlled operating method.

Up to a load of 45 %, the adsorption of petrol is complete (100 %); the adsorption of Gasol, in case of new carbon amounts up to 95 %

if the petrol load is smaller, i.e. about 3 - 5 %. The capacity of the carbon was per ton guaranteed with 1.25 kg of hydrocarbons. This value has been surpassed considerably. Supercarbon 75 in the form of little round rods of 4 mm diameter and 4 - 6 mm length was used principally.

Plant II comprises 4 adsorbers of 3.5 m diameter with a carbon layer of 2.20 m thickness, a carbon charge of 18 tons and works in the same way. Instead of separate circulations for cooling and drying a series connection is provided so that the synthesis residual gas flows through all adsorbers except that which is being rinsed. This gas current is superimposed by a circulation of 35 000 cu.m.p.h. for drying and cooling. The condensation and the separation of petrol are arranged at the adsorber platform as in plant I. Below the platform are the pipings and gas valves with hydraulic operation. The blowers are at a level with the ground. The measuring station is provided between plant I and plant II. In plant II is a large direct cooler placed before the indirect cooler of the drying circulation which takes up the heat accumulated in changing-over.

Condition and reconstruction of the plant.

Each plant has been damaged by a direct hit. Pipe systems and 4 hydraulically controlled gas valves have been destroyed; the blowers, coolers and heaters of plant I and pipe systems of both plants have been damaged. The Gasol gasholder has been slightly damaged.

For construction stage I the complete repair of plant I including Gasol gasholder is provided.

Construction time: 4 months.

II/3 **Further processing.**

Drawinget Folder II/3

In the steps of further processing, the products of the synthesis plant are worked up by distillation.

II/3 Further processing.

Washing of Diesel fuel with caustic soda solution.

The Diesel fuel, having a neutralization number of about 0.3 mg KOH/g is neutralized by this washing with caustic soda solution to obtain a neutralization number of about 0.02 mg KOH/g. The plant works under a pressure of about 2 atm. above normal and at a temperature of 40° C.

The following apparatusses are used:

1) 1 container of 25 cu.m. for caustic soda solution. In the process, caustic soda solution of 8 - 10 % strength is used, which must be renewed with a NaOH-content of about 1 %.

2) 1 separator of 6 cu.m., for separating caustic soda solution and Diesel fuel.

3) 1 separator, of 6 cu.m., for separating water and Diesel fuel.

4) 1 pump for feeding Diesel fuel, with an output of 6 cu.m. p.h.

5) 1 pump for caustic soda solution with an output of 1 cu.m.p.h.

6) 1 water pump with an output of 1 cu.m.p.h.

II/3

Further processing.

Washing of gas oil with caustic soda solution.

The gas oil having a neutralization number of about 0.5 mg KOH/g is neutralized by this washing with caustic soda solution to obtain a neutralization number of about 0.05 mg KOH/g. The plant works under a pressure of about 2 atm. above normal and at a temperature of 60° C.

The following apparatuses are used:

1) 1 container of 25 cu.m. for caustic soda solution. In the process, caustic soda solution of 8 - 10 % strength is used, which must be renewed with a NaOH-content of about 1 %.
2) 1 separator, of 6 cu.m., for separating caustic soda solution and gas oil.
3) 1 separator, of 5 cu.m., for separating water and gas oil
4) 1 pump for feeding gas oil, with an output of 6 cu.m.p.h.
5) 1 pump for caustic soda solution with an output of 1 cu.m.p.h
6) 1 water pump with an output of 1 cu.m.p.h.

Further processing.

Stabilization.

Description of the process.

The unstable primary petrol coming from the absorption plant having a vapor pressure according to Reid of 1.2 abs. atm. and the gasol gas from the absorption plant containing carbon dioxide (25 - 30 % CO_2) and conducted into a container for gasol gas, are jointly freed from gas and petrol respectively. The gasol from the gasholder is compressed up to 35 atm. above normal by means of two two-stage gasol compressors with an output of 850 cu.m.p.h. each, and is conducted, by way of a condenser (2 x 35 sq.m.) into a separator, kept at a pressure of 35 atm. above normal and a temperature of 25° C. The portions remaining in the gaseous state, chiefly products having 1, 2 and 3 carbon atoms and about 55 % of carbon dioxide, are led back to the absorption plant by way of an automatic pressure regulating valve. The liquefied product is furthermore released and led by way of a steam trap into a receiver of 25 cu.m., kept under a pressure of about 20 atm. above normal. This intermediate release is carried out in order to disengage a further portion of the carbon dioxide dissolved in the liquid. The released gas from this receiver has a content of about 50 % of carbon dioxide and is also conducted back to the absorption plant by way of a regulator. The liquid remaining in this receiver, called crude gasol, is then conducted by means of pumps together with the unstable primary petrol through a mixer and an inserted heat exchanger (50 sq.m.), wherein the stable petrol leaving the stabilization column with a temperature of about 185° C, transfers part of its heat to the material to be processed, to a pre-heater (3 sq.m.) wherein the feed product is heated to about 110° C., and then into the column. The operating pressure of this column is 15 - 18 atm. above atmospheric pressure. In the column, the material is separated into the reflux distilling off as head product by way of a condensing device (4 x 40 sq.m.) containing hydrocarbons with 2,3 and 4 C-atoms and about 8 % of carbon dioxide, into the fuel gas with a content of carbon dioxide of at most 2 % being removed as side flux through a side column with re-boiler (1 sq.m.), and into the stable petrol forming the bottom product with a vapor pressure according to Reid which can be regulated from 0,55 to 0,80 abs. atm.

The fuel gas leaving the side column is conducted by way of a cooler (10 sq.m.) to the fuel gas storing receiver and then by way of a pump station to the filling station for fuel gas. The stable petrol leaving the bottom of the main column is laid through the heat exchanger mentioned above and inserted coolers (2 x 35 sq.m.) into a receiver and then by way of a pump station to the tank store. Neutralization of this petrol is not necessary, because the neutralization number of 0.005 - 0.01 meets the requirements of the engine. The gas components not liquefied in the reflux container still containing beside 25 - 30 % of carbon dioxide essential portions of hydrocarbons with 3 and 4 atoms, are led back to be sucked again by the Gasol compressors.

In a second existing side column, the hydrocarbons with 4 C atoms can be drawn off separately.

The throughput of the plant is 8 tons of primary petrol p.h. and 2.5 tons of crude Gasol p.h.

The main column contains 50 bubble cap bottoms and has a diameter of 800 mm. The side columns have 15 bottoms and a diameter of 300 mm each.

The re-boiler of the main column has a heating surface of 80 sq.m. and is indirectly operated with steam of 18 atm. above normal.

Two weeks are necessary for newly starting the plant.

II/B

Further processing.

Redistillation.

In this plant, there are processed the light petrol coming from the fractionation, boiling between 80 and about 200° C., the light petrol from the distillation under atmospheric pressure of the oil plant, having a boiling range of 40 to about 210° C., and the heavy petrol II of the distillation under atmospheric pressure of the oil plant, boiling from 100 to 300° C. The cut is laid in such a way that a petrol boiling up to about 160° C. distils over as top product, and a Diesel fuel boiling from 160 to about 270° C. is obtained as bottom product. The top product is added to the motor petrol. The bottom product, having a flash point of +40° C. and a solidifying point of -42° C., is added to the heavy petrol of the fractionation, having a similar solidifying and flash point, and is used as Diesel fuel.

The technical data and guarantees of the plant are seen in the following statement:

Petrol distillation for a throughput of 1 900 - 2000 kg p.h.
Operating pressure up to ½ atm. above normal pressure.

1 fractionating column, diameter 600 mm., with 12 + 4 fractionating bottoms with bubble caps,
1 steam heated pre-heater, heating surface 20 sq.m.
1 re-boiler, heating surface 20 sq.m.
1 cooler for residue, cooling surface 25 sq.m.
1 condenser, cooling surface 40 sq.m.

Guarantee:

a) Output 2000 kg/h.
b) Fractionating E.P. (endpoint) capable of being adjusted with an accuracy of ±5° C., highest E.P. = 140° C.

The next page (page 44) of the German type-written report is missing.

II/4) **Paraffin plant**

(cf. drawing: folder II/4)

The following finished products are manufactured, starting from the distillation residues boiling above 320° C. of the crude paraffin of the medium-pressure synthesis, and of the condensed oil, including the hard paraffins coming from the extraction of the contacts (catalysts) of synthesis, obtainable in the topping or fractionating plants of the processing factories:

Plate paraffin
Soft paraffin
Hard wax
Plastic wax
Contact paraffin.

The two residues named above are stored in a heated tank of 100 cu.m. each, which are placed in an elevated position, and are separated in a continuously operating vacuum distillation plant into two distillates and a residue. For this purpose two columns are used containing the heating serpentines in the upper part and the heat exchanger and the cooler in the lower part. Heating is carried out by means of a warm water circulation system. The vacuum is produced by a horizontal two-stage pump. Feeding of the starting material is effected by a pump with a capacity of 1.5 cu.m. p.h. Drawing off of the distillates and residues from the stills is performed by gear pumps. Distillation is carried out in the first column at 160 - 180° C., in the second column at 280 - 300° C. under a pressure of 6 mm of mercury, the temperature of the off-runnings being measured.

For the production of plate paraffin the distillate II of the paraffin from the medium-pressure synthesis is used. It is first freed from acid in disintegrators with addition of 1 % of hot caustic soda solution of 5 % strength, and is washed with water. The acid-free product is brought into heating chambers at 70 - 80° C. and is cooled by air down to some degrees below the solidifying point within 24 hours. By slow re-heating of the resultant paste of crystals in the course of 48 hours it is possible to separate the paraffin crystals from the adhering oil. The portion of the oils with a high solidifying point drawn off is led back for the preparation of a new charge. The crystalline material is melted by

Further processing.
Alkazid-plant.

Description of the process.

The Alkazid-plant is destined for the production of carbon dioxide from the residual gas of the synthesis. This carbon dioxide is used as protective gas in the synthesis and for the saturation in the manufacture of catalysts in the catalyst factory for Fischer-Tropsch plants.

In the plant, Alkazid liquor is used, i.e. a special liquor prepared by I.G.Farbenindustrie for the production of carbon dioxide.

The Alkazid liquor has the property to absorb carbon dioxide at low temperature and to disengage it again at boiling temperature. Therefore, the process on a technical scale is carried out in such a way that the Alkazid liquor is conducted between a washing tower and a gas expeller in continuous circulation with the following steps: The regenerated liquor (fresh liquor) enters first the washing tower (diameter 1.5 m, height 12 m), working under a pressure of a water column of 4 m and filled with Raschig rings made of procelain, passes it from above to below in countercurrent to the residual gas to be freed from carbon dioxide, and is loaded with carbon dioxide. The most suitable working temperature in this washing tower is about 45° C.

The liquor is saturated with carbon dioxide taken from the residual gas of the synthesis, and is then pressed by means of a pump out of the sump of the washer through a heat exchanger into the gas expelling tower (diameter 1.5 m, height 17 m), working under a pressure of a water column of 4 m and filled with Raschig rings made of procelain. In the heat exchanger mentioned above, the saturated liquor is heated by the regenerated liquor running the reverse way, and the latter is cooled down correspondingly.

The saturated, pre-heated liquor passes the gas expelling tower from above to below, is heated more and more and is finally boiled for a short time in the lowerst part of the gas expelling tower at a temperature of about 110° C, by means of a boiler indirectly heated with steam of 2.5 atm. above atmospheric pressure.

By action of the elevated temperature and of the water vapors rising from below to above, the carbon dioxide, previously absorbed in the washing tower, is separated from the liquor in the expeller. The carbon dioxide leaves the gas expelling tower at the head, together with the water vapors. In order to compensate the loss of water steam is directly introduced into the lower part of the expelling tower for regulation of the density of the Alkazid liquor. The carbon dioxide is conducted, after cooling and condensing the water vapors, into a gasholder of 300 cu.m. A piston compressor for 6 atm. above normal pressure sucks the carbon dioxide out of this holder and presses it, by way of two drying towers filled with silica gel, which can be used alternately, to the places of consumption. The regenerated liquor freed from gas is run in the hot state to a second pump whereby it is conveyed by way of a heat exchanger and a cooler connected therewith, to the washing tower again. Thus, the circulation of the liquor begins again.

In the plant, about 900 cu.m. of residual gas of the synthesis are partly freed from their content in carbon dioxide. The maximum production of carbon dioxide amounts to 230 cu.m.p.h.

directly blowing in steam, dehydrated by means of a separator, refined by means of about 1 % of bleaching earth and a small quantity of decolorizing carbon, pressed through a heated frame filterpress and finally brought to a cooling press. The plates obtained here are cut into a size suitable for dispatch. The solidifying point of the material is 50 - 52° C.

In the same way, by heating of the distillate I of the paraffin coming from the medium-pressure synthesis, a material with lower solidifying point is obtained. If this product together with plate paraffin is mixed with hard wax the latter becomes less brittle. The elastic plastic wax thus obtained has a solidifying point of about 75° C. and is formed into scales by means of a cooling roller.

4 heating chambers are present and each contains 48 heating cells, double cylinders with a height of about 3 m, an interior diameter of 450 mm and an exterior diameter of 600 mm. The circular room with an inner width of 150 mm is destined to take up the material to be heated. Heating is carried out by steam pipes arranged below; for melting, steam inlets are provided in the height of the bottom of every cell. One chamber can take up 15 cu.m. of material. 5 cu.m. of finished product are obtained, and 3 cu.m. of oil are led back. For refining, a mixing vessel of 10 cu.m. is used. Mixing is carried out by compressed air. Frame filter press and cooling press contain 36 frames each, their measurements are 800 x 800 x 25 mm and 1000 x 800 x 40 mm.

The residue of the vacuum distillation of the paraffin from the medium-pressure synthesis is refined, at 80° C., after adding about 10 % of bleaching earth and 1 % of decolorizing carbon, filtered and formed to scales by means of a cooling roller. The pure white hard wax obtained has solidifying points of 90 - 95° C. For refining there may be applied two mixing vessels of 10 and 15 cu.m. and a further frame filter press of the measurements named above. Furthermore, two cooling rollers with a daily output of 15 tons each are present.

The residue obtained in the fractionation contains the high boiling paraffins of the normal pressure synthesis and the contact paraffin coming from the extraction of the synthesis catalysts. The residue obtained therefrom by vacuum distillation can only be refined with

relatively great difficulties. Hitherto the non-refined material could be sold as it was. Seales are also produced on a cooling roller with a daily output of 12 tons.

All the distillates and oils obtained by vacuum distillation or by the heating process, which cannot be processed, in the said manner or by mixing with suitable portions of soft paraffin with a solidifying point of 30 - 35° C., to produce marketable goods, are used in our plant as starting material for the manufacture of cracking petrols and, thus, of synthetic lubricating oils.

The spent bleaching material obtained in the refining process, contains about 50 % of paraffin and is also vendible.

Condition of the plant.

The cooling press and the corresponding pressure vessel for the manufacture of plate paraffin are destroyed. Smaller damages exist in a container for caustic soda solution, in a receiver for residues and a mixer. There are missing two plunger pumps with an output of 3.5 cu.m.p.h., which have been sent for repairs to the furnishing firm, Maschinenfabrik Dahne at Halle/Saale. The hot water oven must be overhauled. The supply of electric current is severely damaged, but the distributing system is generally in order. Also the piping damages are small. Some insulations must be repaired. Buildings have to be repaired to a pretty large extent.

II/5

Lubricating oil plant

Stabilization

This stabilization plant, originally built for the stabilization of the light hydrocarbons of the synthesis is used for the stabilization of the low temperature petrol obtained in the Dubbe cracking plant. Low temperature petrol and cracking gas are processed with a throughput of about 1.2 cu.m. of low temperature petrol and about 800 cu.m. of cracking gas p.h. As top product a fuel gas with a liter weight of about 1.65 and as bottom product stable petrol with a vapor pressure according to Reid of 0.7 absolute atmospheres is obtained.

For the compression of the cracking gas, which is fed in the gaseous state into the upper third of the column, a tandem compound compressor having a sucking capacity of about 1000 cu.m. is available The conditions of temperature and pressure are as follows:

Column pressure: 16 atm. above normal
Pre-heater temperature: about 120° C.
Re-boiler temperature : " 180° C.
Bottom temperature : " 175° C.
Head temperature: about 62° C

The fuel gas obtained is mixed with the fuel gas of the stabilization II and is conducted to the filling station. The stabilized low temperature petrol is mixed with the condensation petrol of the first stage of the cracking plant and after drying is conducted to the polymerization plant of the lubricating oil synthesis. The waste gas from the stabilization containing about 25 % of ethylene, is sent to Chemische Fabrik Holten in order to utilize the ethylene.

The technical data and guarantees of the plant are seen in the following statement:

Gas petrol stabilization and gas petrol recovery. Throughput: 1 900 kg of petrol + 1000 cu.m. of gas p.h. Operating pressure: maximum 25 atm. above normal.

1 fractionating column, diameter 600 mm,
35 fractionating bottoms with bubble caps at a distance of 600 mm. Operating pressure 25 atm. above normal.

1 pre-heater, 15 sq.m.
1 heat exchanger, 25 sq.m. heating surface
1 re-boiler 25 sq.m. heating surface
1 petrol cooler, 20 sq.m. cooling surface
1 condenser, 60 sq.m. cooling surface

Guarantees:

a) Output: 2000 kg of petrol and 1000 cu.m. of gas (gas condensed by means of active carbon)
b) The gas freed from petrol is practically free from pentene (below 0.05 %)
c) The stabilized petrol is free from propane (below 0.05 %)
d) The vapor pressure may be adjusted to 0.4 - 0.6 abs.atm. according to Reid at 40° C. ± 10 %.

The plant can be ready for use within two weeks if sufficient workers are available.

II/5 Lubricating oil plant

Synthesis

Description of the manufacture of lubricating oil.

The starting material for the production of synthetic lubricating oils are two fractions of the primary products of the Fischer synthesis, to wit the fraction boiling from 230 to 320° C. and the fraction boiling above 300° C. The cracking petrol produced in the Dubbs olefine cracking plant consisting of about 72 % of olefines having the double bond at the end of the chain, is dried by means of calcium chloride, as the moisture content of the petrol shall not surpass 0.01 %. The drying with calcium chloride is carried out in a tower containing about 12 tons of calcium chloride. The throughput of this tower is about 3 cu.m.p.h. After drying, the cracking petrol is conducted into the stirring vessels for synthesis. After addition of aluminium chloride as a catalyst, two new products are formed during stirring by polymerization and condensation at a temperature rising from 20° C. up to 100° C., the so-called contact oil and a polymerization product, called "upper layer". From this "upper layer", after dechlorination and filtration the lubricating oil is distilled by means of a normal pressure distillation followed by a vacuum distillation. The aluminium chloride is granulated and, as the process is continuous, is introduced into the synthesis vessel previous to every charge in a quantity of 1.2 %, calculated upon the cracking petrol used.

The stirring vessel for synthesis has a volume of 32 cu.m. For one charge are used:

150 kg of aluminium chloride,
13 cu.m. of cracking petrol and
8 cu.m. of contact oil (in the case of manufacturing motor oil).

The stirring vessel for synthesis is provided with steam heating and water cooling.

As a reaction takes place the increase of pressure during conversion is not proportional to the increase of temperature because of the change of the boiling range of the reaction product. The pressure rising quickly in the beginning up to 3 atm. above norma'

decreases to about 1.5 atm. above normal at the end of polymerization. Reaction time is 12 - 14 hours.

In the following an example is given for a favorable temperature course during polymerization:

1 hour at 40° C.,
2 hours " 60° C.,
3 " " 80° C.,
8 " " 100° C.

The velocity of polymerization increases with rising temperature.

The number of revolutions of the stirrer in the synthesis vessel is 150 per minute.

As time for cooling down after the polymerization, about one hour is necessary.

The contact oil formed during polymerization is a complex compound of aluminium chloride and hydrocarbons, and must be designated as an intermediate compound of a homogeneous catalysis. It is composed of about 70 % of organic and 30 % of inorganic components. About 2 - 3 % of the cracking petrol used form contact oil with the aluminium chloride present. The most favorable proportion for the polymerization to motor oil is 1 : 2 for contact oil and cracking petrol. After polymerization the reaction product consists of about

60 % of lubricating oil,
25 % of Diesel oil and
15 % of petrol,

having a boiling range up to 200° C. About 78 % calculated upon the about 72 % of olefines of the cracking petrol are polymerized to lubricating oil. The remainder is used up for the formation of polymerizates like Diesel oil and light oils.

The boiling range of the "upper layer" is about the following:

boiling-up to 120° C.	0.2 %	by weight
" from 120 to 140° C.	0.9 %	" "
" " 140 " 160° C.	1.9 %	" "
" " 160 " 180° C.	3 %	" "
" " 180 " 200° C.	12 %	" "
" " 200 " 300° C.	10 %	" "
" " 300 " 370° C.	7 %	" "
" above 370° C.	65 %	" "

After polymerization, the reaction product is pressed out of the stirring vessels for synthesis by means of nitrogen and is separated from the accompanying contact oil in settling towers. The time of settling in the settling vessels of 25 cu.m. is about 7 hours. The duration of settling essentially influences the degree of separation of the contact oil and, thus, the effect of the following dechlorination. The separated contact oil is drawn off in the lower conical section of the settling tower into a receiver. It is returned to the synthesis at the mixing rate of petrol and contact oil given above. The "upper layer" is led by way of a receiver into the dechlorination vessels. These vessels have a volume of 32 cu.m. and are provided with stirring devices with a number of revolutions of 105 per minute. The dechlorination vessels are provided with steam heating and water cooling like the vessels for the synthesis. One charge comprises 24 cu.m. of "upper layer" which are mixed with about 1.2 % of bleaching earth (Tonsil) and about 1.2 % of zinc oxide, calculated upon the "upper layer", and is stirred for about three hours at a temperature of 180° C. The time of heating up to this temperature is about 2½ hours, the maximum pressure attained during the dechlorination at 180° C. is about 12 atm. above normal. The water content of the auxiliary agents Tonsil and zinc oxide must not surpass 1 %.

When leaving the settling tower, the "upper layer" contains chlorine in the following form:
Carriers of chlorine are:
1) The particles of contact oil still present,
2) the hydrogenchloride dissolved in a small quantity,
3) chlorinated hydrocarbons formed during polymerization.

The total chlorine content of the "upper layer" after settling is about 7000 mg /kg; about 4000 - 4 500 mg/kg thereof are chlorine capable of being split off. The neutralization number is 2 - 3. After dechlorination the total chlorine content has sunk to about 100 mg/kg; 15 mg/kg are chlorine capable of being split off. The neutralization number is about 0.02. The dechlorinated product is separated from the added auxiliary agents in a frame filter (Kelly-filter). The capacity of these filters with a filter surface of 25 sq.m. is about 25 cu.m.p.h. Filtration is carried out at 80° C. In order to completely remove the auxiliary agents, the product is filtered clear in a supplemental frame filter press. The de-

chlorinated and filtered product is subjected, for further processing, to a distillation under normal pressure in order to distill off the portions of petrol and Diesel oil up to a boiling limit of about 300° C. The maximum throughput of this plant is 5.5 cu.m.p.h. The outlet temperature of the tubular oil heater, having a heating surface of about 80 sq.m. is 250° C. The outflow temperature of the heavy petrol running into a side column for heavy petrol is 160° C., the head temperature of the main column is 120° C. The bottom product of the main column is conducted, by way of a siphon, into a second column, wherein the product called heavy petrol II is distilled off by means of steam. The products obtained by distillation under normal pressure are about as follows:

10 % of light petrol boiling, up to 200° C.,
20 % of heavy petrol I (Diesel oil), boiling from 180 to 330° C.,
5 % of heavy petrol II, boiling from 100 - 300° C. and
65 % of partly finished oil (residue).

The partly finished oil of the distillation under normal pressure is further processed in a supplemental vacuum still distillation under a pressure of 2 mm of mercury. The products obtained in the still distillation with addition of steam are as follows:

1) first runnings,
2) distillate I,
3) " II,
4) " III,
5) residue.

The temperature and operating data as well as the boiling range of the said products depend on the types of oil to be manufactured. All sorts of spindle oil, machine oil, motor oil and superheated steam cylinder oil can be made. The throughput of the plant amounts to about 3 cu.m.p.h. The oils produced are already pale and are further brightened by means of bleaching earth (Tonsil) in stirring vessels at about 140° C.

The filling of one bleaching vessel is: 8 cu.m. of oil,
Heating up time: 1 hour,
Bleaching time: 20 minutes,
Addition of Tonsil per charge: about 15 - 20 kg.

The bleached oil is freed from the added Tonsil without previous cooling in a frame filter (Kelly filter) with a filter surface of 25 sq.m. with an output of 8 cu.m.p.h. A second filtration follows in a frame press, having a filter surface of 24 sq.m.

The output of the plant, with the existing four non-damaged synthesis vessels and with 5.5 charges per day corresponding to a consumption of cracking petrol of 100 cu.m., is about 1000 tons in 25 working days monthly.

The yield of the lubricating oil production from cracking petrols beginning with 5 C-atoms, corresponds to the following numbers:
40 % of lubricating oil, calculated upon feed for cracking plant,
51 % " " " " " cracking petrol.

The viscosity pole height of the lubricating oil produced is about 1.72.

The distribution of the whole products obtained in the polymerization, calculated upon
1) feed for cracking plant
2) cracking petrol
is as follows:

1) <u>feed for cracking plant:</u>

40.0 % of lubricating oil
17.6 % of Diesel oil
4.7 % of rest petrol up to 9 carbon atoms
1.8 % of contact oil and
1.3 % of loss.

2) <u>cracking petrol:</u>

60.0 % of lubricating oil
27.1 % of Diesel oil
6.0 % of rest petrol up to 3 carbon atoms
3.0 % of contact oil and
2.0 % of loss.

For the repairing of the plant in order to reach an output of 1000 tons per month, about three months are necessary until newly starting the plant if a sufficient number of workers is available.

) Dubbs olefine cracking plant.

Description of process.

The plant is divided into the following parts:

1) Cracking furnace:

consisting of a heating zone with a heating surface of about 300 sq.m. and a cracking zone with a heating surface of about 265 sq.m. The furnace must be considered as a pure convection furnace. The pipe system of the furnace consists of pipes of 3½ and 4 inches, heated for a length of 10,500 mm. 80 pipes of the heating zone in the cold section consist of normal carbon steel, whereas the two lower series (10 pipes) of the heating zone and the 80 pipes of the cracking zone, connected in 5 parallel strands, consist of the material Marwe 215 ESV. The furnace is heated with the residual gas of synthesis.

2) Coke chamber:

The coke chamber is a cylindrical container, diameter 2 100 mm and height 10 500 mm, without insertions. In order to cool the vapors leaving the furnace, the bottom product taken from the sump of the fractionating column, cooled down to 90° C., is introduced into the coke chamber as cooling oil in a quantity of 5 - 6 cu.m.p.h.

3) Fractionating column:

The vapors leaving the coke chamber are conducted into the fractionating column, diameter 1 800 mm, height 18 m.

This column is constructed as follows:
18 bubble cap bottoms in the upper part for separating petrol and return product (recycle), a receiver for drawing off the recycle built into the column, below it two further bubble cap bottoms and 6 perforated bottoms for separation of recycle and cooling oil and the cooling oil sump.

4) Condensation:

The vapors of the fractionating column are subjected to a two-stage condensation. The first stage is carried out indirectly by water and the second stage indirectly by brine. The cracking petrol cooled down to about 70° C. in the condensation stage I is

returned to the column as reflux and the product proper is conducted after supplemental cooling or washing with caustic alkali solution to the storage tank.

The condensation stage I, consisting of 2 groups with condenser of 100 sq.m. and cooler of 80 sq.m., can be operated alternately on account of the corrosion occurring during condensation caused by the organic acids existing in the cracking petrol.

The so-called low temperature petrol obtained in the condensation stage II is cooled down to about 10° C. and is conducted by way of an inserted pressure receiver to the stabilizer already described. In the gas section of the storage vessel for the low temperature petrol, the final pressure regulator is installed, adjusted to 4 atm. above normal. Thereby the cracking gas produced can pass off into a container for propane-butane-mixture. This cracking gas is conducted into the stabilizer together with the low temperature petrol in order to remove petrol.

In the plant, the gas oil coming from the fractionating process, boiling range 230 - 320° C. (material I) is processed or, separated thereof, the distillates beginning to boil above 300° C. and coming from the paraffin plant.(material II). The cracking petrol produced has always a final boiling point which corresponds to the initial boiling point of the raw material used, i.e. with a feed material I beginning to boil at 200° C., the final boiling point of the cracking petrol is 200° C., and with a feed material II beginning to boil at 300° C., the final boiling point of the cracking petrol is 300° C. The cracking petrols produced have an olefine content of 70 - 76 %. The double bond is chiefly at the end of the molecule. The consumption of the plant amounts to about 100 tons per day, if material I is processed, and at most 135 tons per day in the case of material II.

The yield of stabilized petrol is about 65 % (material I), and about 72 % (material II).

In the process, 1000kg of steam superheated to 500° C. are added per hour between heating and cracking zone.

The pressure course from the entrance of furnace to the final pressure regulator is as follows:

	with material I and II
inlet of furnace	14 atm. above normal
outlet of heating zone = inlet of cracking zone	7 " " "
outlet of cracking zone	5,8" " "
outlet of coke chamber	5,5" " "
outlet of fractionating column	5.2" " "
final pressure in the storage tank of low temperature petrol	4 " " "

The course of temperature is as follows:

	material I	material II
inlet of heating zone	200° C.	240° C.
outlet of " "	460° C.	450° C.
" " cracking zone	550° C.	530° C.
" " coke chamber	380° C.	410° C.
" " fractionating column	200° C.	275° C.

With a total feed of the furnace of 20 cu.m.p.h., there is used in the case of

material I = 1 part of fresh oil for 2.65 parts of recycle
material II = 1 " " " " " 1,85 " " "

The operating data of the pumps are as follows:

	cu.m.p.h.	gauge pressure	temperature
hot oil pump	20	14	260° C.
fresh oil pump	5.5 - 7.5	14	60° C.
cooling oil pump	6	12	90° C.
reflux pump	10 - 14	12	70° C.

For repairing of the plant, six weeks are necessary until new start, if a sufficient number of workers is available.