THE ARC PROCESS FOR ACETYLENE PRODUCTION

1. Introduction.

(a) The synthetic rubber plant of I.G. Farbenindustrie at Huls, Germany, has what is believed to be the first successful commercial plant in existence for the production of acetylene by the thermal cracking of low molecular weight hydrocarbons. The plant produced around 200 metric tons per day of a 97 percent acetylene for subsequent use in other parts of the plant in the manufacture of acetaldehyde, aldol, buylene glycol, and butadiene. By-products of the acetylene production were hydrogen, carbon blac, and some small amounts of ethylene. The plant is at present time intact and capable of immediate operation as soon as raw materials are supplied. Dr. Baumann, former chief chemist of the plant, has assumed direction of the entire works.

(b) The process in brief consists of high temperature, normal pressure cracking of natural gas using an electric arc, and the subsequent purification of the acetylene by scrubbing it from the gas stream with water at 18 atmospheres. Hydrogen and ethylene are removed from the tail gases by low temperature liquefaction and fractionation, and the remaining gases are recycled to the arc. Carbon black formed in the arc is removed by water sprays, cyclones and bag filters, and is used in the compounding of synthetic rubbers.

(c) The plant formerly operated on a refinery tail gas consisting largely of C_2 and C_3 hydrocarbons. At that time the yields were much superior to the present methane feed stock. However, subsequent bombings so destroyed surrounding refineries that the acetylene plant was forced to the use of natural gas. In the following sections of this report an attempt will be made to describe in detail the operating conditions and equipment used when operating on natural gas, and to point out what differences were encountered when using the refinery tail gas.

2. Arc Converters.

(a) The plant had seventeen (17) pairs of high temperature arc converters for the thermal cracking step. Only ten (10) pairs of these were ever used because of bottlenecks further along the process, and because

2. Arc Converters (a) (Cont'd.)

of power shortages in the area. Each converter pair was equipped with its own rectifiers, coolers, and minor appurtenances. One converter was operated while the other was shut down for repair or overhaul.

(b) Feed to each arc converter was 2800 m^3/hour of a mixed gas consisting of 50 percent by volume natural gas, and 50 percent by volume recycle gas. The natural gas contained about 92 percent volume methane, 5 percent nitrogen, and 3 percent carbon monoxide. There was about 10 grams of surfur per cubic meter present in the form of hydrogen sulfide. The recycle gas contained about 85 percent methane, and the balance hydrogen, nitrogen and heavier hydrocarbons. The gas was fed to the arc at normal temperature and around 1.8 - 1.5 atmospheres pressure.

(c) Each arc was powered by a mercury vapor rectifier system operating on 6000 volts three-phase line power. Where at all possible, two rectifiers operated in series, each giving 1000 amperes at 3500 volts, and rectifying from a twelve phase secondary for smooth rectification. This gave a 7,000 volt, 1,000 amperes feed to the arc. Because of the shortage of rectifiers, some of the arcs were forced to use a single one with 7,000 volt output. This was found to be quite unsatisfactory, and there was continual trouble with backfires, etc. Figure 1 shows the electrical hookup of the rectifiers when working in series for one pair of arcs. The rectifiers were made by Brown-Boveri Company.

(d) The arc converter proper is shown in Figure 2. Feed gas enters the distributing ring tangentially near the top of the apparatus. It then flows through slots in the annular distributor and swirls down through the arc tube. On leaving the tube it is immediately quenched with a water spray in the bottom section. The distributing ring, arc tube, and spray section are all made of ordinary steel. They are grounded, as is one side of the rectifier output, and water jacketed for proper cooling.

(e) The upper electrode is made of copper and separated from the converter proper by a porcelain insulator. It is water jacketed to prevent local overheating. A pneumatic operated steel shortingrod starts the arc, and is withdrawn automatically when the right current is flowing.

(f) Control of the arc synthesis is affected by control of the power factor. At the design current and voltage, the feed gas flow is in-

2. Arc Converters (f) (Cont'd.)

creased until the power factor is 0.7 to 0.75. Under these conditions the arc flame extends the full one meter length of the arc tube. Higher powerfactors cause the arc to go out; lower ones give lower conversion. The arc tube proper, or the center one (1) meter section of the apparatus, has a life of about 250 hours. At the end of this time the internal diameter is so enlarged as to prevent further satisfactory operation. This tube can readily be replaced by disconnecting flanges at either end and springing down the lower section through its bellows expansion joint. During this time the other arc of the pair is in operation, thus avoiding the necessity for shutting down the synthesis.

(g) Mean gas temperature in the arc is estimated as 1,600 degrees centigrade. Temperatures nearer 1,300 would be preferable, but are difficult to obtain in the apparatus without seriously affecting its operation. The exit gas from the apparatus, after quenching with water, has the approximate composition (carbon black free) as given below:

C_2H_2	13% by volume
H_2	52%
C_2H_4	1%
sat. HC's (mostly methane)	25%
CO	2%
N_2	7%

In addition about 3 percent of the hydrocarbon input is converted to carbon black and leaves the arc entrained in the gas stream.

(h) When operating on refinery tail gases it was found that the exit acetylene concentration was much higher and hydrogen also up accordingly. Saturated hydrocarbons took care of this increase by being correspondingly lower. This means that when operating on natural gas the yield was 50 percent of theoretical, while on refinery gases it was stated to be even better. No figure was given to indicate the magnitude of the improvement.

3. Preliminary Purification.

(a) The plant had a rather elaborate preliminary purification plant which was admittedly not all necessary for acetylene production. However, the management stated that in view of the fact that the entire process was built hurriedly during the war without all necessary piloting, and in view of war-time shortages of certain materials such as HCN, the elaborate system was installed. This seems entirely reasonable, and experience has shown the lack of necessity for many of the items.

(b) In Figure 3 is shown a process flow diagram of the preliminary purification, including the carbon black plant. Gas from the arc, which has been cooled to 150 degrees centigrade by the water quench, is passed down through a water seal to four (4) cyclones, two (2) operating in series, two (2) in parallel. High density carbon black is removed here, and sent through a series of hoppers and conveyors to an air classifying tower. Oversize material is removed, and the remaining black is tumbled in drums to increase its packed density for shipping. It is then bagged and shipped to rubber plants throughout Germany.

(c) The gas from the cyclones is then cooled to 70 degrees centigrade in a spray cooling tower, where low density carbon black is removed as a slurry in the spent cooling water. This black is then settled in a Dorr classifier, filtered in a vacuum filter, dried, and bagged. It is used in mixture with the other black in rubber goods manufacture.

(d) For final cleanup the last traces of black are removed in eight (8) bag filter towers. These towers are heated slightly with steam to prevent the saturated entering gas from wetting the bags and ruining their efficiency.

(e) So far the system described has applied to each pair of arc converters. At this point however the gas streams from all converge in a common header, and goes to two (2) water spray coolers operating in parallel. It is cooled to 25 degrees centigrade with a minimum of water so as to prevent the solution of much of the HCN present. The gas then passes to two (2) oil scrubbers operating in parallel, using revolving trays. Here the benzene and higher hydrocarbons are removed. The wash oil used was a light tar oil, although an aliphatic oil was preferable but not obtainable. The scrubber bottoms were heated to 70 degrees centigrade and stripped in two (2) parallel towers using CO from subsequent purification steps. These towers were operated under 600 millimeters vacuum, and the overhead from them was water-washed and sent to the boiler house for fuel.

3. Preliminary Purification (3) (Cont'd.)

The stripper bottoms were in main recycled to the oil scrubbers, but a small part was continuously withdrawn and distilled in one (1) of four (4) tar stills. Here the heavier hydrocarbons and sludges were removed from the system, and fresh wash oil returned.

(f) The main process gas is next passed through two water scrubbers in parallel, where HCN is removed. The bottoms from these scrubbers, along with the water from the previous water spray after the carbon black cyclones, are sent to two (2) vacuum stills for HCN recovery. Bottoms from these stills are aerated in a cooling tower and returned to the system. HCN overhead, still in crude form, is worked up in a small batch still. Much trouble was encountered in this recovery because of the presence of higher acetylenes in the HCN cut. Nevertheless HCN was recovered and used elsewhere for acrylonitrile production.

(g) The gas stream was finally sent to six (6) large "Luxmasse" or iron oxide towers where sulphur was removed. These are similar to towers used in the U.S. manufactured gas industry. The gas is now ready for acetylene recovery.

4. Acetylene Recovery.

(a) In Figure 4 is shown the acetylene recovery system. Raw gas from desulfurization is combined with recycle gas from the recovery system and compressed in four (4) stages to 18 atmospheres. Four stages were deemed necessary to prevent local interstage overheating of the high pressure acetylene-containing gas. There were six (6) complete acetylene recovery units similar to the one herein described, all operating in parallel.

(b) The high pressure gas was scrubbed in a 66 perforated-tray tower with pure water, and the overhead gas from the tower contained less than 0.1 percent acetylene. Its composition was approximately as follows:

25%	CH
55%	H_2
1%	C_2H_4
4%	CO

4. Acetylene Recovery (b) (Cont'd).

10%	N2
4%	C_2H_6 and higher
1%	O_2

This gas is sent to units for hydrogen and ethylene recovery which are described later.

(c) The acetylene containing water is expanded to 2 atmospheres through a turbine, and flashed in a vessel. The off-gas, containing percent acetylene, is recycled to the compressor mentioned above. The water is then flashed to one atmosphere and a 92 percent acetylene off-gas obtained. It is further flashed to 0.15 and then 0.05 atmospheres, using suitable vacuum pumps, and these gas streams combined with the one atmosphere flash to give a 96 percent acetylene, containing N_2, CO_2, CH_4, C_2H_4, H_2, and small amounts of higher acetylenes. Before acetaldehyde synthesis it was necessary to remove the higher acetylenes, which was done in one of two (2) fashions and will be described below. The water from the last flash is pumped to one atmosphere, stripped with gas being recycled to the arc, and aerated in a cooling tower to remove last traces of acetylene before returning to the water scrubber. Summer water temperatures into the top of the scrubber never exceeded 26 degrees centigrade.

5. Acetylene Purification.

(a) Removal of higher acetylenes was accomplished in one of two (2) ways. The first method made no attempt to recover this material, but was much cheaper to build and operate. The second attempted recovery for further synthesis, but was quite expensive to build and operate.

(b) In Figure 5 is shown the first or oil scrubbing method. Here a light wash oil of unknown composition (stated to be aliphatic) was used to scrub out the higher acetylenes. This oil was then stripped with recycle gas to the arc, first of acetylene in the upper section of the column, then of higher acetylenes in the lower. However, the selective stripping proved unworkable, and the column was finally run as a single stripper. The off-gas from the oil scrubber, containing about 97 percent acetylene, was considered purified material and sent to the acetaldehyde synthesis unit. It was essentially free of all higher acetylenes.

5. Acetylene Purification (Cont'd)

(c) In Figure 6 is shown the second or liquefaction method of higher acetylene removal. Here the raw acetylene gas is first cooled in a water scrubber and then the higher acetylenes condensed by cascade refrigeration to -78 degrees centigrade using ammonia and ethylene. Counter current heat exchangers assure maximum recovery of refrigeration from the purified acetylene. The condensed higher acetylenes are quickly flashed in a stripper by the use of warm recycle gas. It is imperative that the warming up and vaporization of the higher acetylenes is accomplished rapidly to minimize the explosion hazards.

(d) Some small amounts of aromatics, particularly benzene, condense from the gas stream when it is cooled to about -10 degrees centigrade. These are steam distilled, decanted, and used as fuel.

(e) It has been found that the use of methanol as a diluent for the liquified higher acetylenes greatly decreases the hazard of explosion. Therefore, the entire system is properly protected with methanol flushing lines to enable quick dilution in the event of trouble.

6. Hydrogen Recovery.

(a) The off-gas from the acetylene purification contains a number of valuable constituents which are recovered before recycling the remainder to the arc. The two (2) principal ones are hydrogen and ethylene. The gas, whose composition has been given in former sections, is first treated for hydrogen recovery in a Linde designed liquefaction plant. Figure 7 shows the flow sheet of the apparatus. In it five (5) cuts are made on the gas; a hydrogen cut, a $CO-N_2$ cut, an ethylene or C_2 cut, a CH_4 cut, and a C_3 cut. The last two (2) are combined within the apparatus and furnish the major source of recycle gas for the arc. The hydrogen cut is used for hydrogenation within the plant, the $CO-N_2$ cut for inert stripping, and the C_2 cut is further processed for ethylene.

(b) The off-gas is first compressed to 16 atmospheres and cooled by counter current exchange with the product streams to -15 degrees centigrade. It is then cooled with ammonia refrigeration to -50 degrees centigrade. Further heat exchange with the cold product gases condenses the C_3 cut, which is caught in a trap, flashed to one atmosphere, and joins

6. Hydrogen Recovery (b) (Cont'd)

the CH_4 stream as the recycle gas cut. This stream returns through the two (2) previously mentioned exchangers, and goes to the arc.

(c) The off-gas, now at -70 degrees centigrade, is further cooled to -140 degrees centigrade by counter current exchange with product gases and sent to a condenser. Here the C_2 cut is liquified by condensing with the cold hydrogen cut and a medium pressure outside nitrogen refrigeration coil. The nitrogen has been previously compressed to 200 atmospheres and condensed by exchange with one of several of the cold gas streams; i.e., cold medium pressure gaseous nitrogen, cold methane, cold atmospheric pressure nitrogen, and cold high vacuum nitrogen. The C_2 cut is flashed to one atmosphere and sent to the ethylene recovery plant through the previously mentioned exchangers.

(d) The off-gas, now free of C_2 and higher, and at -145 degrees centigrade, is further cooled to -170 degrees centigrade with the H_2, $CO-N_2$, and CH_4 streams. The CH_4 cut is then condensed with a one atmosphere outside nitrogen cooling cycle, and is flashed to one atmosphere and returned through suitable exchangers as shown to the arc, mixed with the C_3 cut just before the C_3 condensing exchanger. The remaining off-gas, now at -185 degrees centigrade, is sent to the base of a double rectification column.

(e) The bottom or high pressure part of the column condenses and removes the $CO-N_2$ cut as bottoms. Liquid nitrogen at one atmosphere is used as reflux for this section, and a liquid hydrogen evaporator at the bottom adds additional cooling. The $CO-N_2$ cut is evaporated by liquifying medium pressure nitrogen and cooling atmospheric pressure nitrogen, and is then sent back through the counter current exchangers mentioned before and used as an inert stripping gas.

(f) The hydrogen cut is liquified in the condenser side of the upper column by using boiling liquid nitrogen at 0.1 atmosphere absolute pressure. It is evaporated in the lower section and returns through the exchangers previously mentioned. Temperature of liquefaction is about -212 degrees centigrade. This hydrogen is used in other sections of the plant for various hydrogenation processes.

(g) The hydrogen cut contains about 2 to 3 percent N_2, and the re-

6. Hydrogen Recovery (g) (Cont'd.)

mainder H_2. The CO-N_2 cut contains about 15 percent CO, 70 percent N_2, and 15 percent CH_4. The recycle gas cut contains about 85 percent CH_4, with the rest C_3's.

7. Ethylene Recovery.

(a) The ethylene recovery system is shown in Figure 8. It is a Linde designed liquefaction system for fractionation of the C_2 cut. The feed gas contains about 15 to 25 percent ethylene, and the purified ethylene product contains over 98 percent C_2H_4. The other cut, containing principally ethane diluted with some methane, is combined with the recycle gas from the hydrogen recovery unit and recycled to the arc.

(b) The feed gas is first compressed to 30 atmospheres and cooled to -35 degrees centigrade by ammonia refrigeration. It is then cooled to -55 degrees centigrade by exchange with cold CH_4 cut, to -95 degrees centigrade in the reboiler coil of the methane column, to -105 degrees centigrade by exchange with the C_2 intermediate fraction, and the CH_4 cut, and is then flashed to one atmosphere and -140 degrees centigrade and put into the top of the methane column.

(c) The methane gas overhead from the methane column passes through the exchangers previously mentioned and is combined with the ethane gas stream and goes to the arc. Bottoms from this column are passed to the center section of the ethylene column.

(d) The ethylene column takes ethylene overhead as product, and after suitable heat exchange this material is sent to the styrene plant as purified ethylene. A 6 atmosphere ethylene refrigeration cycle, with 0.7 atmospheres suction, is used for reflux and cooling for the column as shown in the flow-sheet. Column bottoms, consisting primarily of C_2H_6, are flashed and after suitable heat exchange combined with the CH_4 cut and sent to the arc.

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The Joint Chiefs of Staff,
by Col. E. W. Gruhn.

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THE WESSEL~~K~~ING SYNTHETIC FUEL PLANT

Mulit & Aldrich

CONFIDENTIAL

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

~~CONFIDENTIAL~~

THE WESSELING SYNTHETIC FUEL PLANT

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CIOS Target No. 30/4.10
Fuels and Lubricants

[1945]

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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THE WESSELING SYNTHETIC FUEL PLANT

ERRATA SHEET

Page	Paragraph	Line	
11	1	2	"meters-hour" should read "meters/hr".
12	4	14	"has" should read "gas"
17	j	3	"CH_y" should read "CH_4".
32	3	2	"5,400 cu. meters/hr." should read "5.4 cu. meters/hr." "6,000 cu. meters/hr." should read "6.0 cu. meters/hr."
42	1	1	"110°" should read "110°C"
42	9	8	Should read "products, which could be supplied by the chemical company at Wesseling."
44	5	3	"Doreaker" should read "Breaker"
49	1	10	"317°C" should read "417°C".
50	g	3	Delete "red earth (Fe_2O_3)".
51	1	1 - 4	"22.5 - 25.0 MV" equals 434° - 476°C. "25 MV" equals 476°C.
55	7	8	"19 MV" equals 374°C. "21.5 MV" equals 417°C.
56	1	8	"21.0 MV" equals 408°C.
58	1	3	"21.5 MV" equals 417°C.
58	1	9	"22.5 MV" equals 434°C.
60	8	3	"25.0 MV" equals 476°C.

ERRATA SHEET (Cont'd)

Page	Paragraph	Line	
60	9	5	"25.3 MV" equals 481°C.
61	1	6	"Valve" should read "value".
61	5	4	"from" should read "by".
64	q	1	"MV^3" should read "MV^2".
64	q	3	Column "Coal Paste" should read "35.0" not "35,000" m^3/hr., etc.
65	1	2	"outlet" should read "inlet".
72	v	7	Sentence should read, "Since initial operating difficulties were anticipated, the 700 atmosphere auxiliary press pumps could be used in place of the middle oil pumps."
73	3	5	"at is" should read "as it".
80	e	3	Sentence should read, "These should be from 0.10 to 0.11 in the prehydrogenator, and 0.075 in the 6434 stage."
80	f	2	"18.5 MV" equals 366°C.
80	f	4	"19 MV" equals 374°C. "21 MV" equals 408°C.
87	j	6	"0.75" should read "75 %".
92	i	6	"for" should read "of".
94	1	3 - 4	"1.34 tons/hr" and 0.42 tons/hr."
95	3	3	"Heating" should read "Heating Value".

ERRATA SHEET (Cont'd)

Page	Paragraph	Line	
95	3	3	"Heating" should read "Heating value"
106	1	1	"1-8" should read "1:8".
106	1	2	"5-7" should read "5 to 7 atm."
108	2	5	"D, K" should read "Dik"
111	1	6	"proprionic" should read "propionic"
117	3	2	"TK_4" should read "dry coal".
121	1	23	API max. is 52°, not 52°C.

THE WESSELING SYNTHETIC FUEL PLANT

SUMMARY

This report describes the inspection trip of the U. S. Naval Technical Mission in Europe to the Wesseling Synthetic Oil Plant, 17 March to 19 March 1945.

It describes the present condition of the plant and includes a detailed account of the processes used. There is also an operating summary, giving the yields produced during the period in which the plant operated and other facts of interest concerning its construction. There is also a product analysis.

May 1945

TABLE OF CONTENTS

Page

1. Introduction. 4
2. Sources of Information. 5
3. Brief History of Plant. 5
4. Present Condition of Plant. 7
5. Description of Process. 7
   - (a) Hydrogen Production. 8
     - I. Pintsch-Hillebrand - Water Gas Unit. 11
     - II. Producer Gas Unit. 15
     - III. Methane Cracking Unit. 20
     - IV. Water Gas Conversion Unit. 25
     - V. CO_2 Purification Unit. 29
     - VI. CO Purification Unit. 32
     - VII. Hydrogen Compressors and Recirculators. 36
   - (b) Hydrogenation Processes. 39
     - I. Coal Mill Unit. 39
     - II. High Pressure Hydrogenation. 46
     - III. Gas Phase Hydrogenation. 78
     - IV. Centrifuging Units. 84

TABLE OF CONTENTS (Cont'd.)

Page

(b) Hydrogenation Processes. (Cont'd.)

- V. Coking Unit. 88
- VI. Distillation Unit A. 98
- VII. Distillation Unit B. 101
- VIII. Alkaline Wash and Stabilization. 104
- IX. Hygas Processing. 106
- X. Sulfur Problem. 115
- XI. Raw and Finished Products. 118
- XII. Steels in the Hydrogenation Unit. 122

(c) Utility Processes. 129
- I. Power Plant. 129
- II. Treatment of Discharge Water 136

(d) Miscellaneous. 141
- I. Met and Not Met Guarnatees. 141
- II. Air Attacks and Counter Measures Taken. 143
- III. Air Attack of 18 July 1944. 146
- IV. List of Important Manufacturers of Machines and Equipment. 148
- V. Fuel Administration in the German Government. 154

THE WESSELING SYNTHETIC FUEL PLANT

1. Introduction.

(a) The Wesseling Synthetic Oil Plant is one of the most important plants in Germany for producing synthetic fuel by the hydrogenation of lignite. As such it was a target of prime importance to those investigators who are charged with fuel problems.

(b) The plant was visited on 17, 18, and 19 March 1945. The team representing the U. S. Naval Technical Mission in Europe were: Lieutenant Commander Lloyd H. Mulit, USNR, and Lieutenant Richard C. Aldrich, USNR. A CIOS team visited the plant the same time consisting of : Colonel J. A. Oriel and Major Tilley of the British Army and Messrs. Weir and Jones, U. S. civilians.

(c) The plant was so badly damaged in the air raid of 18 July 1944, that it was never put back into operation. Nearly all the workers were removed and only three men of the administrative staff remained behind and were available for questioning at the time of the visit. These men were:

Dr. Muller von Blumencron - Director.
Dr. Ernst Peuckert - Chief Operating Engineer.
Dr. Sustmann - Chief Chemist in the Analytical Laboratory.

The other important former members of the plant staff who had left were:

Dr. Meissner - Chief Chemist.
Herr Moll - Chief Engineer.
Dr. Miedelmann - Engineer charged with installing machinery.
Herr Neubaner - Assistant to Moll.
Herr Heinrich - Chief Civil Engineer - belonging to the Todt Organization.
Herr Dietsch - Responsible for moving machinery to new sites after the air raid of 18 July.

There were also thirty-five (35) Russian workmen left in the barracks, and a few plant guards.

2. Sources of Information.

(a) As previously mentioned, three of the principal men of the plant's administrative staff remained behind. They were able to give fairly complete information on the plant. However, they were not able to discuss the use of the products after they left Wesseling, as all such matters were kept highly secret by the German Army.

(b) The best source of information was a synopsis written by Dr. Peuckert, containing very complete information on the design, operation and yields of the plant. This most complete and detailed volume is now in the hands of CIOS. The U. S. Navy members were able to get a microfilm copy of this book which has been used as the basis of this report. Other facts were taken down from conversations with the above mentioned Germans.

3. Brief History of the Plant.

(a) The plant is owned by the Union Rheinischen Braunkohle Kraftstoff A. G. It is situated on the west bank of the Rhine, about one mile south of Wesseling, a village roughly half-way between Cologne and Bonn. This company consists of the principal lignite mine owners of the Rhine area (Knapsack). The plant was pushed by the German Government as an important part of the Four-year Plan.

(b) The company was founded on 27 January 1937. Plant construction started on 4 April 1938. The outbreak of war interrupted construction from 8 November 1939 to 1 February 1940. A few pieces of equipment remained to be installed but were all operating by the middle of 1943. These consisted of the last high-pressure stall, the water gas conversion system, the mud coking units and the second butane unit.

(c) The equipment was put on stream in the following order:

(1) Power House.
(2) Hydrogen Production.
(3) Hydrogenation.

3. Brief History of the Plant. (c) (Cont'd.)

The boiler house was ready 10 January 1941 and its electric power units were soon running and able to supply energy to the outside net-work.

(d) While waiting for the hydrogen stalls to be completed, a large quantity of middle oil for pasting was made by the distillation of Esthonian oil shale.

(e) The originally proposed gasoline de-hydrogenation unit and the alkylation unit were never completed due to the 19 July air raid. The methane cracking plant was added to the original design due to the demands of the high pressure hydrogenation unit.

(f) Fuel production began on 31 August 1941 and continued to increase steadily until 18 July 1944, when an air raid occurred that completely stopped production.

(g) Serious thought was given toward reconstructing the plant, but Dr. Geilenbarg, the man personally responsible to Hitler for the construction and rebuilding of hydrogenation plants, decided against it. Accordingly, the workmen were moved away to other jobs, as was some of the equipment. Just what the Germans intended to do to the remaining part of the plant is not clear, but Dr. von Blumencron states that he was ordered to stay behind when the German Army retreated at the specific order of Major General Erdmann.

(h) Some of the hydrogen compressors were to have been shipped to a secret place, known only as JACOB II, supposedly and underground installation in Pinch, Kosswig near Dresden. These were never moved.

(i) Documents were removed from the plant and sent to Fabrikant Projahn in Waldbrohl, fifty (50) kilometers from Wesseling. Essential parts of the compressors were hidden at Alster, west of Bonn, in a tube working plant.

4. Present Condition of the Plant.

(a) The plant is very badly wrecked. The great bombing attack of 18 July 1944 reduced its capacity to twenty (20) percent of the total production which was rebuilt to forty (40) percent when a small attack on 6 October completely shut it down. At that time. the German Government decided to abandon it.

(b) Nearly every building has been damaged to some extent, while some are completely destroyed. The gas holders are all smashed. The boiler house was not too badly damaged, but much of its equipment had been removed. The hydrogenation stalls are standing, but the pre-heaters are badly injured. The pipe lines for steam, water and product are wrecked. The laboratories are standing, though damaged, and their equipment had been removed. The compressor house was also hit, but many compressors can be used.

(c) It would be impossible to operate this plant without a very complete reconstruction and replacement of equipment.

5. Description of the Process.

(a) The Wesseling Plant works on the I. G. high pressure hydrogenation process, as shown in the accompanying flowsheet (Fig. 1). In this, heavy hydrocarbon molecules and hydrogen are combined to produce light hydrocarbons. The process uses very high pressures, seven hundred (700) atmospheres, and various catalysts to obtain these results. The transformation occurs in two or three steps depending on the final products; two for Diesel oil alone and three if motor gasoline and aviation gasoline are to be made.

(b) The first stage is known as the "sump" phase. Here coal is pulverized and mixed into a paste with heavy oil and catalyst. The heavy oil is either obtained from the outside, i.e. tar distillation, or is a recycle product from the process itself. The product from this sump phase is distilled into two cuts, middle oil and heavy oil. The average coal through-put in this stage is approximately twelve hundred (1200) tons per day. This coal upon hydrogenation yields about six hundred fifty (650) tons of

5. Description of the Process. (b) (Cont'd.)

middle oil, which is further processed in the gas phase. The heavy oil is recycled for pasting of coal in the sump phase.

(c) The gas phase operates at three hundred twenty-five (325) atmospheres pressure. The catalyst is in pellet form and is kept on screens in the reactor, as opposed to the sump phase catalyst which is injected in the paste. Three types of gas phase catalysts are used:

(1) Hydrogenating Catalyst.
(2) Cracking Catalyst.
(3) Catalyst combining both the above operations.

This phase of the process had a total capacity of about six hundred eighty (680) tons of oil in the first gas phase stage per day. The gasoline production of one hundred eighty (180) degrees centigrade E. P. averaged about three hundred ninety (390) tons/day. The final product from this process is high in aromatics, naphthenes and iso-paraffins, thus making it an extremely valuable fuel for aviation engines.

A. HYDROGEN PRODUCTION.

(a) Hydrogen is produced by two methods:

(1) The Fintsch Hillebrand system - yielding water gas by directly gasifying lignite briquettes.

(2) A methane cracking unit which cracks the residual methane and ethane from the hydrogenation into raw hydrogen.

(b) The products of the two above processes are converted to raw hydrogen over a conversion catalyst and are then purified by CO and CO_2 removal. To supply the endothermic heat of reaction required in the conversion process, fuel gas is manufactured in a producer gas generator. The CO from the final gas purification is also used in this gas stream.

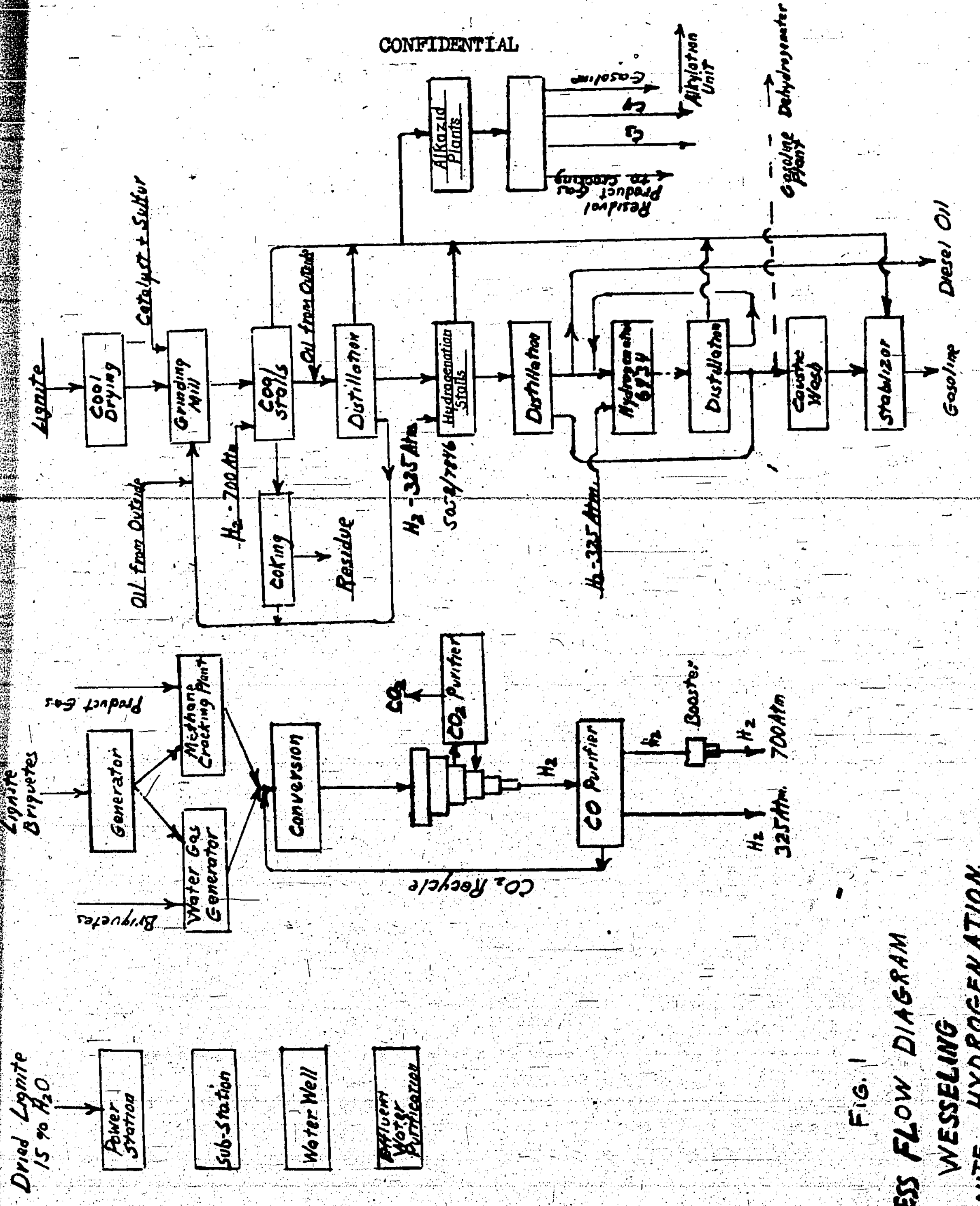
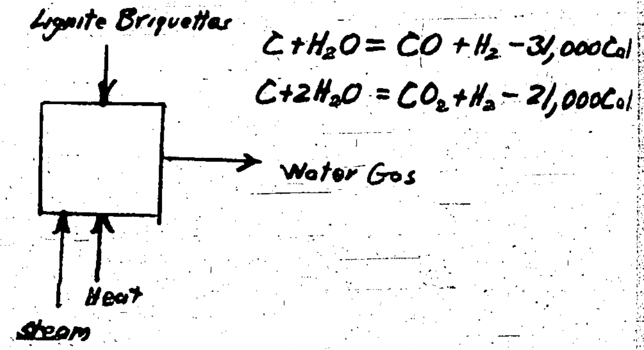


FIG. 1
PROCESS FLOW DIAGRAM
WESSELING
LIGNITE HYDROGENATION

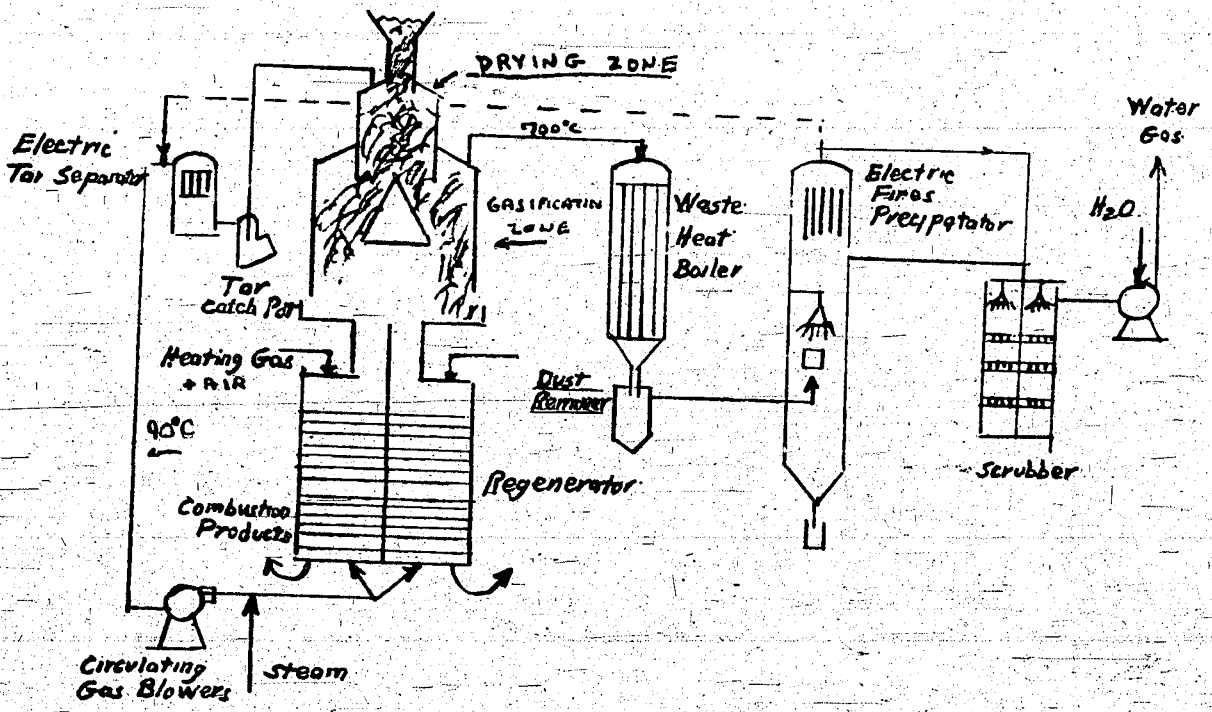
FIG. 2

PINTSCH-HILLEBRAND UNIT

Principal



Flow Sheet



I. PINTSCH-HILLEBRAND UNIT.

(a) The Wesseling Plant has eleven generators producing a total five thousand five hundred (5,500) cubic meters/hour of process gas. This is a fairly recently developed German process and is shown on the attached diagram (Fig. 2). It consists in destructively distilling the lignite briquettes by circulation of a superheated steam-water gas mixture. The gas used is part of the already distilled product, which after mixing with steam is passed over a regenerative type, fire brick lined heater, acting batchwise at fifteen (15) minute intervals. The general reaction is as follows:

$$C + H_2O = CO + H_2 \quad - \; 31,000 \text{ cal.}$$

$$C + 2H_2O = CO_2 + 2H_2 - \; 21,000 \text{ cal.}$$

The endothermic heat of reaction is supplied by burning fuel gas in the section of the regenerator not in use. The distillation of the briquettes is in two stages: a preheating or drying stage, and a gasification stage. Part of the recirculated gas and steam from the gasification zone passes upward through the freshly charged briquettes, driving off the moisture in the drying zone. All of the gas from the drying stage is used in recirculation as is also part of the gasification product. The former goes first through a tar catch pot, and then to an electric tar remover. This gas is always kept above the dew-point of its water content, i.e. ninety (90) degrees centigrade. On leaving this, it is joined by the second gas stream and enters the circulation blower. Steam is then injected, and the combined streams enter the regenerative heat exchanger where they are heated to one thousand three hundred (1,300) degrees centigrade.

(b) All valves controlling the circulating gas, heating gas, and air for combustion are located outside of the unit. The valves are hydraulically operated from a central control. In order to avoid burning-cycle exhaust gas from contaminating the product, a small quantity of circulating gas is allowed to leak continuously into the exhaust gas stream.

(c) The circulating gas and steam gives up all its sensible heat to the briquettes. The gasification product stream leaves

I. PINTSCH-HILLEBRAND UNIT (c) (Cont'd.)

The unit at seven hundred (700) degrees centigrade. It passes through a waste heat boiler, and a dust remover, before entering a water saturator which cools it down to seventy (70) degrees centigrade. It then enters an electric dust remover and is split, part going to the circulating stream as mentioned, and part to a subsequent water scrubber where it is further cooled to twenty (20) degrees centigrade. A final wash to remove very fine dust completes the process.

(d) The unit has worked very well during its three years of operation. The only point of interest is the need for briquettes of constant quality, as variation in the same cause trouble. They should all have the same compression in manufacture, which should give them a specific gravity of 1.23. The water content should remain between 13.5 and 14 percent.

(e) Since the main cause for the Wesseling Plant not meeting its guaranteed capacity was in its gas production, means were constantly being taken to remedy the lack of briquette consistency.

(f) The capacity of a generator is fixed by theporosity of the briquette bed. By good standardization of the briquettes, four thousand (4,000) cubic meters/hour of circulating gas and three thousand five hundred (3,500) kilogram/hour of steam can be handled. From this is produced nine thousand four hundred (9,400) cubic meters/hour of water gas which is split into five thousand four hundred cubic meters/hour of production and four thousand (4,000) cubic meters/hour of circulating gas. If the circulating gas stream is substituted by hydrogenation tail gas, the regenerator section of the furnace can be used as a cracking furnace, as shown in the diagram (Fig.3). Hence, four hundred (400) cubic meters of tail gas gives one thousand (1,000) cubic meters of cracked gas. Furthermore, the tail gas acts just as well as the circulating has-stream mixture for preheating briquettes, provided the quantity of gas circulated or injected is reduced from four thousand (4,000) to three thousand (3,000) cubic meters/hour. This results in an increase of total gas produced from five thousand four hundred (5,400) to six thousand four hundred (6,400) cubic meters/hour.

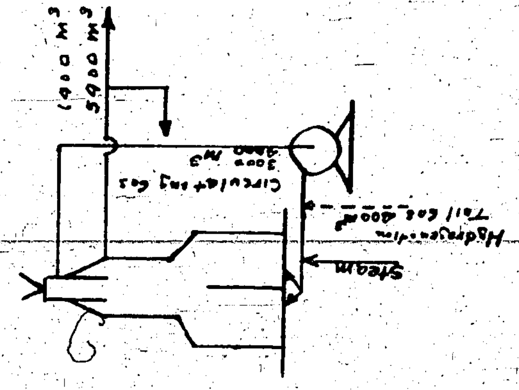


FIG. 3.
Cracking of Hydrogenation Tail Gas
In the Preheater of the Generator

Production and Circulating Gas
Solid Lines — Normal operation
Broken Lines — Addition of Hydrog. Tail Gas

I. PINTSCH-HILLEBRAND UNIT. (Cont'd.)

(g) The regenerator is well fitted for cracking, as its temperature is one thousand three hundred (1,300) degrees centigrade and sufficient steam is on hand in the gas itself so that other steam injection is not necessary. Naturally, more heat has to be added to the regenerator than before in order to account for the required heat of cracking. It is theoretically possible to substitute hydrogenation tail gas for all of the circulating gas, but this was actually only approximated. The increase in gas yield is one thousand (1,000) cubic meters/hour which raises the total output of the Pintsch-Hillebrand units up to ten thousand (10,000) cubic meters/hour.

(h) As previously stated the main operating difficulties of the unit were in not getting briquettes of constant quality. When normal briquettes were obtained, the unit ran well.

(i) Another source of trouble was in the tar recovery. Calcium carbonate deposits were also troublesome, but by proper operation of the water treating plant this difficulty was removed.

(j) In conclusion, air raid damage which shut down the heating gas plant, caused the water gas unit to be used as heating gas unit. Due to its higher heating value, two thousand two hundred (2,200) as opposed to one thousand four hundred fifty (1,450) W. E., the generators were only run at sixty (60) percent full load capacity. The operation under these conditions was satisfactory.

OPERATING DATA ON GENERATOR - FULL CAPACITY

Lignite required
Total steam required 3,000 Kg/h
4,200 Kg/h
of which 60% was steam added
40% from saturation of circulation gas
Fuel gas required 4,4 Mil. W.E./h
Gas produced 3,000 nm3/h (H_u1460 W.E.)
Circulating gas 5,400 nm3/h
4,000 nm3/h
of which 70 - 90% preheat gas
30 - 10% product

I. PINTSCH-HILLEBRAND UNIT. (j) (Cont'd.)

TEMPERATURES

Circulating gas before heat accumulator
Circulating gas after heat accumulator
Distilled gas leaving chamber
Distilled gas leaving waste heat boiler

II. PRODUCER GAS UNIT.

(a) The plant is equipped with eighteen generators producing three thousand (3,000) cubic meters/hour each. In these units lignite briquettes are first incompletely oxided to a mixture of CO_2 and CO by an air blowing (Fig. 4). The completion of the reaction to produce water gas or CO is then accomplished by injecting either steam or CO_2 and carbon. The reactions are as follows:

$$\begin{aligned} C + \tfrac{1}{2}O_2 &= CO + 26{,}000 \text{ Cal.} \\ C + O_2 &= CO_2 + 94{,}000 \text{ Cal.} \\ C + H_2O &= CO + H_2 - 31{,}000 \text{ Cal.} \\ C + CO_2 &= 2CO - 41{,}000 \text{ Cal.} \end{aligned}$$

(b) The resulting gas is a mixture of CO_2, CO, H_2 and N_2 with a lower heating value of one thousand four hundred fifty (1,450) heat units.

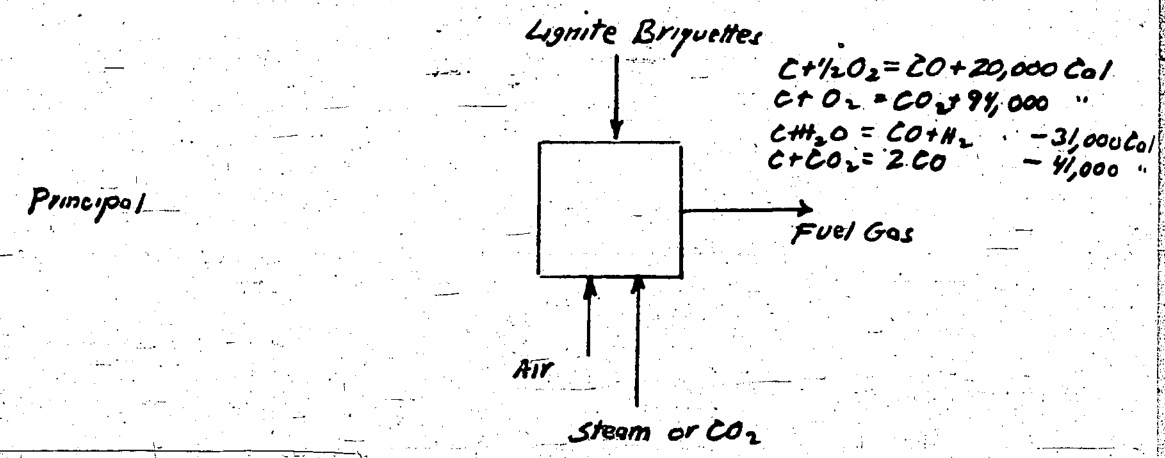
(c) The gas generator has both a preheating and distillation section. Its diameter is three (3) meters.

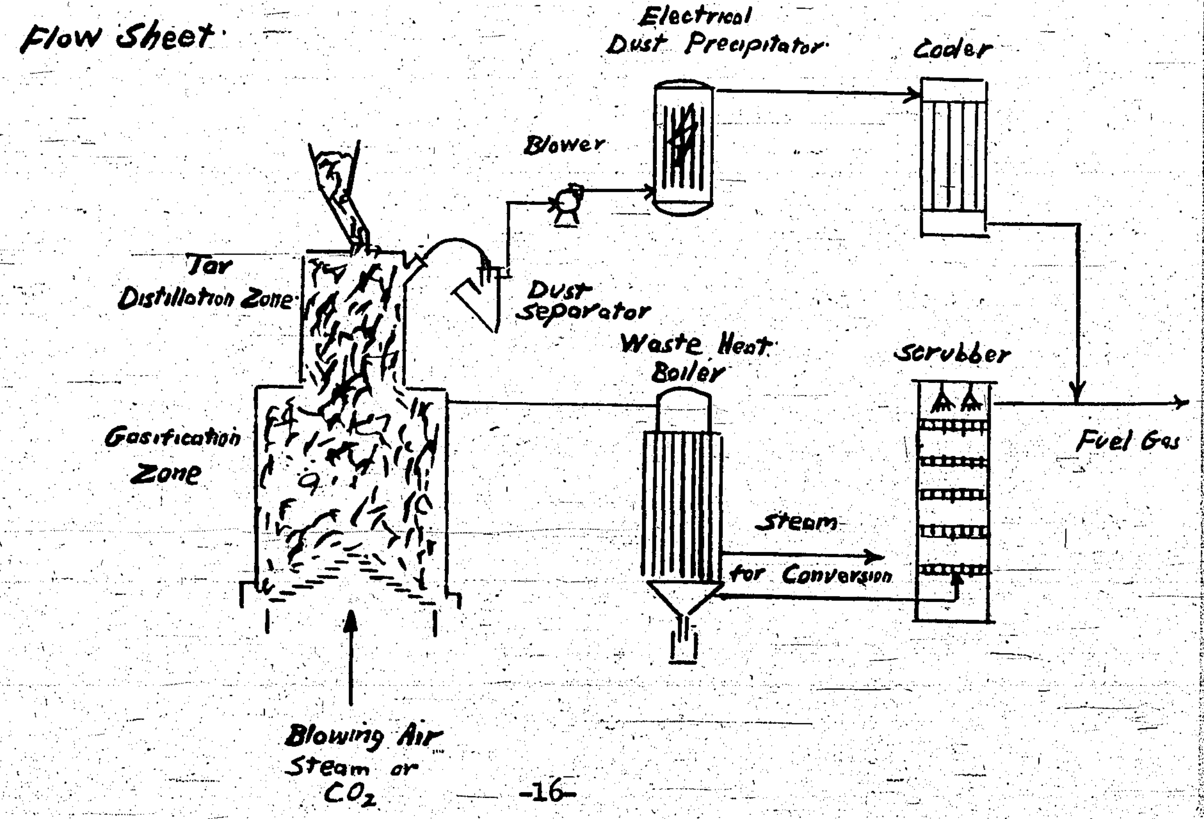
(d) The gas quantities from preheating and distillation are equal. They are cleaned and cooled in separate units, and then as heating gas are either stored in a gas holder or used directly.

(e) The blast air is saturated with water vapor or blended with CO_2 to maintain a proper outlet temperatures as follows:

with steam - 750° C
with CO - 650° C

FIG. 4
Fuel Gas Unit





II. PRODUCER GAS UNIT. (Cont'd.)

(f) The gas quantity from the preheating stage is the result of properly drying the briquettes. The outlet temperature of the preheat gas is around one hundred (100) degrees centigrade. This gas stream passes over a dust remover, an electric oil filter and a tubular cooler. The second cooler, planned for in the original design, was omitted to save material, and results in an oil loss in the outlet gas of over one (1) gram/cubic meter. The gas leaving the idstillation zone is passed through a waste heat boiler and a scrubber before joining the preheater gas stream.

(g) As orininally planned, this unit was to have been small. Due both to an increase in the total hydrogen plant, size, and also to the diversion of propane and butane from a heating gas to crack gas feed stock, the fuel gas unit had to be enlarged. This was also due to the fact that the gas resistance through the generator was higher than anticipated, because of the clogging up of the ashes in the lower part of the generator, through which the blowing air had to pass.

(h) This clogging of the ashes was greatly improved by substituting CO_2 for H_2O in the blowing air mixture. The former was available in large quantities from the final gas washing step

(i) In this way, the CO_2 was converted to CO and H_2 while the steam formerly used was available for other uses. The ash clogging was greatly reduced with its resulting pressure drop and the capacity of the whole unit greatly increased.

(j) The analysis of the heating gas was altered by the CO_2 substitution but its heating value did not change.

Gas Analysis	CO_2	Heavy Hydrocarbons	CO	H_2	CHy	N_2	Hu
With H_2O addition	4.4	0.3	31.5	15.9	1.9	45.4	1440
With CO_2 addition	6.3	0.3	38.6	8.3	1.6	45.1	1430

(k) From an operational view point, the substitution of CO_2 was as follows: The capacity of the unit was raised thirty-five (35) percent, that is up to eighteen thousand (18,000) cubic meters/hour. As mentioned, the hydrogenation tail gas become avail-

II. PRODUCER GAS UNIT. (k) (Cont'd.)

able for cracking, hence producing both more hydrogen, C_3 and C_4. This was equivalent to twenty thousand (20,000) tons/year of aviation gasoline on a lignite basis and thirty-five thousand (35,000) tons/year on an oil basis.

(l) The steam from the waste heat boiler was made available to the conversion unit at 2.5 atmospheres pressure, and was equivalent to sixty thousand (60,000) cubic meters/hour of heating gas or six (6) tons/hour of steam.

(m) Firthermore, the finished heating gas outlet temperature can be dropped from seven hundred seventy (770) degrees to six hundred seventy (670) degrees centigrade, resulting in a considerable saving of heat and fuel briquettes. A further saving occurs in the use of CO_2 to make CC and H_2. Each kilogram of briquettes produces:

With H_2O injection - 2.4 cu. meters heating gas
With CO_2 injection - 2.55 cu. meters heating gas

(n) This saving of briquettes is equivalent to sixty thousand (60,000) cubic meters of gas production, or twenty-five (25) tons per hour of briquettes.

(o) The dust and oil content of the heating gas is ten (10) milligrams of dust/cubic meters and one hundred fifty (150) milligrams of oil/cubic meters. Such quantities can cause trouble in the gas holders.

(p) After storage, the quantities drop to three (3) milligrams of dust and twenty (20) milligrams of oil/cubic meter gas, which indicates the extent of material deposits that can occur. This could be avoided by including the second oil filter as originally planned, and a fine dust separator.

(q) Another operating difficulty was the stopping up of the bottom baffles in the generator. After two years, these all had to be replaced.

(r) This was caused by both the corroding and the eroding influence of the ashes. The composition of the metals used also

II. PRODUCER GAS UNIT. (r) (Cont'd.)

played an important part in the trouble. The main point to be watched is keeping the temperature of the ashes low. It is also helped by hardening the steel, through heating to nine hundred (900) degrees centigrade, quenching followed by reheating to five hundred fifty (550) degrees centigrade and then slowly cooling in air.

OPERATING SUMMARY

Quantities	1943	1944
Heating gas production	48,000 m3/hr	60,000 m3/hr
- Average per generator	2,815 m3/hr	3,600 m3/hr

UNIT QUANTITIES

Heating gas/briquettes	2,480 m3/ton	2,580 m3/ton
Tar/Briquette	4.8 %	4.8 %
Heating value of gas	1,444 WE/m3	1,430 WE/m3

TEMPERATURES

Distilled gas leaving saturator	70°
Pre-heater gas leaving preheater	90°

ANALYSES OF DISTILLED AND PREHEATER GAS

	CO_2	Heavy H. C.	CO	H_2	CH_4	N_2
Distilled gas	14 %		28%	56%	1 %	1 %
Preheater gas	17.5%	0.8%	26%	48%	6.5%	1.2%

Dust content in water gas: 1 - 2 mg/nm^3
Carbon content in ash : 40 - 50%

II. PRODUCER GAS UNIT. (r) (Cont'd.)

OTHER RESULTS

WATER GAS PRODUCTION BY MEANS OF INCREASE GENERATOR CAPACITY

1943	1944
48,600 nm^3/h	52,000 nm^3/h
4,950 nm^3/h	5,100 nm^3/h

UNIT QUANTITIES

	1943	1944
Water gas production/briquette	1,938 nm^3/kg	1,900 nm^3/kg
Heating gas required/water gas	828 WE/nm^3	825 WE/nm^3
Tar recovery	4.8%	4.9%
Outside steam/water gas	0,126 kg/nm^3	0,125 kg/nm^3
$CO + H_2$ in water gas	83.5%	84.%

III. METHANE CRACKING UNIT.

(a) This unit has four furnaces, each with sixty-six (66) catalyst tubes. In this unit the tail gas from the hydrogenation process is cracked in the presence of steam to CO_2, CO, and H_2 as follows:

$$CH_4 + H_2O \quad CO + 3H_2 - 49,500 \text{ Cal.}$$
$$CH_4 + 2H_2O \quad CO_2 + 4H_2 - 39,500 \text{ Cal.}$$

(b) The process at Wesseling is one using catalysts at six hundred (600) to seven hundred (700) degrees centigrade, as opposed to the other well known thermal cracking process operating at one thousand (1,000) degrees centigrade. The former was developed by I. G. at Ludwigshaven. The Wesseling process is shown on the attached flow diagram (Fig. 5).

(c) The catalyst used is of a nickel base and therefore sensitive to sulfur poisoning. Hence the feed gas must be very free from sulfur.

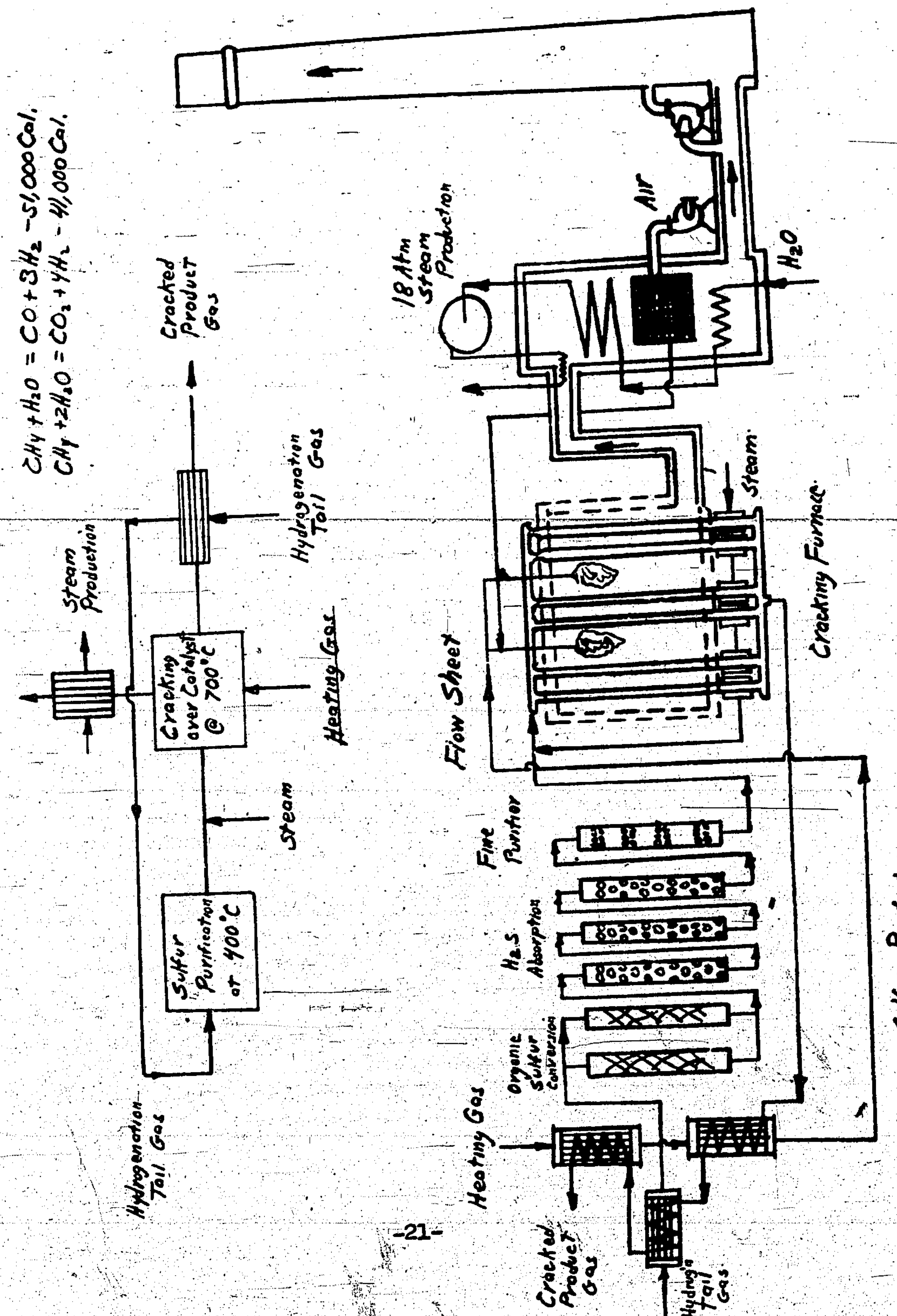


FIG. 5 Hydrogenation Tail Gas Cracking

$$CH_4 + H_2O = CO + 3H_2 - 51,000 \text{ Cal.}$$
$$CH_4 + 2H_2O = CO_2 + 4H_2 - 41,000 \text{ Cal.}$$

(d) The reaction is endothermic and therefore requires heat other than that obtained by heat exchange. Hence the process requires a fairly complete sulfur removal unit and large heat exchangers as well as the furnace. The gas is passed first through three exchangers heated against the finished product at about four hundred twenty (420) degrees centigrade. It then enters a catalyst chamber where the organic sulfur is converted to H_2S over an Fe_2O_3 catalyst. Next, it enters a chamber containing ZnO which removes the H_2S, still at the same temperature of four hundred twenty (420) degrees centigrade. Finally it enters a "fine sulfur" removal chamber having alternate beds of Fe_2O_3 and ZnO.

(e) Steam amounting to 1.2 kilograms/cubic meter of feed is injected into the gas steam, of which 0.5 kilograms is for conversion and 0.7 kilograms maintains equilibrium. The gas now enters the cracking catalyst chamber where a temperature of six hundred fifty (650) to seven hundred (700) degrees centigrade is maintained by external heating of the catalyst tubes. The exhaust heat from the burners is used to produce steam and to preheat the combustion air.

(f) The final cracked product has a CH_4 content of 1 - 2 percent. It leaves the unit in giving up its sensible heat to the incoming gases through the exchangers.

(g) In order to insure the complete conversion of the methane, and to maintain the proper cracking catalyst life, the sulfur content must be below five (5) milligrams/cubic meter. The tail gas normally contains fifty (50) to one hundred (100) milligrams/cubic meter of H_2S and fifty (50) milligrams/cubic meter of organic sulfur. The Bamag unit, using first Fe_2O_3 for the organic sulfur, and then ZnO for the $H_2S$is insufficient in scope, as it only reduces the sulfur content to ten (10) to twenty (20) milligrams/cubic meter.

(h) After many experiments, the present "fine sulfur" removal system was devised using spent Nickel catalyst from the cracking chamber at four hundred (400) degrees centigrade. This reduces the final sulfur content to the operating requirements.

(i) The waste heat boiler was originally fed with water fromthe Permutit water purifier. Due to scale formation on the tubes, the water source was changed to condensate from the distilling unit. This water had to be filtered, as well as given a further treatment by injecting NaOH to neutralize the free oxygen and CO_2 therein.

(j) Through the above improvements, as well as adding an economizer to the unit, it was possible to equal the quantity of 3.5 atmosphere steam with an equal quantity of eighteen (18) atmosphere steam.

(k) Another cause of operating trouble on the cracking unit was the dust and oil content in the heating gas. As already mentioned in describing that product, its dust content was ten (10) grams/cubic meter and its oil content one hundred fifty (150) grams/cubic meter. This led to fouling of the feed gas preheater tubes, requiring quarterly cleaning, lasting one week with a corresponding loss in production.

(l) The cost of producing water gas and cracked gas are almost the same. The water gas costs 2.82 Pfg/cubic meter and the cracked gas 2.60 Pfg/cubic meter when the hydrogenation tail gas has a heating value of 0.45 Pfg/1000 W.E.

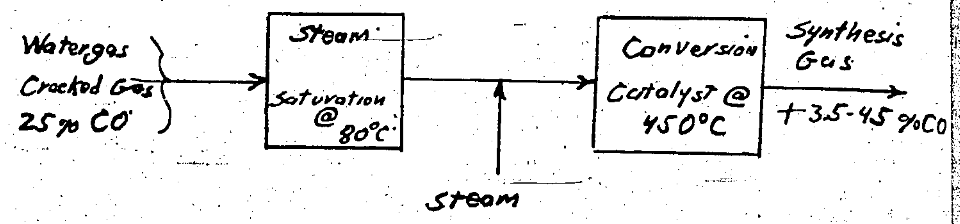
	Water Gas	Cracked Gas
Raw material and energy	16.15 R.M./1000cu.m.	21.74 R.M./1000cu.m.
Credit as by-product	H (2.27)	- (2.23)
Operating cost	3.33	1.66
Fixed charges	7.64	3.75
Other charges	3.11	1.07
	28.16 R.M./1000cu.m.	25.99 R.M./1000cu.m.

OPERATING RESULTS

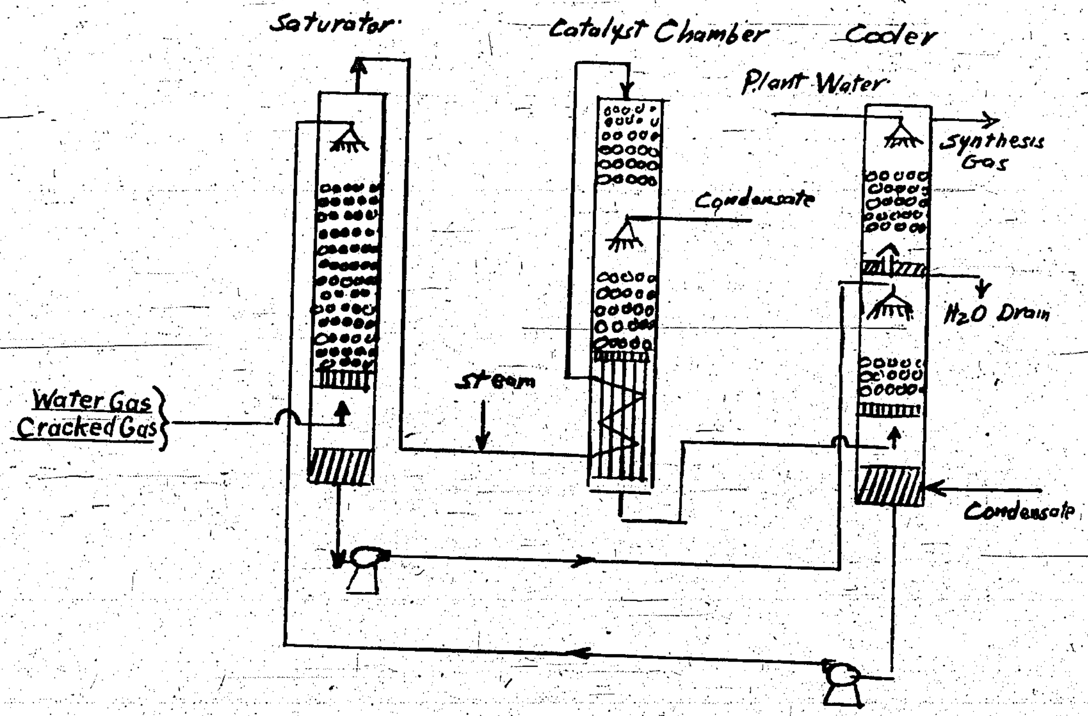
	1943	1944
Cracked gas produced	33,600 cu.meter/hr	40,000 cu.meter/hr

FIG. 6
Water Gas Conversion

Principal



Flow Sheet



III. METHANE CRACKING UNIT. (1) (Cont'd.)

UNIT FIGURES

	1943	1944
Cracked gas/tail gas	2,717	2,400
Heating gas/cracked gas	902 W.E./cu.m.	850 W.E./cu.m.
3.5 Atm. steam req'd/cracked gas	0.554 Kg/cu.m.	0.550 Kg/cu.m.
18 Atm. steam req'd/cracked gas	0.320 Kg/cu.m.	0.450 Kg/cu.m.
CH_4 in cracked gas	2.7 %	1.5 %

IV. WATER GAS CONVERSION UNIT.

(a) This unit consists of fourteen (14) catalyst chambers for each seven thousand (7,000) cubic meters of gas processed and three (3) towers for saturating the incoming and cooling the outgoing gas, as shown in the diagram (Fig. 6).

(b) The CO from the water gas and cracked methane is converted to hydrogen in the presence of steam as follows:

$$CO + H_2O = CO_2 + H_2 + 10{,}000 \text{ Cal.}$$

(c) The reaction occurs at four hundred fifty (450) degrees centigrade over an iron catalyst in the presence of steam. It is strongly exothermic and hence requires no heat.

(d) The steam is either used to saturate the incoming gas or enters the reactor directly. At the same time, a small quantity of condensate is injected over the catalyst to regulate its temperature.

(e) The saturation step takes place in the saturator tower where hot water enters and is sprinkled through the gas steam. The water is heated by the outgoing gas and is circulated between the saturator and the cooler. The highest temperature possible is obtained in this water and usually reaches eighty (80) degrees centigrade.

IV. WATER GAS CONVERSION UNIT. (Cont'd.)

(f) The gas is saturated as much as possible with water, but a limit is reached due to resistance through the catalyst chamber. In order to save steam, a lower catalyst temperature is used since the

$$CO + H_2O \rightleftarrows CO_2 + H_2$$

equilibrium cells for a definite water quantity. The temperature used at Wesseling is four hundred twenty (420) degrees centigrade.

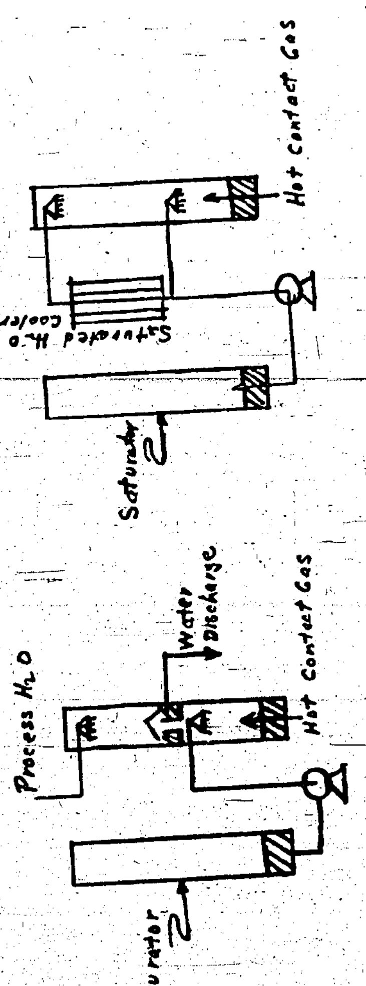
(g) The capacity of the unit was thirty (30) percent over the guarantee. Further increase was impossible due to the limitation of the water gas blower outlet pressure and also the size of the saturator.

(h) In order to overcome this bottleneck, the cracked gas which formerly joined the water gas stream before the saturator, was fed directly into one of the conversion furnaces. This led to an increase in capacity because the cracking gas has a naturally higher outlet pressure, than the water gas blower, thereby reducing the load on the saturator and increasing the throughout by fifteen (15) percent. This also lowers the CO content of the feed to the CO purifier from 4.5 percent to 3.5 percent and reduces the load on the latter. It should be emphasized that as long as the cracked gas has a CO content of twelve (12) percent, the process will run on its own heat energy.

(i) Because the sulfur content of the Rhine lignite was so low, no sulfur purification was attempted on the feed to the conversion step. The sulfur content of the water gas was four hundred (400) milligrams/cubic meter (three hundred (300) milligrams H_2S and one hundred (100) milligrams organic sulfur) only slightly above the amount of purified gas in other plants. The conversion catalyst was not affected by this sulfur and ran well for three (3) years. However, the product or synthesis gas would often contain high quantities of sulfur due to the releasing of the absorbed material in the conversion catalyst bed.

(j) The reaction is

$$FeS + H_2O \rightleftarrows Fe_2O_3 + H_2S$$



Normal Process

Process after Installation of the Cooler

FIG. 7

c

IV. WATER GAS CONVERSION UNIT. (j) (Cont'd.)

and varies with equilibrium conditions, especially the ratio of cracked gas to water gas in the feed. Hence a condition, dangerous to the hydrogenation catalyst, would occur often. Another important feature in the conversion catalytic process is the need for clean injection water, in order not to contaminate the catalyst. The minimum quantities are five (5) milligrams/liter of condensate residue and three (3) milligrams/liter of chlorine. A definite contamination was found on the top of the catalyst after three (3) years operation, even after meeting the above requirements. The deposition of solids comes only from entrainment in the saturation water and continues to build up with the addition of condensate. This addition is necessary as the saturation requires more water than is condensed in the cooler tower.

(k) By introducing a tubular cooler for the circulating saturation water, the required quantity of water injection can be reduced and an improvement in the saturator efficiency obtained. This is shown on the attached diagram (Fig. 7). In the previous arrangement, the hot exit gas was first cooled by sixty (60) degrees centigrade water and then given an after cooling of plant water. Hence the gas carried a large part of the cooling water with it which was lost.

(l) This loss was greatly reduced when the saturation water was indirectly cooled. The re-cycled water also contained less solids. It was found after a test of several weeks that the addition of fresh condensate was unnecessary and that the solid entrainment in the circulating saturation water fell below that of the plant condensate.

(m) If part of the circulating water is diverted to the lower section of the column, a two (2) degree higher saturation temperature is possible (eighthy-one(81) instead of seventy-nine (79) degrees centigrade) which results in greater saturation of the gas and a considerable saving in steam requirements.

IV. WATER GAS CONVERSION UNIT. (m) (Cont'd.)

OPERATING RESULTS

	1943	1944
Total Catalyst Gas Feed	103,300 cu. met/hr	120,000 cu.met/hr
Avg. chamber load	7,000 cu. met/hr	7,200 cu.met/hr
CO in entering gas	24.9	26.4
CO in finished exit gas	4.1	4.4
Finished exit gas/entering gas	1.202 m^3/m^3	1.205 m^3/m^3
Steam added/exit gas	0.187 Kg/cu.meter	0.220 Kg/cu.meter
Steam in Methane cracked gas	0.118 Kg./cu.meter	0.100 Kg/cu.meter
Vaporized saturation water	0.274 Kg/cu.meter	0.250 Kg/cu.meter
Condensate added	0.015 Kg/cu.meter	0.020 Kg/cu.meter
	0.594 Kg/cu.meter	0.590 Kg/cu.meter

V. CO_2 PURIFICATION

(a) The unit consists of eight scrubber columns, each two (2) meters in diameter and twenty (20) meters high. In this purification step, the CO_2 content of the synthesis gas is stripped down to a trace. The operation is carried out at twenty-eight (28) atmospheres pressure.

(b) Simultaneously with the CO_2 purification, the remaining H_2S is also removed. The absorbed gases are released from the water by expansion. The quantity of wash water is fixed by the required purity from CO_2 in the synthesis gas and by the allowable combustible gas quantity in the CO_2. The process is carried out in such a way that the CO_2 content of the washed hydrogen is from 1.5 to two (2) percent, while the $CO + H_2$ content in this CO_2 is never above 7.5 percent. The water containing the absorbed gases is expanded in Pelton turbines which drive the fresh water pumps. The process is shown on the attached flow sheet (Fig. 8).

(c) The expansion releases eighty (80) percent of the absorbed CO_2, the inert gases and the largest part of the H_2S. The expanded CO_2 normally contains six (6) to six and one-half (6.5)

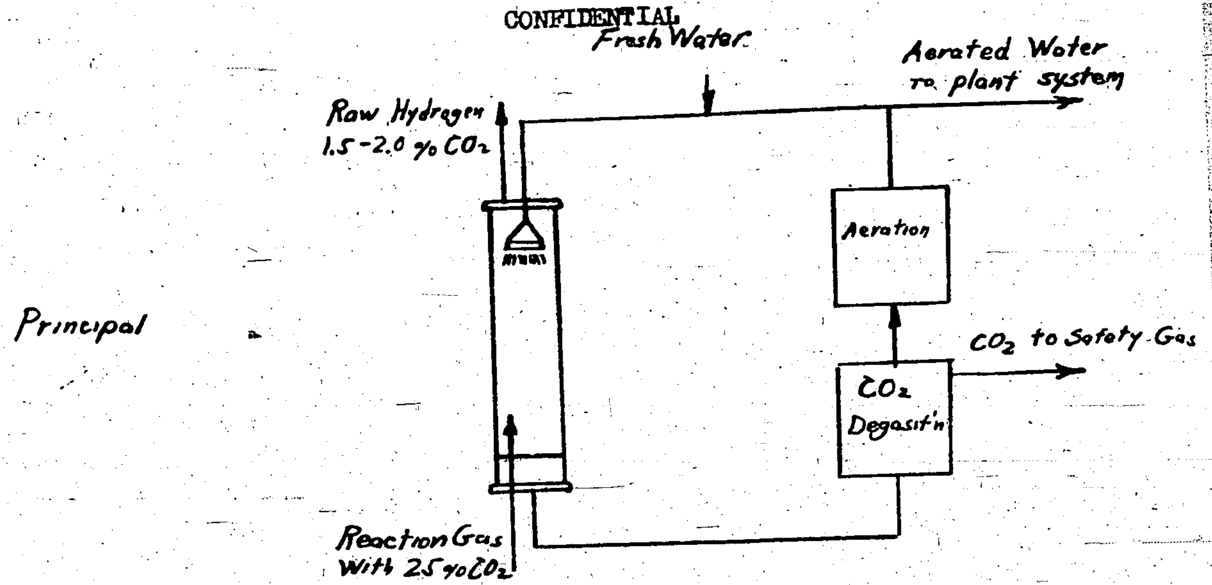
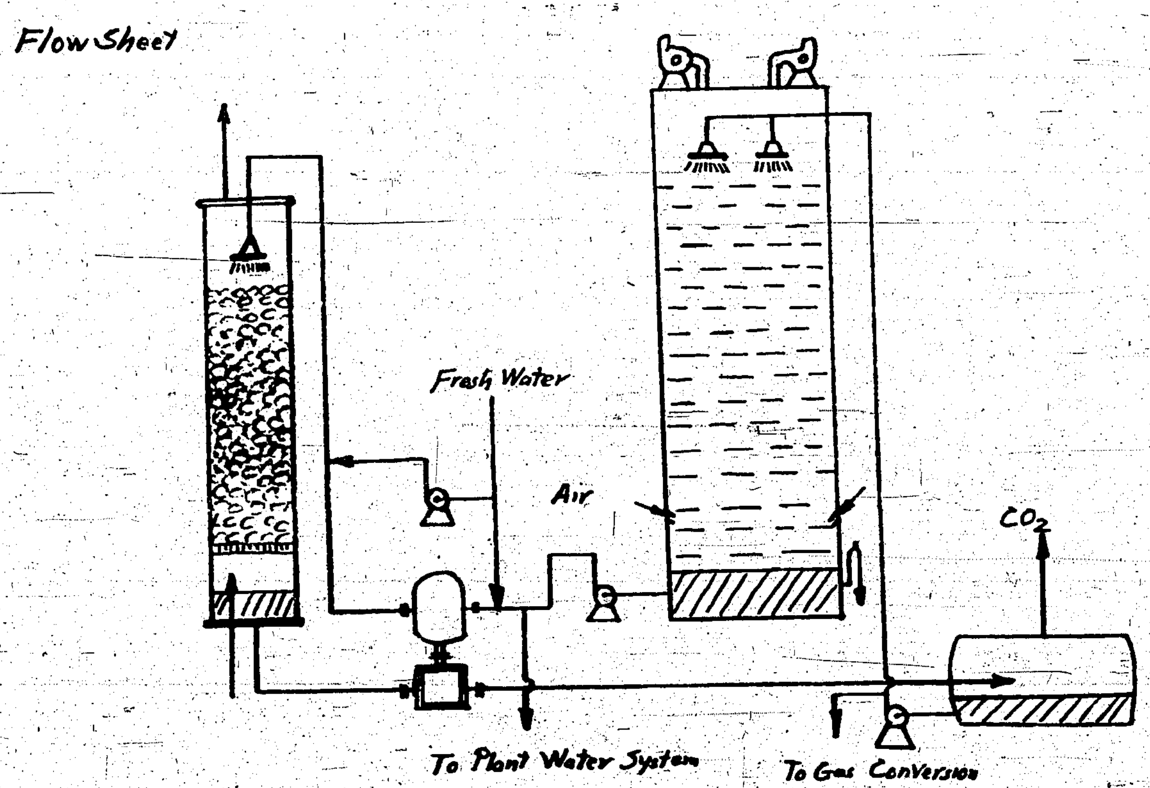


FIG. 8
CO_2 Removal



percent of H_2 and CO, and approximately five hundred (500) milligrams/cubic meter of H_2S.

(d) The expanded wash water is aerated to remove the remaining CO_2 and H_2S, and used again, partly for CO_2 washing and part returning to the plant water system.

(e) The re-using of aerated water in the CO_2 removal, serves to introduce the proper amount of O_2 in the raw hydrogen, as it tends to maintain the proper copper salt balance in the cupric caustic wash to follow in CO removal.

(f) Since the plant water, coming from wells, has only two (2) to three (3) milligrams/cubic meter of O_2, which is insufficient, the introduction of the ten (10) milligrams/cubic meter of O_2 is important. The plant well water and the re-used aerated water are mixed 1 : 1.

(g) The CO_2 wash towers and the H_2O regeneration gave adequate capacity for both the gas producing units and the methane cracking plant. This pre-supposes that no operating difficulties exist in thw wash towers. These can be clogged up by algae growth on the packing rings. Due to this, the capacity of the CO_2 wash towers have sometimes been reduced to half.

(h) In order to maintain production, definite steps had to be taken. Since the central water pumping station could not assist, the algae had to be removed from each wash tower individually. This was done by circulating chlorine water in each tower.

(i) The towers were filled with water at twenty-five (25) to thirty (30) degrees centigrade. The chlorine was injected in batches every two (2) hours and circulated. After each injection, the chlorine content ran from one hundred (100) to two hundred (200) milligrams/liter. Each washing required fifty (50) to eighty (80) kilograms of chlorine and took two (2) days to complete. The algae were completely removed and the towers were able to run as if new.

(j) Another possibility for improving operation is in using larger tower packing. By increasing the size of the ceramic

V. CO_2 PURIFICATION. (c) (Cont'd.)

rings from sixty (60) millimeters to eighty (80) millimeters size, the capacity of the unit was increased fifteen (15) percent.

OPERATING RESULTS

HOURLY QUANTITIES

	1943	1944
Synthesis gas flow	103,300 cu. meters/hr	120,000 cu.meters/hr
Avg. wash tower load	15,700 cu.meters /hr	17,000 cu.meters/hr
CO_2 REMOVAL		
CO_2 - gas absorbed in	23,600 cu.meters/hr	26,000 cu.meters/hr
H_2O solution	5,400	6,000
CO_2 in synthesis gas	27.4 %	26.8 %
CO_2 in washed H_2	1.8 %	1.8 %
$CO + H_2$ in CO_2	7.1 %	7.1 %
WASH WATER		
Water used	4,520 cu.meters/hr	4,800 cu.meters/hr
Fresh Plant Water	55 %	55 %
Re-circulated Water	45 %	45 %
Water needed/100 cu.meters of	44 m^3	42 m^3
Synthesis gas water temperature	9-18°C.	8.5-15°C.

VI. CO PURIFICATION.

(a) The CO purification unit consists of seven wash towers and equipment. In this step, the raw synthesis gas is stripped of the remaining CO and CO_2 by absorption at three hundred twenty-five (325) atmospheres (Fig. 9). The absorption medium is an ammoniacal copper solution. After use, it is regenerated by a mild heating both at atmosphere and sub-atmosphere pressures. The gases removed are next washed to remove NH_3 entrainment and used in the gas conversion process. The expanded gas energy in

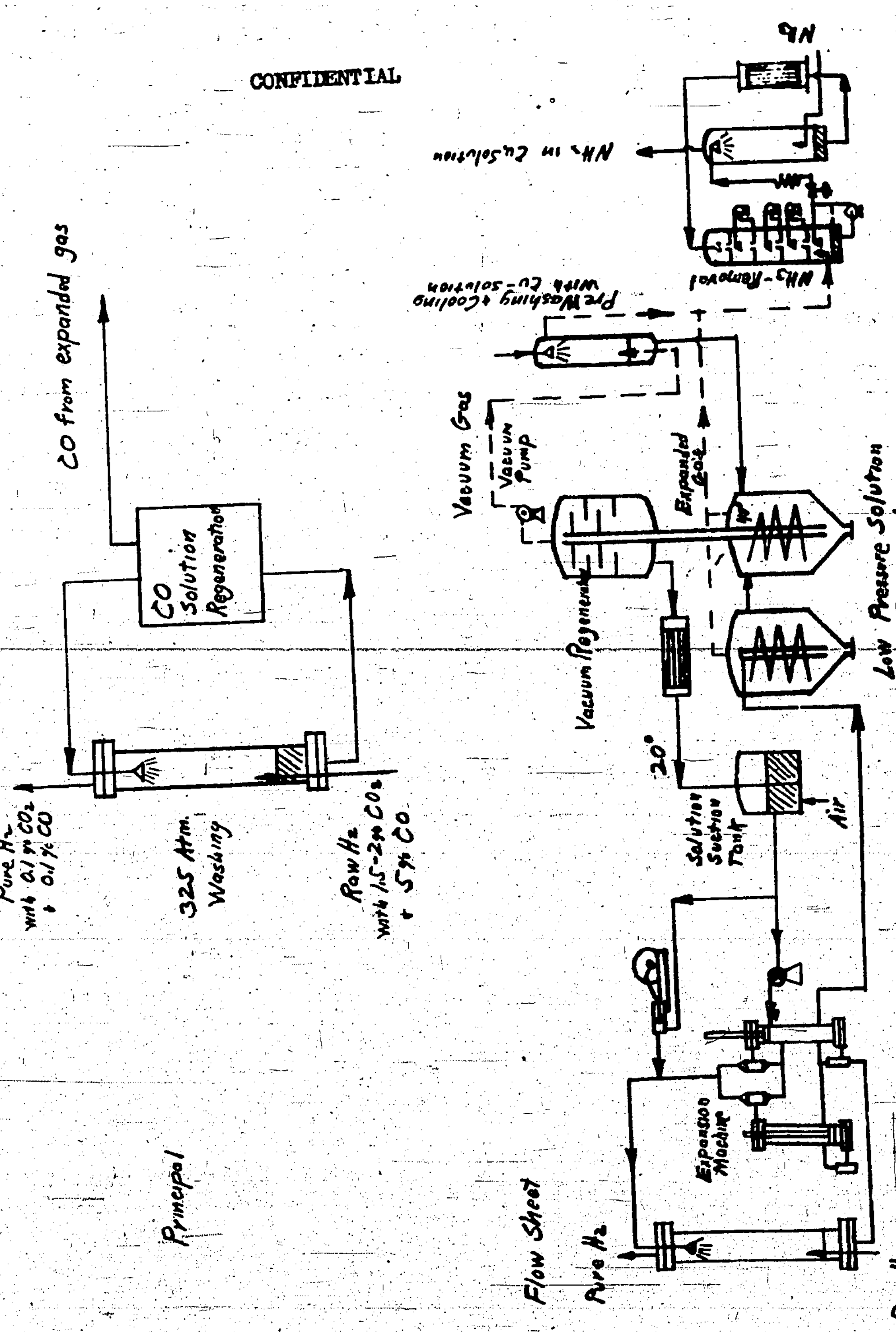
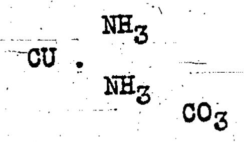


FIG. 9
Schematic Flow of CO Removal

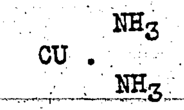
VI. CO PURIFICATION. (a) (Cont'd.)

the stripped gases is recovered one hundred (100) percent by driving the pumps which handle the fresh copper solution.

(b) The copper solution contains CU. NH_3 and CO_2. In its operating state it is as follows:



$$CU \cdot \begin{matrix} NH_3 \\ NH_3 \end{matrix} \; CO_3$$



$$CU \cdot \begin{matrix} NH_3 \\ NH_3 \end{matrix}$$

It is cuprous tetramine carbonate. Hence one (1) molecule of copoer can absorb one (1) molecule of CO. This absorption is increased as the ammonia concentration is increased. However, a limit of the allowable NH_3 is reached because of the base with which ammonium bicarbonate drops out of solution.

(c) On heating, the CO acts as a reducer, leaving a solution of metallic copper. In order to arrest this tendency a definite quantity of cupric copper must be on hand. However, since cupric copper can not absorb CO, its content must be minimized, and the rest made cuprous copper. The optimum solution has the following specifications:

Sp.gr. @ 20°C.	1.15	
Cuprous salt content	12	mols/10 liters = 6.6 %
Cupric salt content	2	mols/10 liters = 1.1
NH_3	0.480	mols/10 liters = 7.1
CO_2	0.240	mols/10 liters = 9.2

(d) Two-thirds (2/3) of the ammonia is in the complex, the rest is tied up with the carbon dioxide. The cuprous containing complex is colorless as the blue color of copper solution is due to cupric rather than cuprous salts. Theoretically, this solution can absorb twenty-seven (27) times its volume of CO but in practice only two-thirds (2/3) of this figure is obtained.

VI. CO PURIFICATION. (Cont'd.)

(e) The old method of making the solution entailed considerable loss of NH_3. It consisted in simultaneously pumping CO_2, air and ammonia water over copper. This generated much heat which had to be removed.

(f) The new method consists in pumping standard copper solution over new copper and results in no ammonia loss. Although the process is slow, it is still sufficiently fast as not to cause delay, and requires no supervision.

(g) The unit started up without difficulty and has since run well. The only operating difficulty was in packing the expansion machine and the pumps, as it was difficult to obtain chrome tanned leather. Buna rubber has proved to be a good substitute.

(h) The capacity of the unit is fixed by the head available on the high pressure pump of the wash system, as high capacity operation produces high resistance due to its complicated piping system. By simplifying the piping, the copper solution quantities was raised from two hundred forty (240) to two hundred eighty (280) cubic meters/hour. With this amount of solution, eighty-five thousand (85,000) cubic meters/hour of raw hydrogen, having a CO content of 5.5 percent, can be treated. By lowering the CO content to 4.5 percent, as should occur by properly balancing the water gas - methane cracking gas quantity, one hundred thousand (100,000) cubic meters/hour can be treated.

OPERATING RESULTS

HOURLY QUANTITIES

	1943	1944
Avg. raw H_2 quantity treated	74,100 cu.m/hr	86,000 cu.m/hr
Avg. H_2finished product	67,500 cu.m/hr	78,000 cu.m/hr
of which @ 325 Atm. H_2	23.5 %	28.5 %
700 Atm. H_2	76.5 %	71.5 %
Avg. Wash Tower load	16,800 cu.m/hr	17,500 cu.m/hr

VI. CO PURIFICATION. (h) (Cont'd.)

CO REMOVAL

	1943	1944
CO removed	5,900 cu.m/hr	7,000 cu.m/hr
CO content in raw H_2	5.4 %	6.0 %
CO content in finished H_2	0.2 %	0.2 %

ANALYSES OF CO REMOVED

	1943	1944
CO_2	21.0 %	21.0 %
CO	63.1 %	63.0 %
H_2	13.8 %	14.0 %
NH_3	3 mg/m^3	2 mg/m^3

COPPER SOLUTION

	1943	1944
Solution used	251 cu.m/hr	255 cu.m/hr
Colution required for 100 cu. meter of pure H_2	3.7 cu.meter	3.2 cu.meter
Temp. of fresh solution	20.5°C	20.7°C
Temp. of regeneration	40.6°C	39.5°C

SOLUTION ANALYSES

	1943	1944
Cu_2O mol/10 liters solution	11.0	12.0
CuO mol/10 liters solution	2.1	2.2
NH mol/10 c. c. solution	0.488	0.490
CO mol/10 c. c. solution	0.248	0.250
Copper loss (% of makeup)	1.83 %	1.3 %

VII. HYDROGEN COMPRESSORS AND RECIRCULATORS.

(a) The hydrogen compressors, eleven in number, operate with six (6) stages and are driven by synchronous motors. In stages one (1) to three (3), the synthesis is compressed to twen- ty-eight (28) atmospheres whence it goes to the CO_2 purification step. The purified gas is then compressed in stages four (4) to six (6), up to three hundred twenty-five (325) atmospheres. At

VII. HYDROGEN COMPRESSORS AND RECIRCULATORS. (a) (Cont'd.)

this pressure, part of the total gas is used for the gas phase hydrogenation. The remainder is compressed to seven hundred (700) atmospheres in seven single stage boosters for the sump phase step. The boosters are hydraulically operated.

(b) The compressors required numerous alteration. The original piston bearings were held in place by springs, these were later shrunk in. The piston rings had to be so fitted, as not to rotate duting operation thereby allowing the gas to by-pass the joints. The "Presko" piston ring spring was substituted in the fourth stage for "Thermit" springs. By the above changes, the compressors were able to operate without repairs for an aver- age operating period of five thousand (5,000) hours.

(c) The boosters experienced no particular difficulties. The most reliable discharge temperature was found to be ninety (90) degrees centigrade, which temperature is controlled by the difference in pressure between suction and discharge. To hold ninety (90) degrees centigrade temperature, the discharge press- ure was made twice the suction pressure.

(d) The lubrication of the compressors called for special oils. An explosion occurred in one unit while being tested on air, due to lubricating oil. The best oil was one having an Eng- ler viscosity from fifteen (15) to twenty (20) at fifty (50) de- grees centigrade. The running parts used the same oil as the cylinders. The oil used amounted to 0.7 grams/H.P. hour or 3.8 kilograms/hour for each compressor and 6.5 grams/H.P. hour or 5.4 kilograms/hour foreach booster. The largest oil loss was in the discharge of each cylinder. This oil was regenerated by cen- trifuging and the loss made up with fresh oil.

(e) The loss in the machine and the centrifuging amounted to twenty (20) percent, which for eight (8) machines amounted to six (6) tons/month. This loss was later cut in half by improving the oil separation through settling in two (2) decanting tanks at sixty-five (65) degrees centigrade every twenty-four hours.

(f) The capacity of the compressors was to have been increased by reboring the cylinders. Only stages four (4) to six (6) would have needed this as stages one (1) to three (3) were already over-

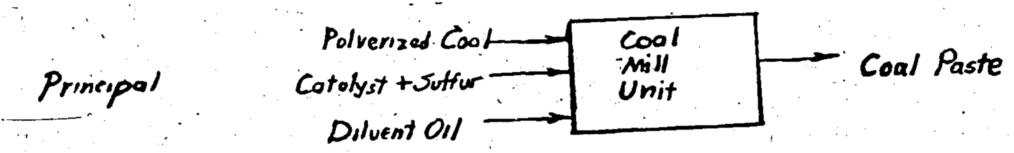
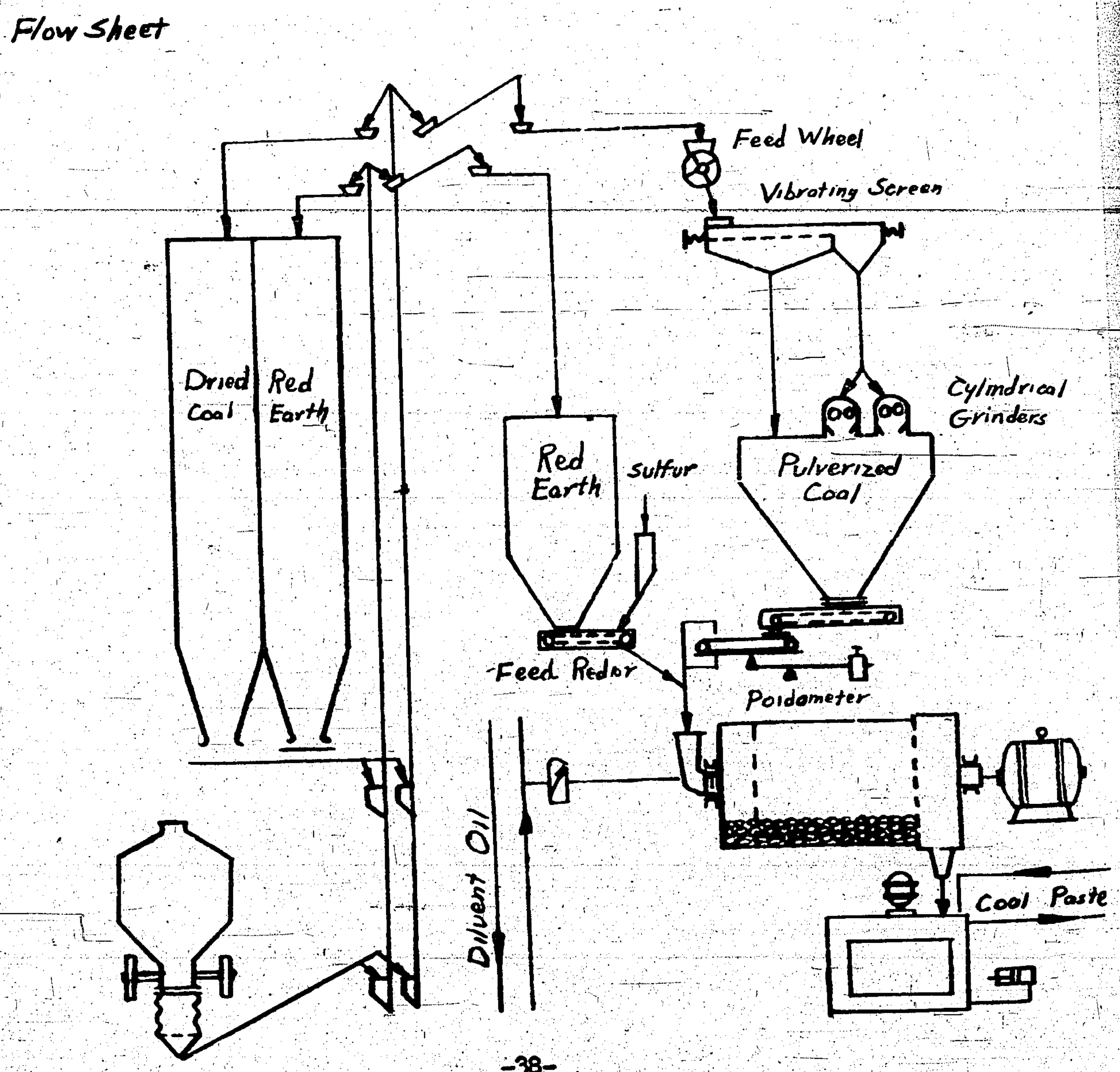


FIG. 10
Coal Preparation Unit



sized. By such changes the compressor output would have increased twenty (20) percent and the booster ten (10) percent. The electric drives were sufficient for the increase.

OPERATING RESULTS

COMPRESSORS

	1943	1944
Total gas throughput	103,100 cu.m/hr	120,000 cu.m/hr
CO_2 purified gas	74,100 cu.m/hr	85,000 cu.m/hr
INDIVIDUAL MACHINES		
Stages 1 - 3	13,300 cu.m/hr	14,400 cu.m/hr
Stages 4 - 6	9,400 cu.m/hr	10,400 cu.m/hr
Avg. working period	4,000 hours	5,000 hours
BOOSTERS		
Avg. 700 Atm. H_2 quantity	51,000 cu.m/hr	55,000 cu.m/hr
Individual machines	10,800 cu.m/hr	10,900 cu.m/hr

B HYDROGENATION PROCESSES.

I. COAL MILL UNIT.

(a) Descriptions and Operation. (Fig. 10)

(1) The dry coal and the catalyst are hauled in. in closed cars. These cars are then connected to the conveying system, which brings the coal and catalyst to the bunkers, by means of section completely dust-proof and equipped with valves operated by compressed air. To eliminate large foreign matter, the coal and catalyst, upon entering, must pass through a grate with bars fifty (50) millimeters apart. The conveying system consists of horizontal and vertical

I. COAL MILL UNIT. (a)(Cont'd.)

Redler conveyors, two for the end and one for the catalyst. For vertical lifting of the catal~~yst~~ it was necessary to replace the Redler by a bucket conveyor, to avoid caking and jamming. The long way that the coal and catalyst must travel required a great multiplicity of conveyors. Altogether there are six (6) Redlers operating in succession, to avoid jamming and breakage caused by faulty sequence to loading and unloading, each conveyor is equipped with an automatic slip clutch connection to its motor.

(2) All the equipment of the grinding plant is protected by an atmosphere of carbon dioxide checked carefully by analysis. The coal is thereafter passed over a vibrating screen where it is divided into two (2) almost even portions. The coal that passes through the screen goes directly to the bunkers which feed the coal paste mill, while the coal that is retained above the screen is first broken up in two (2) roller breakers. To avoid spilling of the coal, there is installed at the bunker outlet a Redler conveyor which can circulate up to eighty (80) percent of its capacity. The only important part of the installation that follows is the rotary grinding mill, with feeding apparatus for coal, catalyst and diluent oil. The mill parts are: a screen conveyor inlet for coal and catalyst, a preliminary chamber and main chambers are separated by a perforated wall. The preliminary chamber contains as grinding elements steel balls of fifty (50) to eighty (80) millimeters diameter; the main chamber steel bars about twenty (20) millimeters diameter and twenty-five (25) millimeters long. The speed of the mill is twenty-one (21) RPM.

(3) The feeding mechanism consists of an automatic scale conveyor for the coal, and for the catalyst, Redler driven by a variable speed drive.

(4) The feeding and measuring of the diluent oil is done through a slide gate with a volumetric feeding control.

(5) The sulfur is weighed and added manually. In the mill coal, catalyst, sulfur, and diluent oil are mixed in

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I. COAL UNIT. (a)(Cont'd.)

a coal paste. This paste goes to a tank, from which it is taken and brought to high pressure by reciprocating pumps, so as to pass under pressure over the vibrating screen and into the seven hundred (700) atmosphere pumps of the finished coal reservoirs.

(b) Development Difficulties and Remedies.

(1) Coal Paste. For smooth operations of the sump phase it is imperative that the coal paste be perfect, and for that purpose any sudden change in its production process must be avoided.

(2) The paste prepared with Rhine lignite, has an abnormally high viscosity.. This characteristic is due to a high swelling property of the coal; it is tied with the high water content of the coal and also with the viscosity of the heavy effluent oil from the coal stall having an initial boiling point of three hundred fifty (350) degrees centigrade. It is not advisable to exceed a concentration of solids in the paste, of forty-two (42) percent. Above that figure the paste loses its pumpability and leaks at the screens under high pressure. In order to obtain a well-prepared coal paste, it is desirable to watch:

((a)) The proper feeding of its component parts, especially the catalyst.

((b)) A low water content of coal and catalyst.

((c)) A constant composition of the diluent oil.

((d)) A low proportion of solids.

The temperature of the diluent oil must be kept as high as possible (one hundred twenty-five (125) to one hundred fifty (150) degrees centigrade) in order to assure the vaporizing of the water brought in by the coal and the catalyst, and to check the viscosity of the paste.

(3) The analysis of the coal paste in 1943 averaged:

I. COAL MILL UNIT. (b)(Cont'd.)

Sp. gravity at 110°	1.175
H_2O	1.9 %
Solids	41.0 %
Coal	35.6 %
Ash in solids	13.3 %
Fe in solids	3.0 %
Sulfur (figured on solids)	1.6 %
Retained on a .3 mm. screen	10.6 %

(4) The coal was generally stripped from the mine in good, constant condition. Mechanical difficulties set in when the screens in the drying plant were operated. The wa- ter content was around 7.5 percent and ran up to twelve (12) percent. The average is 3.5 percent over the estimate. Although water is not desired in the coal, because it tends to disturb the viscosity of the paste and requires addition- al heat in the high pressure phase, a high water content was accepted to reduce fire and explosion hazards. The dry coal has a tendency to "run" with the result that a greater quanti- ty of fines is suddenly discharged out of the bin. Effective counter measures consist in avoiding all possible friction of the coal in the Redler, which would tend to increase the proportion of fines, and to empty the bunkers once or twice every week to eliminate the accumulation of fine coal.

(5) The cellular Redler installed under the intermedi- ate bunker is of value here. It restricts the "running" of coal to the chutes between Redler and Hilburg scale. Eventually these chutes were to be reduced in size.

(6) Dry coal dust is unpleasant and dangerous. A seri- ous explosion took place in the drying plant of the mine, and in one grinding plant a number of fires and "puff-backs". The use of CO_2 scavenging of the inside of the apparatus proved satisfactory. After a few preliminary difficulties, there were no more accidents. The dust escaping to the out- side caused further difficulties . . . . (words missing in microfilm . . . . led to the install- ation of a funnel shaped restriction in the chute from the scale to the mill, which was a great improvement. Attempts to wet the coal with diluent oil, as they had done at Leuna,

I. COAL MILL UNIT. (b)(Cont'd.)

to reduce the dust production, produced good results. The difficulty against building a spraying plant, however, was one of available space.

(7) The catalyst used was an extraction residue from Bauxite decomposition. called "Bayer Schlamm" dried and in large pieces up to thirty (30) millimeters supplied by Gerde mann in Quadrat, Ichendorf and by Chemical Fabrik Hösch, Düren. The Fe_2O_3 content of the Gerdemann product was alway above fifty (50) percent, in the Hösch product it was about five (5) percent lower. Besides, this Red Earth contains $CaCO_3$ alkalies, aluminum oxide and silicate. The H_2O con- tent of the catalyst was averaging six (6) to eight (8) per- cent and occasionally went up to twelve (12) percent. The high water content. found especially in the Gerdemann pro- duct were reduced by installation of a steam line for dry- ing of the "Red-Earth".

(8) This Earth tends to form crusts in the high bunker which should consequently be emptied and cleaned every three (3) months.

(9) Sulfur. Heretofore, weighing and addition of sul- fur was done manually The installation of an automatic measuring apparatus was stopped on account of war restrict- ions. To reduce the cost and also to make the measuring easier through handling of larger volumes (inasmuch as only one hundred (100) kilograms of pure sulfur per ton is need- ed) thoughts are given to the use of sulfur-containing re- sidue of gas cleaning products. All particular inconsive to do this comes from the fact that the Chemical Company at Wesseling could supply such products. In order to study the mechanical possibility of the use of these gas cleaning products, especially in the Redler conveyors, the one of the mill's units was operated for two (2) days with such products and no difficulties of operation were encountered.

(10) The diluent oil. This oil is a mixture of coal- stall effluent heavy oil, centrifuged oil, low temperature oil, and outside oil. The mixing is done in the heavy oil tanks by means of Eckard measuring recorders.

I. COAL MILL UNIT. (b) (Cont'd).

An average composition for 1943 would be:

Coal stall effluent heavy oil	34.2 %
Centrifuged oil and low temperature oil	56.7 %
Outside oil	9.1 %

An analysis of the important data in 1943 is as follows:

Sp. gravity @ 110°C	1.062
Solids	4.0 %
Ash in solids	50.7 %
Asphalt in oil	8.8 %
Fraction boiling below 350°C	10.6 %

(11) In these data, the content of solids is of particular importance, because it controls the content of solids of the coal paste when the preparation of coal remains unchanged. The centrifuged oil is the carrier of the solids; it is therefore important that the making of this centrifuged oil and its addition be kept strictly uniform.

FINENESS OF THE COAL

Remaining ~~over~~ screen of	3.	2.	1.	.5	.3	.1	.06 m/m
On dry coal as received	10.	24.	46.	60.	71.		

HIGH SPEED SCREEN

52 % through	-	.3	9.	25.	43.5		
48 % above	21.	49.5	86.1	98.	100.		
Doreaker outlet	.8	1.8	25.2	45.7	64.8		
Mill inlet (paste)	-	-	-	3.5	13.	16.7	36.

(12) Loading of the mill. The maxmum capacity of the mill corresponds to a production of twenty-five (25) tons/hours of paste at forty-one (41) percent dry coal. The bottleneck comes from the passage through the perforated plate between the preliminary and the main chamber. But the conveyor belt does not . . . . . . (words missing here on microfilm) . . . . . either permit a capacity of twenty (20) ton/hours. To get a better fineness the weight of milling bodies

I. COAL MILL UNIT. (b) (Cont'd.)

was enlarged; in the preliminary chamber, the balls were increased from nine (9) to twelve (12) tons, and in the main chamber from fifty-five (55) to sixty (60) tons. The wear of the balls was .65 kilograms per hour of operation and the wear of the bars .75 kilograms per hour, meaning about four (4) percent loss of balls and one (1) percent loss of such as the liners, made of harder material than the balls, is low and after three (3) years of operation does not come into the picture.

IMPORTANT FIGURES

Use factor of the mills	60.1 %	
Number of mills operating	2,445.	
Average load of all mills	54.	tons/hour dry coal
Hours of operation	21,416.	tons/hours/month

COAL PASTE

Fineness through the .3 m/m screen	10.6 %
Solids	41. %
Viscosity (500 cm3/100°C/6m/m orifice)	48 - 60 sec.
Ash in solids	13.4 %
Fine coal in the paste	32.4 %

DILUENT OIL

Coal stall effluent heavy oil	34.2 %
Centrifuged oil and low temp. distillation oil	56.7 %
Outside oil	9.1 %
Portions boiling below 340°C	10.5 %
Solid	4.05%
Asphalt	8.9 %
H_2O in the dry coal	7.86%
Ash in the dry coal	5.83%
Red Earth (%of pulverized coal)	5.88%
Fe in Red Earth	35.5 %
H_2O in Red Earth	9.3 %
Sulfur (% of pulverized coal)	1.19%

I. COAL MILL UNIT. (b) (Cont'd.)

QUANTITIES

Dry coal	48.98 tons/hour
Red Earth	2.88 tons/hour
Sulfur	.58 tons/hour
Diluent Oil	79.67 tons/hour
Coal paste produced	130.57 tons/hour

II. HIGH PRESSURE HYDROGENATION.

(a) The unit consists of four (4) stalls, five (5) gas circulating pumps, sixteen (16) paste presses, and pumps for seal oil and water, as shown on the attached diagram (Fig. 11). Also an oil, recycle gas washing unit with preliminary circulating gas cooler, intermediate expansion tanks for both the effluent and wash oil.

(b) Each stall has four (4) chambers and includes two (2) heat exchangers sixhundred millimeters diameter, a single burner preheater having eighteen (18) to twenty (20) hairpin tubes ninety (90) to one hundred (100) millimeters diameter, and two (2) circulating gas blowers. Two of the reactors are eighteen (18) meters long and two are fifteen (15) meters long, all one thousand (1,000) millimeters in diameter. There is also in each stall an air and water cooler for the muds, and after-coolers for the effluent product. The first reactor chamber has a desanding device.

(c) Due to packing trouble, the gas circulating pumps were changed from plunger to piston type, piston diameter equal to two hundred (200) millimeters. The circulating pumps handle seventy-two thousand (72,000) cubic meters/hour at a differential pressure of eighty (80) atmospheres.

(d) The coal paste is injected by sixteen hydraulically operated presses, each handling ten (10) cubic meters/hour. Lately, two more twenty (20) cubic meters/hour presses were added. The circulating gas is washed in three towers each twelve (12) meters long, one thousand (1,000) millimeters in diameter, and attached are three (3) expansion machines handling sixty-five

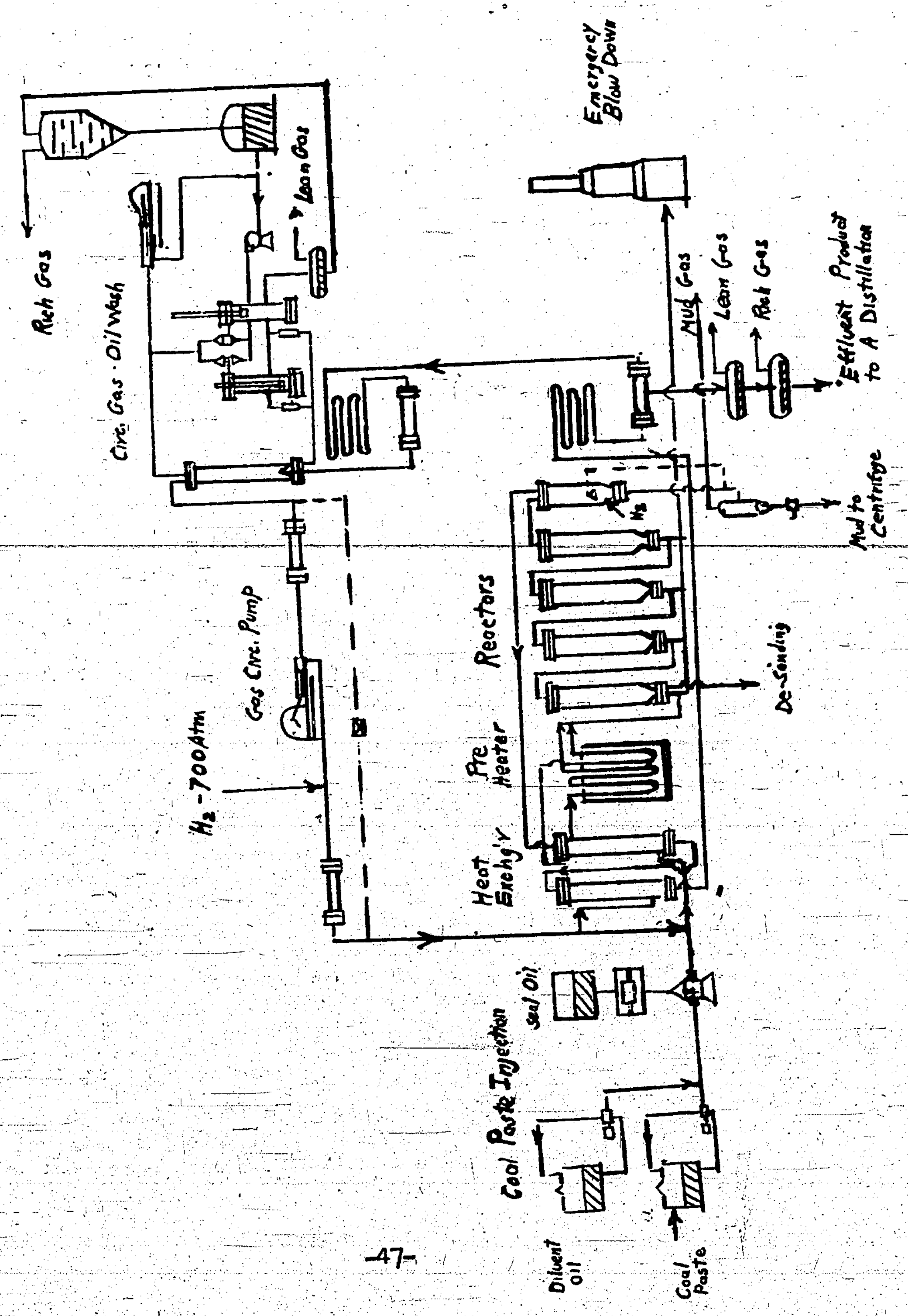


FIG. 11
High Pressure Hydrogenation
Sump Phase

(65) cubic meters/hour, four (4) feed pumps of ten (10) cubic meters/hour capacity each and one wash oil degasifier pump operating at atmospheric pressure.

(e) The intermediate expansion units for effluent and wash oil are designed for fifty (50) atmospheres operating pressure.

(f) In the sump phase lignite outside oil and H_2 are converted to middle oil as follows:

CRACKING AND HYDROGEN ADDITION IN THE PROCESSING OF COAL IN THE SUMP PHASE

	LIGNITE	MIDDLE OIL
avg. mol. wt.	over 1000	220
Carbon atoms	over 100	14
H_2 content	2 %	10 %

(f) Description of the Process.

(1) The coal paste from the mill unit is first screened in a vibrating screen. The oversized reject normally runs 0.3 kilograms per ton of paste. Most of the rejects can be mixed with screenings of dry coal along with heavy grinding substances in preportion of 2.0 kilograms /ton paste. As it contains forty (40) percent oil it is easily marketed as fuel. The paste is then taken to the high pressure paste injection pumps by piston type pumps. The former delivers the paste to the reactors. The suction lines of the paste presses are connected to the diluent oil circulating system. A quick change over from paste to diluent oil is possible by merely manipulating two (2) valves. This is done at every starting and shutting down of the unit because the evaporation of the diluent oil can quickly reduce the chamber temperature. For protection, the glands of the paste presses are sealed with this oil.

(2) The paste and gas, rich in H_2, pass through heat exchangers to a preheater and then into the catalyst chambers. The circulating gas flows through a cooler, and then

is split into equal streams. One-half joins the paste entering the hot heat exchanger and there through the preheater to the first chamber, while the second half goes through the other preheater section directly to the first chamber. The preheater uses gas fuel whose smoke is removed by blowers. The temperature of the circulating gas must remain below the maximum allowable creep temperature of the heater tubes. The temperature of the paste gas mixture is held as low as possible, not only to save heating gas, but because temperatures over three hundred seventeen (317) degrees centigrade (21.5 M.V.) cause salt deposits.

(3) The cool paste is decomposed in the catalyst chamber at four hundred seventy-eight (478) degrees centigrade (25M.V.). This temperature is controlled by injecting cold circulating gas.

(4) On the underside of the first chamber is a de-sanding device, through which one thousand (1,000) to one thousand five hundred (1,500) liters/hour of material is removed. This greatly reduces "caviar" formation in the form of spherical lumps of $CaCO_3$. The conversion products are carried off in vapor phase in the circulating gas stream. The ash content tends to mix with the heavy boiling oil and accounts for the discharge mud leaving the last chamber. The solid content of the mud runs around twenty (20) to twenty-two (22) percent and sets the temperature of the chamber. The solid content of the mud tends to coke up in the heat exchangers due to lack of hydrogen. The coking can hence be eliminated by injecting fresh H_2. This also helps to strip the dissolved gaseous hydrocarbons from the mud.

(5) The mud is cooled to one hundred fifty (150) degrees centigrade and expanded to atmospheric pressure by special valves, having seats and gloves of tungsten carbide. It is then pumped over to the mud disposal plant. The gas released from the mud contains over ninety (90) percent H_2 which can be used over again as fresh hydrogen.

II. HIGH PRESSURE HYDROGENATION. (f) (Cont'd.)

The product stream from the catalyst chambers passes through heat exchangers and coolers. The liquid cut is condensed and enters the coal liquid stripper. The temperature in the latter must be kept above one hundred twenty (120) degrees centigrade in order to prevent foaming. The Bottoms product from this is next expanded and distille. The expansion occurs in two stages and the released gas separated into a rich and a poor hydrocarbon fraction.

(7) The non-condensable gases are given an oil wash under high pressure and the subsequent hydrogen remaining in the gas is used over again in the re-cycle stream along with fresh hydrogen make-up.

(8) The oil wash unit consists of a high pressure wash tower, a wash oil stripper, and an injection pump for the tower. The wash oil used is the product from the gas phase gasoline reactor. The washing is regulated to give a gas of gravity of 0.35 which corresponds to a hydrogen content of seventy (70) to seventy-five (75) percent. The wash oil regeneration is done by expansion in two stages.

(9) The circulating gas temperature is always kept above thirty (30) degrees centigrade to prevent the deposition of hydrates and ammonium becarbonat.

(g) Operating Data and Product Analyses.

Paste feed	40 cu.meters/hr. (in chamber 31 cu. meters)
Catalyst added	5 % red earth (Fe_2O_3) based on dry coal.
Sulfur added	1.25% red earth (Fe_2O_3) based on dry coal.

(h) Paste Analyees.

Solids	41.0 %
Pure Carbon	35.6 %
Ash	13.3 %
Iron	3.0 %

II. HIGH PRESSURE HYDROGENATION. (h) (Cont'd).

Sulfur	1.6 %
Sp.Gr.@ 110°C	1.175
H_2O	1.90
Screen oversize (above 3 mm. mesh)	10.6 %
Gas feed	28,000 cu.meters/hr.
(Paste gas)	13,000 cu.meters/hr.
(Direct to chamber)	15,000 cu.meters/hr.
Cold Gas (quench)	12,000 cu.meters/hr.
H_2 content (Stall entrance)	73.0 %
Pressure	650 atm.

(i) Temperatures.

(1)	Chamber 1	22.5 - 25.0 MV
	Chamber 2	25.0
	Chamber 3	25.0
	Chamber 4	25.0
Coal converted		99.6 %
Middle oil produced		0.275 tons/cu.m. reaction space.
Effluent product based on coal paste		50.0 %
Mud product based on coal Paste		33.3 %
Middle oil - 360°C end point - in effluent product		39.0 %
Middle oil - 350°C end point - in mud		11.0 %
Solids in mud		20.0 %
Asphalts in mud oil		16.0 %

(2) Important difficulties in the sump phase prevented continuous perfect operation. These were:

(a) Difficulties with water separation, causing foaming in the product streams.
(b) $CaCO_3$ deposits causing caviar and salt crust formation.
(c) Injurious asphalt formation and coking in the product cooler.

(d) Preheater tube erosion from sudden product stripping.

(j) The Effluent.

(1) The coal stall effluent when processing pure Rhine lignite has a high specific gravity. It runs from 1.00 to 1.03 at twenty (20) degrees centigrade. This makes ordinary separation of the water of reaction (which must occur before distillation) impossible. Instead, ten (10) to fifteen (15) percent of a special light oil must be added which is the product from the gasoline chamber of the subsequent gas phase operation. The resulting specific gravity of 0.97 at fifty (50) degrees centigrade allows for complete and easy separation.

(2) The effluent also tends to foam, causing trouble in the circulating gas pump and wash system. The foaming can be eliminated by maintaining an effluent temperature of one hundred twenty (120) degrees centigrade.

(k) Caviar and Salt Deposits.

(1) In hydrogenation, the term "caviar" refers to the formation of inorganic products of coal decomposition in the form of small spheres. At Wesseling, this caviar formation took on a size not seen before in other plants. Frequently all four (4) catalyst chambers in a stall would be filled up after ten (10) days operation. At the same time the caviar was forming, salt encrustation of baffles and chamber interiors was taking place. Caviar and salt deposits are chemically alike and contain almost the same amount of $CaCO_3$.

	Residue after roasting	Ash Analyses							
		CaO	CO_2	MgO	S	Fe	Al_2O_3	S_1O_2	C
Caviar	58.4%	46.3	31.8	2.9	1.4	1.8	3.1	0.1	7.1
Salt Crust	59.4%	47.0	26.0	4.5	1.5	4.0	1.7	0.5	5.6

(2) Caviar formation results in poor operation as it quickly reduces the available catalyst chamber volume and leads to all kinds of mechanical difficulties. The loose caviar also gets baked into the material being processed, and finally results in a solid mass, requiring a complete overhaul and cleaning of the unit. Such cleaning is difficult and requires time and materials.

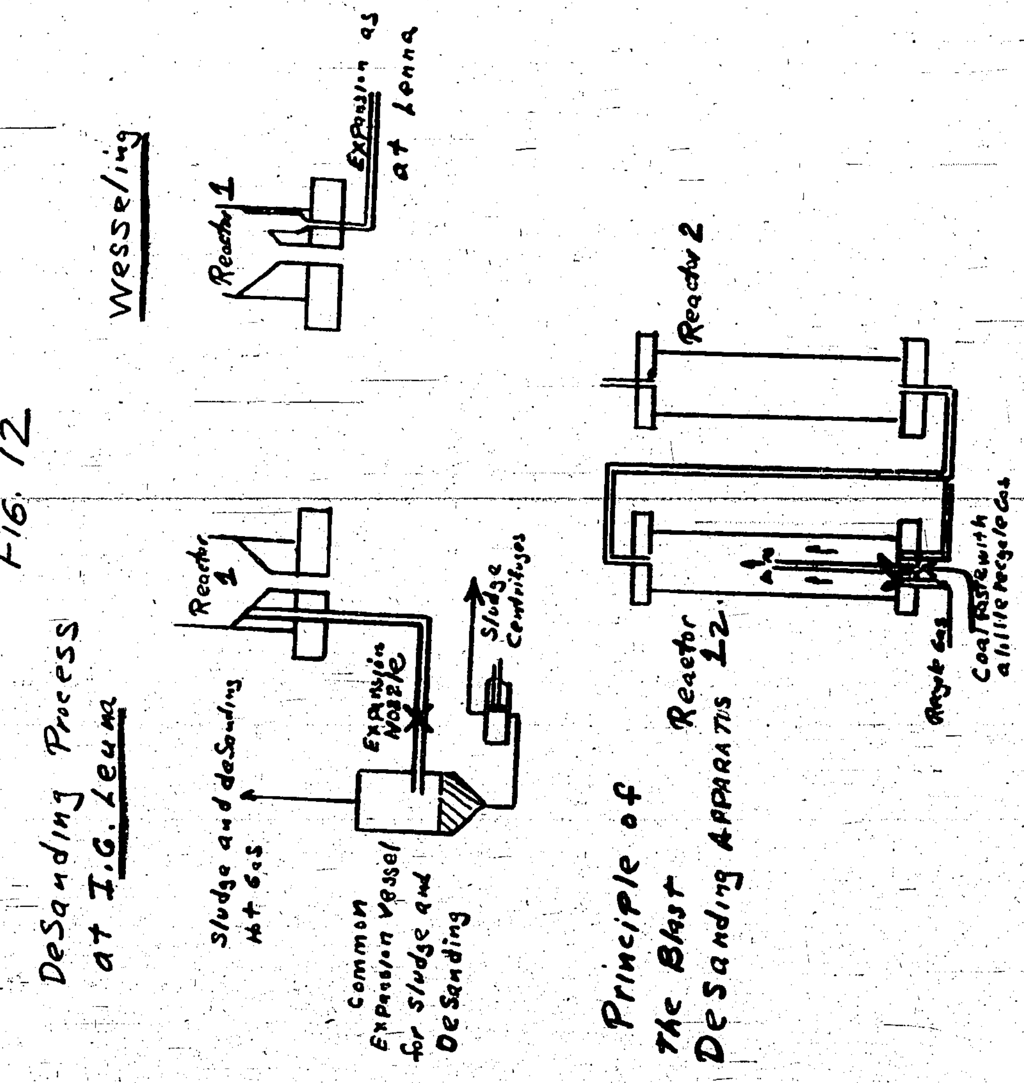
(3) The exact cause of caviar formation has often been discussed. Means used at Wesseling for preventing its formation indicate its origin, namely that it is definitely related to the disintegration phase of coal, which occurs in a zone at the lower part of the first chamber. Hence its growth can be prevented if the crystallization nuclei and the small particles themselves are removed. This is done in a de-sanding apparatus through which part of the product stream from the first chamber is passed, under a long soaking time which breaks down the crystal structure of the caviar. The process can be seen on the attached diagram (Fig. 12).

(4) The method has been used at Leuna for some years. The exact Leuna arrangement could not be used here and had to be changed. In Leuna, one hundred (100) to two hundred (200) liters/hour of product were removed, while at Wesseling one thousand (1,000) to two thousand (2,000) liters/hour had to be withdrawn. Such quantities could not be withdrawn in the Leuna system without causing very large gas losses.

(5) The reason for this is as follows:

The expansion of the de-sanding product takes place batchwise in the horizontal part of the piping through orifices. To do this, the orifice is fully opened for one (1) to two (2) seconds allowing the pipe to become completely empty, while at the same time material is coming into the pipe from the catalyst chamber. The material contains a large quantity of gas, because its high discharge velocity does not allow for a separation of gas and liquids. In the time interval until the next blow-down, liquid fills the gas space resulting in the wrong proportions of gas to liquid.

FIG. 12



DeSanding Process at I.G. Leuna

Wesseling

Principle of The Blast DeSanding APPARATUS 12

II. HIGH PRESSURE HYDROGENATION. (k)(Cont'd)

Hence gas and liquid expand through the orifice into the next catalyst chamber. In Leuna, this difficulty did not occur because the chambers are raised on supports and also because the vertical pipe is proportionately long. To solve the problem at Wesseling, a six (6) liter bottle was attached to the forty (40) millimeter diameter blow down line under the funnel of the first catalyst chamber.

(6) Because of the excessive erosion of the expansion orifices, and also the difficulty in replacing the parts due to material shortage, a de-sander without orifices was developed. A further reason for this new design was the large product and gas loss formerly entailed. In the new design, the de-sanding product was no longer expanded but put into the second catalyst chamber. The diameter and length of the connecting pipe was fixed to maintain a constant pressure drop of one (1) atmosphere between the two (2) chambers. The flow velocity was not allowed to drop below 1.5 meters/second hence preventing the settling out of heavy products. Means were taken to remove the causes of caviar formation in the second chamber, by putting a vertical internal coal paste feed pipe in the first chamber. After a fifty (50) day operating period, no caviar was found in the second chamber.

(7) Salt deposits like caviar also cause trouble despite the results of Leuna and Ludwigshaven for avoiding the same. In the experimental pilot plants, heavy deposits were found on all hot parts of the unit, especially in the catalyst chambers. Attempts were made to eliminate this by chemical means adding H_2SO_4 or S, with no success. It was definitely proven however, that the formation is a function of temperature. This starts at nineteen (19) M.V. and increases with higher temperatures. At Wesseling the limit was somewhat higher, 21.5 M.V. The heaviest deposits occur in the caviar forming zone which is the lower section of the first chamber. Deposits as thick as fifty (50) millimeters were found on the chamber sides and on both the hot and cold hydrogen tubes. The deposit on the piping between chambers one and two was ten (10) millimeters thick, and in subsequent piping only slightly less so. The effect on the

II. HIGH PRESSURE HYDROGENATION. (k)(Cont'd).

preheater tubes is serious as it cuts down the heat transfer and results in overheating the tubes. At Wesseling, the relatively high formation temperature of 21.5 M.V. makes the elimination of the deposits easy. The relatively high partial pressure of hydrogen at seven hundred (700) atmospheres in the first chamber results in a high heat of reaction so that the preheater outlet can be held below twenty-one (21) M.V.

(8) The salt deposits in the first chamber were really serious from a war viewpoint because of the time delays needed to remove them and the freeing of the clogged de-sander nozzles.

(l) Influence of Diluent Oil on $CaCO_3$ Deposits.

(1) Since salt deposits tend to increase in cold, non-insulated pipes rather than in hotter insulated ones, it is fairly safe to assume that this again is a crystallization of $CaCO_3$, and led to attempts to remove it by injecting cold diluent oil rather than cold gas. This produced a delay of the initial formation from three (3) or four (4) days to three (3) or four (4) weeks and resulted in doubling the operating period. The nature of the material removed at the end of this time was not different from the previous operation.

(2) The oil injection was finally given up as its results were not good in that it overloaded the chamber and did not remove the caviar from the first chamber.

(m) The Use of Oil Feed in Cold Regenerators.

(1) Recently, a catalyst chamber using oil feed was built. In this way extra quantities of outside oil could be converted, increasing total production, as oil feed only requires half the hydrogen required for coal.

(2) At the same time, the salt deposits on the first chamber in the stalls could be removed by dilution.

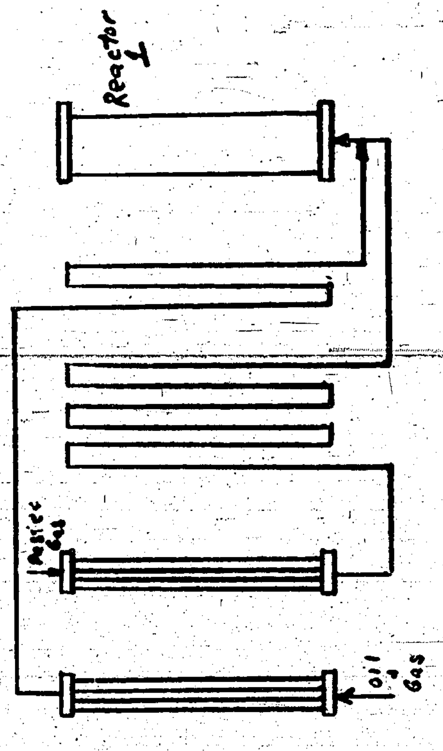


FIG 13

Apparatus Layout

II. HIGH PRESSURE HYDROGENATION. (m)(Cont'd.)

(3) The oil conversion takes place with smaller heat of reaction than with coal and requires higher conversion temperatures. Since the maximum allowable temperature (21.5 M.V.) in the coal paste preheater tubes must not be exceeded (on account of salt deposition), the heating of the oil must take place in special separate tubes. Hence part of the circulating gas stream is used with coal paste as before, and part with the oil. The latter is highly superheated so that the mixture of the two final streams is 22.5 M.V.

(4) The arrangement led also to a saving of heat in the cold heat exchanger. Also the preheater tubes could be overheated without danger because no deposits occur with oil circulation. Further the deposits on the coal tubes were less, due to the lower temperatures of the exhaust gas circulated around the same. The arrangement is shown in the accompanying sketch (Fig. 13).

(5) The catalyst chamber operated fifty (50) days with a feed rate of from forty (40) to forty-five (45) cubic meters/hour and resulted in an increase in total production of one thousand (1,000) tons/month.

(6) The heat released in the cold heat exchanger amounted to five hundred thousand (500,000) kilogram-cal./hour. The resulting scale on the oil tubes in the first chamber was only two (2) to five (5) millimeters thick as opposed to forty (40) to sixty (60) millimeters in the coal paste tubes, which is harder and sticks to the walls. By increasing this oil quantity it is definitely possible to decrease the salt deposits or even eliminate them entirely. It is also important from the number of preheater tubes required, especially since the hairpin tubes are very costly.

(7) In conclusion it can be said that caviar formation can be eliminated by withdrawing part of the product stream from the first chamber and that salt deposits can be reduced by simple means, but that these steps are not of practical importance.

II. HIGH PRESSURE HYDROGENATION. (Cont'd).

(n) Asphalt Decomposition.

(1) Asphalt is precipitated in the sump phase production of gasoline. The analysis of asphalt is very difficult since the formation of asphalt is not easily understood. It was impossible to obtain reliable results with the I. G. Ludwigshafen and Leuna methods. Therefore the following analysis was developed at our laboratory.

(2) Two (2) grams of the solid free oil is placed in a three hundred (300) cc. Erlenmeyer flask, and dissolved in ten (10) cc. pure benzol at room temperature; one hundred ninety (190) cc. of n-hexane are added and the flask shaken vigorously for about a minute. After standing one (1) hour in the cold, the contents of the flask are filtered through an extraction thimble and rinsed with n-hexane. Occasionally a thin layer of asphalt adheres to the wall of the flask and this is dissolved in benzol and added to the rest of the contents.

(3) The extraction is continued for one (1) hour in a cold extraction apparatus so that the asphalt is completely freed of the adhering oil. To prepare the asphalt for weighing the air dried thimble is extracted hot in a Soxhlet apparatus with pure benzol for one and one-half ($1\frac{1}{2}$) hours. After the above mentioned extraction the asphalt residue is placed in a tared flask. After evaporation of the benzol, and standing one-half ($\frac{1}{2}$) hour in a dessicator, it is weighed.

(4) The asphalts prepared from lignite oils are very resistant to hydrogenation. The principles of the hydrogenation process are applicable to asphalt decomposition. This decomposition is a function of the outside oil feed, the catalyst and sulfur contents, the contact time, and the operating difficulties.

(5) The outside oil was formerly used as road oil. No more than five (5) percent of the latter could be used in the diluent oil, since it would raise the asphalt content and cause difficulties in the centrifuging of the mud residue.

Catalyst and sulfur to the extent of five (5) percent and 1.25 percent, based on dry coal, were sufficient.

(6) The principal operating difficulties were:

((a)) The chamber temperature;
((b)) The H_2 partial pressure in the gas throughput was estimated;
((c)) The contact time in the reactor determined the thickening of the mud discharge since the latter carried the asphalt.

(7) These operating difficulties were interdependent as for example an increase of the temperature at a constant mud residue concentration allowed the recycle gas to be decreased, while the thickening of the mud residue was influenced by the temperature and the flow of the gases in the paste.

(8) In operation the inlet temperature was highest, principally to permit a high reaction temperature. It was held between twenty-five (25) and twenty-five and three-tenths (25.3) M.V. maximum. The mud residue concentration was also held constant, between twenty (20) and twenty-two (22) percent. It was necessary to have a positibe inlet gas flow which was about twenty-eight thousand cubic meters/hour, disproportionately low, and gave an H_2 partial pressure of about four hundred fifty (450) atmospheres at the reactor inlet and three hundred (300) atmospheres at the exit. The asphalt control of the mud residue contained fourteen (14) to sixteen (16) percent and met the operating specifications.

(9) In an emergency on account of a quick shut down of the heat exchangers the recycle gas to a reactor was about fourteen thousand (14,000) cubic meters/hour. The reactor temperature was raised to the highest permissable degree of twenty-five and three-tenths (25.3) M.V. The asphalt content of the mud was sixteen (16) percent only slightly over normal. A slight H_2 pressure change can be controlled by a high conversion temperature.

(10) It was felt on this basis that the effect of the H_2 partial pressure was not appreciated. In short tests with our coal at Leuna at various H_2 partial pressures it was proved that the influence was very pronounced. For an increase of partial pressure of the H_2 of one hundred (100) atmospheres, the asphalt content was lowered about one-third (1/3) of the previous valve.

(11) This observation resulted in the introduction of fresh H_2 in the hot heat exchanger and an improvement of several percent in the asphalt plant.

(o) The Introduction of Fresh H_2 in the Hot Separator.

(1) In connection with the difficulty of decomposing the asphalt, the sludge tended to coke in the separator. The tendency was influenced by increased solids concentration. In operation the upper limit was twenty (20) to twenty-two (22) percent.

(2) A precaution against coking was used. Asphalt-free oil was fed to the sludge and recycle gas added to the outlet cone in the separator. The oil injection was reduced while the extremely effective gas introduction was maintained. Later through the substitution of fresh gas for recycle, the operation was improved.

(3) The fresh gas addition, apart from the more effective prevention of coking of the sludge, lead to the following improvements. The hydrocarbons dissolved in the sludge would be removed from the recycle gas stream and in the normal operation added to the sump chamber products.

(4) Propane and butane were produced mostly as power gas and the higher hydrocarbons as middle oil fraction. The residue gas had a H_2 content of over ninety (90) percent as against sixty (60) percent previously and it was as suitable as new H_2 for hydrogenation.

(5) A part of the heat removed with the sludge was carried in the circulating gas, and this was used in the heat exchanger.

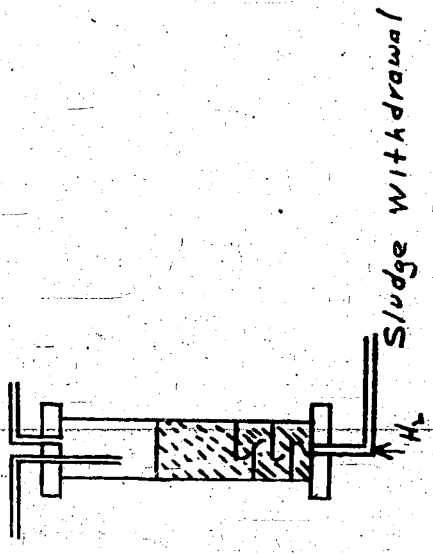


FIG. 14
Countercurrent
Hot Separator

II. HIGH PRESSURE HYDROGENATION. (o)(Cont'd.)

(6) The addition of the sludge gases to the fresh gas disturbed the hydrocarbon content of the resulting sludge-desanding gases. This liability was one of the reasons for the rebuilding of the de-sanding apparatus in the so called reactor 1 - reactor 2 - de-sanding. With its use, desanding product did not accumulate and therefore no de-sanding gas was lost any longer.

(7) The high H_2 partial pressure of the fresh gases caused a hydrogenation of the sludge oils and likewise they were cooled by the inlet gas. The hydrogenation caused an increased oil yield in the chambers. The hydrogenation had to be greatly intensified to maintain the temperature in the hot separator. An apparatus was constructed for this to contact the liquid and gas counter-currently in a small space. The installation of the small hot separator increase the reaction space (Fig. 14).

(8) It is used for heat exchange purposes and contains several indented baffle plates which have a gas space on the lower side. This produces a good gas distribution. Gas and liquid are thoroughly contacted in their counter-current flow between the plates. By keeping the velocity of the liquid phase lower than the gas phase a back flow of the cooled liquid is prevented. Accordingly a good heat exchange is obtained between the sludge and fresh gas. The temperature is controlled in the upper part of the separator by cold gas inlet.

(9) The total requirements of a chamber can be met by a fresh gas flow of about fifteen thousand (15,000) cubic meters/hour. The discharged sludge is cooled about one hundred fifty (150) to two hundred (200) degrees centigrade. The heat exchanger and cooler may be omitted in the sludge discharge.

(10) A separator of this type was used several days before operation of the plant ceased. In this short time, only the heat exchange operation could be investigated and was found to be exceptionally good.

(11) The hydrogenation was tested in a similar but packed separator, but the heat exchange was not complete as only four thousand (4,000) cubic meters/hour fresh gas could be used. The asphalt content of the sludge oil was reduced from sixteen (16) to twelve (12) percent. It is possible that the asphalt decomposition may still be improved by the use of higher H_2 flows past the baffle plates.

(p) Erosion in the Preheater Tubes.

At the beginning of 1943 the inside surface of the preheater tubes was noticed to have cuts six (6) millimeters deep. In previous operations no erosion had been observed. Meanwhile the charges to the chambers had been increased, and the erosion was related to the increased throughputs. It followed that it conformed approximately to the MV^3 value of the mass and velocity.

(q) MV^2 in the Preheater at Differant Feed and Gas Rates.

(at 420°C. preheater temperature, 650 atm. pressure, 0.380 gas density and 1.000/420°C. sp. grav. coal paste)

Gas m^3/hr	Coal Paste m^3/hr	Velocity m/sec	= Mass Coal Paste + Inlet Gas	MV^2
27,000	35,000*	6.4	12.6 (units?)	516
27,000	40,000	6.6	14.0	610
27,000	45,000	6.8	15.4	712
13,000	40,000	4.1	12.5	210
13,000	45,000	4.3	13.9	257
13,000	50,000	4.5	15.3	310
10,000	40,000	3.6	12.1	157
10,000	45,000	3.8	13.5	195
10,000	50,000	4.0	14.9	239

* These values obviously should be 35.000, etc.

(1) The table shows that the inlet gas flows must be reduced when the coal paste throughput is raised. Therefore the paste throughput was held constant, and of the twenty-seven thousand (27,000) cubic meters/hour outlet gas, twelve thousand (12,000) cubic meters were added directly to the hair pin bends and to reactor. This method of operation stopped the erosion.

(r) The Heat Exchangers.

(1) The operation of the heat exchangers in the sump phase was unsatisfactory when operated with only coal paste. The chief reason was the deposition of heavy solid matter out of the coal paste onto the walls of the tube bundle. It resulted in plugging of the inner passages of the tubes. Analyses showed that the deposits were chiefly catalyst, while the inside of the tubes were corroded by H_2S. Part of it was traced to the sludge discharge.

(2) Analyses of the deposits of a heat exchanger compared with the analyses of the catalyst and ash analyses of coal paste and sludge.

	Non-combustible	Analysis of non-combustibles			
	%	CaO	Fe	S	Al_2O_3
Deposit outside of tubes	78.5	12.7	31.5	15.4	8.8
Deposit inside of tubes	91.5	7.3	43.9	20.6	6.3
Coal paste + Solids	14.	23.9	23.5	9.5	10.7
Sludge + Solids	84.5	23.9	23.5	9.5	10.7
Catalyst	100.	2.2	38.2	21.8*	18.6

* Calculated

(3) The original remedy of operating both exchangers with paste-gas mixtures was stopped. Only the hot exchanger is run with paste-gas mixture, and the cold one is operated with gas.

(4) Additional studies were made with smaller tube bundles in an attempt to decrease the solid deposit through increase in the fluid velocity. Instead of the usual one

hundred ninety-nine (199) tube bundle, ones with one hundred fifty (150) and one hundred eight (108) tubes were made and so the velocity of the paste-gas mixtures were raised from 0.4 meters/ second to 0.6 and 0.8 meters/second. A further study was made of a modification of the upward flow of paste, and gas in a down-flow, process.

(5) The following table shows the operation times of the bundles with paste-gas mixtures with various arrangements.

Average Operating Periods of Heat Exchanger Bundles

	Total days	Days when K value was over 100
1. Charging both exchangers with paste-gas mixtures in down flow	55	41
2. Charging only the hot exchanger with paste-gas mixture		
199 tube bundles up flow	95	70
150 " " " "	95	67
108 " " " "		61
199 " " down flow	104	81

(6) The distribution in the gas-paste exchanger caused an increase in efficiency, while the shortening of the bundles caused a decrease. Nevertheless even by this method of operation special results may be expected. This reverse of fluid flow led eventually to a greater improvement, and since the results were not satisfactory, the answer was sought through change of the exchanger construction.

(7) An Exchanger with Paste Down-Flow and Gas Up-Flow.

((a)) This method of operation differed from the previous paste-gas down-flow, since in gas up-flow the exchanger is filled with liquid while in gas.

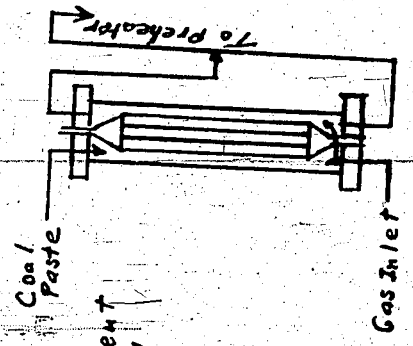


FIG. 15
Heat Exchanger With Counter-Current Flow of Paste and Gas

down-flow it is empty. In the gas down-flow method heavy solids in the liquid settled out.

((b)) In the exchanger full of liquid a drying and agitation of the paste by the gases occurred. The pressure of H_2 is indispensable from a chemical standpoint to prevent flow stoppages (due to deposits). During the operation it was noticed that the exchanger must be kept full of paste and that the paste must not be allowed to overflow the top or the gas to discharge from the bottom. The arrangement is shown in Fig. 15.

((c)) The joining of the gas and paste outlets, half way up the column prevented the paste from being carried out in the gas streams, and the liquid-gas interface was kept constant.

((d)) A direct injection of the gas into the paste outlet is only done with large gas flows.

((e)) A heat exchanger of this construction was in operation twelve (12) days before the closing of the plant. As opposed to this, the previous exchanger had to be cleaned after twelve (12) days operation to restore the K value. The new construction did not require cleaning. It operated well and used two million eight hundred thousand (2,800,000) kilograms cal./hour less. For this the nine (9) meters long hot separator should find application, since by remodeling the last reactor for fresh gas injection, the counter-current separator would be freed. Heavy oil is added to the regular gas stream at the entrance to the exchanger.

(8) The Cleaning of Plugged Exchangers.

((a)) This is costly and time consuming. Previously it was necessary to build the exchangers separately so that by taking off the heads the tube bundle could be withdrawn.

((b)) The repair methods of other plants were not suitable for Wesseling. It is obvious that the cleaning of plugged exchangers with a suitable flushing oil would simplify matters. This method could be used to good advantage by all hydrogenation plants. It was desired to inject CO_2 at high velocity through the exchanger. At the same time high pressure steam would be passed through the tubes. (Fig. 16).

((c)) For a dissolving medium, diluent oil is desirable since it has been shown by laboratory tests to be a good cleaning agent for the deposits on the tube bundles. The flushing would be continued for several days with repeated oil changes.

((d)) The cleaned exchanger could not be tested on account of the plant shut down. The solid matter lost was not great as in the previous method. The cleaning left the exchanger with a pressure drop no greater than in a new one, and the K value was increased to a serviceable figure.

((e)) If the tubes on the inside are plugged too tight, and a high pressure steam or the previously mentioned flushing does not produce the desired results then a mechanical cleaning is necessary. The tube bundles are removed, new ones installed, and the old ones cleaned. This saves production time.

((f)) The flushing with oil and CO_2 played a large part in reducing costs of exchanger maintenance.

(s) Machines.

In spite of the war and the supply difficulties of all of the stuffing box materials, all machines operated trouble free so that no production loss resulted on this account.

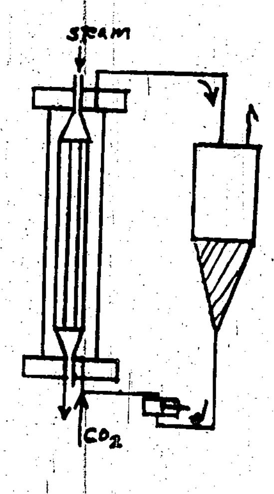
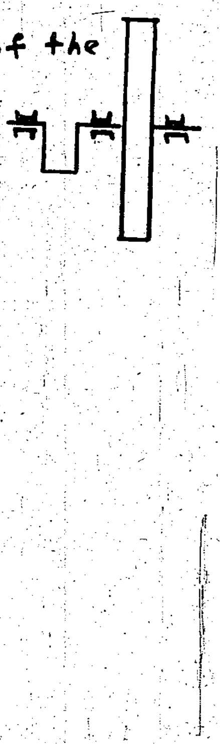
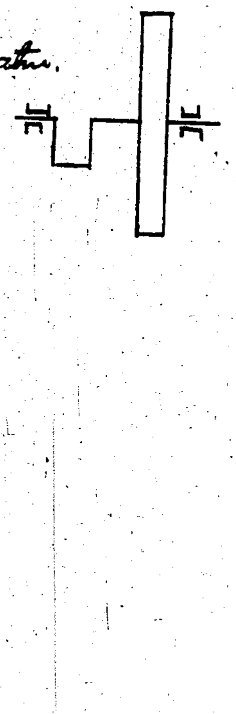


FIG. 16
Heat Exchanger
Flushing with oil
and CO_2 Injection

Fig 17



Bearing
Arrangement of the
700 Atm
Circulating
Pumps



do
325 atm.

(t) Circulating Pumps - Seven Hundred Atmospheres.

The circulating pumps were plunger type machines of one hundred seventy (170) millimeters diameters. They were converted piston type machines. The diameter of the piston was two hundred (200) millimeters, and the piston rod was one hundred (100) millimeters in diameter. The capacity varied from fifty-two thousand (52,000) to sixty-two thousand (62,000) cubic meters/hour. The working life of a set of packing was from sixty (60) to two hundred (200) days. The oil consumption per machine was from eighty (80) to about thirty (30) liters per hours. In starting, entrainment of frothy effluent caused difficulties, but these stopped after raising the effluent temperature to one hundred twenty (120) degrees centigrade and starting the recycle gas washer (Fig. 17).

(u) Circulating Pumps - Three Hundred Twenty-five Atmospheres.

These are in contrast to the seven hundred (700) atmosphere pumps arranged two (2) ways and are also of lighter construction. They required frequent maintenance of the heads and crank shafts. The average working period of the packing was one hundred sixty (160) days.

(v) Oil Feed Pumps.

The middle oil feed pumps (325 atmospheres) are packed with Götz metal. The packing metal contained ten (10) to twelve (12) percent tin. Numerous difficulties of the packing glands could be reduced by careful installation and regular maintainance. Lubrication of the center of the plunger with coal effluent residue mixed with middle oil prevented rust formation by water sprayed for cooling. Diesel oil was fed to the packing. The seven hundred (700) atmosphere auxiliary press pumps of the recycle gas oil washer worked opposite, because of the fear of initial operating difficulties.

(w) Expansion Machines.

They are adversely affected by the solid matter carried with frothy effluent into the wash oil. The removal of the froth delayed getting the plant into normal operation and made frequent

changes and filtrations of the wash oils necessary. Safety precautions in operation proved wise. The reversing valve for reversing the piston from working stroke to discharge stroke caused considerable difficulty. The valve was replaced by a bored hole to the operating cylinder. The engine then operated without difficulty.

(x) Installation, Maintenance, and methods of Operation to Avoid Difficulties.

(1) The starting of the seven hundred (700) atmosphere sump phase was without incident. Later difficulties arose sporadically.

(2) The technical maintenace was placed in the most competent hands; however, the actual installation was done by unskilled people, and yet the apparatus was fully tight when starting up. Those charged with maintenance of the high pressure apparatus used utmost care. Repairs were made quickly and competently carried out. The operational supervision was good in spite of the cosmopolitan set of workmen. Noteworthy is the fact that no tube ruptures occurred through overheating in the coal stalls. Only once did a lens joint between the first and second reactor fail. It caused a fire, but was not serious. In the construction of other hydrogenation works, where chamber fires occurred with powerful fine pointed flames, brick work of the chamber walls were built higher than the reactors, and the windows opposite the paste press houses were walled up. Likewise, for fire protection the windows on the chamber operating walkways and those over the recycle pumps were walled up. Here one mistake was made, an intermediate expansion was not sealed off. The escaping gases ignited and damaged the buildings. For further operating protection the expansion piping, which was especially dangerous, was carried outside overhead.

(3) In the installation of the plant the emergency blow down lines of all of the chambers were led out of the operating chambers into a common line for economy sake. This line meant a serious hazard to the operating crew and apparatus as it was exposed to fluctuations of temperature.

A serious fire developed once at a remote place on the line during a chamber blow down. On this account a second alarm system was installed by the first.

(4) Special attention was paid to the quick opening valves of the high pressure, and the control switches of all driving pumps were connected to one master switch. By this means the high pressure apparatus can be stopped within a few seconds. The arrangement is for airraid protection, but it is useful in normal operation.

(5) The production rate may be estimated by the H_2 generation except in sporadic cases. After elimination of the caviar difficulties (October 1942) the only things that could stop operations were gas production and very heavy demands on the technical section. The average chamber operating time was usually forty-two (42) days. It rose in 1943 to sixty-six (66) and in 1944 to eighty-four (84) days.

(6) The relatively short operation time is largely dependent on the war. A total stoppage or flow caused coking in the chambers. When air raid warning sounded, diluent oil was pumped into the chambers and caused the temperature to drop and often the de-sanding failed to work. The apparatus would then become plugged up from salt crusts.

(7) In normal operation the average chamber operation time was over one hundred (100) days. The simultaneous use of more chambers reduced the hydrogen capacity, especially the high chamber charges, to fifty (50) m^3/hour. These peak loads was later abondoned on account of erosion in the preheater tubes and the asphalt decomposition became too difficult. Later they succeeded in avoiding erosion through bypassing part of the chamber inlet gas that used to be added to the coal paste and passed through the preheater tubes. It was shown by experience with both de-sanding reactors to be possible to use charges about fifty (50) m^3/hour. By use of both chambers the asphalt decomposition was practically complete after the first two reactors. The cold gas charging to the reactors as previously described was used. It is worth noting that in the lower part of the first reactor

the gas and paste were in counter-current flow. The result is that it should be used in conjunction with the counter-current hot separator to obtain similar throughputs at a wide change of reactor volumes.

(8) In Example I (Fig. 18) is shown a chamber with three (3) pieces. In the first reactor the main flow of the paste is down. A small paste flow is allowed to be carried out to the top to keep the reactor full. In the second reactor both gas and past flow upwards. The third apparatus is a counter-current hot separator which must work well, because fresh gas is injected into it. Example II (Fig. 18) shows a straight counter-current apparatus.

IMPORTANT OPERATING RESULTS - 1943

Stream efficiency	sump chambers	84.4 %
(Actual operating hours)	paste presses	75.5 %
(Possible operating hours)	circulating pumps	81.9 %
Avg. Reaction volume (sum of all chambers)		98.1 cu.meters
Avg. No. of chambers used		3.00
Avg. Paste injected/chamber		37.5 cu.met/hr.
Coal Paste fed		1.43 t m^3reaction volume/hr
Middle Oil made		0.265 t m^3reaction volume/hr
Coal converted		99.2 %

FRESH GAS USED

Fed to middle oil reforming	2038.5 cu.meter/ton
Fed to paste	373.6 cu.meter/ton
Circulating gas (Chamber entrance)	575. cu.meter/ton
H_2 in Circulating gas (Chamber entrance)	71.9 %
Pressure	625 atm
Solids in coal paste	41.0 %
Ash content of solid	13.3 %
Pure carbon in coal paste	35.6 %

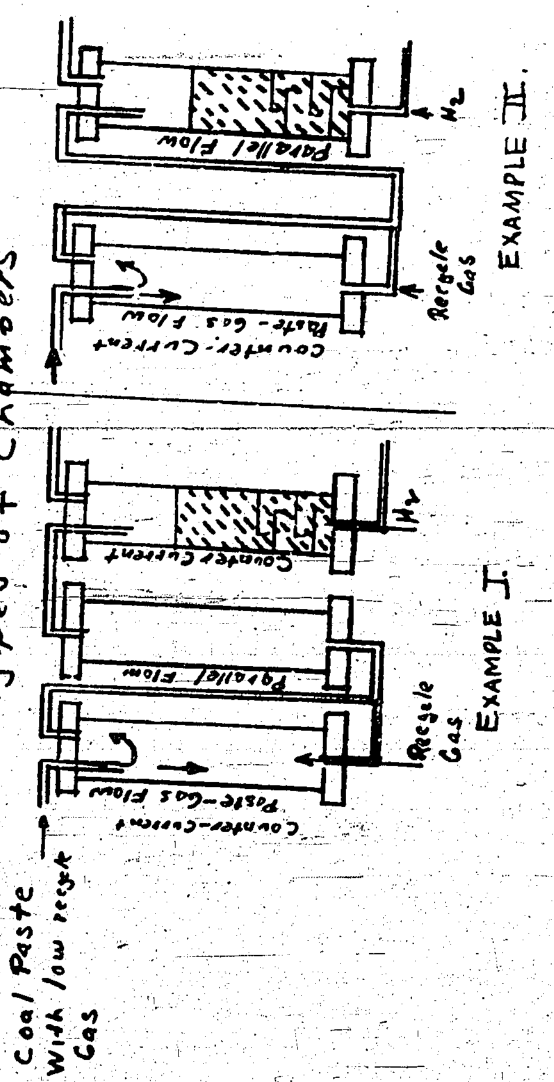


FIG. 18 — Two Types of Chambers

II. HIGH PRESSURE HYDROGENATION. (Cont'd.)

FRESH GAS USED (Cont'd.)

Red earth based on dry lignite/water free	5.76	%
Sulfur - dry lignite/water free	0.129	%
Screen reject over 400 mesh	10.6	%
Screen reject over vibrating screen	0.6	kg/ton

COMPOSITION OF DILUENT OIL

Sump phase residue	34.2	%
Centrifuged oil	56.7	%
Outside oil	9.1	%
Boiling range-up to 340°C	10.6	%
Solids	4.0	%
Asphalt	8.85	%
Effluent (based on paste and seal oil)	51.0	%
Mud " " " " " "	33.8	%
Middle oil - up to 340°C in effluent	38.8	%
Middle oil - up to 340°C in mud	11.2	%
Solids in mud	18.5	%
Asphalts in mud oil	15.6	%

DISSOLVED GAS

In effluent	lean	74.9	cu.met/cu.
	rich	14.2	
In mud		122.5	
In wash oil	lean	151.3	
	rich	46.9	

INTERMEDIATE EXPANSION PRESSURE

Effluent	38.	atm.
Wash oil	35.	atm.

HOURLY QUANTITIES

Coal paste	112.6	cu.met/hr
Cold oil and seal oil	8.8	cu.met/hr
Water injection	6.4	cu.met/hr
Effluent	65.5	

HOURLY QUANTITIES (Cont'd.)

Mud		41.0	
Middle oil reformed		25.4	tons/hr
Fresh gas required		51,583.	cu.met/hr
Gas in effluent	lean	4,902.	" " "
	rich	929.	" " "
Wash gas	lean	7,851.	" " "
	rich	2,274.	" " "
Gas in mud		5,027.	
Wash oil		50.1	cu.met/hr

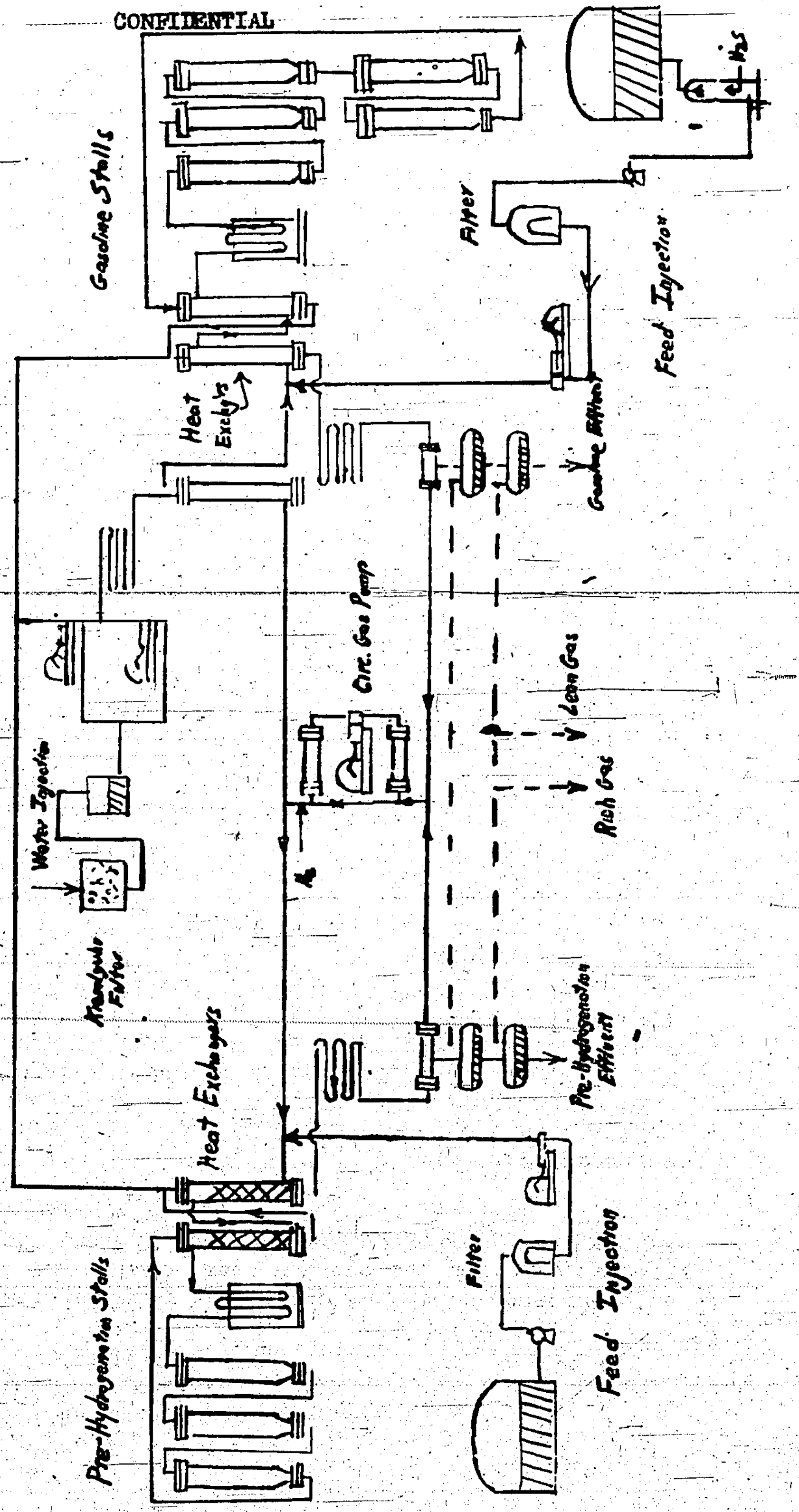
III. GAS PHASE HYDROGENATION.

(a) This unit runs at three hundred twenty-five atmospheres pressure. It consists of four (4) stalls, four (4) gas circulating pumps, six (6) feed pumps for middle oil, three (3) feed pumps for water injection, two (2) water wash towers and sulfurizing equipement for the prehydrogenation and gasoline stalls (Fig. 19).

(b) The stalls each have two (2) regenerators, six hundred (600) millimeters diameter, and catalyst chambers. The latter are one thousand (1,000) millimeters diameter, eighteen (18) meters long and have a catalyst volume of eight (8) cubic meters. Two of the stalls have electric preheaters and the other two gas-burning preheaters. The latter have one (1) oil burner and two (2) circulating blowers. At this time, two (2) stalls of three (3) chambers and one (1) stall of five (5) chambers are filled with the prehydrogenation catalyst 5058/7846 W. The fourth stall of five (5) chambers is filled with gasoline catalyst 6434. Each circulating pump handles seventy-five thousand (75,000) cubic meters/hour at a differential pressure of thirty-five (35) atmospheres. All middle oil injection pumps are enclosed in the stalls. In order to prevent the products of the prehydrogenation and gasoline stalls from mixing, all piping is equipped with double valves and intermediate expansion. The pump suction lines are fitted with porous filters. Three (3) of the six (6) feed pumps have variable speed motors. The feed pumps will handle twenty-five (25) cubic meters/hour and the condensate pumps six (6) cubic meters/hour. The condensate is filtered through kieselguhr.

FIG. 19

High Pressure Gas Phase



(c) The process, as mentioned, works in two (2) steps; the prehydrogenation of the sump-phase middle oil, and the gasoline production step. The former is accomplished by a simultaneous cracking and hydrogenating of the middle oil to a liquid suitable for the production of gasoline. The latter catalyst is more of a cracking catalyst, producing aromatics and iso-paraffins. The former catalysts are designated 5058 and 7846. Catalyst 5058 contains tungsten, an element difficult to obtain in Germany, so 7846 was developed as a substitute. The final catalyst is 6434. All chambers in one stall are connected in series. The operation and arrangement of the stalls is almost identical with the sump phase units.

(d) It must be emphasized that special precautions must be used with catalyst 6434, as it is extremely sensitive to alkaline nitrogen compounds as well as oxygen, both free and combined. Hence the prehydrogenation product must be well refined. Its phenol content can not exceed 0.03 percent and the alkaline nitrogen five (5) milligrams/liter. By water wash, the NH_3 content is reduced to from 0.02 to 0.03 grams/cubic meter. The catalyst must also have H_2S added, up to from 0.4 to 0.5 percent.

(e) The degree of refinement of this feed is measured by the difference in specific gravity between the feed and effluent streams of the stall. These should be from 0.10 to 0.11 entering the prehydrogenator, and 0.075 leaving.

(f) From these values, the catalyst temperatures are established. Using catalyst 5058 this should be 18.5 M.V. and 7846 one (1) M.V. higher. The 6434 catalyst chamber uses nineteen (19) to twenty-one (21) M.V. The increased value is due to slow loss of catalyst activity through refining requirements which the 6434 catalyst requires. The loss of activity in the prehydrogenation chamber took three (3) years to occur and in the 6434 chamber one and one-fourth (1¼) years. This may be considered a normal life.

(g) The A middle oil used in the gas phase consists of seventy-five (75) percent liquid from lignite and twenty-five (25) percent oil from the outside (tar, etc). The composition varies with the specifications of the oil from outside. It normally contains fifteen (15) percent phenols but has gone up as high as twenty (20) percent. The high phenol content, attained during the early days of operation, caused high heating of the chambers with resulting loss in production. Later, when the phenol content dropped, the original catalyst activity was restored and full capacity regained.

(h) In the beginning of 1943, a 6434 stall was built to produce aviation gasoline. This required increased purity from the prehydrogenation unit which in turn lowered the capacity of the same. The average capacity was 0.6 kilograms/liter catalyst space (going up at times to one (1) kilogram/liter.). The capacity of the 6434 stall was higher, and averaged 0.75 to 0.8 kilograms/liter (maximum 1.1 kilograms/liter of space).

OPERATING RESULTS - 1943

Stream efficiency	(actual operating hours) (possible " ")		
Gasoline stalls	75.2		
Middle oil stalls	51.8		
Circulating pumps	53.2		
	Catalyst		
No. of operating stalls (avg)	5058	2.08	
	6434	0.80	
Avg. reaction vol.	5058	37.4	cu.meters
	6434	12.4	" "
Avg. injection feed	5058	0.824	cu.met RV/H
	6434	1.120	" " " "
Avg. throughput	5058	0.764	tons/cu.met RV/H
	6434	1.070	" " " " "
Gasoline capacity (180°C end point)	5058	0.280	" " " " "
	6434	0.710	" " " " "

III. GAS PHASE HYDROGENATION. (Cont'd.)

OPERATING RESULTS - 1943 (Cont'd.)

	Catalyst		
Fresh gas/injection feed	5058	432.0	cu.met/ton
	6434	255.0	" " "
Cooling gas/injection feed	5058	700.0	" " "
	6434	262.5	" " "
Circulating gas (stall inlet)	5058	4325	" " "
	6434	1610	" " "
H_2 in circulating gas (stall inlet)		83.9 %	@ 306.5 atm
Gasoline (180 C end point) in A middle oil		22.8 %	
Phenols in A middle oil		15.4 %	
H_2S in injection feed	5058	38.2 %	
	6434	68.0 %	
Freed gas in effluent	5058 lean	24.9	cu.met/cu.met
	5058 rich	11.5	" " " "
	6434 lean	58.2	" " " "
	6434 rich	29.3	" " " "
Intermediate expansion pressure	5058	28.	atm.
	6434	28	"
Hourly rates - Middle oil injection	5058	29.9	cu.met/hr
	6434	12.1	" " "
Water injected (NH_3 wash water)	5058 + 6434	6.85	cu.met/hr
Fresh feed gas	5058	12,340	" " "
	6434	3,670	" " "
Effluent gas	5058 lean	818	" " "
	5058 rich	377	" " "
	6434 lean	1,660	" " "
	6434 rich	750	" " "

III. GAS PHASE HYDROGENATION. (Cont'd.)

AVERAGE PROPERTIES OF ENTERING AND EFFLUENT PRODUCTS - 1943

PREHYDROGENATION

A Middle Oil

Sp. gravity	0.927	@ 20°C	(Aromatics 62.5 %
API (Phenol free prod)	4.0	?	(Olefines 17.2 %
Phenol content	15.4		(Naphthenes 9.2 %
Mat'l boiling up to 180°C	20.5		(Paraffines 11.4 %

5058 Effluent

Sp. gravity	0.825	@ 20°C	(Aromatics 15.0 %
API	52.0		(Olefines 2.0 %
Phenol content	0.03		(Naphthenes 29.4 %
Alkaline nitrogen in B middle oil	5	mg/liter	(
Gasoline content up to 180°C	38	%	(

Properties of the Gasoline - 180°C End Point

Sp. gravity	0.765	@ 20°C	(Aromatics 8.4 %
API	43.4	?	(Olefines 1.9 %
Cut below 100°C	22.	%	(Naphthenes 30.4 %
Octane number (motor method)	60.5		(Paraffines 59.3 %

Samples taken after the 5058 stall had the following inspections:

Sp. gravity	0.825	@ 20°C
API	20.0	?
Phenol content	6.0	%
Gasoline up to 180°C	25.0	%

GASOLINE MANUFACTURE

Feed to 6434 Stall

Prehydrogenation middle oil	56.0	%
6434 recycled middle oil	44.	
Sp. gravity	0.824	@ 20°C

III. GAS PHASE HYDROGENATION. (Cont'd.)

Feed to 6434 Stall (Cont'd.)

API	54.0
Alkaline nitrogen	5.0 mg/liter
End point	328. °C.

6434 Effluent

Sp. gravity	0.768 @ 20°C	(Aromatics 7.2 %
API	54.	(Olefines 2.6 %
Gasoline - 150°C end point	49.0 %	(Naphthenes 58.0 %
Gasoline - 180°C end point	68.0 %	(Paraffines 32.2 %

Gasoline properties - 150°C End Point

Sp. gravity	0.724 @ 20°C	(Aromatics 6.1 %
API	53.5	(Olefines 1.9 %
Gasoline - 100°C end point	49.0	(Naphthenes 43.5 %
Octane number (motor method)	73.3	(Paraffines 48.5 %

IV. CENTRIFUGING UNITS.

(a) The units consists of thirty-six (36) continuously operating De Laval centrifuges, each handling two (2) cubic meters/hour throughput (Fig. 20).

(b) The mud in the coal stall effluent is separated in the centrifuging units into an oil free from solids, suitable for diluent oil, and a solid residue which is later coked in coke ovens.

(c) The mud is first de-sanded in a conical settling tank, forty (40) cubic meters capacity. The feed enters tangentally and passes down through a fine screen (0.8 millimeter mesh). The sand is removed through the bottom cone.

(d) The de-sanded product is pumped from a recirculating pipe system to the centrifuges. These rotate at three thousand two hundred (3,200) revolutions per minute and have a drum diameter of four hundred thirty (430) millimeters, and a factor:

IV. CENTRIFUGING UNITS. (Cont'd.)

c = 2,500 kilograms. The vertical shaft carries on its upper end the so called plate block, consisting of two hundred twenty-five (225) superposed plates, two hundred eighty (280) millimeters in diameter and 0.5 millimeters apart. The plate block rotates in the drum which has a discharge orifice at the center made of Widia metal (tungsten carbide). Between the plate block and the drum is a latticed basket with Widia edges which scrapes off the residue from the drum sides. Through cyclic operation, a difference in speeds of fifteen (15) revolutions per minute is set up between the basket and the drum.

(e) The feed enters the centrifuge from the top through a throat to a measuring device and then through the hollow shaft. In the narrow space between the plates, the separation of oil and mud takes place. The oil flows upwards through holes in the plates near the shaft. The solids contained in the residue pass through orifices in the drum wall.

(f) The resistance offered by the basket has an important influence on good operation. This normally amounts to 1.5 kilograms but rises occasionally to from 3.0 to 3.5 kilograms. As a safety device, shear pins are installed. After numerous experiments, it was possible to arrange the motor circuit to cut out if the resistance on the basket became too great.

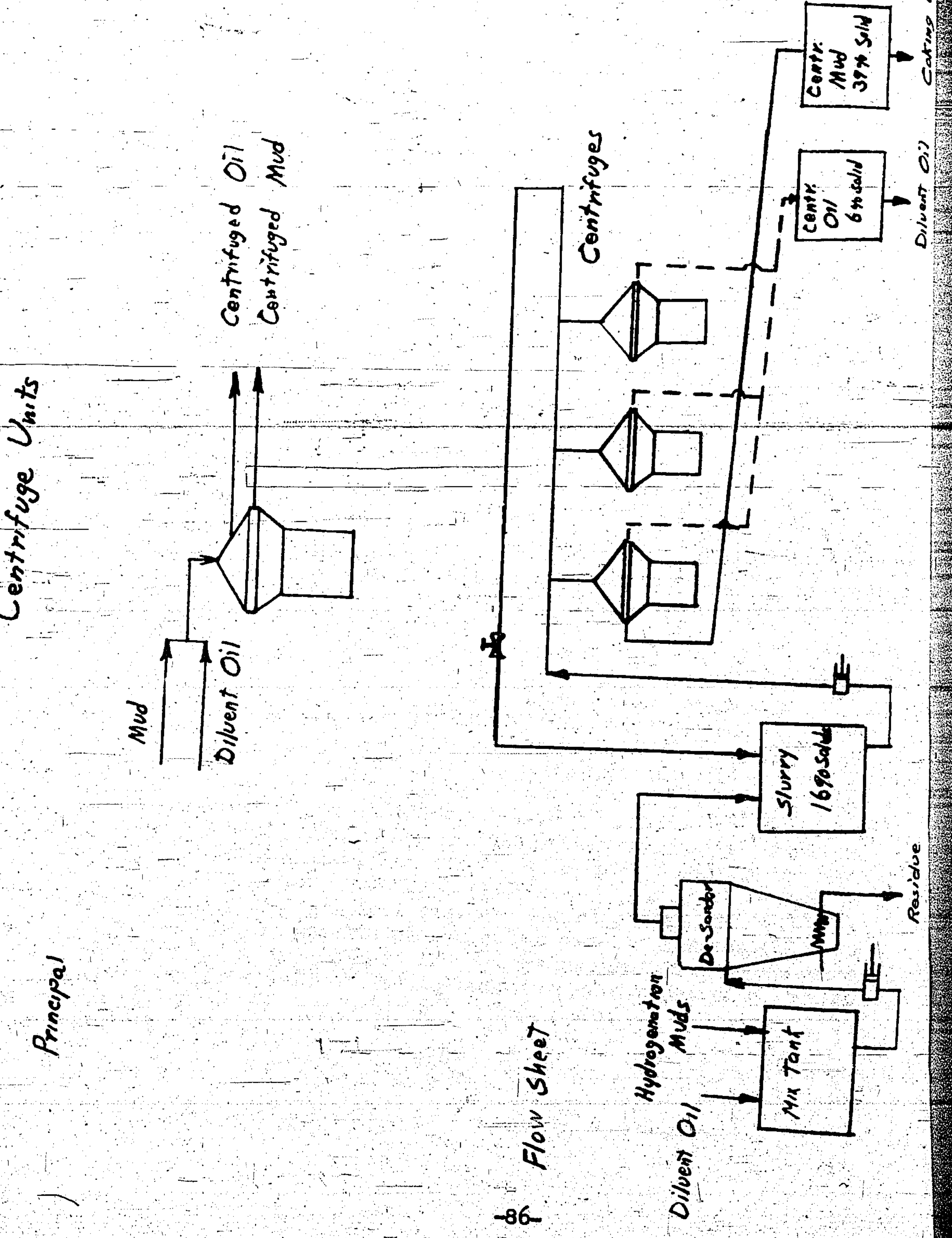
(g) Proper operation of the centrifuges depends on the following factors:

(1) Pressure in recirculating lines;
(2) Solid content;
(3) Temperature of feed 140°C.

Hence the throat and orifice diameters in the centrifuge must be so chosen as to give the highest possible solid content in the residue and the lowest in the centrifuged oil.

(h) The solids in the mud vary from eighteen (18) to twenty-two (22) percent and can be lowered to sixteen (16) percent by dilution with effluent from the coal stalls. By doing this, the coarse particles can be separated easier in the de-sanding step.

Fig. 20
Centrifuge Units



IV. CENTRIFUGING UNITS. (Cont'd.)

(i) The capacity of the machine can be altered by varying the pressure in the feed pipe system. This pressure is normally maintained at one (1) atmosphere but can be raised to 1.2 atmospheres. A variation in feed rate is accomplished through changing the neck in the feed pipe. These run from 1.8 to 2.2 millimeters. By the above changes, the following operating results can be had:

Load on one centrifuge	3.2 tons/hr -	100 %
Product: Centrifuged oil	2.2 tons/hr -	69 %
Product: residue	1.0 tons/hr -	31 %
Solids in centrifuged oil		6 %
Solids in residue		39 %

By increasing the size of the residue exit orifice and keeping the same entrance throat size, the quantity of solids both in the residue and in the oil is lowered.

(j) Ash, Asphalt and Sand.

The solids in the mud contain from ninety (90) to ninety-two (92) percent coal ash. The ash content in the centrifuged oil is seventy-five (75) percent and in the residue ninty-five (95) percent. The asphalt content of the feed to the centrifuge unit is five (5) percent. The discharge from the de-sander contains twelve (12) percent which removes 0.75 of the total solids in the feed. The discharge is finally pumped back to the residue stream.

(k) Operating Difficulties.

The highest allowable solid content in the residue is forty-two (42) percent. By further concentration the drum and basket become clogged with solids. It is impossible to operate the centrifuges without de-sanding. The biggest operating trouble was in starting up after a coal stall had been idle, as in stopping the nozzles would become clogged with material then in the unit. This was eliminated by thoroughly washing before the machines come to rest. The originally installed pneumatic valves for pressure control were replaced by hand operated ones as the former did not work well. This was due to the colgging up of instrument piping with the product.

IV. CENTRIFUGING UNITS. (Cont'd.)

IMPORTANT OPERATING RESULTS

Stream efficiency	53.0	%
Avg. No. of units	19.1	%
Avg. capacity	2.86	tons/hr
Operating hours	116,906	hrs/month

FEED COMPOSITION

Mud	88.51	%
Diluent oil	11.49	%

SOLIDS IN:

Mud	18.5	%
Feed	16.4	%
Centrifuged oil	6.8	%
Residue	38.8	%

HOURLY QUANTITIES

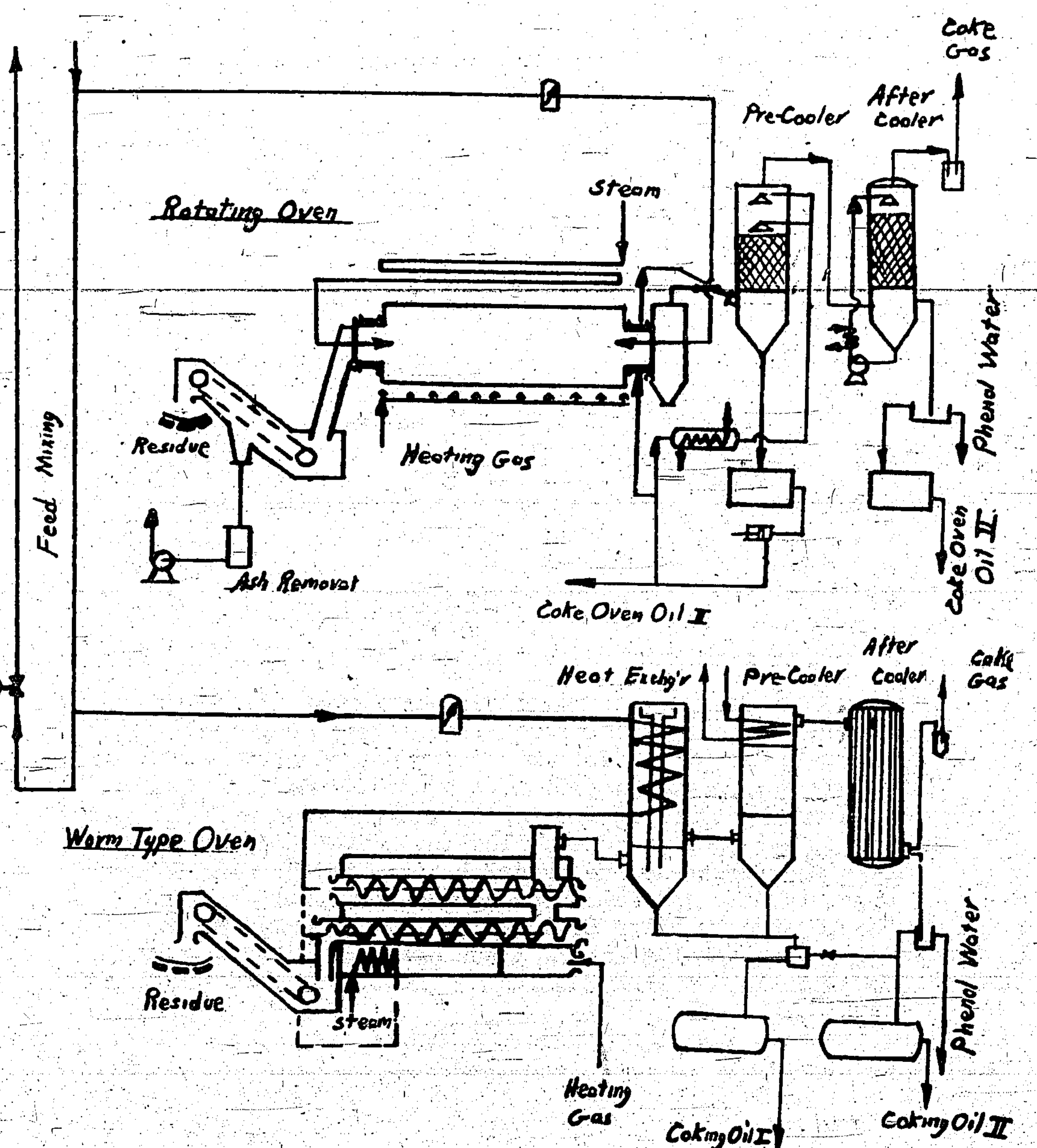
Mud to centrifuges	48.3	tons/hr
Diluent oil	6.3	tons/hr
Total feed	54.6	tons/hr
Centrifuged oil	39.7	tons/hr
Residue	15.4	tons/hr

V. COKING UNITS.

(a) The muds from the centrifuging units are coked in an atmosphere of steam which allows for the recovery of seventy-five (75) percent of their oil content, which is split into two (2) fractions (Fig. 21). The coke oven gas wither goes to the flare or is used as heating gas. The residue consisting of sixty-five (65) percent ash-containing solids and thirty-five (35) percent oil is discarded. The unit consists of:

(1) Six rotating furnaces;
(2) Six worm type furnaces.

FIG. 21
Coking Ovens



(b) Rotating Furnace.

(1) The rotating oven consists of a horizontal, slightly inclined, drum eleven (11) meters long and 2.2 meters diameter. The inner lining of the drum has a worm welded to it of "guronite" (chrome content twenty-five (25) percent). The drum normally rotates nine (9) revolutions per minute but can be raised to eighteen (18) revolutions per minute. It is heated by twelve (12) gas burners. A second row of burners over the drum, preheats the feed and produces superheated steam. The coking section of the drum, which is approximately three-fourths (3/4) of its length, has nine (9) tons of mill balls, of 1.8 kilograms weight each, while the rear section of the drum has twenty-four (24) mill balls, of ten (10) kilograms each.

(2) The feed after leaving the preheater, passes through a feed orifice and is continuously fed to the forward part of the oven. The mill balls prevent the formation of coke on the hot sides of the drum and assist in heat transfer from the sides to the material. The product passes through the drum to the rear of the furnace, or exit chamber, where it is conveyed to a water washer by a worm. A scraper then feeds it to a loader which fills it into railway cars. Steam is injected through the whole length of travel. The released oil vapors leave the drum and enter a dust catcher. They are then condensed and separated in a system of towers and coolers.

(c) Worm Furnaces.

(1) The worm ovens consist of two stationary drums, one above the other, sixteen (16) meters long and one (1) meter in diameter. They are joined together at the front end by a connecting section. In both frums is a worm, each run by a motor, turning it one (1) revolution per minute. The upper worm is hollow and serves to feed material into the furnace. The material moves to the rear of the upper furnace and falls through the connecting section into the lower drum where it again is moved forward to the exit. From here it falls into a water bath and then is lifted by conveyor into railway cars.

(2) The furnace is heated by a burner in the front section, under the lower drum. The heat is controlled by a heating gas regulating valve. The steam superheater tubes are in the rear sections of both the upper and lower furnaces The oil vapors leave through a vertical pipe at the forward end of the upper drum. It gives up its heat to the incoming feed through a heat exchanger and is then condensed in two (2) stages by a precooler and an after-cooler, both water cooled. The former is a packed dephlegmator and the latter a tubular condenser. The condensate from the precooler and dust catcher is mixed to make coke oil I. Coke oil II comes from the after-cooler, and passes through a water separator. The gas is used either for heating or discarded.

(d) Operation.

(1) The operation of the coking units depends on the following factors:

((a)) Feed rate;
((b)) Furnace temperature;
((c)) Amount and temperature of injection steam;
((d)) Top temperatures of the coolers;
((e)) Furnace pressure.

(2) The two types of furnaces require different operation due to their design. The coked product from the rotating furnace easily goes to dust. In order to prevent stopping up the equipment, only small quantities of scrubber steam can be used, one hundred (100) kilograms/ton of feed as opposed to five hundred (500) kilograms/ton in the worm oven. To obtain the same coking effect the temperature in the former must be kept higher, namely at five hundred ninety (590) degrees centigrade at the exit while the worm oven is only kept at five hundred sixty (560) degrees centigrade. Higher temperatures tend to coke up the worms. The products are approximately the same, except that the rotating oven residue has more coke due to the higher temperature.

(3) The top temperatures of the pre- and after-coolers require careful attention if a good separation of water in

coke oil II is to be had. The former runs around two hundred twenty (220) to two hundred forty (240) degrees centigrade and the latter one hundred ten (110) to one hundred thirty (130) degrees centigrade. The pressure in the furnace differs in both types, eighty (80) to one hundred twenty (120) millimeters for H_2O for the rotating oven and forty (40) to sixty (60) for the worm type oven.

(e) Operating difficulties.

(1) The rotating furnace started up without difficulty. Frequent stopping of operation due to air raids would often cause the tubes in the preheator to coke up, requiring the by-passing of the same. The resulting objections were eliminated by reducing the throughput fifteen (15) percent and raising the furnace temperature from five hundred seventy-five (575) to five hundred ninety (590) degrees centigrade. Further, a new type of preheater was considered which would work with the central burner and avoid the errors in the former type. The stopping up of steam superheater tubes, made of Sichromal, was frequent because the flow through three (3) parallel streams is difficult to regulate. By building a single stream superheater, much better operation resulted. The coking residue tends to become slimy due to pulverization by the mill balls and small remaining oil content. For this reason, conveyance by a rubber belt was impossible, and a slurry had to be used. The residue is passed over a grate by the water stream. The grate removes the coarse particles which can then be moved in the normal way. The remainder falls through the grate in fine particles and is sluiced with much water in a tank. It then is pumped through pipes to the ash dump where it is handled by cranes.

(2) The worm type furnaces started up without difficulty. Difficulties were met in the even distribution of heat over the drums. The heating up of a furnace required forty-eight (48) hours as opposed to the six (6) hours of the rotating type. By constructing baffles and by diminishing the size of the combustion chamber a small improvement was obtained. It was finally necessary to build circulating gas blowers as with the rotating ovens. The operating period

of a furnace is fixed by the coking up time of the worms. This was seventeen (17) to twenty (20) days for the lower and one hundred (100) to one hundred twenty (120) days for the upper worms. An improvement was obtained by not shutting down during alerts and by constructing circulating gas blowers. The heat exchangers were also stopped up due to air raid alarms. For this reason a furnace without preheat was used. In this way the heavy oil condensed in the uncooled exchanger. By circulating coke oven oil I in the exchanger, the process was able to operate and resulted only in a slight loss of capacity and a ten (10) percent loss of possible heat recovered.

OPERATING RESULTS

	Rotating Furnace	Worm Furnace
Dist. of load	70.0 %	30.0 %
Capacity - ton/hr/furnace	3.2	2.0
Capacity oil - ton/hr/furnace	2.0	1.25
Coke oven oil I - ton/hr/furnace	1.4 - 70 %	0.75 - 60 %
Coke oven oil II - ton/hr/furnace	0.1 - 5 %	0.20 - 16 %
Oil recovery	75.0 %	76.0 %
Furnace temperature	590.0 °C	560.0 °C
Heat required/charge	350,000.0 kg cal /ton	350,000.0 kg cal/ton
Steam required/charge	100.0 kg/ton	500.0 kg/ton
Solid content of coke oven feed	39.0 %	39.0 %
Asphalt content of oil in oven feed	13 - 15%	13 - 15%
Analysis of Residue:		
Gasoline soluble	2.0 %	20.0 %
Coke	33.0 %	16.0 %
Ash	65.0 %	64.0 %
Heating valve	3,300.0 kg cal	3,450.0 kg cal

TOTAL COKE UNIT

Feed	16.0 tons/hr
Coke oven oil I	5.69 tons/hr
Coke oven oil II	1.34
Coke oven gas	0.42
Oil less from injected oil	27.29 %

ROTATING OVEN

Stream efficiency	57.59 %
Total hours in operation	30,269.0
Avg. No. of operating furnaces	3.46
Avg. furnace load	3.26 tons/hr

Charge

Solids	39.54 %
Asphalt content in oil	24.2

Residue

Gasoline soluble	2.4 %
Ash	64.2 %
Heating valve	3,498.0 K cal/kg

Hourly Quantities

Feed	11.28 tons/hr
Coke oven oil I	4.03 tons/hr
Coke oven oil II	0.84 tons/hr
Gas	0.35 tons/hr
Outside oil injected and lost	28.5 %

WORM OVEN

Stream efficiency	34.2 %
Operating hours	17,973.0
Avg. No. of operating furnaces	2.05 %
Avg. furnace load	2.30 tons/hr

Monthly Quantity

Oil lost by injecting outside oil	24.4 %

Charge

Solids	34.49 %
Asphalt in oil	24.2 %

Residue

Gasoline soluble	18.9 %
Ash	80.31 %
Heating	3,449.0 K cal/kg

Hourly Quantities

Feed	4.72 tons/hr
Coke oven oil I	1.66 tons/hr
Coke oven oil II	0.50 tons/hr
Coke oven gas	0.07 tons/hr

(f) Production of Briquette Binder.

The large demand for briquette binder material for French hard coal led to experiments using the coke oven residue. In these, only a part of the light oil was removed at low temperature operation. As Fig. 22 indicates, the product from the oven enters a conical tank and is then circulated around by a pump, through a cooler, or pumped directly to a tank car. The experiment was conducted under the following conditions.

	Feed	Product
Solid content	16.8 %	32.1 %
Ash content in solids	85.3 %	79.6 %
Asphalt in oil	26.3 %	41.0 %
Sp. gravity at 80°C	1.2 kg/liter	1.42 kg/liter
Penetration point (Krämer Sarnow)		74.5 °C

FIG. 22

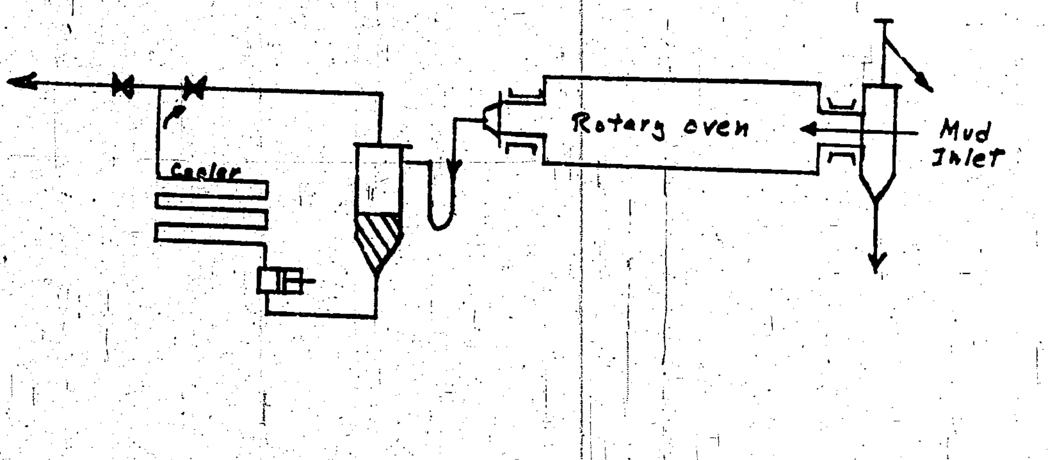
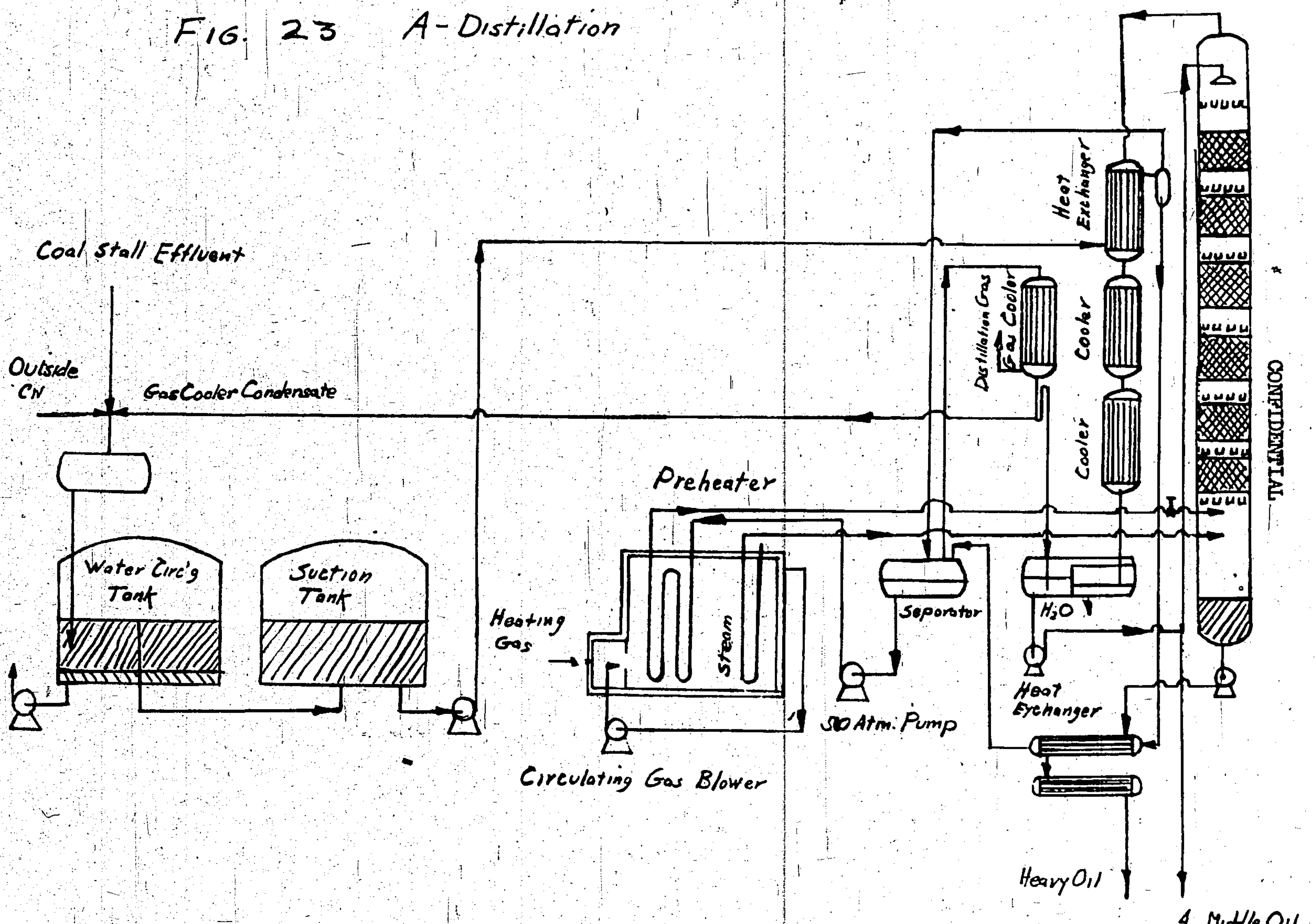


FIG. 23 A-Distillation



V. COKING UNITS. (Cont'd.)

Capacity	3.6	tons/hr
Oil	3.6	tons/hr
Solid	0.6	tons/hr
Steam added	0.21	tons/hr
Furnace temperature	500.0	°C

YIELDS

Coke oven oil I + II	1.36	tons/hr
Residue	2.13	
Oil	1.44	
Solid	0.69	

VI. DISTILLATION UNIT A.

(a) The A distillation unit consists of two (2) separate columns and equipment. In this unit the total coal hydrogenation product and outside oil for pasting are separated into the A-middle oil for the gas phase hydrogenation step and a heavy oil to be used again in the sump phase. (Fig. 23). Both the product and pasting oil are mixed together, as only one storage tank exists for A distillation feed.

(b) The main tower is equipped with packed sections, four (4) meters apart. The feed is preheated under fifty (50) atmospheres pressure, thereby keeping it in liquid phase. This prevents erosion and clogging of the packing through too high vapor velocity. Two (2) pump stages are used. By the first the feed is pumped at atmospheric pressure over a heat exchanger into an intermediate storage tank, while the second pumps the feed through the pipe still into the tower. The gas resulting from this preheating is condensed and used as explained later. All pumps are centrifugal.

(c) The feed may not contain more than one (1) percent water, otherwise it will cause foaming with a resulting flooding of the tower. The water removal is accomplished by settling in two (2) storage tanks. In order to obtain a complete dehydration, sufficient gas condensate is added to the feed to keep its specific

gravity under 0.970 at fifty (50) degrees centigrade. This normally amounts to fifteen (15) percent of the feed.

(d) The middle oil for the gas phase must have an end point of three hundred fifty (350) degrees centigrade. This high boiling range material causes a heavy loading of the gas phase catalyst. Hence material boiling above three hundred fifty (350) degrees centigrade must be absolutely avoided. The distillation requirement therefore produces a gap between the end point (Engler) of the middle oil and the initial boiling point of the heavy pasting oil. This gap has to be paid for by burning extra heating gas in the preheater and using extra stripping steam in the base of the column. Hence the utility requirements, originally anticipated, are somewhat over the guaranteed figures.

(e) The unit started up without difficulty and has run well ever since. Water entrainment caused some trouble due to the emusifying effect of hydrogenation mud and paste. This was remedied by improving the sump phase operation and by blending tower feed with outside pasting oil.

(f) The tower can be operated with good separation and ordinary utility requirements at between thirty (30) and one hundred fifty (150) percent of the guaranteed capacity figures.

OPERATING RESULTS

Avg. operating capacity	85.6	
"Number" of units used	1.715	
Avg. unit capacity (including circulation)	48.6	tons/hr

OPERATION WITHOUT GAS CONDENSATE

A*middle oil	40.05	%
Residue (bottoms)	59.32	%
Overhead gas K	0.58	%
Loss	0.05	%
Middle oil end point	342.0	°C
Residue - I.B.P.	349.0	°C
Gap	7.0	°C

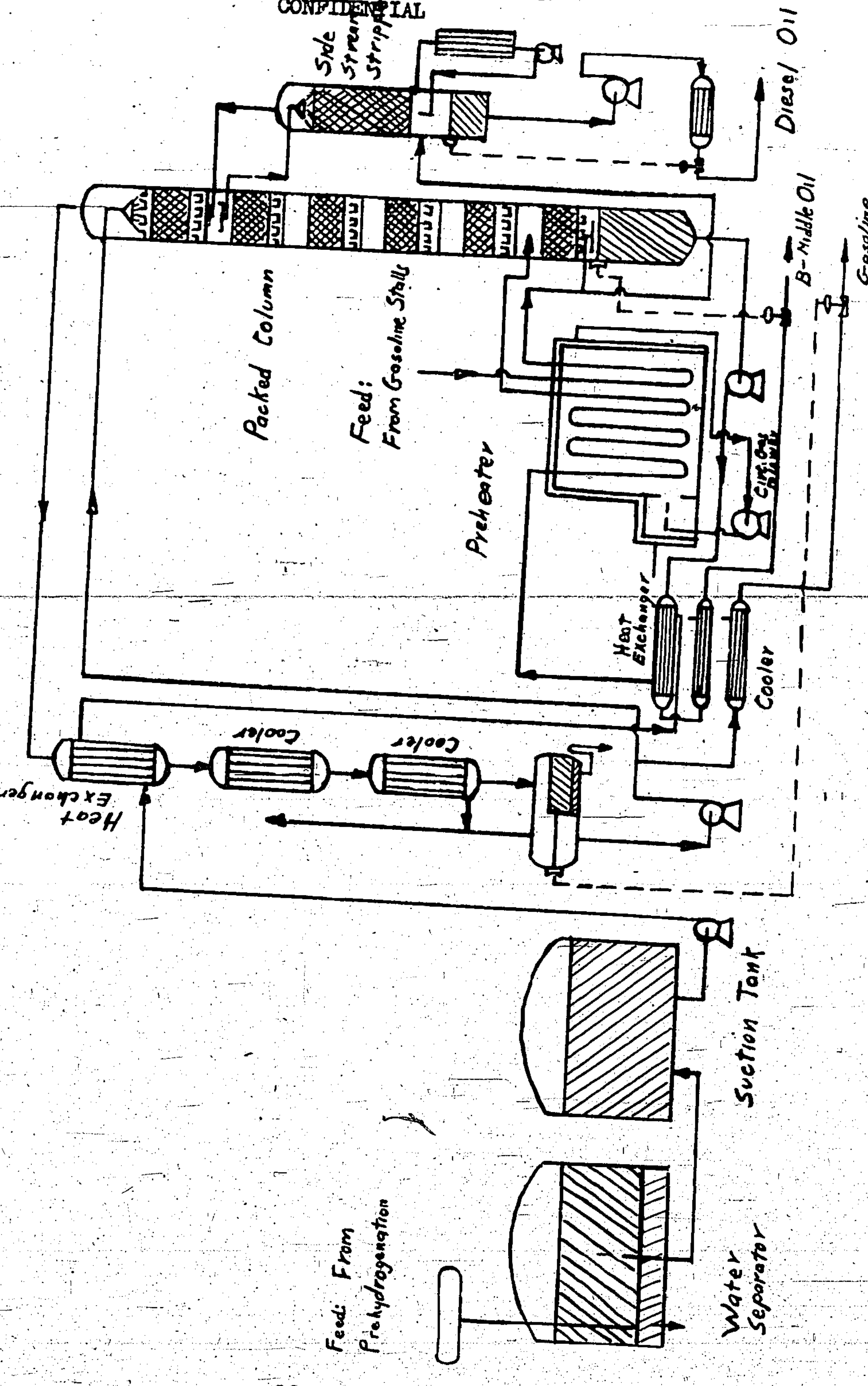


FIG. 24
B-Distillation

VI. DISTILLATION UNIT A. (Cont'd.)

OPERATION WITHOUT GAS CONDENSATE (Cont'd.)

Circulation quantity/total throughput	14.6 %
Distillation factor - without circ.	2.92 %
Distillation factor - with circ.	2.50 %

HOURLY QUANTITIES

Throughput (no recirculation)	71.3 tons/hr
A-middle oil	28.5 tons/hr
Residue	42.3 tons/hr
Tail gas	27.8 cu.met/hr
Gas condensate - returned	5.92 cu.met/hr

ENERGY REQUIREMENTS

Heating gas/ton throughput	0.125 mill kg, cal.
Steam/ton throughput	0.078 mill kg, cal.

VII. DISTILLATION UNIT B.

(a) In the B-distillation unit, the products from the gasoline stalls are separated. The apparatus greatly resembles that of the A-distillation. The tower packing is higher because a side stream stripper is connected to the column for removing intermediate products (Fig.24).

(b) The products of prehydrogenation and the gasoline stalls are separated by distillation. To produce the normal products of aviation gasoline and diesel oil, the following conditions, are used. The effluent from the prehydrogenation step is cut at one hundred twenty-five (125) to one hundred thirty (130) degrees centigrade. If a part of the distillation residue is to be sold as diesel fuel, an intermediate fraction of from one hundred thirty-five (135) to two hundred five (205) degrees is withdrawn. The part boiling above two hundred five (205) degrees is diesel oil. The intermediate cut plus some diesel oil is then fed to the gasoline stalls. In using the whole distillation residue from the gasoline stalls, the yields of intermediate product is assured. The gasoline stall effluent is separated into

VII. DISTILLATION UNIT B. (b)(Cont'd.)

gasoline with an end point of one hundred fifty (150) degrees centigrade and a bottoms product which after mixing with the prehydrogenation residue is reused as feed to the gasoline stalls. The gasolines from both distillations are treated, and then blended with the condensed cut from C_4 unit followed by a caustic wash and stabilization.

OPERATING RESULTS

PREHYDROGENATION DISTILLATION - 5058

Stream efficiency	81.8 %
No. of operating units	0.83 %
Avg. load/unit (including circulation)	37.3 tons/hr

Yields

Gasoline 5058	31.7 %
Heavy gasoline 5058	2.28 %
B-middle oil 5058	64.66 %
Distillation gas 5058	1.27 %
Losses	0.09 %

F.B.P. - gasoline	150°C
I.B.P. - Heavy gasoline	136°C
F.B.P. - heavy gasoline	192°C
I.B.P. - B-middle oil 5058	155°C

Recycle product/total throughput	10.3
Distillations factor without recycle	3.26
Distillations factor with recycle	3.63

Hourly Quantities

Throughput without recycle	26.73 tons/hr
Gasoline 5058	8.21 tons/hr
Heavy gasoline	0.61
B-middle oil 5058	17.55
Distillation gas	180.0 cu.met/hr

VII. DISTILLATION UNIT B. (Cont'd.)

Utility Requirements

Heating gas/ton throughput	0.190 mill kg. cal.
Steam/ton throughput	0.139 ton

6434 - DISTILLATION

Stream efficiency	79.3 %
No. of operating units	0.55
Avg. load/unit (including circulating)	29.0 tons/hr

Yields

Gasoline 6434	48.08 %
B-middle oil 6434	49.57
Distillation gas 6434	2.35
Losses	0.00

F.B.P. gasoline	149°C
I.B.P. middle oil	152°C
Gap	3°C

Circulating product/total throughput	8.33 tons/hr
Distillation factor (throughput - distillation)	
With recycle	2.27
Without recycle	2.08

Hourly Quantities

Throughput without recycle	15.99 tons/hr
Gasoline 6434	7.69 tons/hr
B-middle oil 6434	7.92
Distillation gas	162.0 cu.met/hr

Utility Requirements

Heating gas/to throughput	0.163 mill kg. cal.
Steam/ton throughput	0.038 ton

VIII. ALKALINE WASH AND STABILIZATION.

(a) The crude gasoline is purified by an alkaline wash using dilute NaOH solution to remove H_2S and Phenolics. This is followed by treatment in the stabilizer to remove the light hydrocarbons and make the vapor pressure correct for a saleable product.

(b) Apparatus. (Fig. 25)

The alkaline wash precedes the stabilizer in order to protect it from H_2S corrosion. A degasser may be installed ahead of the alkaline wash. The alkaline wash is done in two (2) stages. For washing entrained alkaline solution, a water wash is employed afterwards. The stabilizer is made with a round-based column, and is built for six (6) atmospheres working pressure. It operates fully automatic except for manual feed injection.

(c) Operation.

Since the crude gasoline contained only one hundred fifty (150) to two hundred (200) milligrams H_2S/liter, degassing was unnecessary. The gasoline was generally stabilized to have a vapor pressure of 0.5 atmospheres by the removal of dissolved C_1 to C_4. The resulting liquid heads product was vaporized and combined with the heads gas from the C_3 plant. With increased production the C_3-C_4 plant was not able to meet its guarantee, and the C_3 head product could not be used as mentioned. Consequently the reflux to the stabilizer had to be increased which was able to maintain the purity specifications for marketable C_3 gas. Thus the C_3 plant was kept supplied. The fraction of the total C_3 production resulting from C_3 heads gas amounted to about thirty (30) percent.

OPERATING CONDITIONS

Gasoline	21.5	ton/hr.
Vapor pressure at entrance of stabilizer	0.95	atm.
Vapor pressure at exit of stabilizer	0.48	atm.
Liquid top product	1.1	ton/hr

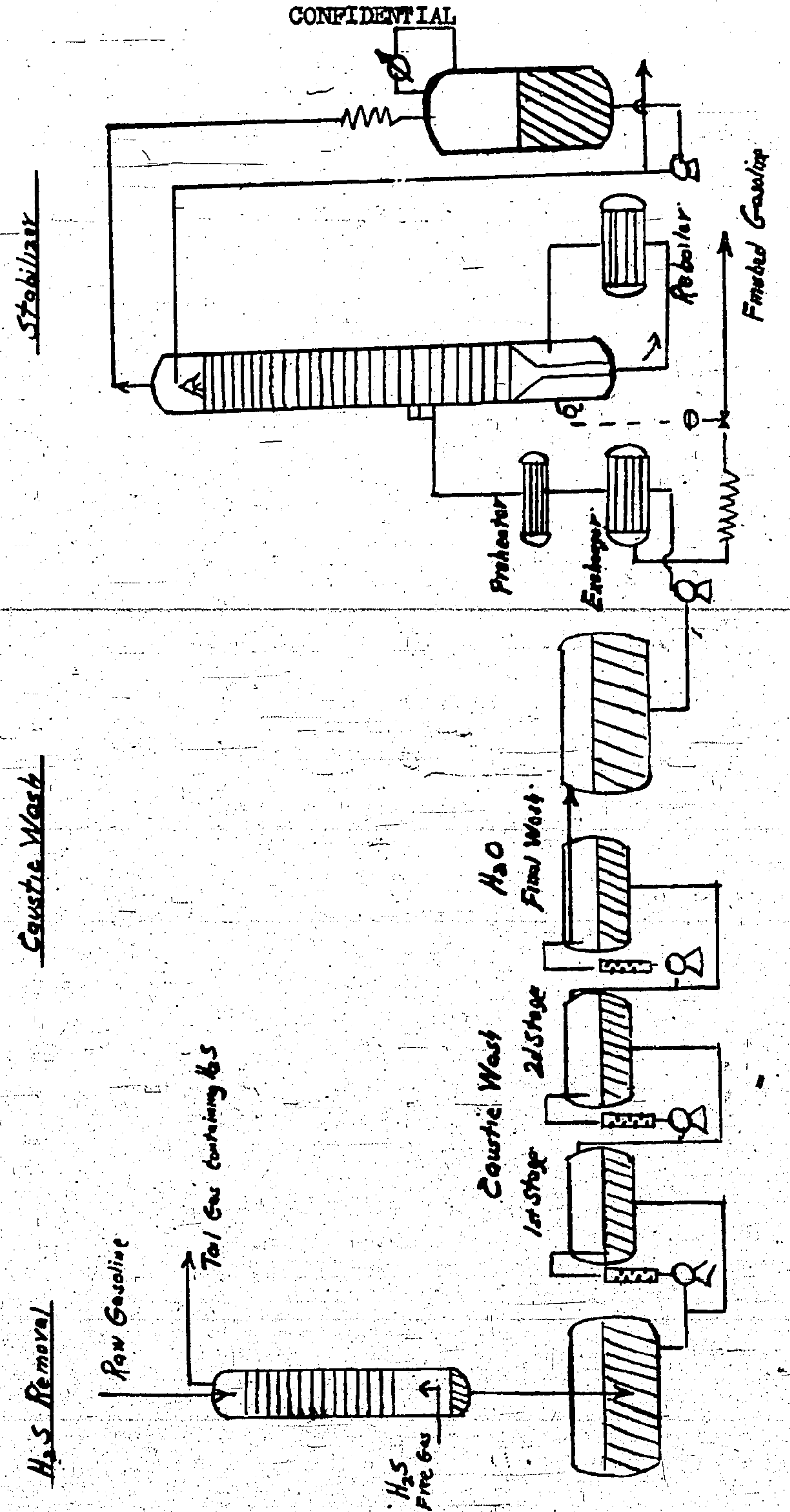


FIG. 25
Caustic Wash

VIII. ALKALINE WASH AND STABILIZATION. (Cont'd.)

OPERATING CONDITIONS (Cont'd.)

Reflux ratio	1 - 8
Operating pressure	5 - 7
Head temperature	45 - 55°
Boiler temperature	150 - 165°
18 atm. steam consumption	0.18 ton/ton inlet against 0.15 ton/ton when based on outlet top C_3 gas

ANALYSIS OF LIQUID TOP PRODUCTS

	Mol %
C_2	nil
C_3	10.
iC_4	45.
nC_4	45.
C_5	nil

ANALYSIS OF HEAD GASES

C_1	2.0
C_2	8.0
C_3	28.0
iC_4	28.0
nC_4	25.0
C_5	8.0

IX. HYGAS PROCESSING.

(a) The outlet gases from the hydrogenation are freed from NH_3, CO_2 and H_2S in the Hygas unit. The gases rich in hydrocarbons are processed for gasoline, and the lean gas is sent to the cracking plant and the utility mains. The apparatus consists of an alkazid unit for removal of H_2S or $H_2S + CO_2$, an oil absorption unit for removing higher hydrocarbons, an organic sulfur converter, a NaOH absorber for final removal of H_2S and CO_2, and a gasoline plant for liquifaction and fractionation of the liquid products.

IX. HYGAS PROCESSING. (Cont'd.)

(b) The outlet gases from the hydrogenation consist of:

(1) Flashed gas from the coal and gasoline chamber effluent product;
(2) Flashed gas from the recycled gas wash;
(3) Flashed gas from the mud of the coal stalls;
(4) Outlet gas from the coal and gasoline stripper distillation;
(5) Outlet gas from the stabilizer;
(6) Low temperature coke oven gas from the residue desulfurization.

(c) The flash from the liquid product is reprocessed; the sludge removal gas and the low temperature coke oven gas are sent to the utility mains or used directly for fuel.

(d) The liquid product and the absorption oil from the high pressure process are degassified by a multi-stage pressure breakdown, and then separated into rich and lean fractions. The individual steps are:

(1) Flashing to 30 - 40 atmospheres: coal and gasoline and washed gas;
(2) Flashing to about 0.1 atmospheres: coal and gasoline and washed lean gas;
(3) Heating at atmospheric pressure in the stripper: A and B distillation gas;
(4) Heating under pressure in the stabilizer: Stabilizer gas.

Amonia is removed from the lean gas by a water wash, H_2S is removed with alkazid, and the gas is sent to the cracking plant or to fuel.

(e) The gasoline rich gas is freed from sulfur with alkazid caustic and further purified together with the pure coal rich gas. The mixture of the two gases treated with NaOH for removal of residual CO_2 and H_2S, and then largely liquified as mixed rich gas in the gasoline plant. The residue gas from the gasoline is sent to the cracking unit.

(f) Amonnia Absorption.

Rich and lean gas must be practically free of ammonia, or else ammonium becarbonate is formed in the presence of CO_2 and water, which plugs the lines. The outlet power gas can contain only traces of amonia. The ammonia absorption results partially from condensation in the gas lines and partially from complete washing with process water, where the water is circulated to a predetermined concentration of NH_3. By adding fresh water and drawing off used water the NH_3 in the cycle is kept low. Condensate or process water previously freed from calcium must be used.

(g) Lean Gas.

In the lines so much condensation from the purified lean gas occurs in the absence of an absorber (up to five (5) m^3/hour), that the original NH_3 cintent of about eight hundred (800) milligrams/m^3 is reduced to about thirty (30) milligrams/m^3. The removal of H_2S follows by means of a wash with D, K caustic (principally dimethyl and amino proprionic acid) at fifteen (15) atmospheres pressure. The H_2S content of the new gas is about ten (10) grams/liter and of the pure gas about one hundred (100) milligrams/m^3. The purified gas is sent to the cracking unit. A part of the lean gas is bled off before the alkazid wash and sent to fuel.

(h) Coal Stall Rich Gas.

The coal rich gas consists of the gases from the coal stripper, the absorption oil, and the A-distillation rich gas. The coal stripping rich gas is reduced in NH_3 content from forty (40) grams/m^3 to thirty (30) milligrams/m^3 by washing with process water in two thorough steps. The absorption oil rich gas is similarly treated and the NH_3 reduced from two hundred (200) milligrams/m^3 to forty (40) milligrams/m^3. After the NH_3 absorption these two (2) gases are combined with the NH_3 free A-distillation gas and the mixture purified. The coal stall rich gas passes through a three thousand (3,000) m^3 gasometer and is compressed in one stage to five (5) atmospheres. The condensation from the compressed cooled gas is added to the raw gasoline. The compressed gas is carefully washed twice with alkazid caustic (principally ethyl amino proprionic acid) in order to remove

CO_2 and H_2S. The raw gas contains about forty (40) grams/m^3 H_2S and about two hundred eighty (280) grams/m^3 CO_2 which are reduced to about two hundred (200) milligrams/m^3 and one (1) gram/m^3 respectively. The gas is finally given a wash with middle oil or diesel oil to remove heavy hydrocarbons so that they will not cake in the sulfur conversion over iron oxide catalyst at three hundred (300) to four hundred (400) degrees centigrade. Oil absorption and organic sulfur conversion which also operates at five (5) atmospheres have been shown to be unnecessary. After the organic sulfur purification the gas is reduced to 0.3 atmospheres and after purification of the gasoline rich gas is sent to the NaOH absorber.

(i) Gasoline Stall Rich Gas.

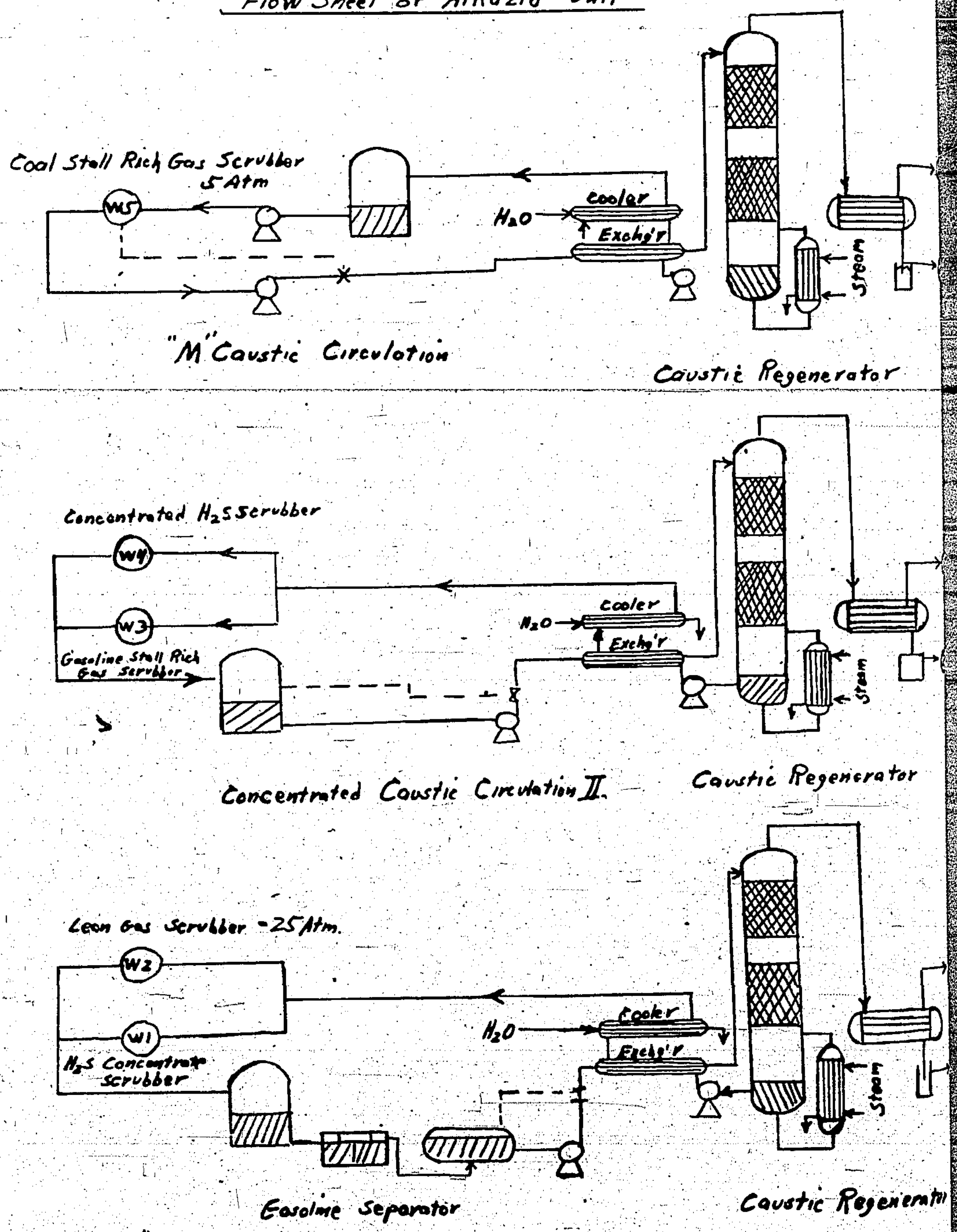
The gasoline rich gas consists of the flash from the liquid product of the primary hydrogenation, the gasoline chamber, and the B-distillation gas. The stabilizer gas is pure and is sent directly to the inlet of the power gas unit. The flash gas from the primary hydrogenation contains about thirty (30) grams/m^3 of NH_3 and must be processed in a packed absorber with process water to reduce this content to about thirty (30) milligrams/m^3. The gas from the gasoline chamber is ammonia free. The two gases from the B-distillation gas are washed with water after the NH_3 removal. The gasoline rich gas passes through a two thousand (2,000) m^3 gasometer and finally after compression to 0.4 atmospheres by a turbe-blower goes to an alkazid wash. The condensate from the compression is added to the raw gasoline in a bell-bottomed absorber; the H_2S is removed with alkazid Dik-caustic. The H_2S is reduced from about forty (40) grams/m^3 to about three hundred (300) milligrams/m^3. The gasoline rich gas and coal rich gas are then purified together in an NaOH absorber, removing the last traces of H_2S and CO_2, after which it is sent to the power gas plant. The H_2S content of the mixed rich gas is 0.0 milligrams/m^3.

(j) Alkazid Plant. (Fig. 26)

The so-called alkazid caustic has the property of almost completely absorbing H_2S or $H_2S + CO_2$ at twenty (20) degrees to forty (40) degrees centigrade, and of releasing it at about

FIG. 26

Flow Sheet of Alkazid Unit



① = Gas containing H_2S
② = Condensate " "

one hundred degrees centigrade. The apparatus consists of an absorber, a regenerating column, coolers, heat exchangers and pumps. The lean gas, the gas from outlet water recovery, and the gasoline-rich gas are washed in two (2) bell-botton absorbers with D.K.-caustic (principally dimethyl amino acetic acid) the co-rich gas with M-caustic (principally methyl amino propri-onic acid) in two (2) packed columns. The saturated caustic goes to intermediate storage in the Dik-caustic system and from there is pumped through a heat exchanger counter current to the hot regenerated caustic from the regenerator column. In the lean gas washing system there is a gosoline separator between the heat exchanger and the tank to remove condensate up to seventy (70) degrees centigrade. The column is filled with rings and the kettle is fed with direct and indirect steam. The steam outlet connection leads to the side of the circulating kettle, which contains the tube bundle for indirect heating. The division between direct and indirect steam is determined by the specific gravity of the caustics, which must be controlled to prevent corrosion. After degasification to an economic degree, where the gas is cooled in a water cooler attached to the column, the hot caustic is pumped from the kettle through a heat exchanger counter current to the cold saturated caustic, through a water cooler, back to the absorber. For the twenty-five (25) atmosphere absorber there is a booster pump between the cooler and the absorber. In the coal rich gas system there is an intermediate storage tank for saturated caustic like the one for regenerated caustic after the cooler. From this tank the caustic is pumped to the five (5) atmosphere absorber.

(k) Production of High Purity Hydrogen Sulfide.

The gas from the stripper column of the lean gas absorber system contains about fifty (50) percent H_2S and fifty (50) percent CO_2, which results from the large CO_2 excess in the lean gas and which will be absorbed in caustic. The gas will be washed once with Dik-caustic in a bell-bottomed absorber to concentrate the H_2S. The saturated caustic from this wash will be regenerated together with the CO_2 free saturated caustic from the gasoline rich gas absorption. The stripper gas from this caustic cycle contains about ninety (90) percent H_2S. The stripper gas from the coal rich gas M-caustic contains about ten (10) percent H_2S

and ninety (90) percent CO_2. The ratio of H_2S and CO_2 approximately corresponds to that of the lean gas and therefore will be concentrated in a washer, and the caustic will be regenerated with the caustic from the lean gas system. In this concentrating washer the exit gas from enriching washer (20% H_2S), the outlet gas from the waste water recovery (5-8% H_2S), and the outlet gas from the middle oil desulfurization unit (10% H_2S) are washed for the production of high purity H_2S.

(l) Report of Operation of the Alkazid Unit.

The alkazid unit was put into operation in June 1943. No basic difficulties have been experienced. Wet aluminum corrosion occurred only on heating tubes of a column, and appears as pitting. Iron corrosion occurred to a greater extent on the water side of numerous coolers. Strong benzin condensation in the lean gas washer caused considerable trouble by pump abrasion and by carry over in the caustic to the intermediate storage and the columns. A benzin separator remedied this in normal operation. By degassing the caustic in the columns, a small but continual caustic loss occurred. A lower caustic inlet temperature and a layer of tower packing above the inlet corrected this condition. Increased oxygen content reduced the effectiveness of the M-caustic, and regeneration with potash made no improvement. The efficiency of the caustic cooler and H_2S condensers was often impaired by water side fouling.

(m) Power Gas Plant.

The power gas plant serves to condense the liquifiable gases and fractionates them into C_3, C_4 and gasoline fractions. The gas mixture containing about fifteen (15) percent CH_4, fifteen (15) percent C_2, H_6, twenty-four (24) percent C_3H_8, twenty-eight (28) percent C_4H_{10}, nine (9) percent C_5 and heavier and ten (10) percent residue gas (H_2O, CO, N_2) is compressed to twenty-five (25) atmospheres in a two stage machine, and the entrained lube oil is then removed with an oil wash. The oil wash consists of a small packed absorber over which liquid product is trickled. The lube oil is knocked down to the kettle. The gas from the oil wash is cooled to about sixty (60) degrees with water. Gas and condensate together are sprayed into column 1. In column 1 the

residue gas, CH_4, C_2, H_6, C_3H_8 goes overhead and the C_4 and heavier fraction is taken from the kettle. The kettle product is fed to column 3 through a heat exchanger, where C_4H_{10} is removed overhead and the remainder from the kettle. This column operates at seven (7) atmospheres, and the pressure is controlled by the water flow to the C_4H_{10} condenser. Part of the condensate is used to reflux column 3 and part is removed as product through a cooler and a displacement meter to intermediate storage. The kettle product is passed through a cooler, a displacement meter, and is added to raw gasoline. The heads from column 1 are partially condensed and are used to reflux the column. The residue gas goes through a NH_3-water wash, a silica gel drier, and an NH_3 refrigerated cooler at $10^{\circ}C$. The condensate and residue gas are sent to a separator, purified, and sprayed into column 2. In this column the residue gas (H_2O, CO, CH_4, C_2H_6) goes overhead and the propane is taken from the kettle. Reflux is obtained with NH_3 refrigeration at -350° and is fed to the top of the column. The propane is removed as product through a cooler and displacement meter to an intermediate tank. The residue gas is expanded to ten (10) atmospheres in the first stage, to five (5) atmospheres in the second stage, and is sent to the cracking plant as power gas residue. Between the first and second stages the gas is sent over a gas heater and finally used for drying the saturated silica gel.

(n) Operation of the Compressor Building.

Because of operating errors and particularly because of flaws in construction a great number of difficulties occurred. By the end of 1942 the coal rich gas compressors were generally satisfactory during their operating time. Both machines were destroyed near the end of 1943 due to the failure of the piston rods, which was traced to faulty construction. Two-stage power gas compressors were substituted as alternates for the Borsig compressors. These had various disadvantages, particularly too weak frames. One machine was strengthened. The refrigerating machine was poorly erected in some respects. Valve plate breakage caused by faulty material was frequent in all machines. One machine sustained damage by failure of lubrication. The rotary blower for Bi-rich gas was often under repair because of the rotor.

(o) Report of Operation of the Power Gas plant.

The power gas plant went into operation in January and April of 1943 without difficulty and has operated without trouble.

IX. HYGAS PROCESSING. (o)(Cont'd.)

The performance guarantees were in no manner met, particularly the A-plant. Principal faults were poor separation in the columns and a poor performance of the ammonia dephlegmator and ammonia precooler. This was partially remedied by increasing the length of column B-1 and exchanging the dephlegmator of column B-2 for two (2) unit coolers, and providing separators and pumps. The Uhde Company of Dortmunde had guaranteed, and to make good it was necessary to build a new plant.

OPERATING RESULTS

Yield		
Nat-gasoline		98.0 %
Butane		93.0 %
Propane		81.0 %
? (illegible) ?		
In nat-gasoline	C_4	5.0 %
	C_5 and higher	95.0 %
In butane	C_3	5.0 % (wt.)
	iso C_4	61.0 % "
	n C_4	33.5 % "
	C_5 and higher	0.5 % "
In propane	C_2	15.0 % "
	C_3	12.5 % "
	C_4	7.5 % "
Power gas	C_2	4.2 % "
	C_3	30.0 % "
	C_4	65.4 % "
	C_5 and higher	0.4 % "
Vapor pressure		
± 40°		10.2 atm
± 15°		1.3 atm
Hourly quantities		
Inlet mixed rich gas		4,450. m^3/hr
Nat. gasoline		0.9 tons/hr
Power butane		2.2 tons/hr
Power propane		1.1 tons/hr
Residue gas		1,500. m^3/hr
Mixed rich gas to cracking plant		550. m^3/hr

X. SULFUR PROBLEM.

(a) In order to remove asphalts in a satisfactory manner, a difinite partial pressure of H_2S is necessary in the recycle gas stream to the coal stalls. Due to the low sulfur content of the raw coal (0.5 to 0.8 percent) and the diluent oil (0.3 percent), and also due to the heavy sulfur requirement of the iron oxide catalyst, the coal paste at Wesseling has elementary sulfur injected into it amounting to 1.25 to 1.5 percent of solids in the coal paste.

(b) After the conversion in the stall, the sulfur becomes bound chemically with: the iron of the catalyst, the tail gas, the discharge water of the effluent, the distillation and amonia wash, and the tail gas. It is also to be found in the caustic wash of the rich gas and gasoline. The sulfur in the coking residue and the caustic is lost, while that in the discharge water and tail gas is recovered to a large extent. The discharge water is freed from H_2S by gasification with CO_2.

(c) In the alkazid unit, the tail gas feed for the gas cracking unit and the rich gas for the fuel gas unit are concentrated and stripped of H_2S. The concentrated H_2S (approximately ninety (90) percent) is used for sulfurizing the feed to the gasoline stalls, hence the sulfur soluble in the feed eventually turns up in the crude gasoline, in the gasoline stall rich gas, and in the discharge water. The non-absorbed sulfur is taken back into the alkazid unit. The sulfur losses can be summed up as follows:

(1) Coking residue;
(2) In the purified gas in the alkazid unit due to incomplete washing;
(3) The discharge water from the gasification;
(4) The caustic wash from the raw gasoline.

(d) In the alkazid unit, either H_2S or $H_2S + CO_2$ are absorbed alkazid caustic at room temperature. Upon reheating, the H_2S is stripped out and the caustic used over again. Two kinds of caustic are used, concentrated caustic and M-caustic. The former consists of N-dimethyl amino potassium acetate ($(CH_3)_2-N-CH_2-COOK$) and this is highly selective in absorbing H_2S in contrast to CO_2.

M-caustic is principally N-methyl amino potassium propionate.

$$\begin{array}{c}(CH_3\text{-}CH\text{-}COOK)\\ (\qquad\qquad)\\ (\ NHCH_3\ )\end{array}$$

and this is equally effective for H_2S and CO_2. The removal and concentration of H_2S occurs as follows (Fig. 26). The lean gas is washed in scrubber W2 with concentrated caustic, removing the H_2S as opposed to CO_2. The absorption in the caustic is determined by titration against H_2SO_4 and the quantity of gas determined. One volume of gas to one volume of caustic is known as "One Point Absorption."

(e) The saturated caustic from scrubbers W2a and W2b is regenerated in columns M1 and M2 by open and coil steam. The relative proportion of the two steam quantities is fixed by the specific gravity of the caustic, which must be kept exact, due to corrosion on one hand and volume contraction on the other.

(f) With regeneration, the H_2S concentration is approximately fifty (50) percent and can be scrubbed within scrubber W4 once again with concentrated caustic. The caustic from scrubber 4 and also from the gasoline stall rich gas washer, W3a and b, are regenerated together, releasing only the H_2S. This concentrate is approximately ninety (90) percent strength and can be used directly for sulfurizing the middle oil.

(g) The gas discharged from scrubber 4 contains approximately twenty (20) percent H_2S. The gas discharged from scrubber W1 goes through the after burning unit in which H_2S is converted into SO_2. The final H_2S content in the SO_2 is very small.

(h) The rich gas from the coal stalls is washed in scrubber W5 with M-caustic which absorbs equally well H_2S and CO_2. The saturated caustic is regenerated in the stripping columns M1 and M2. Due to the high CO_2 content in this gas the H_2S amounts to only fifteen (15) percent and must be concentrated in scrubber W1.

IMPORTANT FACTS ABOUT THE ALKAZID UNIT

H_2S content	g/cu.met	mg/cu.met	Vol/% H_2S	Quantity cu.met/hr
Lean gas raw	8.7			15,000
Lean gas pure		80		
Coal stall rich gas raw	35.			4,000
Coal stall rich gas pure		221		
Gasoline stall rich gas raw	39.			2,400
Gasoline stall rich gas pure		330		
Blower gas	78.			700
Tail gas W1a/b	4.			2,000
Tail gas D1/2			48	800
Tail gas D2/3			91	120
Tail gas M1/2			18	1,000

	Custic type	Scrub-ber	Feed In-jected cu.met/hr	Recirc. quantity cu./met/hr	Gas cc/cc	Steam direct tons/hr	Steam indirect tons/hr	Sp.Grav. @ 20°C.
	Concent.	W1	32		16			
Fresh	"	W2	22		23			
caustic	"	W3	16		15			
	"	W4	16		18			
	M	W5	64		—			
	Concent.	D1/2		54	4 - 6	4	3	1,155
Regen.	"	D2/3		32	4 - 6	3,5	2,5	1,155
caustic	M	M1/2		64	7 - 10	4	3	1,175

SULFUR BALANCE

Entrance

Elem. Sulfur	350 tons
S in TK4	276
S in oils	22
	648

	Exit
Coking residue	406 tons
Tail gas from after burner	72
S lost in phenol removal	52
Expansion loss	24
S residue in heating gas	18
S residue in mud gas	17
S residue in tail gas from methane cracker	16
S in NH_3 wash	16
S in exhaust water not recoverable	22
	648 tons
S in 6434 feed	183 tons

XI. RAW AND FINISHED PRODUCTS.

(a) The raw materials used in making the final product are pure lignite, outside oil (tar oils, etc.) and petroleum. Seventy-eight (78) percent of the finished product comes from lignite and the rest from oil. The outside oil is partly used as diluent oil, and partly as feed to the cool stall effluent distillation unit, the boiling range, specific gravity, water and solid content, are fixed. All oils containing either water and more than twenty (20) percent middle oil are passed through the A-distillation unit. The remainder is mixed with diluent oil.

(b) Variations in quality of final product can be made in operational changes: reactor throughput, reactor temperature, coal paste injection, amount of catalyst and boiling range of middle and heavy oil.

(c) Properties of Pure Lignite Charge Stock.

The pure Rheinish lignite is easily decomposed chemically. However, the resulting oils are difficult to hydrogenate, requiring the high-pressure seven hundred (700) atmosphere operation in the sump phase. This is apparently due to the high carbon content in the lignite. The lignite seems to be mixed with decayed stumps and plants. The latter did not seriously affect operation after properly sifting the raw coal.

ANALYSIS OF THE LIGNITE

Water content	7.5 %
Ash content	6.0 %
Tar content (Fischer Test)	8.0 %

ULTIMATE ANALYSIS		ASH ANALYSIS	
C	67.76 %	SiO_2	2.9 %
H_2	5.10 %	Fe_2O_3	22.5 %
O_2	25.30 %	Al_2O_3	4.1 %
N_2	1.10 %	CaO	45.1 %
S	0.75 %	MgO	4.9 %
		SO_3	20.1 %

The underlined values play an important part in the process.

(d) CaO Content.

This is converted into $CaCO_3$ by the chemical decomposition of the lignite. This results in salt crust or "caviar" formation leading to serious operating difficulties.

(e) O_2 Content.

Due to its high value, twenty-five (25) percent, it uses up large amounts of hydrogen, two-thirds of it being converted into water in the process.

(f) Sulfur content.

It amounts to only 0.75 percent which is relatively low compared to lignites from middle Germany. Extra sulfur has to be injected to meet the sulfur content requirements of the catalysts.

XI. RAW AND FINISHED PRODUCTS. (Cont'd.)

(g) Outside Oil.

This consisted of shale oil, petroleum residue, hard coal tars, and lignite tars of aliphatic, naphthenic and aromatic character. Variations in the same produced different products. However, these variations were hardly felt in the final product due to the large part of hydrogenated lignite effluent used and the proper operation of the gas phase catalyst. By using too much road asphalt (over five (5) percent) as diluent oil, a highly asphaltic product resulted. Best results from hard coal tar occurred when the resulting middle oil end point was reduced from three hundred fifty (350) degrees to three hundred thirty (330) degrees centigrade.

(h) The final products were:

(1) Ordinary Diesel oil;
(2) Marine Diesel oil;
(3) Cold service Diesel oil for winter campaigns in Russia;
(4) Crude gasoline for the DHD plant of I. G. at Ludwigshaven - prehydrogenation only;
(5) Ordinary aviation gasoline;
(6) Ordinary $C_3 - C_4$ gas for automobiles;
(7) Special aviation $C_3 - C_4$ gas for use in aviation engines.

INSPECTIONS OF PRODUCTS

(1)	Ordinary Diesel oil	
	Sp.gr. @ 15° C	0.8 - 0.885
	Viscosity - Engler @ 20° C	1.1 - 2.6
	Pour point	
	Summer not over	- 10° C
	Winter not over	- 20° C
	Flash point over	21° C
	Cetane No. - not under	35

INSPECTIONS OF PRODUCTS (Cont'd.)

(2)	Marine diesel oil	
	Sp. gr. @ 15° C	0.840 - 0.870
	Viscosity - Engler @ 20° C	1.2 - 2.6
	Cloud point	± 0° C
	Flash point not under	± 55° C
	Cetane No. - not under	35
(3)	Diesel oil (Russian quality)	
	Viscosity, Engler @ 20° C - not under	1.1
	Cloud point, under	- 30° C
	Pour point, under	- 35° C
	Flash point, over	21° C
(4)	Gasoline for D.H.D. unit	
	F.B.P.	165° C
(5)	Ordinary aviation gasoline	
	Sp. gr. @ 15° C	0.725 - 0.750
	I.B.P. not under	40° C
	10 % min. vol. 10	70° C
	50 % " " 10	100° C
	90 %	145° C
	F.B.P.	165° C
	Max. R.V.P.	0.5 @ 37.8° C
	API max.	52° C
	Octane No.	
	Motor method - clear	- 70
	+ 0.09 vol. T.E.L.	87
(6)	Normal $C_3 - C_4$ gas for automobiles	
	Vapor pressure	
	1.4 - 31.8 - minimum	0.7 atm. @ 0 °C
	- maximum	16.7 atm. @ 40°C
	1.9 - 31.3 - minimum	1.5 atm. @ -15°C
	- maximum	16.7 atm. @ +40°C
	H_2S - not over	0.2 mg/cu.met.
	Organic sulfur - not over	250
	Elemental sulfur	negative
	Mercaptan (Doctor test)	"
	NH_3	"

XI. RAW AND FINISHED PRODUCTS. (Cont'd.)

(7) Aviation $C_3 - C_4$ gas for aviation motors

C_4	70 - 80 %
C_5	20 - 30 %
Max. H_2 content	5 %

Vapor pressure not over 2 atm @ 0° C
Other specs. as per normal power gas requirements
All the above specs. were met. only the winter quality Russian Diesel oil failed to meet its cloud point test.

XII. STEELS IN THE HYDROGENATION UNIT.

(a) The great technical development of the high pressure hydrogenation process would be impossible without the existence of suitable steels for the high pressure apparatus. The steels must be safe in regards to the high pressure requirements, but in addition the apparatus must not be too heavy and unmanageable. It must in addition be resistant against H_2 and the corrosive influx of the products of hydrogenation, especially of the contained H_2S. In zones of high temperatures, they must be able to meet the special properties.

(b) These requirements are met only by alloy steels, which have been given a special treatment. Especially suitable are the V_2A steels, which are mixtures containing over twenty-five (25) percent Ni and Cr. The faults of these aloy steels forced the development of other steels with lower impurities. It is to the credit of the I. G. Farbenindustrie that they produced such special steels, having strength meeting the requirements of the high pressure hydrogenation with a metal of lower alloy content. Through a specific heat treatment, the steels are given a structure, which gives them great mechanical strength and high resistance against chemical attack. The treatment consists in prolonging the heating in the austenitic region, quenching and short tempering. The heating and the quenching make the steels hard, but brittle. Through the tempering the hardness and brittleness again will be partially raised. The steels receive by this means a sufficient toughness for working safety.

(c) The added tabular groupings give an insight into the hitherto developed steels and their distribution for individual

HIGH PRESSURE 325 Atm

Applications of the Working Materials

Temp. Stage	Operating Temp. C	Nominal Range	Applicable Material for						
			Tubes	Formed Pieces	Blind Flanges	Screw Flanges	Bolts	Nuts	Lenses
I	0-200	6-16 16-160 200	St.45.29 St.35.29	S2	S2	S1	S3 K4Ms K1,K1MS	S3	S2
II Now 200 - 400°C	200-400	6-200	N8A	N8A	N8A	K4MS,K1, K1MS	K4MS,K1, K1V,K1CV K1MS	S3	N5A
II Previously 200-480°C	200-480	6-200	N8	N8	N8	K3,K3CV	K3,K3CV	S3	N5
III Now 400-510°C	400-510	6-200	N9	N9	N9	K3CV K3	K3CV K3	S3	N5A N5
III Previously 480-510°C	480-510	6-45 58-200	N8 N8V,N10	N8 N8V,N10	N8 N8V,N10	K3,K3CV K3,K3CV, K5,K5V	K3,K3CV K5,K5V	S3 K1MS,K3 K1V,K1CV	N8 N8A

HIGH PRESSURE 700 Atm

Applications of the Working Materials

Temp. Stage	Operating Temp. °C	Nominal Range	Applicable Working Materials for Tubes	Formed Pieces	Blind Flanges	Screw Flanges	Bolts	Nuts	Lenses
I	0-200	6-16 24-160 except 135 135	K2M K2	S3 S3	S3 K4MS K1 K1MS	S1 K1 K1MS	S3 K4MS K4MS K6	S3 S3,K1, K1V,K1CV	S2 S3
II Now 200-400°C	200-400	6-45 58-160 except 135 135	N8A	N8A	N8A	K4MS,K1 K1MS K4MS,K1 K1,K3 K3CV	K4MS,K1 K1V,K1MS K4MS,K1,K1V K4MS K6	S3 S3,K1,K1V K1CV	N5A N8 N8A
II Previously 200-420°C	200-420	6-160	N8	N8	N8	K3,K3CV	K3,K3CV	S3	
III Now 400-510°C	400-510	6-16 24-45 58-160	N9 N10,N8V N9	N9,N8V N8V	N9,N8V N8V	K3CV K5,K5V K7	K3CV K5,K5V K7	S3 K3,K1V K1CV K1MS	N5A N8
III Previously 420-510°C	420-510	6-160	N10W	N10	N10	K7 K5,K5V	K7 K5,K5V	K1MS K3,K1CV	N8A

working purposes. An attached formulation shows the timely development of the steels, their composition, strength properties, the methods of their testing, as well as the art of the heat treatment. First named are the steels, which could be employed for the cold parts of the apparatus of the original two hundred (200) atmosphere working pressure apparatus. These are ordinary carbon steels, in part with a low Cr addition. For the hot parts of the apparatus were developed (see in part) the N1 to N8 steels. N1 is the standard steel for the high pressure vessels. For all other purposes, N8 will be employed especially for piping at high temperatures. The principal characteristics of these steels is their Cr content which usually amounted to three (3) percent. The remaining elements are Mo and V, which are used in the amount of 0.5 percent. These steels have the advantage of needing only a local tempering after welding, while the later developed steels must be heated in a furnace.

(d) After the introduction of the higher pressures of seven-hundred atmospheres, the strength qualities of S-steels no longer sufficed for cold piping, one bought steels, which had a Brinell hardness of seventy (70) to eighty (80) instead of the hitherte fifty (50) and sixty (60). Such steels would be made under the designation K-steels. They differed from the S-steels through a Cr content of about one (1) percent and a lower Mo composition.

(e) For the hot seven hundred (700) atmosphere apparatus the N8A to N10 and some K-steels were used. Of these K5 and K7 are outstanding. All of these steels contain V. Another characteristic is the somewhat higher C content of the K-steels.

(f) The best developed steel for high temperatures is the N10 steel with a creep strength of sixteen (16) at five hundred fifty (550) degrees centigrade. It is employed for the most stressed tube piping and bends of the seven hundred (700) atmosphere apparatus, expecially for the hot preheator tubes.

(g) The lack of tungsten and molybdenum forced the preparation of substitute steels, such as the N8A, N8V, N9, K1CV, K_3CV steels. In these, the contents of V and Mn was increased.

XII. STEELS IN THE HYDROGENATION UNIT. (Cont'd.)

(h) When chrome also became scarce, Si was added. Thus originated the seven hundred (700) atmosphere steels K1MS and K4MS. Al of the last named steels could not be used under normal conditions. The added summary about the availability of the material for the separate apparatus of high pressures are presented to help installation men. The adjacent table "Material summary for high pressure 325 and 700 atmospheres" gives the classification indicating the steels, that would be available for the duration of the war.

THE CONSTRUCTION STEELS IN THE WESSELING HIGH PRESSURE WORKS

For 325 atmospheres	
High pressure hollow vessels	N1
Cold tubes	S2
Hot tubes	N8, N8A, N10
Gas preheater	N8, N8V
Electric preheater	N8, N10
Formed pieces	S2, N8, N8A, N9, N10
Lenses	S2, N5, N5A, N8
Flanges	S1, K1, K3, K3CS, K4MS
Bolts	S3, K1, K3, K3MS, K5

For 700 atmospheres	
High pressure hollow vessels	N1
Cold tubes	K2
Hot tubes	N8, N8A, N10
Gas preheaters	N8, N8V, N9, N10
Formed pieces	N8, N8A, N9, N10
Lenses	S3, N5, N5A, N5C, N8
Flanges	K1, K3, K3CV, K5, K4MS
Bolts	S3, K1, K3, K3CV, K4MS, K5, K6, K7

(i) The preceding expose of the correct application of the steels is a reliable control for average strength values. The minutest detail must be watched, since the improper application of the smallest parts of the apparatus such as bolts and lens packings can cause trouble.

(j) The supervision starts with the analyses. There are two methods available: the quantitative complete analysis; and the rapid, or so-called spot method. The latter consists in a

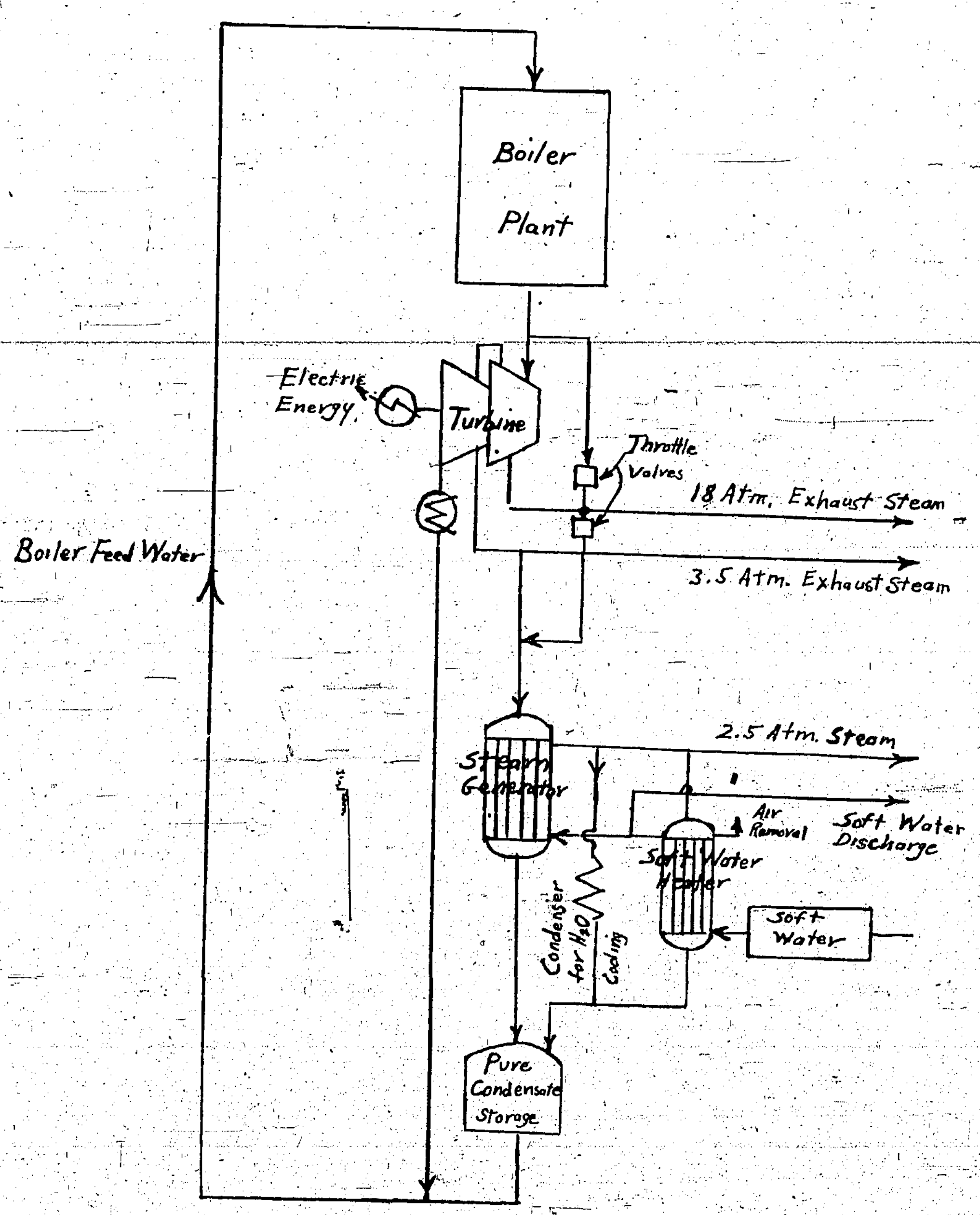
STEELS FOR HYDROGENATION I.G. QUALITY

C O N F I D E N T I A L

Number	Alloys	Treatment Conditions	C *	Mn	Si	Cr	W	Mo	V	Strength Kg/mm²	Min yield point kg/mm² @ °C.				Min Elongation %	Notch Toughness mKg/cm²	Min Creep Strength Kg/mm² @ °C *					Heat Treatment Forging °C	Heating °C	Hardening °C	Cooling Medium	Tempering °C
											20	300	350	400			400	450	500	550						
S1	C	Normal	0,28-0,34	0,60-0,80	0,20-0,35					50-60	27				22	6						1050 - 850	700	870	Air	
S2	C	"	0,15-0,23	0,45-0,60	0,20-0,35					40-48	-				26	10						1100 - 850	690	900	"	600 - 650
S3	Cr	"	0,20-0,25	0,85-0,95Max	0,30	0,30-0,50				50-60	30				26	8						1100 - 850	690	850	"	"
[illegible]	C	"	0,18-0,25	1,10-1,30	0,30-0,40					50-60	30				24	6						"	650 - 700	"	"	500 - 650
[illegible]	Cr	"	0,22-0,29	0,85-0,95Max	0,30	0,30-0,50				50-60	30				26	6						1150 - 800	650 - 700	"	"	600 - 650
[illegible]	C	"	"	"	"					50-60	30				24	6						1050 - 800	"	"	"	500 - 550
E1	Cr Mo	Oil Temper	0,20-0,30	0,40-0,60	0,25-0,35	2,50-3,00		0,20-0,25		65-77	45	35			15	6						1100 - 850	700 - 750	900 - 950	"	650 - 700
E5	Cr Mo	Air "	0,12 Max	0,30-0,50	0,20-0,40	"		0,2[illegible]-0,30		50-60	30				22 lg	maximum						1050 - 850	750	"	"	700 - 730
E5A	CrV	" "	"	0,40-0,60	0,25-0,35	"			0,10-0,20	50-60	25				22 lg	10						1100 - 850	"	980 - 1000	"/oil	"
E5B	Cr	" "	"	0,30-0,50	"	"				50-60	-				22 lg	maximum						1050 - 850	"	920 - 950	"	"
E5C	CrV	" "	0,17-0,20	"	"	"			0,10-0,20	50-60/55-65	30-35		20-27		20-17-18	8 6						1100 - 850	"	980 - 1000	"	"
[illegible]	CrMoV	" "	"	"	"	5,5 Min.		0,35-0,45	0,10-0,20	60-75	35				18	maximum						"	"	960 - 970	Air	680 - 750
[illegible]	CrWMoV	Oil "	"	0,30-0,40	"	2,50-3,00	0,40-0,60	0,40-0,60	0,05-0,15	65-80	45			33	18	8	33	20	10			1100 - 900	700 - 720	950 - 970	oil	680 - 730
[illegible]	CrV	" "	0,20-0,25	0,60-0,80	"	"			0,25-0,35	70-85	50			33	16	8,1/5 qu	33					"	"	950 - 980	"	680 - 700
[illegible]	CrMoV	Air "	0,18-0,23	0,40-0,60	"	"		0,40-0,60	0,40-0,50	80-95	55-50				16	10,6 - 8,5			17	12	7,5	1050 - 850	"	1000 - 1030	"	660 - 680
E9	CrMoV	" " "	0,19-0,24	"	"	3,00-3,50		0,20-0,30	0,45-0,55	80-95 75-90	55-50				16	10,6 - 8,5			17			"	"	1000 - 1030	OilorAir	660 - 660
E10	CrWMoV	" " "	"	0,30-0,40	"	2,50-3,00	0,35-0,45	0,35-0,45	0,70-0,85	80-95 70-85	55-50				14	10,6 - 8,5		25	20	16	11	1100 - 900	700 - 740	1020 - 1050	"	690 - 730
E12A	CrWMoV	" " "	"	"	"	"	0,20-0,30	0,25-0,35	0,80-0,90	80-95 70-85	55-50				14	10,6 - 8,5		25	20	16	11	"	"	"	"	690 - 730
K1	Cr	Water "	0,28-0,33	1,00-1,30	0,50-0,80	1,00-130				70-80	50				16 lg 13qu	7 lg 6 qu						1050 - 850	700 - 720	830 - 850	Water	620 - 660
K1CV	Cr	" "	0,2			1,0			0,2	70-85	50	38	34	30	17	8	22	15	8			"	700 - 730	950	Oil	600 - 650
[illegible]	MnSi	" "	0,25	1,2	1					70-85	50				16	6						"	"	"	"	"
K2	CrMo	Oil-Air "	0,23-0,28	0,60-0,90Max	0,40	0,90-1,10		0,20-0,25		60-70	38				15	maximum						1100 - 860	"	920	OilorAir	"
K2A	Cr	" "	"	0,60-0,80	0,20-0,30	0,90-1,10				60-70	38				15	maximum						"	700 - 720	"	"	"
[illegible]	Mo	" "	"	1,2 -1,5	0,4					55-60	40				18	maximum						"	"	"	Oil	600 - 660
K3	CrMo	Water "	0,25-0,30	0,50-0,70	0,20-0,30	1,0 -1,2		0,14-0,20		70-80	50				16	8						"	"	930 - 950	Water	640 - 660
K3CV	Cr	" "	0,2	0,7		1,2			0,25	70-80	50				17	8	30	20	10			1050 - 850	700 - 730	950	Oil	600 - 650
[illegible]	MnSi	" "	0,33-0,40	1,1 -1,4	1,1 -1,6					80-90	60	45		40	14	6	15	10	5			1100 - 850	680 - 720	850	"	620
K5	CrMoV	" Oil "	0,20-0,28	0,60-0,80	0,25-0,40	1,3 -1,6		0,50-0,60	0,30-0,40	80-95	65	60	55	50	16	8	45	36	26	8		1100 - 900	700 - 750	980 - 1000	" or Wat.	650 - 680
K6	----	" " "	0,4 -0,5			1			0,2	90	60	45			14	6						1050 - 850	700 - 720	950	Oil	600 - 650
K7	CrMn	" " "	0,3	1,0		1,5			1,2	80-95	60				16	8 - 6	40	30	20	10		"	700 - 720	1000-1030	" orAir	660 - 680

* S X 10-70 / 25 - 35 loaded hours.

FIG. 27


Boiler
Plant
Electric
Energy.
Turbine
Throttle
Valves
18 Atm. Exhaust Steam
3.5 Atm. Exhaust Steam
Boiler Feed Water
2.5 Atm. Steam
Steam
Generator
Air
Removal
Soft Water
Discharge
Soft Water
Heater
Soft
Water
Condenser
for H_2O
Cooling
Pure
Condensate
Storage

XII. STEELS IN THE HYDROGENATION UNIT. (Cont'd.)

moistening of the piece under observation with H_2SO_4, HNO_3, or HCl, and observing the formation of colored spots. In conclusion, all installed apparatus was tested first by the quantitive method and second by the spot method.

(k) Another point of interest is the characteristic of the materials. All larger pieces of apparatus, formed and tube pieces, are marked with stamps, showing the type, the current application, and tests used to show the correct application and testing. With smaller parts, as lense packing, bolts, etc., the material is recognized through grain or similar ways.

(l) The supervision of the correct installation of single pieces of working apparatus is followed closely by outlines and sketches, on which the technical material data of each construction part is recorded. The lists would be changed after each repair.

(m) It should be stressed that the nearly four (4) years of plant operation not a single working stoppage occurred through material failure or incorrect material application.

C. UTILITY PROCESSES.

I. THE POWER PLANT.

(a) The power plant supplies the factory with steam, power, soft water, and condensate (Fig. 27). The maximum demands of each of the above quantities is as follows:

(1) Electric energy - 80,000 kilowatt;
(2) Demand of 18 atmospheres steam - 25 tons per hour;
(3) Demand of 3.5 atmospheres steam - 30 tons per hour;
(4) Demand of 2.5 atmospheres steam - 180 tons per hour;
(5) Feed water at 185° C - 50 tons per hour;
(6) Pure condensate 50 - 80° C - 25 tons per hour.

(b) From the above steam quantities, one hundred (100) tons per hour of condensate are returned to the power plant.

I. THE POWER PLANT. (Cont'd.)

(c) The power plant contains the following units:

(1) Boiler house with coal conveyors and preparation;
(2) Turbo-generators, low pressure steam generator, water purification and pump unit;
(3) Electric control station.

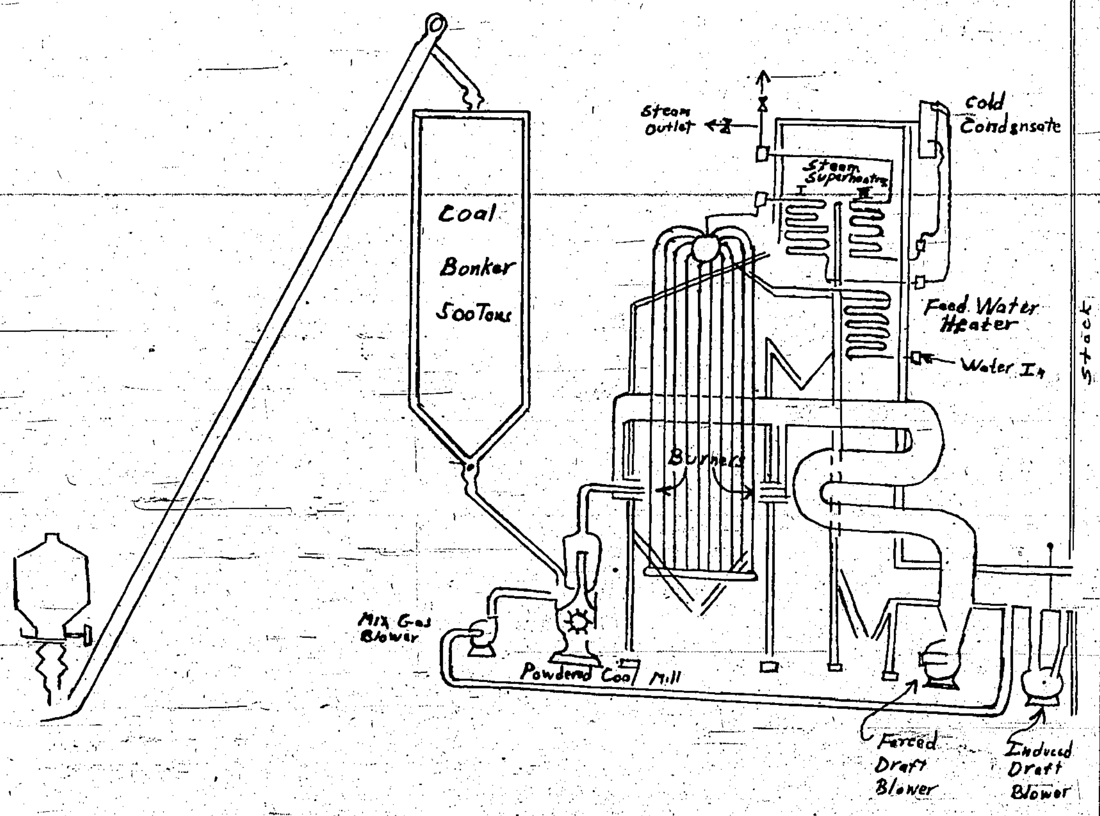
(d) In the boiler house high pressure steam at seventy (70) atmospheres pressure is produced. This is run through turbines, producing electric energy in three (3) stages: at eighteen (18), 3.5, and 0.4 atmospheres. The largest quantity is expanded to 3.5 atmospheres whence it goes to an exchanger-generator. By indirect exchange the 3.5 atmosphere steam in contact with 2.5 atmosphere soft water converts the latter into the steam. In this way the total condensate from the turbines is saved for boiler use and is not contaminated with other water streams. The loss and make up of other steam takes place in distillation unit.

(e) The 2.5 atmosphere steam is made from soft water which is made from treated plant water. This is done by decarbonization through milk of lime, Permutit softener, and a heating of above one hundred ten (110) degrees centigrade in order to remove the dissolved oxygen.

(f) In order to care for variations in electric load the turbines were equipped with condensers which allow for increasing the power from the turbines with the same boiler capacity.

(g) The boiler house (Fig. 28) is equipped with five (5) boilers, each capable of producing sixty-four (64) to eighty (80) tons of steam per hour at eighty (80) atmospheres and five hundred (500) degrees centigrade. Four (4) more boilers were almost completed. The boilers are of the flash type (one drum) with integral feed water heater and steam superheater. They also have air preheaters and pulverized coal burners. The temperatures are controlled by Askania regulators, which inject condensate into the superheated steam flow to maintain five hundred (500) degrees centigrade. Boiler feed water is fed through two (2) types of pumps, electric or steam turbine driven. In order to remove scale forming components, a constant blow down of two (2) percent is maintained.

Fig. 28
Boiler House



I. THE POWER PLANT. (Cont'd.)

(h) The fuel is dried lignite having fifteen (15) percent water content. Also, provision is on hand to use briquette fines and semi-coke dust from the gas plant, as well as excess tail gas from the hydrogenation unit. The coal is transported in special railway cars, in an atmosphere of CO_2. Each boiler is equipped with five (5) pulverizing mills. The combustion gases are removed from the boilers at one hundred eighty (180) to two hundred (200) degrees centigrade through a smoke stack one hundred (100) meters high whose natural draft is augmented by an induced draft fan. The expansion of the boiler house called for a second stack. The ashes are removed from the combustion chamber by a water slurry and pumped into a catch basin.

(i) The original plan called for four (4) steam turbines with condensats. Later two (2) back pressure machines were installed. The turbines discharged ninety-five (95) tons per hour of exhaust, of which thirty-two (32) tons per hour passes through the condensers. In straight condensing operation, a maximum of eight thousand (8,000) kilowatts can be produced. A maximum load requires ninety-five (95) tons per hour of steam which amounts to eighty (80) tons per hour of exhaust at 3.5 atmospheres. Under such operations thirteen thousand (13,000) kilowatts can be produced. For generating one thousand (1,000) kilowatts, twenty (20) tons of steam at eighteen (18) atmospheres or seventeen (17) tons of steam at 3.5 atmospheres or four (4) tons of condensate are required. The generators are so built that when loaded to thirteen thousand (13,000) kilowatts, the resulting power factor is 0.8. The voltage of the generators is six thousand (6,000) volts.

(j) Each turbine has three (3) stages. The first expands from forty (40) to eighteen (18) atmospheres and as such produces the eighteen (18) atmospheres plant steam and the superheated steam for the 2.5 atmosphere steam operation. The temperature of the eighteen (18) atmosphere exhaust is held constant at three hundred (300) degrees centigrade by injecting condensate. The second expansion stage produces 3.5 atmosphere steam which also indirectly produces the 2.5 atmosphere steam for the generator. To cool the condensate the compressor cooler water from the gas plant is used. Its temperature is approximately seventy (70) degrees higher than the temperature of the plant water system.

I. THE POWER PLANT. (Cont'd.)

(k) All turbines are equipped with over speed trips which automatically operate above three thousand (3,000) revolutions per minute.

(l) The boiler house can continue to supply plant steam even if the turbines are not in operation by means of the throttle stations which reduce the boiler pressure to the various plant requirements.

(m) In order to convert the 3.5 atmosphere extraction steam from the turbine to 2.5 atmosphere plant steam, six (6) exchanger-type steam generators are used. Each can produce forty (40) tons per hour of 2.5 atmosphere steam, and by raising the pressure from 2.5 to 4.5 on the high pressure side, the out-put can be raised from forty (40) to fifty-five (55) tons per hour.

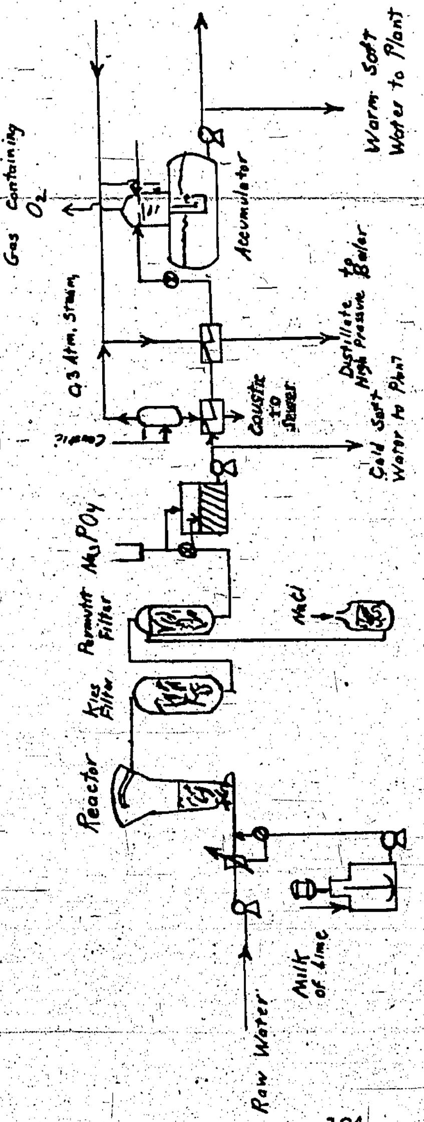
(n) The generator is fed with soft water. In order to remove scale formation, approximately three (3) percent of the soft water is blown down constantly. The soft water is stripped of its oxygen before entering its generator by heating to one hundred five (105) degrees centigrade and then further warmed to one hundred thirty (130) degrees. This heating is done by 2.5 atmosphere steam.

(o) The soft water used in making the 2.5 atmosphere steam is made from plant water which passes through the calcium softener, Permutit unit and de-aerator (Fig. 29).

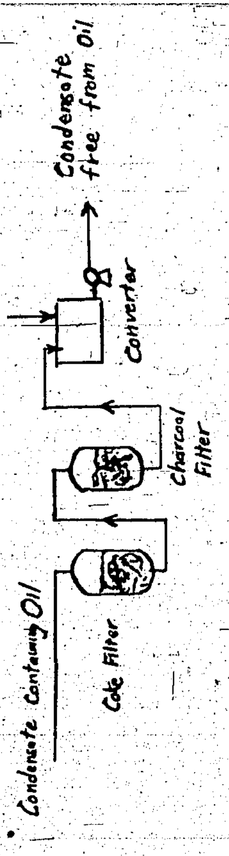
(p) The softening is done by adding milk of lime in the so-called agitation reactor which is filled with small particles of marble. The plant water is quickly mixed with calcium hydroxide which combines with the total CO_2 content and converts the calcium bicarbonate to calcium carbonate. The result calcium carbonate settles out on the marble balls in solid form. When the marble particles become too large they are renewed.

(q) After leaving the agitation reactor the water passes through a Kies filter in which the large particles are removed, and enters the Permutit filter. This is filled with a zeolite which works by a standard well known process. Finally to remove traces of calcium carbonate, Na_3PO_4 is added. The water is then

FIG. 29
Flow Sheet of Water Softener



Flow Sheet of Plant Water Unit



I. THE POWER PLANT. (q)(Cont'd.)

freed of oxygen by heating. The degassed soft water is now taken to the generator and vaporized. In order to prevent scale formation on the tubes, two (2) to three (3) percent of distilled water is constantly added while the same quantity is blown down.

OPERATING DATA 1943

Avg. No. of boilers on stream	4.02
Avg. load boilers	60.5 tons/hr
Avg. No. of turbines on line	3.05
Avg. Load each turbine	11,542 kw.

FUEL DEMAND

Boiler coal	38.45 tons/hr
H_2O content of coal	15.07 %
H_u - Kg. cal/kg	4,692

PRODUCTION
70 Atm. Steam

Production	244.11 tons
Used - boiler feed pumps	1.10
Used - turbines	235.39
Used - reduction - throttle	1.92

TURBINE EXHAUST

Condensate	40.58 tons
3.5 atmosphere steam	170.11 tons
18 atmosphere steam	24.70 tons
Loss	
Total exhaust	235.39 tons

STEAM TO FACTORY

18 atm. steam	14.66 tons
3.5 atm. steam	17.99 tons
2.5 atm. steam	110.88 tons
Condensate - de-oiled	33.46 cu.met.

I. THE POWER PLANT. (Cont'd.)

STEAM TO FACTORY (Cont'd.)

Turbine condensate	6.24 cu.met.
Soft water - worm	90.61 cu.met.
Soft water - cold	26.10 cu.met.

ELECTRIC ENERGY

Produced in plant	34,862	kwh
Outside power	26,615	kwh
Total required	61,477	kwh
Used in power house	3,015	kwh
Used in plant	54,904	kwh

OTHER FACTS

Steam/coal (ton/ton) (cool 4,500 W.E./kg)	5.78	
70 atm. steam demand/kwh	6.752	
Total coal required/kwh	1.217	
Condenser steam energy	10,144	kw
Total power capacity	34,862	kw

II. TREATMENT OF DISCHARGE WATER.

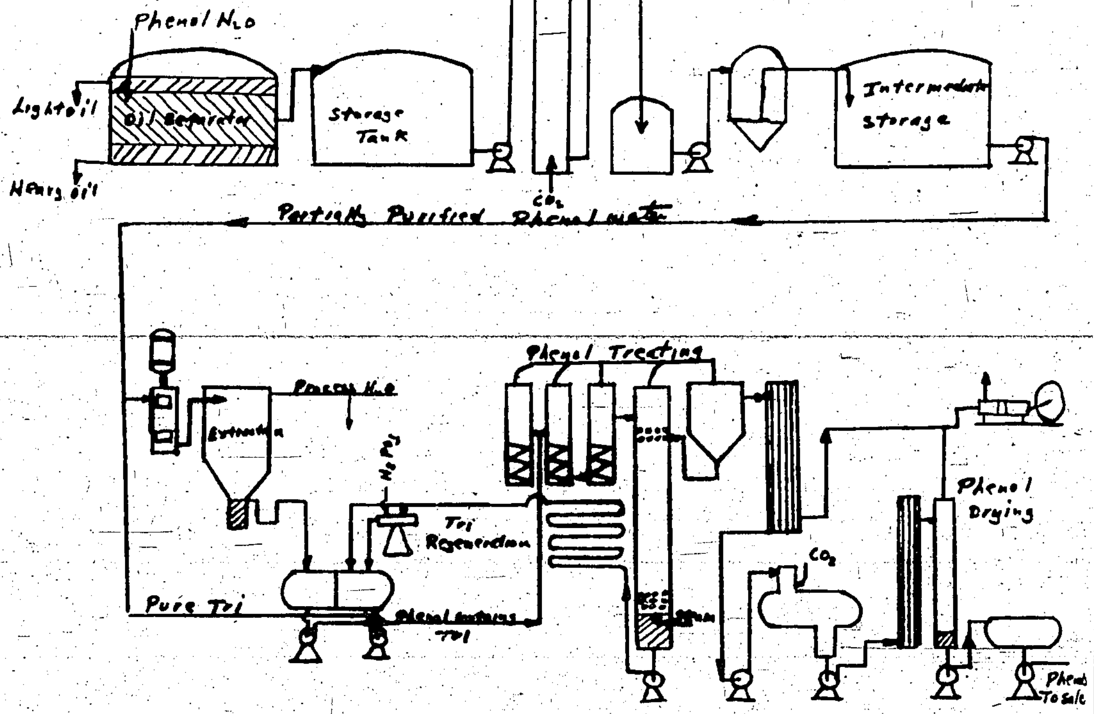
(a) The discharge water treatment removed the largest part of the impurities in the process water, and thereby prevented the pollution of the Rhine. The discharge water streams are:

(1) Water separated from the coal and gasoline stall effluent chamber products in the discharge water tanks;
(2) Water in coal and gasoline effluent which is obtained in the overhead cuts from distillation;
(3) The condensate from the coking residue;
(4) The wash water from the rich gas washer;
(5) The rinse water from all of the oil treating units.

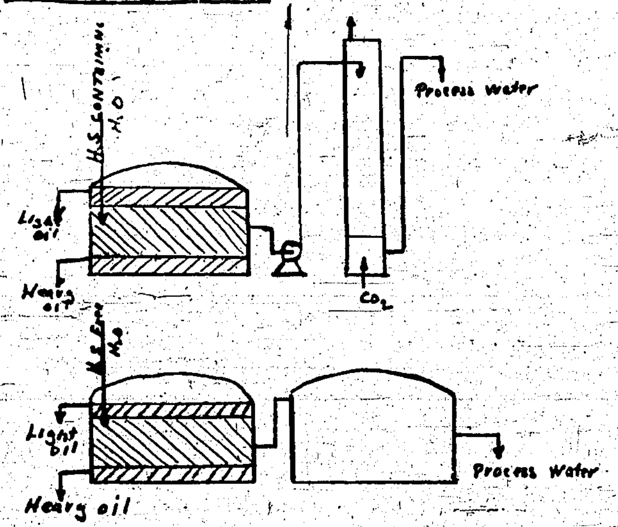
The impurities are: oil, H_2S, and phenols. The discharge waters were divided into two groups for separate treatments. These are the phenol water and dirty water containing oil (Fig. 30).

FIG. 30

Phenol Water



Oil-Containing Water



II. TREATMENT OF DISCHARGE WATER. (Cont'd.)

(b) In the first group belong all waters containing more than one (1) gram/liter of phenol. These are effluent waters of the coal and prehydrogenation chambers, the condensate from the coal effluent and prehydrogenation distillation, and the water produced at the gas plant and the coking kilns.

(c) The second group contains the effluent and distillation water of the gasoline chambers, the wash water from the rich gas washer, and all of the oil-containing rinse waters.

(d) The purification treatment for the oil-containing dirty water is simple. For H_2S-free water it consists in allowing the oil to settle out. For water containing H_2S the oil is first separated and then it is treated with CO_2 to remove most of the dissolved H_2S.

(e) The water containing phenol is given a somewhat extensive purification which consists of extraction with tricresyl phosphate in addition to the oil separation and the CO_2 treatment. The tricresyl phosphate, called "Tri" for short, is a mixture of triphenyl, tricresyl, and trizylenyl phosphates, whose, absorption power depends on the triphenyl phosphate content.

(f) A special characteristic of the Tri process is that the extraction medium boils higher than the dissolved phenols, hence these latter must be distilled out of the absorption medium. This operates so that the phenols produced are very pure, while the impurities remain behind as still residue with the Tri. The Tri must next be purified.

(g) In general the operation of the dephenolating unit is: The phenol-containing water was treated at forty (40) degrees centigrade in three (3) mixing chambers and separators located one behind another. Fresh extraction medium was added periodically. The mixing chambers were constructed with slow moving agitators because the Tri tended to emulsify with water when violently agitated. All together the concentration of Tri was about ten (10) percent of the water. The phenol-containing Tri was vacuum distilled to remove the phenols. The Tri remained as still bottoms. The phenols were condensed, the water separated, and

II. TREATMENT OF DISCHARGE WATER. (g)(Cont'd.)

then vacuum distilled a second time. They were then sold.

(h) The Tri was cooled to about forty (40) degrees centigrade and used for further extraction of phenolic containing water. A fraction of about six (6) percent of the product was centrifuged out to remove impurities, and a second smaller fraction was treated with 0.5 percent phosphoric acid by stirring several hours followed by centrifuging. The dephenolated water was led by a special pipe to the discharge water collection at the Rhine.

(i) Many operating difficulties occurred in the purification of the phenol-containing water from the coal stall effluent, especially when the sludge from the coal chambers overflowed into the effluent. The coal stall effluent could become as dense as the water through the cooling of the latter and then either passed on through the separator or the gravel filter. These disturbances usually led to increased quantity of impurities in the Rhine, since the only remedy was to discharge the water into the Rhine. Once, oil passed through the gravel filter to the extractive vessel and contaminated the tricresylphosphate.

(j) The remedy consisted in preventing the occurrence of the disturbances, for example, as described in the sump phase and coal effluent distillation, the addition of diluent oil to coal effluent produces a deposit. Other points are: the improvement of the apparatus and increased supervision of the high pressure operation, avoiding the addition of emulsion promoting oils, such as heavy oil from the coking residue and unsuitable outside oils to the coal effluent product.

(k) On account of the special properties of the Tri, most higher boiling substances are removed from the water besides the phenols. These materials raised the viscosity of the Tri tenfold inside of three (3) months from the original of three and one-half ($3\frac{1}{2}$) E°. This was in spite of the mechanical and chemical purification of a small bleed. The concentrationof the impurities then were from forty (40) to fifty (50) percent. At the same time the solubility of the phenolies in the Tri, especially phenol itself dropped sharply, so that the phenolic content of the washed water rose from seven hundred (700) milligrams/liter to nearly three (3) grams/liter.

II. TREATMENT OF DISCHARGE WATER. (Cont'd).

(l) Furthermore, the impurities in the Tri tended to cake on the walls of the apparatus. The crust deposits are difficult to remove. It was tried to remove these cakes by circulating hot concentrated NaOH solution, but usually it was necessary to take the apparatus apart.

(m) These difficulties demanded that the Tri be renewed at least every three (3) months. The change was made at suitable intervals to keep the formation of tightly-adhering cokes in check. The product which is drawn off can be regenerated by a high vacuum distillation, which must be used for want of another suitable distillation apparatus. The purified water in the discharge water treatment contained NH_3. This led to the formation of thicker crusts, in particular $CaCO_3$, in the discharge water sewer through its mixing with process water. On this account a pipe line for the impure water was laid to the Rhine in the process water drain.

(n) The disadvantages of the Tri process led to its removal in favor of the "Phenosolven" dephenolation process of Lurgi. Phenosolven is chiefly isobutyl acetate in a mixture of esters. that boils lower than the extracted phenols. It is distilled out of the extraction mixture and therefore remains clean. The impurities remained in the phenolic extract and were sold with them. This change in the process produced a far better phenol yield. The phenol content of the treated water was one hundred (100) to two hundred (200) milligrams/liter.

IMPORTANT OPERATING RESULTS

Quantities

Dirty oil from dirty water	8,900	annual tons
Dirty oil from phenol water	2,600	annual tons
Oil containing dirty water	47	M^3/hr
Phenol water	32	M^3/hr
CO_2 used for injection in the water	600	M^3/hr

II. TREATMENT OF DISCHARGE WATER. (Cont'd.)

H_2S Content

Of the un-gassed phenol water	1.2	g/liter
Of the gassed phenol water	0.2	g/liter
The outlet gases	4	Vol %
Production of phenol oil	950	annual tons

Analysis of Phenol Oils

H_2O	1.2
Mentul oil	2.0
Phenol	31.3
Cresols	43.0
Xylenols	11.5
Rest	11.0

Phenol Content

Of the phenol water	5.2	g/liter
Of the dephenolated water	1.9	g/liter
Phenol production	63.0	%

Tri Content

Of the outlet water	0.086	g/liter
Of the phenol oils	9.5	g/liter

(d) Miscellaneous.

I. MET AND NOT MET GUARANTEES.

(a) Water Gas Unit	Grarantee Figures	Operating figures at the end of 1943
CO + H_2 - content of the water gas	84 %	84 %
Capacity of the generators	5,500 nm^3/hr	5,250 nm^3/hr
Water gas/briquettes	2,030 nm^3/ton	1,900 nm^3/ton
The heating gas required	780 WE/nm^3 water gas	820 WE/nm^3 water gas
Outside steam required		0.12 kg/nm^3

I. MET AND NOT MET GUARANTEES. (a)(Cont'd.)

The guaranteed quantities on gas manufacture were never reached. The deficit amounted to approximately five (5) percent, for which the firm of Pintsch was charged with a penalty. This was particularly due to steam requirements.

(b) Fuel Gas Unit.

The guarantee figures for fuel gas unit were completely met when using H_2S steam injection, and when using CO_2 injection the figures were surpassed. At the same time excess steam in the amount of 0.1 kilograms per cubic meter of heating gas was produced.

(c) Gas Cracking Unit.

No guarantee figures on this unit were set up by the manufacturer, but the operating results exceeded those values anticipated and designed. The most noteworthy points were the high production yields and steam production through waste heat recovery which allowed for steam production equal to the steam requirements.

(d) Hydrogenation.

The hydrogenation plant of I. G. was never covered by a guarantee. However, the operating yields from both unites exceeded the design quantities. The coal requirements were somewhat higher than anticipated. The hydrogen requirement which had been estimated from pilot plant studies of I.G. were originally set too high. The operating differences expected from using a Rhine lignite instead of mid-German lignite were correctly anticipated.

(e) Other Operations.

In all other operations anticipated full loads were either reached or bettered. This is especially true of utility demands. One exception was the C - C unit which did not meet its guarantees; this failure amounted to forty (40) percent of the designed figures. The firm Uhde was going to build another unit in order to meet the original requirements.

(f) Capacity of the Plant.

The production-time curve shows that in 1944 the unit was capable of producing twenty-seven thousand seven hundred (27,700) tons/month of diesel oil and gasoline or two hundred sixty thousand (260,000) tons/year of gasoline and diesel oil. This meant using the complete gas producing capacity. The raw materials were approximately seventy-five (75) percent coal and twenty-five (25) percent oil. From coal alone, two hundred twenty-five thousand (225,000) tons/year of gasoline and diesel oil were produced which was the designed capacity of the unit. The bottle neck was always the hydrogen production. By the addition of outside oil, the capacity of both sump and gas phases rose to the total of two hundred sixty thousand (260,000) tons/year. This use of oil saved hydrogen, as it requires only half as much hydrogen as coal. The sump phase capacity was raised fifty (50) percent by a slight addition to the gas preheater. The gas phase had sufficient reserve capacity for this additional material since the middle oil made from outside oils has less phenols than the coal oil and could even take a throughput thirty-five (35) percent over the designed figure. In using coal, the capacity of the unit is two hundred twenty-five (225,000) tons/year diesel oil and gasoline while with outside oil it is three hundred sixty thousand (360,000) tons/year of the same. To convert the latter quantity to aviation gasoline, a fifth gasoline stall would have to be built.

II. AIR ATTACKS AND COUNTER MEASURES TAKEN.

(a) Protection for Men and Machines.

When the plant was built, many air raid shelters were included in the work. Besides these, a considerable number of walls were built both inside and outside of buildings to protect machines. The turbines and boiler plant were given special protection. All tanks containing inflammable material were specially constructed and had extra heavy dykes and run off ditches.

(b) Blackout.

Special means for blacking out the plant were undertaken, either semi- or complete. Under normal blackout conditions,

special lights kept buildings in semi-darkness, while only the repair shops were kept fully lit. At the approach of enemy planes, the blackout was completed by cutting off the lighting current. Discipline among the workmen during air raids was excellent. No damage was received due to blackout errors.

(c) Air Raid Wardens and Their Work.

Suitable air raid defence was always available. No one shirked his respnonsibility and the posts were always completely manned. The air raid protection procedure was minutely worked out and was constantly being improved. Warning from the outside was conveyed quickly and efficiently to the central station by the Army, with whom good cooperation always existed. The information was relayed to the whole plant by a siren alarm except to the high pressure unit and boiler house which was by telephone, giving the latter instantaneous notice.

(d) Operation Procedure.

The most important detail is the blowing down of the unit. This was done in two stages "Safety Operation" and complete depressuring. The former assumed on receipt of warning that planes were near, while the latter was only resorted to just before or after bombs started to fall. "Safety operation" requires isolating the units in the plant from one another, hence making indirect damage impossible or unlikely. It disconnects the main plant from outside power supply and stops the feed to the gasoline stalls of the gas phase. It also stops the coal paste injection in the sump phase, and substitutes diluent oil for the same. Under extreme conditions even these units can be disconnected from the power plant in a few seconds. The procedure was so well executed that "safety operation" could be carried out in two minutes and complete blow down in another few seconds. Such results were made possible by placing all control valves and power switches in one air raid shelter and at one switchboard in the power house. The same reliability in blowing down the unit existed in resuming operations. Production was completely restored after four (4) to five (5) hours. Hence operations were broken and resumed thirty-one (31) times. The methods were carried out that damage was limited to one gas holder and one small explosion in the gas plant.

II. AIR ATTACKS AND COUNTER MEASURES TAKEN. (Cont'd.)

Loss of Production Due to Enemy Air Raids		Loss of Finished Product
5/29/42	Safety operation (attack on Cologne, loss of outside power - several days)	300 tons
10/15/42	Bombs dropped on gas plant	8,300 "
1/23/43	Safety operation (outside power off)	600 "
2/14/43	Safety operation (outside power off)	250 "
2/23/43	Loss of all outside power	450 "
6/27/43	Blow down complete (1 stall coked up and had to be taken down	700 "
7/4/43	Safety operation with loss of power	300 "
7/9/43	Safety operation with loss of power	100 "
7/30/43	Safety operation with loss of power	150 "
8/12/43	Safety operation with loss of power	150 "
8/13/43	Complete power failure (all stalls coked up despite quick operation)	5,000 "
8/17/43	Safety operation - twice	150 "
8/22/43	Safety operation	100 "
9/10/43	Outside power failure	100 "
9/15/43	Several days outside power lost due to damage to public utility	800 "
10/3/43	Damage to briquette conveyor	20 "
10/14/43	Safety operation	120 "
10/20/43	Safety operation	80 "
10/23/43	Several days lack of briquettes (main water conveyor in mine damaged)	200 "
11/3/43	Safety operation	150 "
11/19/43	Safety operation	150 "
11/27/43	Outside power failure (oil circulating pumps out)	200 "

II. AIR ATTACKS AND COUNTER MEASURES TAKEN. (Cont'd.)

Loss of Production Due to Enemy Air Raids		Loss of Finished Product
11/30/43	Safety operation	150 tons
1/7/44	Safety operation	100 "
1/24/44	Safety operation	150 "
3/4/44	Safety operation	150 "
4/22/44	Safety operation	150 "
5/20/44	Safety operation	100 "
5/24/44	Safety operation	100 "
5/26/44	Safety operation	100 "
5/28/44	Safety operation	100 "
6/6/44	Safety operation	150 "
6/13/44	Safety operation (failure of outside power transformers)	2,080 "
6/22/44	Safety operation and blow down	970 "
6/27/44	Safety operation	250 "
7/10/44	Safety operation	150 "
7/11/44	Safety operation	150 "
		23,220 tons

The total production during this time was 455,356 tons, so that air raids caused a loss of 5.1 percent.

III. AIR ATTACK OF JULY 18, 1944.

(a) In the night of July 18-19, 1944 a heavy air attack was made on the plant. About one thousand (1,000) high explosive bombs, mostly five hundred (500) pound, fell inside the manufacturing plant. On the south west the boiler house was heavily hit.

(b) The account of the attack was as follows: at 12:55 a.m. first alarm, at 1:03 a.m. "safety operations" was sounded and word was passed, "Attack headed for the plant."

(c) 1:07 a.m. full blow down was given after a red flare was dropped over the plant. Seconds later the bombs started to drop. They came in three waves and lasted about twenty (20) minutes.

(d) The attack was made easy for the aviators by the lighted conditions, since the artificial fog was started too late. The reason for not starting the fog is unknown. The manufacturing units were brilliantly lighted up by the flares. Brilliant fires were started after the first bomb attack.

(e) Safety operations and emergency shut down proceeded satisfactorly, despite the short interval of four (4) minutes for doing same and taking air raid shelter.

(f) The same satisfactory conditions obtained for the several men in the power house, who had to remain at their posts as long as possible. Nevertheless, they were in their shelters at the start of the bomb falling. Of the six hundred (600) workmen, there were three (3) deaths. These all occurred through one explosion.

(g) On account of the severity of the attack, the water and electric utilities and telephone lines were down. Likewise the destruction of the radio tower made it necessary to send for aid from Cologne.

(h) The aid men and wardens were brought partly by Intelligence and part came by themselves. The fire was spread by numerous explosions, and through the many hours of fighting there was an extremely thick fog. It followed from the Rhine back to Urdeld. Fire water was taken from DHD tanks filled with water, out of the Rhine through a short finished water line, and out of the ash settling basins of the gas works. Great fires spread in the tank farm where four (4) full tanks were located. Also fires spread in the coal mill and the bunkers of the gas works. Besides, there were a series of small fires in the vicinity of the high pressure, the distillation, and the power gas units. The little fires were quickly extinguished, but the large ones lasted to the following noon.

(i) The following damage occurred: Great destruction in the gas production unit, in the coal mill, in the water gas conversion, in the compressor building, the CO purification, in the recycle pumps used for hydrogenation, in the tank farm, and the pump house for heavy oils. Almost all gasoline was destroyed.

The smaller gas holder for N_2 and H_2S remained almost undamaged. The cracking, distillation, power gas, dephenolating units were less heavily damaged. Four (4) large tube breaks and a larger number of splits occurred. The fresh water, drinking water, and discharged water lines were broken in many places, also the telegraph and telephone lines.

(j) About twenty (20) percent of the plant was destroyed. The capacity was greatly hurt because the buildings had been hit so heavily.

(k) In ten (10) weeks of accellerated repair work, the plant was restored to forty (40) percent of production.

(l) On October 3 and 11, 1944 two more attacks followed and at each about sixty (60) bombs of one thousand pounds fell on the plant. These caused damage in the high pressure, tank farm, and power house units.

(m) On October 6, 1944 the plant was closed. Of all the bombs dropped on the July 18, 1944 raid, two hundred (200) were duds.

IV. LIST OF IMPORTANT MANUFACTURERS OF MACHINES AND EQUIPMENT.

GAS PRODUCING UNIT

Water gas unit	Pintsch - Berlin
Producer gas unit	Pintsch - Berlin
Piping and steel work	Kolsch - Folserwerke, Siegen
Furnace lining	Dr. Otto, Bochum
	Keppers, Essen
Gas scrubber	Theissen, Munchen
Gas and air blowers	Jager and Co., Leipzig
Dust removal and cleaning	Lurgi, Frankfurt
Tar pumps	Weise, Sehne, Halle
Ash removal	Naeher, Chemnitz
Conveyers	Pohlig, Koln-Zollstock
Producer and water gas blowers	Schiele, Eschborn/Taunus

IV. LIST OF IMPORTANT MANUFACTURERS OF MACHINES AND EQUIPMENT. (Cont'd.)

Fittings and cocks	Zimmermann u. Jansen, Duren
Gas holders	Neumann, Eschweiler
Tanks	Neumann, Eschweiler
Cooling towers	Otto Estner, Dortmund
Redler conveyors for dust and ashes	Gebr. Buhler, Dresden
Conveyor belt	Clouth, Koln-Nippes

BOILER HOUSE

Steam boilers	Surr A.G.,Ratingenb. D'dorf
	Walther, Koln-Dellbruck
Boiler feed pumps	Halberg, Ludwigshafen
Boiler feed turbines	Borsig, Berlin-Tegel
Coal conveyors	Maschinenfabrik, Magdeburg,K.S.G. Berlin
Fire brick lining	Karrenberg, Dusselforf
Steam lines	Seiffert, Eberswalde
Burner control	Askania, Berlin
Temperature control	A.E.G., Berlin
Flow regulators	V.A.G., Mannheim
Superheated steam equipment	Amag-Hilpert, Nurnberg
	Schaffer u. Budenberg, Magdeburg
Boiler feed water treatment	Permutit, Berlin
Blowdown (?)	Natorp U. Eberhardt, Hohenturm B. Halle
Turbines	A.E.G., Berlin
Water pumps	Rits U. Schweitzer, Schwabisch-Gemund
Condensate	Vogel, Wien-Stockerau
Low pressure tanks	Feld U. Halm, Bendorf

CONVERSION, COMPRESSORS CO_2 AND CO PURIFICATION

Mathane cracking	
Organic sulfur removal	Bamag, Berlin
Exhaust gas blowers	Kuble, Kopp U. Kausch Frankenthal
Air blowers	Schiel, Eschborn i. Ta.
Boilers	Oschatz, Merane i. S.

IV. LIST OF IMPORTANT MANUFACTURERS OF MACHINES AND EQUIPMENT. (Cont8d.)

Boiler feed units (pumps)	K.S.B. Frankenthal
CO_2 compressor o.4 atm.	C. Enke and Co. Schkeuditz
	Balcke, Frankenthal
Combustion unit for N_2 production	Bamag, Berlin
N_2 compressors	Maschinenfabrik Surth
Conversion	Bamag, Berlin
Pump units	ther Amag-Hilpert jetzt
	K.S.B. Frankenthal
Water gas blowers	C. Enke and Co., Schkeuditz
Heating gas blowers	C. Enke and Co., Schkeuditz
Heating gas blowers Bau 144	C. Enke and Co., Schkeuditz
Heating gas blowers Bau 38	C. Enke and Co., Schkeuditz
	I. Jager, Chemnitz
Compressors	Halberg A. G. Ludwigshafen
Boosters	Ehrhard u. Schmer, Saarbrucken
	Demag, Duisburg
CO_2 purifier	Gutehoffnungshutte, Sterkrade
Pump units	Halberg A. C. Ludwigshafen
Pelton turbines	Escher Wyss, Ravenburg/W.
CO purifier	PreB-u. Walzwork, Reisholz
Press pumps	Gutehoffnungshutte vorm. Haniel u. Lueg, Dusseldorf
Expansion engine	Leuna-Werk, Merseburg
Vacuum pump	K.S.B. Frankenthal
Tanks	Wilhelmshutte, Sprottsu
Cooling unit	Mansfelder Maschinenfabrik, Mansfeld
Booster	Eslinger Maschinenfabrik, Eblingen
	Demag, Duisberg
Oil purification equipment	Bergedorfer Eisenwerke, Bergedorf b. Hamburg

HYDROGENATION - HIGH PRESSURE

Coal reactors and washers - 700 atm.	Friedr. Krupp, Essen
Coal reactors	D.H.H.V. Dortmund-Horde
Gasoline reactors - 325 atm.	D.H.H.V. Dortmund-Horde

IV. LIST OF IMPORTANT MANUFACTURERS OF MACHINES AND EQUIPMENT. (Cont'd.)

Regenerators - 700 and 325 atm.	Friedr. Krupp, Essen
	D.H.H.V. Dortmund-Horde
	Bocumner Verein, Bochum
	Pres. u. Walzwerk, Reisholz
Heat removers - 700 atm.	D.H.H.V. Dortmund-Horde
Product collectors	Bochumer Verein, Bochum
	Pres. u. Walzwerk, Reisholz
Surge tanks	Deutsches Rohrenwerk, Dusseldorf
Preheater construction	Eisenwerke, Kaiserlautern
Hairpin tubes - 700 atm.	I. G. Ludwigshafen, Leuna
Hiarpin tubes - 325 atm.	I. G. Ludwigshafen, Leuna
Exchanger bundles	Leuna-Werke
	Hydrierwerke, Politz
	Chemische Werke, Huls
Appurtenances	I. G. Ludwigshafen
Circulating blowers	Schiele, Escheborn i. T.
Gas coolers - 700 atm.	I. G. Ludwigshafen
Air coolers	I. G. Ludwigshafen
Mud coolers	I. G. Ludwigshafen
Gas cooler - 325 atm.	Deutsche Rohrenwerke, Dussledorf
Roactor crane	Demag, Duisburg
High pressure fittings - 700 atm.	I. G. Ludwigshafen
High pressure fittings - 325 atm.	I. G. Ludwigshafen
High pressure piping	I. G. Ludwigshafen
	Rheinmetall-Borsig, Dusseldorf
	Ruhrstahl, Witten
	Henrichshutte, Hattingenu, Wolking
	Mannesmann, Dusseldorf, Remscheid
	Deutsche Rohrenwerke, D Dusseldorf
Tool wagons	Fuchs Waggon, Heidelberg
Electric preheater	I. G. Ludwigshafen
Electric transformers	Koch und Sterzel, Dresden
Electric equipment	Ges. f. Elektroschweisung, Dortmund
Other cranes	Fredenhagen, Offenbach
Conveyors	Gebr. Wohr, Unterkochen/Wttbg.

IV. LIST OF IMBORTANT MANUFACTURERS OF MACHINES AND EQUIPMENT. (Cont'd.)

Rotating furnaces	Krupp, Grusonwerk, Magdeburg
Furnace lining	Tonwerk, Beibrich-Wiesbaden
Reducing gears	Friedr. Krupp, Essen
Burners	Wistra G.m.b.H.
Preheater and steam superheater	Walther, Koln-Dellbruck
Crane units	W. Fiebig, Waldenburg/Schl.
Residue pumps	I. H. Naher, Chemnitz
Other cranes	E. Holscher, Rechlinghausen
Circulating and fresh gas blowers	Kleinschanzlin & Becker, Frankenthal

DISTILLATION

Distillation units A1, A2, A3	Uhde, Dortmund
Distillation units B1, B2, B3	Uhde, Dortmund
Distillation unit B4	Maschinenfabrik Brunn, Konigsfeld
Stabilizers 1 & 2	Lurgi, Frankenthal/M.
Hot oil pumps up to 40 atm.	K.S.B. Frankenthal
Water pumps	Hesse, Chemnitz
Pumps for cold products	Weise, Sohne, Halle
A3 column	Ganzler, Duren
Tanks 1000, 2000, 3000 cu. meters	Munk & Schmitz, Koln
Tank 500 cu. meters	Dingler-Werke, Zweibrucken
Centrifugal blowers	Schiele, Eschborn, i. Ta.
Power gas storage tanks	Bamag, Bayenthal G.m.b.H. Sterkrade
Power gas scales	Dopp, Berlin
Weighing lorry	Schmidt, Dusseldorf
Other lorries	Schmidt, Dusseldorf

HYGASVERABEITUNG und PHENOLWASERREINIGUNG

Alkazid unit	Dr. Otto, Bochum
Hot and cold caustic pumps	Amag-Hilpert, Nurnberg
Oil scrubber	Bamag, Berlin
Organic sulfur purifier	Bamag, Berlin
Sodium hydroxide wash	Bamag, Berlin
Caustic pumps	Amag-Hilpert, Nurnberg
Hard steel-nozzles	Wallram, Essen
Electric controls for valves	A.E.G. Berlin

De-pressure vessels 50 and 100 atm.	Pintsch, Berlin D.R.W. Dusseldorf Mittledeutsche Stahlwerke Riesa
Repair of tube bundles	Chemische Werke, Huls Leuna-Werke
Hairpin tubes - 700 atm.	Leuna-Werke
Hairpin tubes - 325 atm.	I. G. Ludwigshafen
Paste and presses	BMAG vorm. Schwartzkofpff, Berlin
Middle oil injection pumps - 350 & 700 atm.	Balcke, Frankenthal
High pressure circulating pump - 60 atm.	Halberg A. G. Ludwigshafen
Expansion engine	Leuna-Werke

HYDROGENATION LOW PRESSURE

Discharge units	Gabr. Buhler, Dresden
Conveyor units	Maschinenfabrik Buckau
Mill units (sieve rolls, & Konzentra mill)	Gebr. Buhler, Dresden Krupp, Grusonwerke, Magdeburg
Automatic balancers	Schenk & Co., Darmstadt
Agitator vessel	W. Dohmen, Eschweiler
Product pumps	K. S. Eddesse, Oschersleben
Mud centrifuges	Bergedorfer Eisenwerke, Bergedorf
Widia-mozzles (Wie dremant)	Krupp, Essen Deutsche Edelstahlwerke, Krefeld
Sand separators	Eisenwerke, Kaiserlautern
Dirty ore centrifuge	I. Haubold, Chemnitz
Circulating pumps	Weise, Sohne, Halle Hasse, Chemnitz
Screw conveyor ovens	Uhde, Leuna
Screw conveyor cranes	Peninger Maschinenfabrik und Liebig, Leipzig
$C_3 - C_4$ unit	Uhde, Leuna
$C_3 - C_4$ pumps	K.S.B. Frankenthal
$C_3 - C_4$ compressors	Maschinenfabrik, Surth

Drives	Westdeutsche Getriebebaugesellschaft (Ges. Existiert night mehr)
Carbon rich gas compressors	Maschinenfabrik, Surth
Cooling unit	Rehinkalte, Dusseldorf
Drives and cooling compressors	Tacke, Rehine Westf. Westdeutsche Getriebebauges
Gasoline rich gas blowers	C. Enke, Schkeuditz, Leipzig
$C_3 - C_4$ gas holders	Opbergen, Neuss Gutehoffnungshutte, Sterkrade
Phenol removing unit	Dr. Otto, Bochum
Gear pumps	Neidig, Mannheim
Vacuum pumps	K.S.B. Frankenthal
Oil separators	Schumann, Leipzig
Packed towers	Dinglewerke, Zweibrucken
Dirty water pumps	K.S.B. Frankenthal Weise, Sohne, Halle Amag-Hilpert, Nurnberg
Water tanks	Dinglewerke, Zweibrucken Kolsch-Folzerweike, Siegen

V. FUEL ADMINISTRATION IN THE GERMAN GOVERNMENT.

(a) The head of allindustry in Germany is Dr. Speare. Under him in charge of the chemical industries is Dr. Krauch, The chairman of I. G., who corresponds to the head of our W.P.B. The industries are broken down in the following groups:

(1) Oil,
(2) Aluminum,
(3) Hydrogenation,
(4) Nitrogen,
(5) Explosives.

(b) The planning for all armaments under Speere is done by Herr Kehl, chairman of the planning division.

(c) As well as the above mentioned persons there is Herr Geilenberg, personally picked by Hitler, and responsible only to him, who is in charge of exploiting all hydrogenation plants.

V. FUEL ADMINISTRATION IN THE GERMAN GOVERNMENT. (Cont'd.)

He is responsible for the Geilenberg Plan which:

(1) Repairs plants,
(2) Builds new ones.

Geilenberg was responsible for the decision not to repair Wesseling and moving the workmen elsewhere.

(d) Product Destination.

All of the men at Wesseling seemed ignorant of the ultimate destination of the plant's output. However, the following information was given:

(1) Motor gasoline - shipped to Berlin, Duisberg, Köln, Mannheim to large tank farms - Standard, Shell, etc. Some lead was apparently added.
(2) Diesel oil - sent by canal to Holland, Antwerp and Ghent. Orders came from the Zentral Büro.
(3) Aviation gasoline - destined for plants called WIFO - Munich and Duisberg. Some went to Ruhr Oil Co. at Bothrop.
(4) Special 165° C aviation gasoline went to D.H.D. unit at Ludwigshafen. Here the naphthenes were dehydrogenated to aromatics.

(e) New Underground Installations of Hydrogenation Plants.

The underground installations go by the code name "Schwalbe". They are supposedly five in number. Two are in the Elbe valley, between Dresden and Aussig, in the Elbe Sandstein Gebirge, on both sides of the river. One is near Buckeburg and Minden in a natural cave. The fourth is in Thuringia at Berga Kelbia and the fifth, belonging to I. G., is in the Harz mountains in the old gypsum quarries at Niedersachswerfen. The latter also has an A. T. unit (Alkylation Plant).

Prepared by:

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FILE No. XXXII-11

Classification Cancelled
by authority of
The Joint Chiefs of Staff,
by Col. E. W. Gruhn.

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INTERROGATION OF DR. HABERLAND

Mumford, S. A.

RESTRICTED

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

RESTRICTED

INTERROGATION OF DR. HABERLAND, DIRECTOR OF I.G. FACTORIES ON LOWER RHINE

Reported by

Lt. Col. S.A. MUMFORD

Min. of Supply

[1945]

CIOS Target No. 22/1751

MISCELLANEOUS CHEMICALS

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear), APO 413

RESTRICTED

7 p.

CONTENTS

PAGE

INVESTIGATORS ON TRIP 66 — 2

1. INTRODUCTION — 3

2. INFORMATION OBTAINED

   (a) GENERAL CHEMICAL PRODUCTION — 3

   (b) SYNTHETIC RUBBER — 4

   (c) CHEMICAL WARFARE ETC. — 5

   (d) MISCELLANEOUS — 6

3. CONCLUSIONS — 7

INVESTIGATORS ON TRIP 66

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[x] Failed to join team.

REPORT ON C.I.O.S. TRIP NO. 66

INTERROGATION OF DR. HABERLAND, DIRECTOR I.G.

1. INTRODUCTION

It is understood that Dr. Haberland was originally contacted by Major King, Chemical Officer, 2nd Armoured Division U.S., and that prior to our arrival he had already been interrogated by Dr. Lane and Dr. Farlig of A.L.S.O.S., and by Lt. Col. Dees and Lieut. Swartz of 12th Army Group.

A first attempt to interview Dr. Haberland at his office in the I.G. Uerdingen factory on 20.3.45 was abortive, as the civilian population had been evacuated from the whole area some days previously for security reasons. He was subsequently located by Lt. Col. Keith, Chemical Officer, 102nd Division, U.S., at a private house in Krefeld, and was there interviewed on 22.3.45. A brief tour of the deserted factory at Uerdingen was made on the afternoon of the same day.

2. INFORMATION OBTAINED

(a) GENERAL CHEMICAL PRODUCTION

Dr. Haberland stated that he was Director of a group of I.G. plants in the Lower Rhine and Ruhr Valleys, i.e. those at Uerdingen, Leverkusen, Elberfeld, and Dormagen. He detailed the products turned out by these factories as follows:

Uerdingen: Sulphuric acid; Hydrochloric acid; Nitrate; Chromate (including dichromate, chromic acid); Zinc oxide (for Buna, see 2 (b) below); Iron oxide (black, yellow and red; 40,000 tons/year); Electrolytic caustic soda and chlorine (10,000 tons Chlorine/year); Liquid oxygen and nitrogen.

Benzene and Toluene distillation; Nitrobenzene; Aniline (largest I.G. plant); Methyl and Ethyl Anilines, diphenylamine, toluidine, and other intermediates; Benzyl chloride (by side chain chlorination of toluene); Benzaldehyde; Benzyl acetate; Benzyl benzoate; Benzyl alcohol; Dibenzyl ether; Benzoic acid and its sodium salt (dichromate oxidation of toluene; 2,000 tons/year); Cinnamic acid and esters; Phthalic acid (4,000 tons/year plant 5 months short of completion).

Synthetic resins of the oil-modified alkyd type (Phthalic acid, glycerol, and linseed oil); Emulsions from synthetic resins; artificial leather coatings and dyes for these; Plasticisers and stabilisers for explosives, in particular diethyl diphenyl urea, asym diphenyl urea, ethyl phenyl urethane, and diphenyl urethane (obtained from phosgene and/or chlorcarbonic ester); Triphenyl methane dyes; Adhesives; Polymerisation of propylene; Dodecylamine and didodecyl phenone (plasticiser for synthetic fibres of nylon type).

Leverkusen: (Last visited by Dr. Haberland on 1.3.45 when they had only 8 days' coal supply left).

Lithopone; Titanium dioxide (part converted to the tetrachloride for smoke shell: last produced 6.10.44: whole plant now destroyed); Fluorides (usual inorganic products); Active charcoal; Guanidine nitrate (produced a long time ago; no carbide available now).

Benzene; Toluene; Nitrobenzene; Dinitro benzene; Di-nitrotoluene; Nitronaphthalene; Diamino toluene; Chlor benzene; Phenol; Salicylic acid; Formamide;

Acrylonitrile (for Buna and Perbunan: plant now destroyed); Synthetic oils, including Chlor naphthalene as Transformer oil; Anthroquinone dyestuff intermediates; Azo, Anthroquinone, and Triphenyl methane dyes; Pharmaceuticals and intermediates (Acetanilide, Atebrin, Sulphonamide, Elladrone etc.); DDT (Geigy, ex chloral and chlorbenzene; 100 tons/month); Photographic paper and chemicals:

Buna Research Laboratory and Experimental Tyre plant (see 2 (b) below); Total of 400 chemists employed at Leverkusen.

Elberfeld: Pharmaceuticals (e.g. Salvarsan); Dyes (Indulines); Benzyl cellulose; Hexamethylene tetramine (have not produced for 1 year as no methanol available since destruction of Leuna and Oppau plants).

Pharmaceutical Research Laboratory under Professor Hörlein (still intact).

Dormagen: Sulphuric acid; Cuprammonium; Collite (Hercules powder).

(b) SYNTHETIC RUBBER:

Dr. Haberland had visited Duponts at Philadephin in 1937.

He confirmed that the Research Laboratory for Rubber and Rubber products and the Experimental tyre plant at Leverkusen were intact on 1.3.45. The former was under the direction of Dr. Konrad and Dr. Pohle, and the latter of Dr. Vormandi. Leverkusen produced only acrylonitrile rubber, and this plant and all other I.G. synthetic rubber plants were now destroyed, except that at Hüls. The plant at Ludwigshafen had been destroyed in July 1944 and again in September, October, November and December, since when it had been derelict.

He had little detailed technical information of the processes involved, but made the following miscellaneous statements in reply to questions:-

Buna S and Buna S III differ in the length of the polymerisation cycle times.

Active Zinc oxide, used as filler for Buna to give strength, is produced by indirect heating from the carbonate.

There is no Tonerde production in I.G.

Thiodibutyric esters are used as plasticisers for Buna.

Koresine is a condensation product of phenol and acetylene in the presence of a catalyst (nature unknown).

(c) CHEMICAL WARFARE ETC.

Apart from incidental preparation and use of phosgene and cyanogen chloride in the production of intermediates etc., and from the Adamsite manufacture described below, Dr. Haberland stated that none of the factories under his direction were, or had been, engaged on the production of C.W. agents, and it became evident in the course of discussion that he had little knowledge of this subject.

At the request of the Wehrmacht a 100 ton/month Adamsite plant had been erected and run at Uerdingen: the product (and subsequently (September 1944) the plant) had been sent to Dyhernfurth, but he had no knowledge of what it had been used for. He gave the impression that the I.G. as a whole had not played an active part in any C.W. development or production, but that the Wehrmacht had set up their own C.W. laboratory and production centre at Dyhernfurth on the Oder, now in Russian hands. He knew of, but had never visited the Kaiser Wilhelm Institute in Berlin.

He had no knowledge of the types of C.W. agents (if any) under consideration or being manufactured in Germany. He was generally acquainted with mustard gas, but had no knowledge of nitrogen vesicants, not even to the extent of recognising that methyl dichlorethyl amine (an intermediate in Dolantin manufacture) was a mustard gas analogue and might be expected to possess vesicant properties. He had no knowledge of the manufacture of any organic fluorine compounds other than refrigerants of the Freon type, and did not appear to be cognisant of Dr. Ruff or his work on such compounds.

He had never heard of chlorcyanuric acid, and had no knowledge of any production of anti-arsenical compounds.

He did not appear to have detailed information of the production of active charcoal at Leverkusen, but maintained that no metallic or other impregnation was carried out.

He had no knowledge of the use of resins etc. for thickening fuel.

The DDT produced at Leverkusen was used against lice and bed bugs; Thiodiphenylamine had been used as a mosquitocide on the Russian front.

He could (or would) not give any information of recent advances in drugs for the control of tropical diseases. He knew of Phasmoquin but not what it was: work onbacteriostatics, in particular Penicillin, had been carried out at Höchst; he had no knowledge of plant growth substances.

(d) MISCELLANEOUS

Dr. Haberland stated that all nitrogen plants in Germany had been destroyed by bombing.

The only I.G. Peroxide plant at Heydebrech was now in Russian hands. Metallic sodium was produced at the Degussa works at Kugesach, S.W. of Cologne.

Phosphate was obtained from Finland and previously N. Africa; Mercury from Florence; Germany had plenty of magnesium and aluminium.

I.G. policy had been to keep their plants, research laboratories, and personnel in situ despite bombing attacks, and no significant transfer to the east had taken place. Every I.G. factory has its own research laboratory, and to a large extent each regional group of factories is self-contained and competes with other groups.

The I.G. had no subsidy from, and did not come under the direct control of, the Wehrmacht, but carried out specific investigations or production for the latter when requested. Dr. Haberland stated that neither he nor the other regional directors of I.G. were strong Nazis, but they were, of course, obliged to be members of the party.

At Leverkusen, Dr. Noack was in charge of Inorganic research, and Dr. Böhme on work on Intermediates; Drs. Lommel and Steinkopf, of Lost fame, were dead. Dr. Haberland had no knowledge of a Dr. Zimmerman.

3. CONCLUSIONS

(i) Dr. Haberland might be able to give further information on the general types and quantities of chemicals manufactured in the four factories under his direction, and of neighbouring I.G. works, but his knowledge of technical details is limited, and his evacuation to U.K. or U.S. for further technical interrogation would serve little if any useful purpose.

(ii) There is no evidence that the I.G. plants on the lower Rhine have been engaged to any significant extent on offensive C.W. preparations.

(Sgd) S.A. Mumford.

Team Leader.

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Report No. 271

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WASHINGTON, D. C.

DIESEL ENGINE RESEARCH AND DEVELOPMENT IN GERMANY DURING THE WAR AND PRE-WAR PERIOD

Reported By:

Mr. G. Murphy
NavTechMisEu

This report has been declassified and released to the Office of Publication Board by the War and Navy Departments.

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TABLE OF CONTENTS

Subject	Page No.
Summary	3
M.A.N. Double-acting Two-cycle Engines	3
Junkers Jumo 205	5
M.A.N. Submarine Engines	6

SUMMARY

Diesel engine development in Germany has been of a restricted nature. The projects to be undertaken were rigidly controlled by the state through the exercise of material, manpower, and fuel oil allotments. The development projects were concerned with the design of a new engine or improvement of an existing engine for a specific military or naval application.

The main development effort has concerned itself with the improvement and weight reduction of conventional four-cycle engines, especially the six-cylinder 40/46 M.A.N. submarine engine. This includes the engine changes necessary for "SCHNORCHEL" operation.

M.A.N. has constructed a twenty-four cylinder Vee-type, double-acting, two-cycle engine in two (2) bore and stroke sizes for surface ship propulsion. The smaller size was to be rated at 9.960 BHP at 600 RPM, while the larger size was to be rated at 14,400 BHP at 450 RPM. None of these engines were actually installed aboard ship.

Mercedes-Benz has brought out a 20-cylinder, Vee-type, four-cycle engine, having extremely light weight and low cubic co-efficient for use in "SCHNELL" boats. The engine is essentially a diesel, marine application of an earlier engine used to power the German rigid lighter-than-air-ships.

The Junkers Jumo 205 (opposed piston, aircraft diesel engine) has been used only experimentally. The engine failed to live up to earlier expectations, and consequently was not built or used in production quantities.

DIESEL ENGINE RESEARCH AND DEVELOPMENT IN GERMANY DURING THE WAR AND PRE-WAR PERIOD

1. M.A.N. DOUBLE-ACTING TWO-CYCLE ENGINES.

(a) This general type of engine is built in four (4) bore and stroke sizes. The essential design information on these engines is listed below:

Engine Designation	65/95	42/58	32/44	19/30
HP Per cylinder (Continuous duty)	1220	600	415	150
Bore (Inches)	25.6	16.5	12.6	7.5
Stroke (Inches)	37.3	22.75	17.3	11.8
R.P.M.	255	460	600	1000
Piston Speed (ft./min.)	1590	1710	1730	1970
Mean BMEP (psi)	49.5	51	64	53
Firing Press (psi)	890	930	970	1000
Cylinder arrangements (in line)	3,8	6	4,7,8	7
(Vee)	-	24	24	-

(b) The fuel consumptions at rated load varied from .355 pounds/HP hour on the 65/95 to .40 pounds/BHP hour on the 19/30.

(c) The following information applies to all of the sizes of engines and is of course only approximate:

Full load exhaust temperature - 600° F. Max.
Heat removed by piston cooling oil - 520 BTU/HP/Hr. minimum
- 600 BTU/HP/Hr. desirable
Piston cooling oil flow rate - 7.5 Gallons/HP/Hr.
Jacket water flow rate - 8.8 to 11.3 Gallons/HP/Hr.
Permissable exhaust back pressure - 14" H_2O Maximum

(d) Cylinder wall was lubricated by a Bosch lubricator. The injection of lube oil was not timed to occur at any specific point in the cycle, but the quantity of oil was varied with the load. About one-third (1/3) of full load lubrication requirements are used at no load.

(e) Lubricating oil that is introduced into the cylinder is all consumed, there being no reclaimation.

(f) Lubricating oil consumption averages about 3,000 HP hours/ gallon.

(g) The three (3) largest engines used conventional cast iron liners. The smallest engine was recently changed over to a chrome plated steel upper and lower section with an unplated cast iron port section. The chrome plating used was hard chromium, with no post treatment or reverse current process to introduce porosity. They stated that their operating experience with this liner construction was rather meager, but it was apparently quite satisfactory.

(h) The 65/95 was a heavy engine (40-50 lbs/HP). Orders were placed for twenty four (24) engines (8 cylinders in line). When these engines were sixty (60) percent completed, all orders were cancelled in order to use the much lighter V-24 construction in the smaller bore and stroke sizes.

(i) The 42/58 was known as the "pocket battleship engine". This engine had been built as a six-in-line for several years. In 1940 an experimental V-6 was built. Following eighteen hundred (1800) hours of operation on this engine, a V-24 was built. This engine was on the test bed and had about three hundred (300) hours of operation. Orders had been placed for forty eight (48) engines (V-24). The frames and crankshafts were completed when construction was cancelled (reason unknown). The V-24 engine weighed 23 pounds/HP with all attached auxiliaries.

(j) The 32/44 was built as an in-line engine for use on the cruiser "Leipzig" and on the school ship "Brenza". In 1938 construction of the V-24 was started. Three engines were finished, tested, and ready to ship. The fourth engine was on the test bed, but no fuel oil was available to test it.

(k) The 19/30 was constructed as a seven-cylinder in-line engine for Schnell boats (motor torpedo boats). The Vee constructions was not contemplated for this bore and stroke size.

(l) The "in-line" double-acting two-cycle engine is of fairly complicated construction. By making a Vee-type engine of this construction the complications are increased considerably. A Blade and fork rod construction is used with the lower connecting rod bearing half being integral with the cap. The upper half of the steel is loose and can be replaced, but with considerable difficulty. The cylinder ports are on the outboard half of the cylinder liner. The proximity of the camshaft and the rotary exhaust valve to this port section makes inspection and cleaning quite difficult.

The inboard injection nozzle on the lower half of the cylinder must be serviced by a man laying on this back on a small trolley that moves back and forth in the space between the legs of the Vee. Main bearings can be serviced but it appears very difficult because of the size of the access hole and the distance from the holt to the bearing itself.

(m) The frame is of welded steel with a separate upper and lower base. The cylinders are bolted to the upper base with tie rods, the upper base transmitting the stresses due to firing impulses to the lower base via the welded structure.

(n) The main moving parts, crankshaft, connecting rods, piston rods, pistons, etc., are machined all over to a good finish. It is apparent that weight reduction has been accomplished at the expense of a considerable amount of maching time. The engine was to be used both with and without a torsional vibration dampner on the free end of the crankshaft as necessity dictated. The dampner itself was of conventional M.A.N. design using several nests of circular shaped springs between the crankshaft and the dampner WR2. This type of construction produces a variable elasticity (varies with the displacement) together with a large hysteresis loss when in motion. This dampner is most complicated, and has given a fair amount of trouble in service.

(o) The injection system was conventional, using two (2) Bosch type pumps per cylinder. One pump operated the Bosch type (water cooled) injection nozzle in the top half of the cylinder, while the other pump operated the two (2) injection nozzles, in the lower half of the cylinder.

2. JUNKERS JUMO 205.

(opposed piston aircraft Diesel engine)

(a) This engine was widely publicized prior to the war. During the war period relatively little was heard of the engine. The basic design information follows:

Engine Designation - Jumo 205
Bore in inches - 4.13
Stroke in inches - 6.3
Number of cylinders - 6
Ratings, Takeoff (5 min) - 600 HP @ 2200 RPM
Continuous - 450 HP @ 2000 RPM
BMEP, Takeoff - 107 psi
Continuous - 88 psi
Piston speed, Takeoff - 2310 FPM
Continuous - 2100 FPM
Weight - 1144 lbs.
Weight per continuous HP - 2.53 lbs/HP
Fuel consumption - .335 to .374 lbs/HP/Hr at optimum point.
Lube oil consumption - .011 to .017 lbs/HP/Hr.

(b) The engine was used in small quantities by the Lufthansa (airlines) but was never used except experimentally by the Luftwaffe (Air Force).

(c) The engine was not adopted by the Luftwaffe because of difficulties when used at high outputs and because of the weight of the engine and fuel was less than current gasoline engines only on planes making extremely long flights, that is, when the fuel was a large proportion of the total weight.

(d) The chief difficulty with the engine at high outputs was piston trouble. The Junkers piston is essentially a three (3) piece piston consisting of an alloy steel plate for the crown (flame shield), a solid but floating ring between the crown and the piston body, and finally the main body of the piston itself. Four (4) through bolts hold the entire assembly together. Originally, bolt breakage was prevalent due to overstressing caused by differential expansion of the steel bolt and the aluminum alloy piston skirt section. This was overcome by using a heavy helical (square wire) spring under each nut. As the piston expanded, the springs compressed and thus did not overstress the bolt.

(e) The main reason for the unconventional piston design was the excellent combustion and low fuel consumption. The extremely hot (1300°F) piston crowns resulted in a combustion chamber that was very uncritical to shape an injection system. The other features of the piston necessary to effect this hot crown were all trouble makers. The solid top ring would work fine when new and accurately fitted. As soon as wear took place (about 200 hours of operation) the ring would not seal properly, throwing the load on the second ring (conventional split ring). The second ring would then run too hot and wear rapidly. Increased blow by followed by piston scoring would result in a short time. At reduced ratings (70 BMEP) the construction was satisfactory, but at these reduced ratings the engine was too heavy to be attractive.

(f) The rating of the engine was reduced to fifty (50) percent of sea level rating at an altitude of 20,000 feet. In an effort to restore this rating the engine was supercharged using an exhaust gas driven turbo-charger in series with the engine driven centrifugal blower. This construction enabled the engines to carry <u>momentary</u> loads of 172 BMEP at a piston speed of 3200 feet per minute. The engine would not stand up at these loads, with the piston again being the chief trouble maker. A few of these supercharged engines were installed in experimental planes, but the state forced the discontinuation of this work. Jot propulsion was in the picture by this time, and most of the research and development talent was concentrated in this field.

3. M.A.N. SUBMARINE ENGINES.

Engine Designation - 40/46
No. of cylinders - 6
Horsepower - 2000 (Supercharged) (See text)
Bore (Inches) - 15.7
Stroke (Inches) - 18.1
RPM - 520
Piston Speed (ft/min - 1570
BMEP (psi) - 145 (Supercharger) (See test)

(a) The engine is a conventional four-cycle supercharged engine. A centrally located injection nozzle with an open combustion chamber was used Two (2) intake valves which seat directly in the head and two (2) exhaust valves whith are caged are provided. The valve seat in the case of the exhaust valve was integral with the cage and of plain cast iron.

(b) A welded lower base with a beded, forged steel crank is used. A [illegible] welded upper base mounts on top of the lower base. Individual cylinder sections mount on the upper base and a one (1) inch thick steel plate running the length of the engine mounts on top of the individual cylinder sections.

carry(c) The entire structure is bolted together with the rods which serve to carry the load due to the firing impulses.

(d) The engine is non-reversible (reversing being accomplished by the electric motors), but a shifting camshaft is provided. The camshaft is shifted to change the valve timing when using "Schnorchel". The valve timing for normal operation is conventional for a supercharger engine using a large overlap between intake opening and exhaust closing. When shifted to the "Schnorchel" position this overlap is reduced to five (5) degrees of crank angle.

(e) Connecting rods have an "H" section shank and are machined all over to a good finish.

(f) The piston is of case aluminum alloy and is uncooled. A conventional full floating piston pin is provided.

(g) Fuel precision bearings are used, the bearing lining being a high lead babbit.

(h) Cylinder liners are of cast iron with a heavy coating of zinc on the outside to protect them from corrosion.

(i) Salt water cooling is employed using large water passages throughout. Since the pistons are not cooled, a very small oil cooler is used to cool the lubricating oil.

(j) The valve gear is conventional except that it is open and is hand oiled throughout.

(k) The supercharger was a conventional Buchi unit manufactured by Brown Boveri Company.

(l) Based on a rating of 2,000 HP, the engine with all attached auxiliaries weighed 22 pounds per HP.

(m) The engine manufacturer stated that 21,000 man-hours of shop and foundry labor were used per engine (the average American manufacturer would not be competitive if he used over one-third (1/3) of this amount of labor, that is, he must use less than 7,000 man-hours of labor for 2,000 HP if he is to compete in the open market). A large portion of the extra labor can probable be traced to the excessive amount of machining in order to pare the weight down to 22 pounds/HP.

(n) Inspection of the engine installed on completed submarines revealed that the supercharger had been removed in most instances. It was stated that "Schnorchel" operation was a failure with the supercharged engine. Inability to carry even a moderate load without excessive exhaust temperatures was given as the primary reason for removing the superchargers.

(o) The unsupercharged rating was not definitely determined but appeared to be between 1,300 and 1,500 BHP. Apparently the German Navy was willing to sacrifice power on the surface in order to gain satisfactory and reliable operation when using Schnorchel.

(p) On the basis of an unsupercharged rating of 1,400 BHP, the BNEP is reduced to 101 pounds/in^2, and the weight of the engine becomes 31.5 pounds/HP.

Prepared by: G. MURPHY,
Technician.

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ITEM No. 30. **COPY No** ~~183~~

FILE No. XXIX-51

Classification Cancelled,
by authority of
The Joint Chiefs of Staff,
by Col. E. W. Gruhn.

Solid Fuels 46

~~RESTRICTED~~

Thyssen'sche Gas und Wasserwerke G. M. B. H.
Duisberg-Hamborn
Krupp Treipstoffwerk
Wanne-Eickel

Newman, L. L.

RESTRICTED

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

RESTRICTED

THYSSEN'SCHE GAS UND WASSERWERKE G.m.b.H
DUSIBURG-HAMBORN
DUISBURG-HAMBORN

and

KRUPP TREIBSTOFFWERK, WANNE-EICKEL
THYSSEN-GALOCSY GAS PRODUCER

Reported by

L.L. NEWMAN, TIIC

On Behalf of U. S. Technical Industrial
Intelligence Committee

[1945]

CIOS Target Numbers 30/118 & 30/159

Fuels and Lubricants

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE,
G-2 Division, SHAEF (Rear) APO 413

RESTRICTED

16 p diagr.

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TABLE OF CONTENTS

SUBJECT .... PAGE NO.

Summary..1

Introduction...................................3

Statement by Dr. A. Rettenmaier................3

Wanne-Eickel Installation......................6

Drawing Showing Cross Section of The Producer..6A

Wanne-Eickel Operating Results.................7

List of Documents..............................9

Appendix I - Thyssen-Galocsy Process

Appendix II - Preliminary Report on the Thyssen-Galocsy Plant at Wanne-Eickel

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THYSSEN'SCHE GAS UND WASSERWERKE G.m.b.H.
DUISBURG-HAMBORN
and
KRUPP TREIBSTOFFWERK, WANNE-EICKEL
THYSSEN-GALOCSY GAS PRODUCER

Summary:

The Thyssen'sche Gas and Wasserwerke at Duisburg-Hamborn and the Krupp Treibstoffswerk at Wanne-Eickel are considered a single target for the purpose of reporting the information secured on the Thyssen-Galocsy Slagging Gas Producer Process.

The following were members of the team:

Mr. L. L. Newman (American)
Dr. H. J. Rose (American)
Dr. E. T. Wilkins (British)

The Germans interviewed were:

Dr. A. Rettenmaier)Thyssen'sche Gas und
Mach. Ing. Albert Breisig)Wasserwerke, Duisburg-
Hamborn

Dipl. Ing. Erich Combles
Gen. Mgr. Wanne-Eickel Plant

Visits were made to Duisburg-Hamborn on June 28, 1945 by Dr. Rose and Mr. Newman, and on July 2, 1945 by Dr. Wilkins and Mr. Newman. A visit to Wanne-Eickel was made on July 2, 1945 by Drs. Rose and Wilkins and Mr. Newman.

This report presents the statement made by Dr. Rettenmaier and Ing. Breisig and includes a sectional drawing of the generator. Appended are (1) a statement (supplied in English) of the Thyssen-Galocsy Process, and (2) a Preliminary Report on the Thyssen-Galocsy Plant at Wanne-Eickel (Translated from the German by Lieut. Young, NAVTECMISEU).

All of the available figures are presented without change. No attempt was made to check the claimed efficiencies and percentages of steam decomposition, nor have any calculations been made on the rate of gasification. The previously published data on the Thyssen-Galocsy Process are not available for comparison with the results presented to this investigating group. It appears, however, that the gas composition is about as previously reported, but that the oxygen consumption - which is one of the major factors in the cost of production - is approximately 50% higher than mentioned in any of the reviews.

THYSSEN'SCHE GAS UND WASSERWERKE G.m.b.H.
DUISBURG-HAMBORN
THYSSEN-GALOCSY SLAGGING GAS PRODUCER

Introduction.

Almost every review of German complete gasification processes, particularly those in which oxygen is used for the continuous production of water gas, includes a description of the Thyssen-Galocsy Slagging Gas Producer. These descriptions have generally been based on pilot plant operations which have been conducted for several years on a unit which was gasifying two tons of coal per day at the Thyssen'sche Gas und Wasserwerke at Duisburg-Hamborn, and one gasifying 10 tons per day at the ammonia synthesis plant at Pecs, Hungary.

The results reported, though sketchy, seemed to indicate that non-coking or weakly coking coals having ash of low fusion temperature were suitable and that the gas generated had a higher quality than in other continuous gasification processes using oxygen at normal pressures. The admission of oxygen at two levels and the use of recirculating gas to spread the reaction zone was claimed as an important advantage because it lessened the intensity of combustion and lengthened the life of the refractories.

On the basis of the foregoing information a visit was made to the plant and the personnel directly concerned with its operation were interviewed.

Statement by Dr. A. Rettenmaier.

On June 28, 1945, Dr. H. J. Rose and Mr. L. L. Newman visited Dr. A. Rettenmaier at the offices of the Thyssen'sche Gas und Wasserwerke, 159 Duisburgerstrasse, Hamborn.

Dr. Rettenmaier stated that he was in charge of the development work and confirmed that a two ton per day unit based on the Galocsy patents was built at the works for preliminary research on the slagging operation using oxygen for gasification. The unit demonstrated the possibilities of the process but was not sufficiently large to furnish conclusive data on the results obtainable in the gasification of various fuels.

A unit capable of gasifying 40 tons per day was, therefore, erected at the Krupp Treibstoffswerk at Wanne-Eickel. It took two years to build the unit because of wartime restrictions on the use of labor and materials and interference by enemy bombing. No use was made of the 2 ton unit at Duisburg-Hamborn during this construction period. It was hoped in the 40 ton unit to demonstrate that it was feasible to gasify any grade of fuel in any size or combination of sizes from 5 to 80 mm., coking or non-coking, and regardless of the ash flowing temperature. It was desired to establish the operating conditions under which any fuel could be best gasified so that adjustments could be quickly made for each grade of fuel.

The fuels available in the Krupp company's own mines range from poorly coking coal with a volatile matter content of 35-40% in the upper seam which is at a depth of 400 meters, through coking coal in one of the lower seams at a depth of 700 meters. Below this depth there is a series of leaner coals all the way to anthracite. The younger coals in the upper zones were considered the most economical to use in the slagging operations.

The tests at Wanne-Eickel were frequently interrupted. One test, however, lasted four weeks and it was established that coke 40 to 60 mm in size could be gasified without any operating difficulty. Although laboratory determinations showed an ash flow temperature of 1300°C, it was found that trouble-free operation could only be maintained at 1600°C with coke containing 8-10% ash and without the addition of limestone or iron fluxing material.

Other tests were tried with the use of refuse from the Demag-Duisburg generators containing 40 to50% ash, and also non-coking coal. Various combinations of fluxing material (20 to 40%) were tried, but the tests were interrupted before any conclusive data could be secured by one of three possibilities, namely: (a) the bombing of their own plant; (b) bombing of the Krupp plant which supplied the steam, electricity and water; and (c) bombing of the Stickstoffwerk Hibernia which supplied the oxygen. Only two days of operation were conducted with coal and no stability of operation was attained.

The best sizes of coke were stated to be 30 to 60 mm. No difficulty was experienced in gasifying 40 tons per day with a yield of 100,000 cu meters. The gasification rate was limited by the available amount of oxygen. The calorific value of the gas was about 2700 calories (290 Btu per cu.ft.) and some means of methanation would be required to make the gas suitable for city distribution requiring about 4200 calories (450 Btu per cu. ft.).

Dr. Rettenmaier was very anxious to resume operations, and hoped to test 3 to 5 mm size coke breeze. He desired also to have at least one month for testing young coals and six additional months for making tests with other Krupp coals.

In commenting on oxygen costs Dr. Rettenmaier stated that the power cost was about 4.3 pf. per KWh at the works and was prohibitive for oxygen production. The cost of purchased oxygen was 2.3 pf. per m^3. He felt that he could obtain cheap oxygen (1 to 2 pf. per m^3) by using the coal screenings to generate steam at high pressure for driving the turboblowers and compressors in the Linde-Fräukl plant and by using the exhaust steam in the gasification process.

Dr. Rettenmaier further stated that Wanne-Eickel was chosen for the installation of the 40 ton unit because the Thyssen'sche Gas and Wasserwerke is a gas company buying coke oven gas which it purifies and distributes through a pressure grid system. Intermittent water gas machines are used for standby purposes only, and gas producers are kept in readiness to transmit gas for heating the ovens in the event of any failure of the supply of blast furnace gas normally used for this purpose. The water gas machines are Demag and the gas producers Koppers.

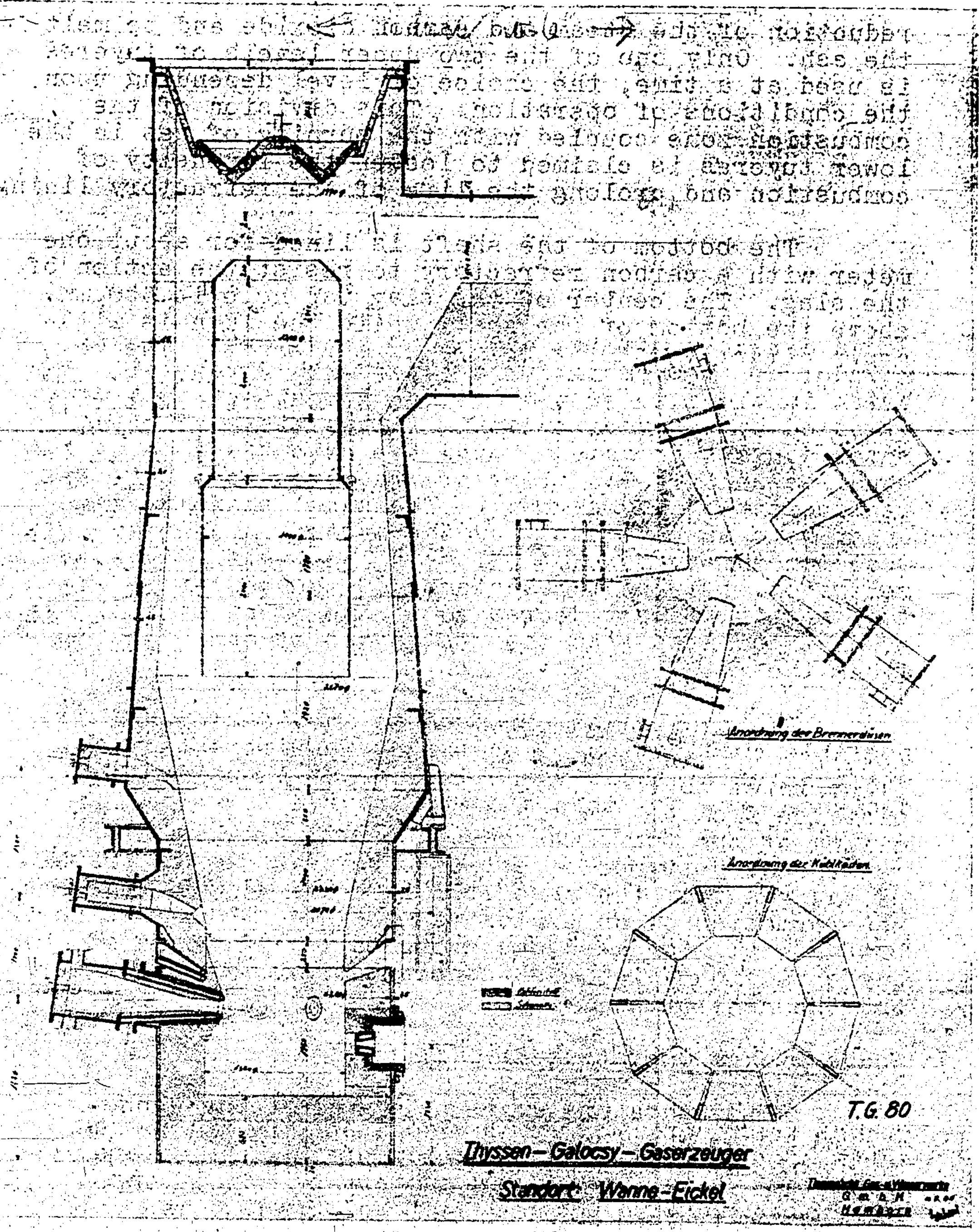
No documents were available. The reason given by Dr. Rettenmaier was that one commission consisting of three American officers had previously visited and evacuated whatever documents remained after the general destruction resulting from Allied bombings. Nevertheless, he promised to provide copies of drawings, which were in process of preparation, and statements regarding the process and operating results.

These documents were secured on July 2, 1945 by Dr. Wilkins and Mr. Newman prior to the visit to the Wanne-Eickel installation on the same day.

Wanne-Eickel Installation.

The Wanne-Eickel installation was visited on Monday, July 2, 1945, by Drs. Wilkins (British) and Rose and Mr. L. L. Newman (American) under the guidance of Dipl.-Ing. Erich Comblés, General Manager of the plant and Dr. Rettenmaier and Herr Breisig, the engineer who supervised the operations. It was found that the instrument house was completely destroyed and there was considerable damage to the piping and the structures surrounding the generator. The generator itself was in fair shape and a good idea of the kind and position of the burners and piping could be secured by inspection.

The generator was found to be blast-furnace shaped, about 35 feet high and about ten feet in diameter. The attached photostat of Drawing No. T.G.80 of the Thyssen-Galocsy Gas Generator at Wanne-Eickel shows the design of the unit at the present stage of development. Note that it has provisions for three levels of tuyeres, with five tuyeres at each level. The tuyeres at the lowest level are actually water cooled burners in which gas from the producer or any other source is burned in a mixture of oxygen and steam and admitted into the fuel bed at a temperature high enough for the steam and carbon dioxide to react with the carbon. (There was no evidence of preheating the gas, steam or oxygen). The tuyeres at the upper levels are not water cooled and are used for the admission of additional oxygen for the purpose of supplying additional heat to balance the amount required for the


Anordnung der Brennerdüsen
Anordnung der Kühlkästen
Schamotte
T.G. 80
Thyssen – Galocsy – Gaserzeuger
Standort: Wanne – Eickel
G. m. b. H.
Hamborn

reduction of the steam and carbon dioxide and to melt the ash. Only one of the two upper levels of tuyeres is used at a time, the choice of level depending upon the conditions of operation. This division of the combustion zone coupled with the burning of gas in the lower tuyeres is claimed to lessen the intensity of combustion and prolong the life of the refractory lining.

The bottom of the shaft is lined for about one meter with a carbon refractory to resist the action of the slag. The center of the slag tap hole is 650 mm. above the bottom of the shaft while the iron is withdrawn directly off the bottom of the shaft. The remainder of the generator is lined with firebrick. The brick in the zone just above the lowest set of tuyeres is cooled by water circulating in two tiers of steel water jackets arranged around the circumference in ten equal segments. Two tiers are required to permit the replacement of any segment. The steel shaft in the upper zone is kept empty by the system of charging the fuel for the purpose of balancing the wall effect of the generator. The unit resistance of the center of the fuel bed is thereby decreased to the point where it is equal to or less than the resistance of the fuel bed along the wall.

The piping was arranged to provide for any possible combination of operating requirements. The steam inlet to the oxygen line was at a considerable distance from the generator to provide for thorough mixing of the two. The oxygen passed through a water seal designed to act as a barrier to the travel of any gas from the generator into the oxygen line and the formation of an explosive mixture. There was no vent, but an explosion head was provided to take care of any explosion which might occur between the water seal and the generator.

Wanne-Eickel Operating Results.

Although a preliminary report of the Wanne-Eickel tests was furnished (See Appendix II) no results of any prolonged operation of the plant were supplied. Below is shown a summary of conditions over three typical days in Test No. 3 which was claimed to have established the feasibility of gasifying coke in slagging operation:

Day	1	2	3
(a) Charge			
Oxygen (pure) Nm^3	32350	24800	25800
Percentage purity	81	90	90
Steam, tons	25.8	17.8	18.9
Auxiliary gas (i.e. gas produced but used in the process) Nm^3	16330	16070	17250
Coke (40-60 mm dia) tons	46.6	39.2	41.3
Analysis:			
Water 5.0%			
Ash 8.5%			
V.M. and S 1.5%			
Heating Value 6865 Kcal/kg			
(b) Yield	1	2	3
Oxygas (Total gas less auxiliary gas) Nm^3	105400	84665	89270
Analysis CO_2	4.6	2.8	2.1
O_2	0.1	0.1	0.1
CO	65.3	70.4	71.0
H_2	24.6	23.1	23.3
CH_4	0.2	0.2	0.2
N_2	5.2	3.4	3.3
Lower Calorific Value cal/m^3	2600	2735	2760
Steam decomposition %	82.1	90.1	90.1
Percentage gasification $\frac{\text{(Total Gas C.V.)}}{\text{(Total Coke C.V)}}$	85.6	85.2	86.7
(c) Requirements for each 1 Nm^3 Oxygas			
Pure oxygen Nm^3	0.30	0.29	0.29
Steam, kg.	0.25	0.21	0.21
Coke, kg.	0.44	0.46	0.46

It was stated that the capacity of the unit was not reached because of the limitation on the amount of oxygen which could be supplied by Stickstoffwerk Hibernia. Herr Breisig was emphatic in his statement that better results might have been obtained if more oxygen were available to operate the unit at a higher rate. He backed his statement by adding that the results were improved when one of the burners was turned off and the available supply of oxygen was divided among the remaining four burners. Although he admitted that the operation was not quite as smooth, he claimed that the efficiency was increased as a result of the decrease of the heat losses in the cooling water circulated through the tuyeres.

Dr. Rettenmaier felt that the use of a slagging gas producer built along the lines of a blast furnace is the best means of providing the large volumes of synthesis gas required for even moderate quantities of synthetic liquid fuels produced by the Fischer-Tropsch process, and that the admission of a controlled amount of steam above the combustion zone would yield the desired ratios of carbon monoxide and hydrogen.

List of Documents.

1. Description of Thyssen-Galocsy Process (in English) See Appendix I.
2. Preliminary Report on the Thyssen-Galocsy Plant, at Wanne-Eickel (in German) See Appendix II.
3. Drawing No. T. G. 60 - Plan of Wanne-Eickel Test Plant, April 24, 1941.
4. Drawing No. T.G. 67 - Elevations of Wanne-Eickel Test Plant, May 29, 1941.
5. Drawing No. T. G. 64 - Cross Section of the Producer - Undated.
6. Drawing No. T.G. 80 - Cross Section of the Producer - July 4, 1945.

These documents are on file in Bag. No. 1481A.

APPENDIX I

Thyssensche Gas-und Wasserwerke Duisburg-Hamborn,
May 1945 G.m.b.H.

Thyssen-Galocsy-(T.-G.-) Process

applying oxygen for total gasification of solid fuel or reduction of ore in a blast-furnace.

The application of oxygen instead of air-blast for the gasification of solid fuel has become econmically interesting, since the Line-Fraenkl-process made possible to produce it cheap, if a great quantity is wanted. Lignite has already been gasified on largest scale with oxygen in Winkler-gasproducers. Technical difficulties, especially unsufficient durability of refractory materials at the attained high temperatures, prevented till now similar large plants for non-baking bituminous coal.

The T.-G.-process overcomes these difficulties with a trick of the proceeding, namely by the division of the extraordinary vehement combustion with oxygen into two steps, either of which can be easily controlled. In the first step a mixture of oxygen and steam is blown into an entrance-chamber of the producer and therein simultaneously such a quantity of any combustible matter is ignited, that the resulting gas mixture is heated up to the temperature, at which steam and carbnic acid are able to react quickly with hot carbon; in the second step this hot mixture is admitted to the solid fuel in the shaft of the producer with such contents of superfluous oxygen, that the heat attained by the combustion of carbon to carbon-monoxide exactly covers the demand of heat for the decomposition of steam and carbonic acid, the smelting of slack and if desired the reduction of ore. Any excess of temperature, which could destroy the refractory shaft lining is however avoided.

The industrial producer is similar to a blast-furnace. The lower part has several burners, each of which having an entrance-chamber for the above mentioned primary combustion. Preferable auxiliary-gas is ignited therein. If for this purpose no cheap gas or other combustible is available, a part of the produced gas is blown in. By such means no surplus of fuel is consumed, because the products of combustion are reduced in the second step to carbon-monoxide and hydrogen, consuming thereby exactly the heat gained by the combustion in the first step.

After having studied the process during several years in a semi-industrial producer, consuming daily 2 tons of bituminous coal, a producer of industrial scale with complete testing installation has been erected during the last war, using surplus oxygen of a synthetic ammonia plant and producing gas for synthesis of benzine. An existing shaft-furnace 12 m high of 28 cbm contents with an internal diameter of 1,35 m at the burners, was adapted to test the system. With the maximum available quantity of 29,800 ncbm with 85.5% oxygen consequently 26,300 ncbm pure oxygen a day, the producer consumed 47 t of metallurgical coke thus producing 105.400 ncbm gas of 2.600 kcal interior calorific value, that is 274 x 10^6 kcal/day. A three day's test at the equal consumption, using only 4 of the 5 installed burners demonstrated, that the maximum output was not yet reached with the available quantity of oxygen. The producer was in operation during several months and has given full satisfaction in spite of many war events, by which sudden interruption often arrived mostly by destroying conduit pipes or wires, by cutting off steam, oxygen, auxiliary gas, electric current or cooling-water, even sometimes these all together. These repeated interruptions have hindered until now the gasification of a supplied stock of 500 t bituminous coal of the same quality, which had given best results in the small testing plant. The producer itself has never been essentially damaged and can start, if the synthetic ammonia plant is able to supply oxygen again.

Technical advantages

Continuous working, simple construction, no change of valves during the run, no mechanical device or work except charing fuel and tapping of slack (and iron if desired). Application of approved proceedings and methods of practical operated gas-producers and blast furnaces.

Highly increased output in comparison with existing plants, highest thermal efficiency on account of low nitrogen contents, total gasification without any loss of combustible in the slack. Recovery of by-products of the low-temperature-distillation type.

Immediate adaptability of the output to the consumption places start from coal to full run in 12 hours.

Economical advantages

Utilization of nearly all available and cheaper combustibles for total gasification, using as an example coal with high contents of ash or not suitable for coke-production, in case of need mixed with ashes in order to prevent caking in the shaft, instead of classified coke or producer-coal.

Cheaper gas for industrial heating and synthetic process.

Production of large quantities of low-temperature tar as by-product of an ecnomical industry.

Suitable sphere.

Production of large quantities of gas, from 100.000 to 1.000.000 cbm/day in a single unit, with low contents of carbonic acid and nitrogen, consequently of high combustion temperature with a calorifique value of 2.700 to 3.100 kcal/ncbm according to the gasified combustible. Especially suitable for the following purpose of

gas production:

Heating-gas for industrial plants,

gas for synthesis of hydrocarbons, securing nearly the theoretical amount of liquid products. Since the waste gas of the synthesis is poor on nitrogen, it can be returned almost totally as auxiliary-gas to the burners. Methane and other not desired hydrocarbons become thereby regenerated to gas for the synthesis.

Gas for hydration-process, the change of carbon-monoxide with steam to hydrogen and carbonic acid being performed in the upper part of the shaft.

Gas for the supply of towns and industries, either indirectly by heating coke-ovens, setting free an equivalent quantity of distillation gas to be additionally supplied, or directly by enriching the calorific value by methane, formed by synthetic method from a part of the produced gas.

In blast-furnaces used for the reduction of iron ore. The process has the following advantages:

Diminution of the costs of plant and operation: Air heaters not needed, since preheating takes place in the entrance-chambers of the burners; smaller furnaces and blowers an account of low nitrogen contents.

Immediate adaptability to change the heat required by change of ore qualtities or moisture or atmospheric conditions.

Increased output of existing blast-furnaces. Saving of coke for the reduction of ore in consequence of the combustion of blast-furnace-gas and of the diminution of heat losses. Most economical use of furnace-gas, since its quality and quantity can be regulated and its use is multiplied on account of the low nitrogen contents.

Thyssensche Gas- und Wasserwerke
G.m.b.H.

APPENDIX II

Duisburg-Hamborn, May 1945

PRELIMINARY REPORT ON THE THYSSEN-GALOCSY PLANT AT WANNE-EICKEL

Translation - by Lt. Yung, Original German Report filed with Documents

The plant was operated in four periods, namely:-

I. Run from 25/10/43 to 22/11/43, end, Hibernia suspended

II. Run from 3/2/44 to 10/2/44, end, off stream, coling chambers burned through

III. Run from 13/4/44 and 29/4/44 to 23/5/44, end Hibernia suspended

IV. Run from 17/6/44 to 4 /7/44, end, bombs on lines and 22/6/44 to 25/7/44

The first and second runs served to teach the operators, who were mostly Dutch workmen who had never worked on blast furnaces or gasification plants before.

The operation confirmed our suspicions that temperatures in this work had to be maintained appreciably higher than in blast furnace operation, but that the burner arrangement first chosen did not meet requirements. The highly heated gasification mixture came out of five burner tips in a ring header, 1.100 metres above the floor, which was placed in a break in the lining and coke bed. With this arrangement, iron coming out of the fuel ash did not fuse with the slag, although in the second run the temperatures went as high as the equipment would stand.

For the third run, these difficiencies were corrected by an arrangement of "tuyeres", similar to the copper ones used in blast furnaces, but made of welded iron because of the heavy duty. Iron and slag ran freely, and every experimental temperature first desired between 1400 and 2000°C could be held constant in front of the tuyeres. The plant was also subject to the unusual wartime accidents, for example, the oxygen failed five times for 2 to 15 hours, twice the main and cooling water failed and during the last run fuel and cooling water fell before the tuyeres in the middle of operation at 1800°C with the plant's being damaged. Gasification, temperature, slag and iron could be fully controlled.

The use of coke was termininated as the new burner system had proved itself in the third run. With the introduction of Baldur coal new difficulties were encountered, chiefly because of enemy action, and the run had to be stopped because Hibernia couldn't supply oxygen. Advantage was taken of the ovens' cooling down to clean them and to examine the condition of the lining, for the fourth run, carbon lining was used instead of the clay used in the third run.

The fourth run was stopped after warming up because Hibernia couldn't supply oxygen, and the two warm-ups, 16 days later, by direct hits which suddenly cut off the gasification material. Both interruptions were too short to allow switching from the coke needed for warming up to hard coal.

Damage which the plant suffered from enemy action could in all cases be fixed on the spot without any particular shop-work and certainly in a shorter time than the fuel plant and Hibernia could again supply operating material. While this was the case, however, a new attack was made, so that from the end of July 1944 to the middle of January 1945 further moving the plant to foreign soil was impossible. Gasification of the local hard coal was not carried out. On 17/1/45 and 23/2/45 all the buildings were smashed by bombs and, with part of their contents, completely destroyed. Work was stopped, but the wrecked machines were cleaned out and the useable parts of the plant, which were difficult to build, were, as far as possible, removed and protected against splinters.

After this, the plant was not further damaged, it was left in its essential parts and can be put in operating condition in 2 to 3 months, without any extensive new construction.

In the following is a summary of conditions over three typical days in the third run.

DAY		1	2	3
(a) **Charge**				
Oxygen, (pure) Ncbm		32350	24890	25800
Percentage purity		81	90	90
Steam, tons		25.8	17.8	18.9
Auxiliary gas (own gas), Ncbm		16330	16070	17250
Coke (40-60 mm. dia.), tons		46.6	39.2	41.3
Analysis	Water 5% Ash 8.5% S and gas, 1.5% Heating value, 6865 kcal/kg.			
(b) **Yield**				
Oxygas (surplus)	Ncbm	105400	84665	89270
	CO_2	4.6	2.8	2.1
	O_2	0.1	0.1	0.1
	CO	65.3	70.4	71.0
	H_2	24.6	23.1	23.3
	CH_4	0.2	0.2	0.2
	N_2	5.2	3.4	3.3
	Hu	2600	2735	2760
Steam decomposition,%		82.1	90.1	90.1
Percentage gasification		85.6	85.2	86.7
(c) **Requirements for each 1 Ncbm Oxygas**				
Pure oxygen, Ncbm		0.30	0.29	0.29
Steam,kg.		0.25	0.21	0.21
Coke, kg.		0.44	0.46	0.46

copy 1

ITEM No. 30
FILE No. XXVIII-57

Copy No. 198

Classification Cancelled,
by authority of
The Joint Chiefs of Staff,
by Col. E. W. Gruhn.

~~RESTRICTED~~

DEUTSCHE ERDÖL A.G. ERDÖLWERKE "NOVA" DACHS II PLANT, EBENSEE, AUSTRIA

Newman + Weir

RESTRICTED

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

RESTRICTED

DEUTSCHE ERDÖLAKTIENGESELLSCHAFT ERDÖLWERKE "NOVA"

DACHS II PLANT

EBENSEE, AUSTRIA

Reported by:

Mr. L.L. Newman, T.I.I.C.
Mr. H.M. Weir, T.I.I.C.

On behalf of U.S. Technical Industrial
Intelligence Committee

CIOS Target No. 30/242
Fuels and Lubricants

June 20, 1945.

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
c/o G-2 Division, SHAEF (Rear) APO 413.

P. 1478

9 p. diagrs.

RESTRICTED

TABLE OF CONTENTS

Subject	Page No.
Location of Target	3
Personnel Interrogated	3
Description of Plant	3
Source of Crude Oil	3
Yields	4
Projected Capacity of Plants	4
Production of Lubricating Oil	4
Number of Employees	4
Conclusions	4

Personnel of Team

L.L. Newman Bureau of Mines U.S. Dept. of the Interior	H.M. Weir Petroleum Administration for War U.S.A.

DEUTSCHE ERDÖLAKTIENGESELLSCHAFT ERDÖLWERKE "NOVA"

DACHES II PLANT, EBENSEE, AUSTRIA

Location of Target

The Dachs II plant of the Deutsche Erdölaktiengesellschaft Erdölwerke "Nova", located underground near Ebensee, Austria was visited on May 28th and 29th by Messrs. L.L. Newman and H.M. Weir.

Personnel Interrogated

The following were interrogated:

Betriebsfuhrer	Dr. Friedrich Staiger
Machine Ingenieur	Egon Kasimir
Kaufmanndirektor	Erich Greiser

Description of Plant

This plant occupies three and one half of eight oil processing drifts which were excavated in the side of a mountain near Ebensee. Each drift is about 10 meters wide by 10 meters high at the top and extends some three hundred meters into the mountain. The drifts are interconnected by counter-gangways of somewhat the same dimensions, and are ventilated by a system of blowers which were designed for ten air changes per hour. The fans were located at the deepest point of the horizontal boring, pulling air in from one drift and discharging it through the next. Most of the details in plan and section are shown in Figs. 1 and 2.

The plant was built by the Reich, most of the excavation labor being supplied from a concentration camp established nearby for political prisoners from occupied countries. The mechanical work was done by German and Austrian labor. The oil refining operations were turned over to the Deutsche Erdölaktiengesellschaft who had been operating one refining plant of similar character near Vienna.

The plant equipment consists principally of topping units which include eight 16 plate columns, each with a capacity of 100 tons per day. The flow diagram of operations is given as Fig. 2 (Labelled #5).

Although a capacity of 800 tons per day of low octane gasoline and diesel fuel was planned the plant never produced over 600 tons per day because of difficulty with railroad service from Gmunden (Austria) in transporting the raw crude.

Source of Crude Oil

The crude oil was pumped from wells in the Zistersdorf Fields near Neusiedl in the Danube region and was transported by river barges to the railhead where it was transferred to rail tank cars. The oil processed was the lighter of two crudes from Zistersdorf, namely a paraffin base oil having a specific gravity of 0.865 to 0.900. The wells range in depth from about 800 meters to 1200 meters. One fourth of the oil reaches the surface by rock pressure and the remainder by pumping.

Yields

The yields were as follows:-

Gasoline	10-12%	58 Octane
Diesel oil	35%	52 Octane
Residue	53-55%	0.920 Sp.Gr.

Ten percent of the residue was used for heating operations in the refinery, 45% sold to the electricity works, and the remaining 45% was sold for fuel oil. The gasoline was heavy (8 to 10% off at 100°C) and was cut with benzol and lighter gasolines.

Projected Capacity of Plants

About 100,000 tons of monthly throughput was planned for all of the projected underground plants. This particular plant was to make one fifth of the production by use of the eight columns, the remaining production being divided in six other plants with two columns in each at the following locations:-

Jürwitz
Weitenegg
Hausenkirchen
Spitz
Statzendorf
Petzenkirchen

Production of Lubricating Oil

The production of lubricating oil was planned in subsequent plant extensions which were to be made in the 4½ excavated but still unused bays. Some of the storage facilities were already installed, but no other equipment as Fig. 3 shows.

Number of Employees

The plant employed 150 operating men, but had only 100 men available for the resumption of operations which the Military Government ordered for the production of 400 tons of gasoline per day to take care of essential transport needs.

Conclusions

Except for the fact that the plant is underground there is nothing remarkable about it. For the relatively small amount of motor fuel that it is capable of producing the amount of investment required

to put it underground would be considered absurd in any peacetime economy.

L.L. NEWMAN
Bureau of Mines
U.S. Dept. of the Interior

H.M. WEIR
Petroleum Administration for War
U.S.A.

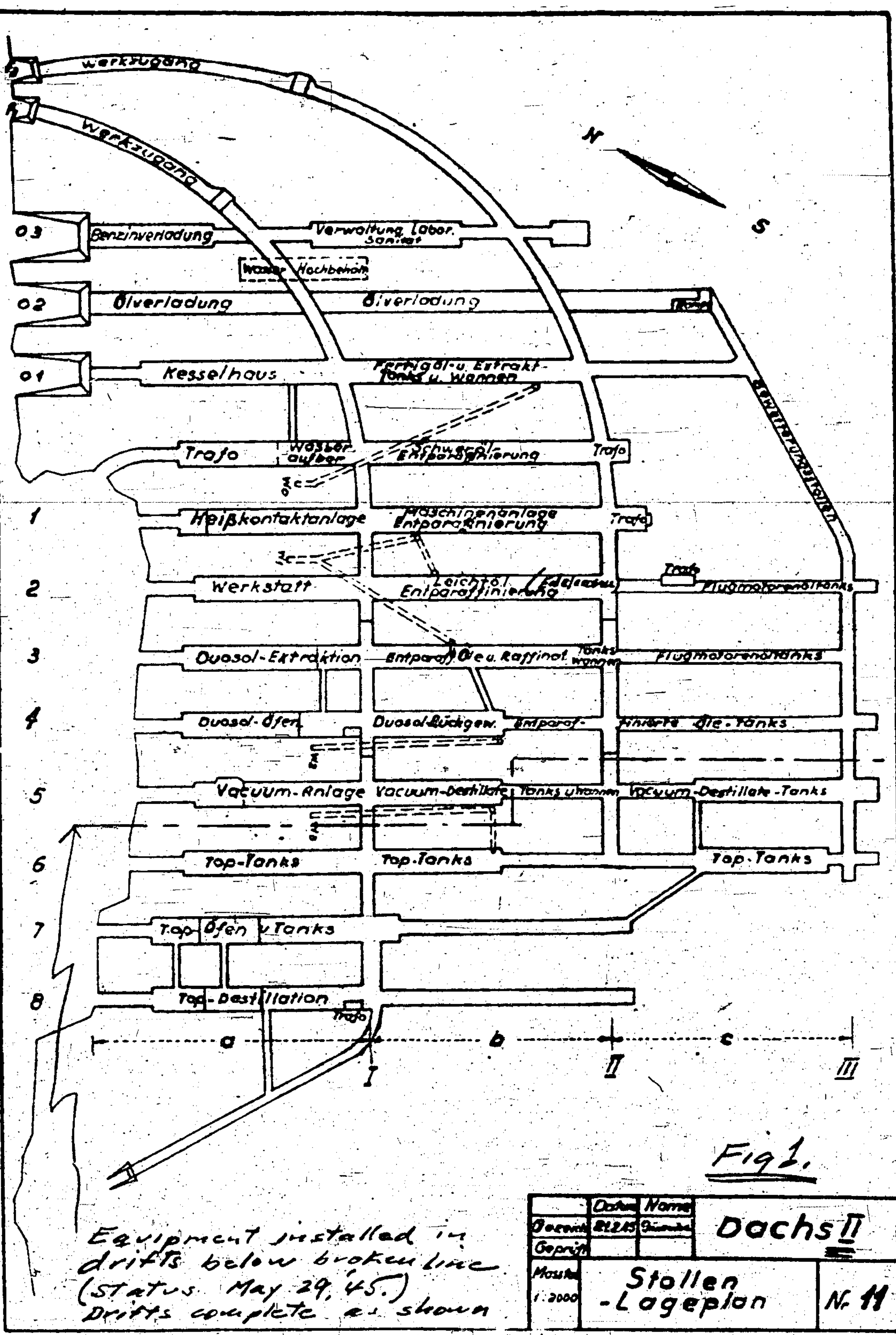


Fig 1.

Equipment installed in
drifts below broken line
(status May 29, 45.)
Drifts complete as shown

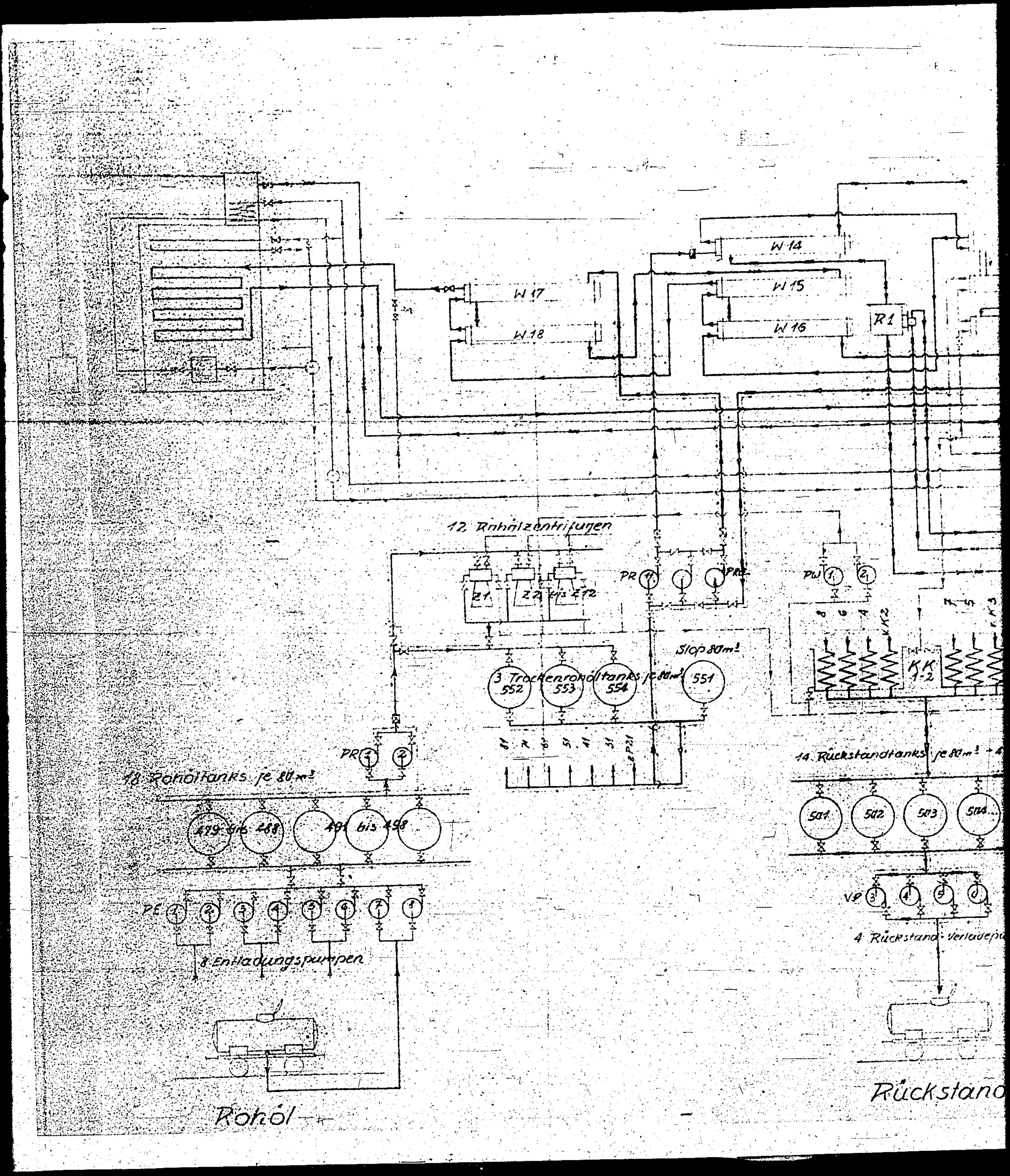
	Datum	Name
Gezeichn.	21.2.45	[illegible]
Geprüft		

Maßstab 1:2000

Dachs II

Stollen
-Lageplan

Nr. 11


W 14
W 15
W 16
W 17
W 18
R 1
12 Rohölzentrifugen
Z 1
Z 2 bis Z 12
PR 11
PR 12
PW 1
2
8
6
4
KK 2
KK 1-2
7
5
KK 3
3 Trockenrohöltanks je 80 m³
552
553
554
Slop 80 m³
551
81
71
61
51
41
31
BP 21
PR 1
2
18 Rohöltanks je 80 m³
479 bis 488
491 bis 498
PE 1
2
3
4
5
6
7
8
8 Entladungspumpen
Rohöl
14 Rückstandtanks je 80 m³ + 4
501
502
503
504...
VP 3
4
5
6
4 Rückstand-Verladepu
Rückstand

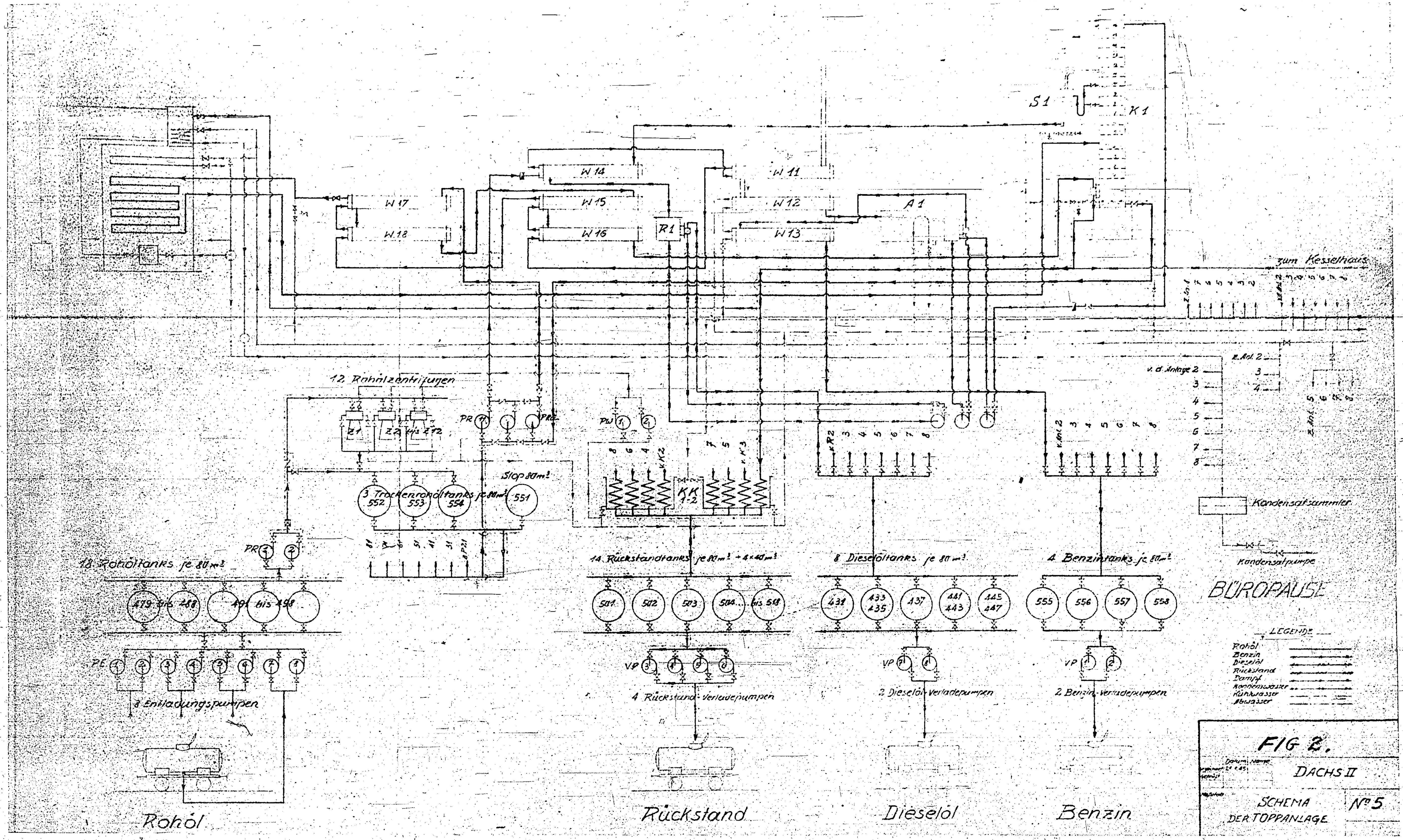


FIG 2.

DACHS II

SCHEMA
DER TOPPANLAGE

Nº 5

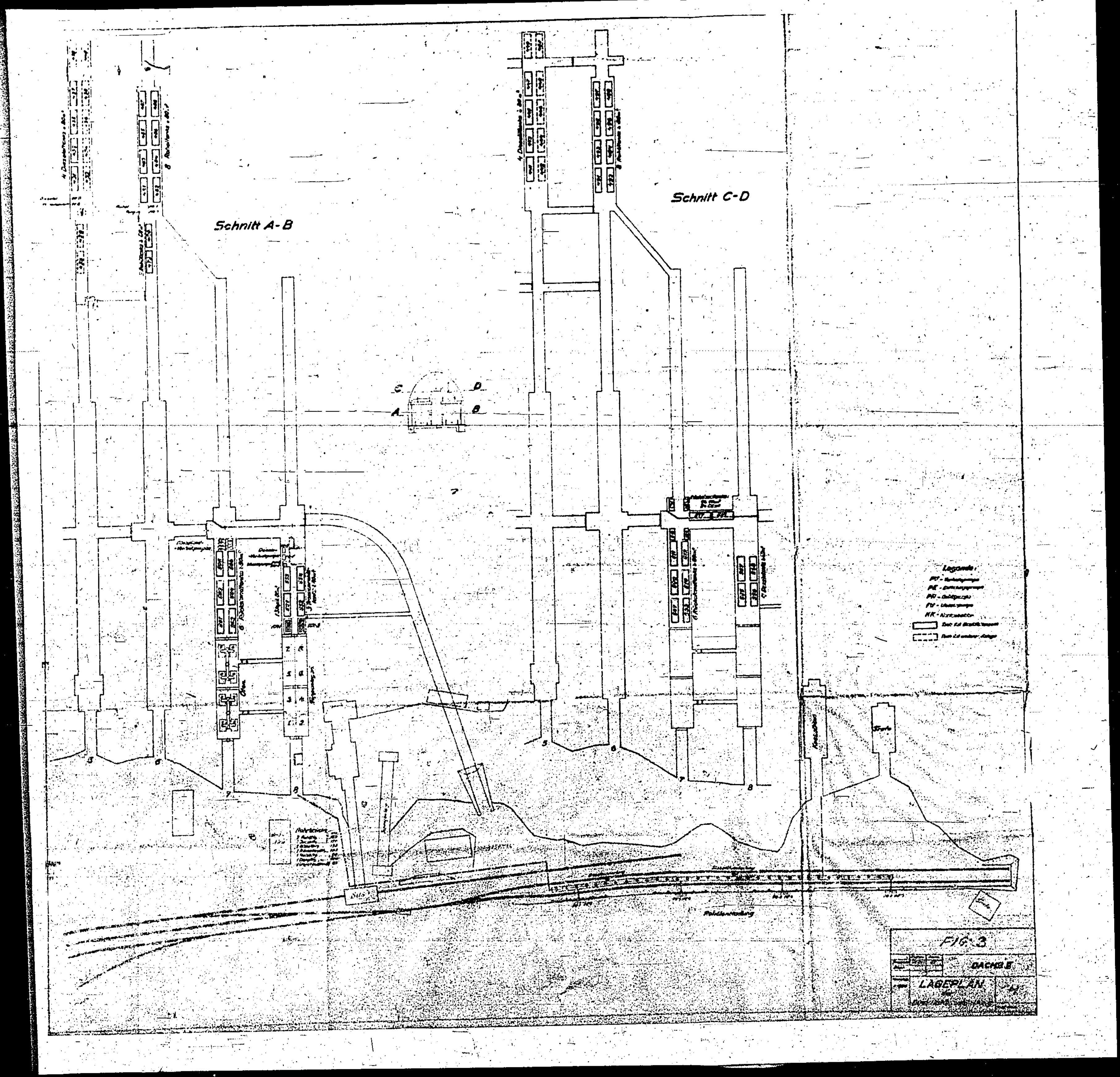

Schnitt A-B
Schnitt C-D
C
D
A
B
Legende:
KK - Kondensator
DACHS II
LAGEPLAN

FIG. 3

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Report No. 285

OFFICE OF THE PUBLICATION BOARD

DEPARTMENT OF COMMERCE

WASHINGTON, D. C.

WIRTSCHAFTLICHE FORSCHUNGSGESELLSCHAFT, M.B.H.

AUSSENSTELLE MUNCHEN I (WIFO I)

STOCKDORF NEAR MUNICH

Reported By:

L. L. Newman - U.S.A.
H. M. Weir - U.S.A.
On behalf of the U.S. Technical
Industrial Intelligence Committee

[1945]

This report has been declassified and released to the Office of the Publication Board by the War and Navy Departments.

NOTE: The Publication Board, in approving and disseminating this report, hopes that it will be of direct benefit to U. S. science and industry. Interested parties should realize that some products and processes described may also be the subject of U. S. patents. Accordingly, it is recommended that the usual patent study be made before pursuing practical applications.

4p

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TABLE OF CONTENTS

SUBJECT	PAGE NO.
Location of Target	3
Personnel Interrogated	3
Description of Plant	3
Source of Fuel	3
Kind of Product	4
Stocks of Fuel and Lube Oil	4
Employees	5
Miscellaneous	5
Conclusions	5

PERSONNEL OF TEAM

L. L. Newman, Bureau of Mines
U. S. Department of the Interior

H. M. Weir, Petroleum Administration for War,
U.S.A.

WIRTSCHAFTLICHE FORSCHUNGSGESELLSCHAFT, M.B.H.

AUSSENSTELLE MÜNCHEN I (WIFO I)

Stockdorf near Munich

Location of Target

The WIFO I underground blending station of "Wirtschaftliche Forschungsgesellschaft, m.b.h., Aussenstelle München I" located near Stockdorf in the Munich area was visited on May 25, 1945, by Messrs. L. L. Newman and H. M. Weir.

Personnel Interrogated

The following were interrogated:

Kaufmanscher-leiter Arthur Henneg
Mach. Ing. Hans Balluncil
Dipl. Ing. Fanta
Chemist Erich Vollbrecht, formerly employed by the Vacuum Oil Company at Hamburg

Description of Plant

The plant was built in 1936. It consists essentially of 30 gasoline storage units, each with a capacity of 3,300 cubic meters, and 20 lubricating oil storage units, each with a capacity of 300 cubic meters. Tanks for 100 tons of tetra ethyl lead were also on hand. All the tanks are buried so that there is a layer of 5 meters of earth above the top of each. All the interconnecting pipe lines are laid 5 meters below the surface. The tanks are scattered in an irregular pattern over an area of 220 hectares (2,200,000 square meters). In spite of the 5 meters of cover and the dispersion of the tanks many were destroyed by Allied air-raid bombs.

Source of Fuel

The fuel was shipped in railroad tank cars to the plant from numerous sources; Lead was supplied by the I.G. Farben from plants near Brandenburg and Frose. The gasoline supplied from Bohlen was 60 Octane, and according to Dr. Vollbrecht was not very good for use in this blending station.

The blending operations were conducted on the basis of orders received, both as respects quantity and quality desired.

Kind of Product

Three aviation gasolines were produced by blending, Type A_3 had and 84 Octane rating and included 0.045% to 0.05% lead and 33% absolute ethyl alcohol; type B_4 (blue) had an 89 Octane rating and included 0.120% lead, but was alcohol free; type Z_3 (green) had a 100 Octane rating and included 0.120% lead and iso-octane. The percentage of lead is expressed in terms of the total volume of finished gasoline. The lead fluid was prepared by mixing 130 liters of tetraethyl lead as received with 70 liters of ethylene dibromide to produce 200 liters blending fluid.

In addition to the aviation gasolines, a diesel-like distillate, J_2, was supplied for aircraft engines which were really turbines of unstated horsepower. In normal flight these consumed 2 tons of fuel every 40 minutes in a two-engine plane.

The lubricating oils supplied were:

Motor oil - winter grade, 4.5 Engler (50°C)
summer grade, 8 " "
Differential oil - summer grade only - 17 Engler at 50°C

All of these had a Pöhl-Höhe of 2.0 - 2.1

Stocks of Fuel and Lube Oil

The following stocks of fuel and lube oils were on hand on the date of the visit:

1100 cu. m. of motor fuel (80 Octane)
No aviation fuel
2700 cu. m. power fuel (Krafft spirit)
Some methyl and some ethyl alcohol the bulk of which (say 90%) is methanol
9000 cu. m. diesel oil, consisting of 6000 cu. m. distillate and 3000 cu. m. Romanian crude not blended.
A quantity of tetraethyl lead consisting of some unknown part of 700 individual barrels plus most of 100 tons in tanks. The bomb damage to these tanks and drums had not yet been fully surveyed at the time of our visit.

Employees

The normal number of persons employed for the operation of this plant was 220, including 40 salaried employees.

Miscellaneous

Construction of "WIFO II" was begun in 1940 but the plant was never completed because of the plant's relatively low priority in the Nazi scheme of utilizing labor and there were frequent starts and stops in construction.

Conclusions

The value of an underground system of storage is questionable for a free peace-time economy. Though the German engineers claim for it the important advantage of uniform storage temperatures and consequent reduction in evaporation losses, these savings may be frequently offset by losses through undetected leaks in the pipe lines buried 5 meters beneath the surface.

L. L. Newman
H. M. Weir

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Item 30
T.O.M-29

copy 1

Report No. 291

OFFICE OF THE PUBLICATION BOARD
DEPARTMENT OF COMMERCE
WASHINGTON, D. C.

FRENCH OIL SHALE INDUSTRY

Reported by
WILLIAM W. ODELL
EMILE L. BALDESCHWIELER

(11 July 1945)
C.I.O.S. Target 30/Opportunity

This report has been declassified and released to the Office of Publication Board by the War and Navy Departments.

NOTE: The Publication Board, in approving and disseminating this report, hopes that it will be of direct benefit to U. S. science and industry. Interested parties should realize that some products and processes described may also be the subject of U. S. patents. Accordingly, it is recommended that the usual patent study be made before pursuing practical applications.

[1945]
9 p.

CONFIDENTIAL

TABLE OF CONTENTS

SUBJECT PAGE NO.

Introduction 1

Data in re: Individual Plants Inspected:

I. Autun 1

II. St. Hilaire 3

III. Lavernhe and Severac-le-Chateau 5

Description of the Petit Retort 7

Personnel of Team:

William W. Odell, U.S. Bureau of Mines,
Emile L.Baldeschwieler, U.S. Petroleum Administration for War.

CONFIDENTIAL.

INTRODUCTION.

This is a brief report of visits to oil-shale distillation plants in France, made by William Odell and Emile L. Baldeschwieler during the period June 26 to July 1, 1945, inclusive. The French Government is exerting efforts to develop the known deposits of shale, particularly those in central and south central France, for the purpose of supplying a portion of their requirements of motor fuel and lubricating oil; that country does not have a natural ready source of supply of petroleum. Because of the efforts being made in France and also on account of the general interest of the Allied Nations in the production of oil from other raw materials than petroleum, it was desirable that information should be obtained as to advancements made in the art in France. The inspection trip was taken with the permission of the French Government and the authors were accompanied by a French engineer, M. Bernard de Resseguier, who also traveled under permission.

The officers and other representatives of the companies whose plants were visited were most gracious and accorded us every facility for obtaining information from their records and notes.

I. - AUTUN.

The first plant visited was that of the Societe Minière des Schistes Bitumineux at Autun. It was found that the latter company is operating 3 batteries of 40 retorts each of Pumpherston type. These retorts are exact duplications of the well-known Scotch units in which shale is distilled in Scotland. Crushed shale varying in size from 4 inches down to about ½-inch is treated continuously by passing it downward through the vertical retorts which are externally heated by burning gases. The upper portion of each retort is made of metal (iron) and the lower portion, which is subjected to higher temperatures, is made of refractory material. The capacity of each retort was initially 3 tonnes per day of 24 hours of oil shale; this was at a time when super-heated steam only was passed upwardly through the mass of shale in process, but later results obtained when both air and steam were introduced adjacent to the bottom of the retort during processing showed an increase to 7 tonnes of shale per day per retort. Thus the total maximum capacity with all retorts operating is 840 tonnes of shale per 24 hours. The air is used internally to burn some of the carbon which otherwise would pass out as carbon in the spent shale; in this manner additional heat is generated for processing without the consumption of additional extraneous fuel. Although there were 350m^3 of gas generated from each tonne of shale treated, an additional amount of gas (producer gas separately generated) was required for heating the retorts. The amount of the producer gas used was the equivalent of 100 kgs. of coal per 100 liters of crude oil recovered; the coal contained 30% ash.

The shale treated at Autun appears to differ chemically from that of most American shales, at least the oil obtained from heat-treating the shale is said to contain less wax. The oil characteristics are not reported here because they are published rather completely elsewhere. However, it is noted that the shale being treated yields 6 to 7 percent of total readily condensable oils by the Fischer assay retort and that in plant operation the yield is 85 percent of Fischer yield. The shale contains 2.0 percent of total sulphur of which 1.5 percent is pyrite and the remaining 0.5 percent is organic sulphur. The softening point of the shale is 1100°C.

The mining is by underground methods, the shale being 325 feet below the surface in a single seam varying from 3.25 to 11 feet in thickness. The mining costs are high.

The number of mean employed operating the plant are given as follows:

Men operating mine	500
Men in other operations	400
Total	900

Thus, at full capacity of 840 tonnes per day, the labor amounts to 1.07 men per tonne of shale mined and treated.

It is noted that the usual condensing and cooling methods did not remove all of the oil and that there was recovered by oil scrubbing the cooled gas 7 to 8 liters (1.85 to 2.1 gallons) per cubic meter of shale, that is, per 1.2 metric tonnes of shale. This amounts to 1.4 to 1.6 U.S. gallons per U.S. ton (2,000 lbs.) of shale treated. The total recovery of oil was 66 liters per tonne, of which 6 liters were obtained by oil scrubbing. In U.S. units the recovery was 15.8 U.S. gallons per ton of 2000 lbs., of which 1.44 gallons was recovered by oil scrubbing. The crude oil has a specific gravity of 0.90.

The shale used had a relatively low lime content and there were no difficulties regarding residue disposal. A representative analysis of the spent shale is as follows:

SiO_2	51.52 % by weight.
TiO_2	0.87
SO_3	1.92
Fe_2O_3	17.50
Al_2O_3	6.61
MnO	Trace
CaO	5.75
MgO	0.50
K_2O	5.07
H_2O	0.70
Loss on ignition.	9.00
	99.44

The company has concluded that the developments in the art of treating oil shale have been such that they are no longer satisfied in building this type of retort and have expressed their intention to replace these retorts as fast as they wear out by a different type of retort which appears to offer substantial economies and increased earnings. The retort which the company has decided to employ, replacing the Pumpherston unit, is that which has developed at St. Hilaire, and which is known as the Lantz retort.

II. ST. HILAIRE.

The next plant to be visited was the St. Hilaire (Allier) installation of the Société Chimique de la Grande Paroisse. A pilot plant has been in operation there since 1938, treating 80 tonnes of shale daily. The apparatus and process employed were unique and different from those employed elsewhere for treating oil shale. The retort which is vertical is comprised of two distinct but connecting sections; each section is traversable by a gas stream. The lower section is the distillation portion of the retort and has a rectangular cross-section. Its outer walls comprise a series of superimposed baffles which support the shale and which function as louvres through which gases can enter the retort. This section is encased in a metal shell and means are provided whereby hot gaseous products evolved in the process are caused to pass horizontally through the shale confined between the baffles while said shale is passing continuously downwardly through the retort. A circulating fan is supplied for propelling the gases and a preheater is also connected in the system whereby the circulating gases are preheated to 450-500°C. before entering the retort. Separately generated producer gas is employed along with the excess retort gas in the preheater to supply the necessary heat energy. The waste or stack gas from the preheater is caused to pass through the shale in the upper section of the retort whereby said shale becomes heated to about 150°C. before entering the distillation step in the lower section. In this process the shale which is initially supplied to the retort in crushed $\frac{1}{2}$-2" size pieces is heated gradually to a temperature not exceeding that of the circulating gases, namely 450-500°C. and the oil and vapors evolved are immediately removed from the shale in process and passed into the circulating system from which 10% is continuously withdrawn for the recovery of shale oil and gasoline.

The spent shale is discharged from the retort continuously into water from which it is removed by a screw conveyor. This quenching step not only cools the spent shale but generates steam through évaporation of water, which steam is mixed into the circulating gas stream. The spent shale discharged from the conveyor still contains 10 to 12% by weight of carbon which ignites in the air and burns in the residue pile in spite of the quenching treatment.

The shale used has a calorific value of 1900 calories per kilogram and that of the spent shale is 950. The oil content of the shale according to the Fischer retort test is 6.8% by weight; substantially all of this is recovered in the processing described. It is noted that the spent shale contains 1.2% of sulphur much of which is burned to sulphur dioxide in the discharging residue. During the processing there is evolved 25 m^3 of gas per tonne of shale and the calorific value of this gas after removing the gasoline vapors therefrom is 6000 to 6300 calories per m^3.

The company felt that the performance of the pilot plant was so satisfactory that they were warranted in in building a larger size unit; one such unit designed to treat 500 tonnes of shale per day is under construction. It is estimated that the water requirements (make up water) will be 125 m^3 per day for each 500 tonne unit.

In other words, 70 U.S. gallons of water are required for each 2000 lbs. of shale treated. The overall power consumption for handling and treating the shale from the mine clear through the plant handling and treating the shale from the mine clear through the plant is estimated to be 9-10 KW per tonne of shale. Two gas producers are installed for a 500-ton unit, one being reserved as spare. Sixty pounds of coke are used in gas producing for every 2000 lbs. of shale treated. The total fuel required for retorting in the 500-ton unit is 60 lbs. of coke and 856 cu. ft. of shale gas per ton of 2000 lbs. of shale. The coke used is: water 5%, combustible 75%, and ash 20%, and it has a calorific value of 5500 calories per kilogram or 9900 B.T.U. per lb.

The future plans of this company include expanding the capacity to 2000 tons of shale per day. A burning device has been developed whereby the heat energy represented by the 12% of carbon in the spent shale will be utilized in the future operations. This will no doubt eliminate the necessity of using coke for producer gas.

The total yield of oil is substantially 132 lbs. per 2000 lbs. of shale.

III - Lavernhe and Séverac-le-Château.

The next and last plants visited were at Lavernhe and Séverac-le-Château in south-central France. The Lavernhe plant was formerly operated by the Société de Bitumes et Schistes, a subsidiary of Laverhe Monthand et Aulnoye, while the Séverac plant was operated by the Compagnie Industrielle des Schistes Bitumineux de l'Aveyron (CISBA), a subsidiary of the Compagnie Alais Forges et Camaigne (AFC). These two present companies have now merged and are being operated under the name of Compagnie Industrielle des Schistes Bitumineux de, l'Aveyron (CISBA).

It was found at Lavernhe that an entirely different type of retort for processing shale had been rather completely developed. This retort is known as the Marécaux retort, after the inventor who is a chemical engineer for CISBA.

This apparatus has been in operation on a pilot scale for more than a year, treating up to 30 tons of shale per day. The retort is vertical and comprises two concentric upright cylindrical shells which confine between them a mass of downwardly moving crushed shale in process. Means are provided for charging crushed shale (3 to 25 mm) into the upper portion of the retort and for discharging the treated shale continuously at the bottom. The shale is externally heated through the outer cylindrical metal casing and the gases are withdrawn adjacent the bottom of the retort. The retort differs materially from the others in that the inner cylinder rotates slowly and has on its outer surface helical members which cause the particles of shale in the annular space to be agitated and turned over during their downward travel through the retort, thereby facilitating heating of the shale. No air is introduced and therefore there is no internal combustion in the operation of this retort. Means are provided, however, for introducing superheated steam at 400°C. into the annular mass of shale at a plurality of levels to facilitate distillation. The distillation gases are withdrawn into the inner cylinder substantially as they are formed in the shale mass, thus minimizing cracking of the oil vapors. All of the gas evolved from the shale is used for heating the retort along with an additional amount of producer gas. The shale charged is previously treated with the hot stack gases for the purpose of economizing heat and raising the temperature of the raw charge to 150°C. The rotating inner cylinder has a water seal connection which prevents air entering the retort at the top.

The total yield of oil from shale from this retort is 41.83 kg. per metric ton of shale treated which amounts to 92% of the standard Fischer retort yield. In addition, there is present in the gas prior to scrubbing 3.70 kg. of gasoline; this gasoline is not now being recovered because of the high content of H_2S in the gas and because the plant is only a pilot plant. The yield of gas per metric ton of shale treated is 13.6 m^3 and 25% of this gas is H_2S. The total amount of producer gas used in the pilot plant per metric tonne of shale treated is 330 m^3. The steam used amounts to 33.4 kg. per metric tonne of shale. Although this furnace has not been tried on a larger scale, the smoothness of the operation of the pilot plant indicates that the operation should be equally smooth on a larger scale with substantially the same operating results. The shale residue contains 4% carbon.

The company is so satisfied that this type retort is economical and an improvement over standard practice that two commercial size units of 120 tons each are under construction at their plant in Severac.

Since the merger of the two companies, plans have been formulated for a large scale study of shale processing. It is proposed that there will ultimately be installed shale processing equipment capable of treating 3000 tons of shale per day. At this plant there is now under construction one Petit furnace, one Lantz retort and two Marecaux retorts as named above. The Petit retort is being installed chiefly because the material is available, and not because it offers any advantages over the other retorts developed in France.

Description of the Petit Retort.

This retort, developed at Grenoble with the help of CISBA on a pilot scale only, has never been operated on a scale larger then 8 tons per day. The process differs from others chiefly from the fact that the shale, prior to the distillation, is wet with the shale oil from previous distillations; the oil-wetted shale passes through a horizontal rotating, externally heated, cylindrical retort comprising two concentric cylinders. The shale in process is caused to pass serially through the inner cylinder and back to the reverse direction through the spaces between the said cylinders, discharging substantially at the inlet end of the retort. The object of the oil-wetting treatment is to cause solvent action and depolymerization of the kerogen.

Due to the fact that only a small portion of the retort heating surface is in contect with the shale in process at any one time, and because the general nature of the methods employed for applying heat to this retort, it is doubtful that it will be of interest in the treatment of American shales.

The writers wish to acknowledge the whole-hearted cooperation of the directors and officers of the companies contacted and their engineers who were most cordial and helpful to us in obtaining the picture of the shale situation in France. Very definitely, advances in the art have been made as a result of the researches of the French scientists.

W. W. Odell.
E.L.Baldeschwieler.

FR.

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OIL TARGETS IN RUHR AND HANOVER AREAS

Oriel, J. A.

SECRET

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

~~SECRET~~

OIL TARGETS IN RUHR AND HANOVER AREAS

Reported By

Col. J.A. ORIEL
Min. of Fuel & Power

CIOS Targets Item 30
Fuels & Lubricants

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear), APO 413

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TABLE OF CONTENTS

Subject .... Page No.

INTRODUCTION AND PART I.
Interrogation of Dr. Brockhaus at Hanover 3

PART II.
The Underground Lubricating Oil Plant at Porta 4

PART III.
Refinery of the Deurag/Nerag at Misburg, and the fields at Nienhagen controlled by these Companies and the Elwerath 7

PART IV.
Nienhagen Field 12

PART V.
Underground Hydrogenation Plant in the Bueckeberg Mountains 13

PART VI.
Ruhr Chemie Installation at Holten 14

PART VII.
Bottrop 18

PART VIII.
Scholwen 18

PART IX.
Gelsenkirchen 19

PART X.
The Topped Crude position in Germany 19

APPENDIX I.
List of "Ofens" 21
List of "Rosts" 22

APPENDIX II.
Code Names 23

Personnel of Team

Col. J.A. ORIEL

REPORT ON A VISIT TO GERMANY FROM 4th TO 21st APRIL, 1945, BY COL. J.A. ORIEL.

INTRODUCTION.

Although several plants were covered in this visit, the report will be written in the order of what I consider to be the greatest importance.

Part of these visits were undertaken with the Ruhr Party under the leadership of Dr. Parker, whilst the Hanover area was investigated together with Mr. Evans of C.A.F.T. and Mr. Vincent, who was kindly released from C.A.F.T. operations to accompany me on this work.

(I) INTERROGATION OF DR. BROCKHAUS AT HANOVER.

Dr. Brockhaus, the Manager of Elwerath, Deurag and Nerag Group, was found to have been Chairman of the Petroleum Planning Committee for the Greater Reich during the war. The other members of the Committee were:-

Vice-Chairman: Dr. Boeder, the Rhenania Company.
Dr. Engel, the Vacuum Oil Company.
Dr. Schlecht, the Deutsche Erdol A.G.
Dr. Brunk, the Deutsche Gasolin.

Dr. Brockhaus gave me a list of the dispersed refineries throughout the country, known as "Ofens". There are forty units in seventeen locations, and the list is attached.

He also gave me a list of smaller batch still distillation equipment, known as "Rosts", which are built in the ruins of factories throughout the country.

Of "Dachs", that is underground lubricating oil plants, he gave us four locations. The one at Porta, Nr. Minden, is being reported on separately as it was visited by the writer. The second was being built by the Rhenania Company and was situated in Osterode in the Hartz Mountains. This one was said to be somewhat larger than the Porta installation, and probably also had furfural extraction. It was mainly to operate on Vienna crude, but would not be completed for another three or four months. The

D.E.A. were erecting one at Ebensee in Austria, and were carrying out dewaxing using dichlorethane. He thought it was operating in conjunction with the "Ofens" 23 to 30. Lastly there was an "Ofen" at Alte Post, Nr. Pirna, operated by the Deutsche Gasolin, again probably working on dichlorethane.

He knew of only two "Taube", which are cracking plants, one by the Wintershall A.G. at Cassel, near Messinghausen, and another by the Continental Oel A.G. on the Danube, but he did not know at the moment the condition of these two plants.

A "Jacob" apparently is a primitave cracking plant working in conjunction with an "Ofen", and these were projected in conjunction with many of the "Ofens", but the one at Boegerhof was the only one that he knew of that had had work definitely carried out upon it.

He had nothing new to offer regarding "Schwalben" or "Kuckuck".

(II) THE UNDERGROUND LUBRICATING OIL PLANT AT PORTA.

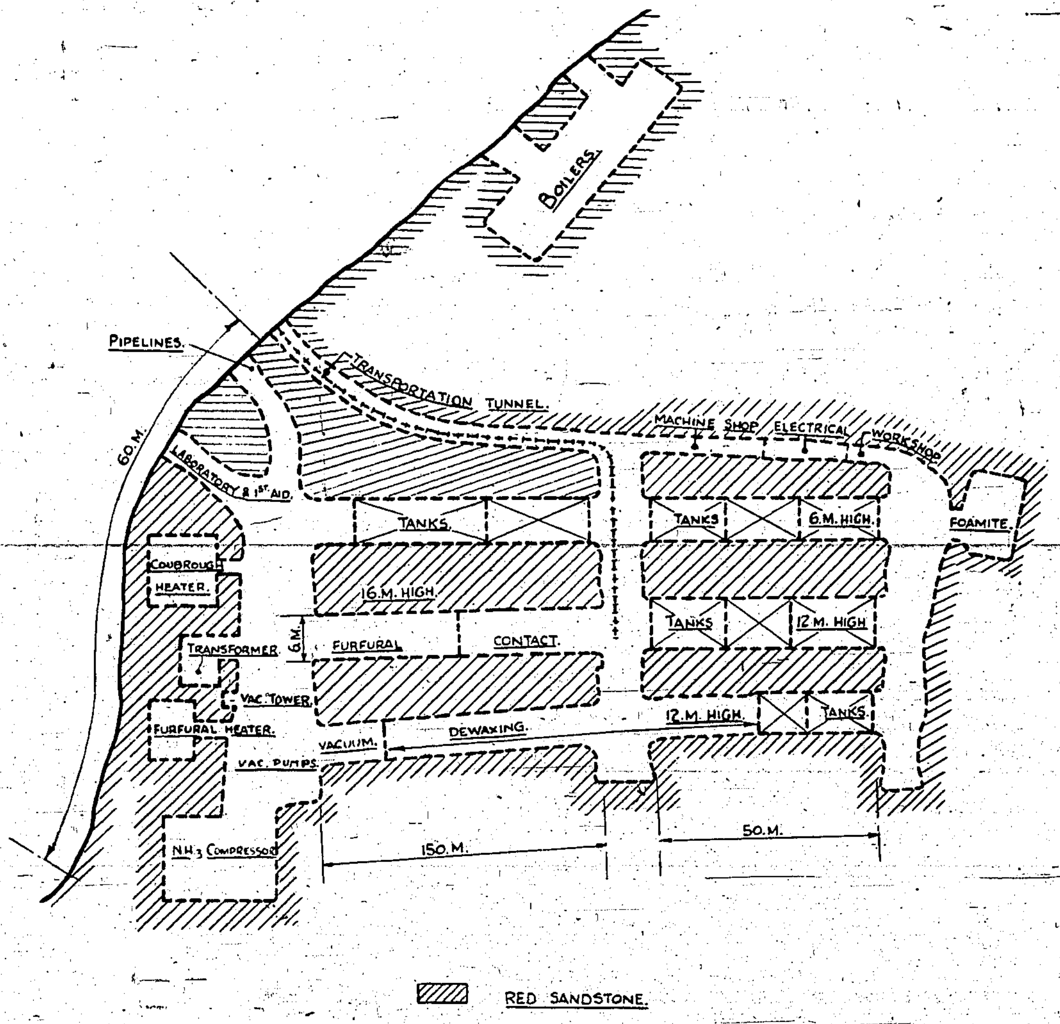
Attached is a sketch plan showing how the lubricating oil plant at Porta is installed in the underground workings. The exact situation of this plant is shown on Army Map P. 3 (Detmold), Scale 1/100,000, Co-ordinates 807052.

The fitting in of the equipment into such a confined space was a really remarkable engineering feat. and as one can see from the drawing, a considerable amount of equipment, valued approximately at £500,000 (erected) has been installed underground. The plant is almost ready for operation, and although it has not been operated could, I think, be operated as soon as the pipelines are completed, that is with about two or three weeks' more work. The capacity of the plant for lubricating oils is 280 tons a day input of topped crude, which is received by tank wagon from various dispersed distillation units. The process is reasonably clear from the drawing, consisting as it does of a Coubrough unit, which takes off gas oil, spindle oil, neutral oil and a light cylinder in the first stage, and in the second stage (using a 1:1 kerosine mixture and a vacuum of 25 mm.) distils off a heavy cylinder, leaving about 20% bottoms. In case of aero-engine oils, a distillate having a 12° Engler viscosity at 70°C. was prepared, leaving a 40/50 penetration asphalt as residue.

The fractions were furfural treated, dewaxed by solution in a mixture of trichlorethane and trichloremethane, and then contact filtered, using clay. No acid treatment was used as during the war colour was considered to be immaterial.

The only interesting technical feature of this underground plant was the dewaxing plant, which was of quite a novel construction. They had decided to give up the conventional Dorr-Oliver filter which they had originally installed at Misburg, chiefly on account of distortion of the filter drums. A new type of filter had been devised by the Edeleanu Gasellschaft, but unfortunately owing to the arrangement under which these plants are built, it was impossible to obtain any drawings. The plant is built entirely under the Geilenberg plan, and the work is all carried out under the Organisation Todt, and until the plant has been completed no drawings are handed over to the operating company. However, all the information should be available with Dr. Grote, at Bingerstrasse 11, Berlin. The plant was designed by Edeleanu, but constructed by R. Wolf of Magdeburg.

The main change in construction from the usual Dorr-Oliver type of filter is that the filtering is done on a type of endless chain, allowing each section to have a more thorough washing than is the case with the Oliver filter. No results were available, but this new construction should be followed up as actively as possible.



Underground Lubricating Oil Plant at

Porta nr. Minden

Westphalia.

(III) REFINERY OF THE DEURAG/NERAG AT MISBURG, AND THE FIELDS AT NIENHAGEN CONTROLLED BY THESE COMPANIES AND THE ELWERATH.

Present on the plants were:-

Refinery Manager:	Herr von Eynatten.
i/c Lubricating oil (Nerag):	Freeherr Herreman.
i/c Cracking (Deurag):	Herr Meditsch.
i/c Laboratory:	Dr. Hundsdoerfer.

The organisation of this Company was somewhat difficult to understand as part of it was owned by the Elwerath Company and part Government owned. Deurag was responsible for the distillation and the cracking part of the establishment, whilst Nerag was responsible for the lubricating oil production.

The plants in existence were as follows:

A. Deurag.

(1) An atmospheric topping plant of the continuous tube still variety, with an intake capacity of 700 tons a day. This is quite a conventional unit and is in condition to operate.

(2) A U.O.P. coking plant, working at 18 atmospheres. Its capacity is 330 tons/day, and the feed consists of a mixture of:-

(a) Excess topped crude;
(b) Furfural extracts;
(c) Asphalt from the propane deasphaltisers;
(d) Wax from the dewaxing plant.

This plant is also in a suitable condition to operate.

(3) A U.O.P. polymerisation plant, designed for 40 tons/day of liquid propane/butane mixture containing 30% unsaturates. The absorber, designed for operation at 17 atmospheres, and the stabliser were in working condition, but the polymerisation plant itself is not in a suitable condition to work and has had an unfortunate life history from the beginning.

It was built in 1939 by the U.O.P., but owing to the difficulties of transport when war broke out no catalyst arrived. Finally it was decided to obtain catalyst from the U.O.P. via Japan, and the order was placed and executed. The catalyst got as far as Russia, when the war between Russia and Germany broke out, so that naturally it never arrived in Germany. A correspondence then began with the Ruhr Chemie regarding the provision of catalyst, and Ruhr Chemie provided a phosphoric acid catalyst which they claimed had proved suitable as a polymerisation catalyst in other plants. Within twenty-four hours of using this catalyst it was caked into a solid block, and considerable difficulty was experienced in digging it out. Ruhr Chemie, however, insisted that this was a suitable catalyst, and further experiments were carried out on the Ruhr Chemie pilot plant, where they claimed to get success. Deurag then built a pilot plant, and again blocked everything up on this in a very short time. However, by perseverence they discovered that one of the reasons for their troubles was air getting in with the fields gas from Nienhagen, and they took steps to exclude air from the gas to the main polymerisation plant, having already had a run of at least 240 hours on the pilot plant. Unfortunately for them, however, at this stage the bombing began to be much more of a nuisance, and every time they were ready to start the polymerisation plant it was bombed. Finally the bombing became so intense and the polymerisation problem the least of their difficulties, that the repairs to the polymerisation plant were always placed at the bottom of the list after each bombing, with the result that what is left of it has still never been operated

(4) A pressure distillate treater. A four-stage, 180 tons/day continuous sulphuric acid treater. The plant is in sound workable condition, but the Doctor sweetening plant has been destroyed and no attempt at repair has been made.

(5) A small rerun unit for treated distillate, capable of distilling 180 tons a day of treated pressure distillate. The light gasoline cut is combined in the rectifying column with Casinghead gasoline from the gas recovery unit. The heavy gasoline has an end point of 210°C. This equipment is all in order.

B. Nerag.

(1) A combined atmospheric topping and vacuum crude distillation unit, intake 600 tons/day. Designed by the Texaco Development Company. The atmospheric unit on Nienhagen crude gives:

15% gasoline of 210°C. end point;
20% kerosine
5% gas oil.

In the vacuum unit, 30% on the crude is distilled over,

consisting of a little gasoil, spindle, neutral and light cylinder oils, leaving a 30% bottoms on crude. This unit is in working order.

(2) Propane deasphaltising plant. It is an I.G. modification of the Kellog system, capacity 180 tons/day, and built by Uhde. It operates with 800% propane by volume, giving 45% cylinder oil on the residue and 55% of asphalt of penetration 15/40, melting point 50/60. The apparatus consists of one settling and one washing stage, and previously was in duplicate in order to remove resins, but only one unit of the equipment is now used. This equipment is in good condition.

(3) Furfural extraction plant. Almost completely destroyed. On this plant they ran to two V.I.'s; a motor oil of 74 V.I. and an aero oil from the cylinder of 90/95 V.I. The work was carried out in two separate units, one on neutrals and the other on cylinder oils.

(4) A Coubrough unit, which was used chiefly for the distillation of crude down to aero oils. In this case instead of distilling off 40% in the atmospheric plant, 32% only was distilled off in the atmospheric unit, the 68% residue going to the Coubrough unit, in the first stage of which gas oil, spindle, neutral and light cylinder were distilled off. In the second stage, with a 1:1 kerosine mixture, and under 25mm. of vacuum, a heavy cylinder distillate was obtained, leaving a 20% bottoms on the crude. This bottoms had a penetration of 40/50 and the heavy distillate was 12° Engler at 70°C. This unit has been completely removed and is now underground at Porta (q.v.)

(5) Contact filter plant, designed by Lummus, used for the decolorisation of aero oils, and the neutralisation of motor oils. It was of the normal pipe still type, using Sweetland presses. All the used earth was recovered, using a recovery plant of a German type, built by Otto Wilhelm Strassund, using triple wash. Four tons of spent clay were washed with 15 cu.M. of gasoline in three washes. The whole plant consisted of three revolving drums of about 8 cu.M. capacity. The vessel revolved at 10 r.p.m. and was stem coiled and jacketed. The gasoline was recovered by continuous distillation, and the gasoline was recovered from the clay by heating in a reverberatory furnace. The gasoline used had a boiling range of 60° to 90°C., and it was claimed that the total loss of gasoline was 1% on the gasoline used. This plant is in a workable condition.

(6) An acid treating plant was installed early in 1945 to replace the Furfural plant which had been destroyed. It

was only capable of the manufacture of motor and industrial oils. It was a continuous plant with three centrifuges, built by Ramisohl and Schmidt in the city of Oelde. The centrifuges had a capacity of 3,000 litres/hour neutral oils and 1,600 litres/hour of cylinder oils. This plant is in almost new condition. The acid sludge from this plant is burned, and generates low pressure steam at 3 atmospheres. This apparatus is also in workable condition.

(7) Dewaxing Plant. This was the original benzol/acetone plant designed by the Texaco Development Company. It originally consisted of three Oliver filters of 30 sq.M. surface each, but was totally destroyed in 1941. On rebuilding, six Oliver filters were put in, but two cast iron filters similar to the Olivers were constructed by R. Wolf of Magdeburg.

The process was run without any repulping, and the yield was 80% of oil, except in the case of aero oils, when the yield was 70%. The oil content of the wax was said to be not more than 5% on the cylinder oils, and about 10 to 12% on the spindle and light machine oils. The dewaxing was carried out to -15°C., except in the case of the hot steam cylinder oil, which is not furfural refined, and the pour point of which was -3°C. This plant is also in a workable condition.

Summary.

Plants available for operation at Misburg are:-

Deurag: Atmospheric topping plant.
U.O.P. coking plant.
Pressure distillate treater.
Small rerun unit for treated distillate.

Nerag: Combined atmospheric topping and vacuum crude distillation unit.
Propane deasphaltising plant.
Contact filter plant.
Acid treating plant.
Dewaxing plant.

Normal Production in tons/month.

Cracked gasoline	210°C. end point	4,200
Straight run gasoline	150°C. " "	3,000
Kerosine	180/285°C.	7,000
Gasoil		3,000

Coke (to Siemens and I.G. for electrodes)	2,800
Aviation gasoline	Nil.

Slack Wax.

From spindle oil:	30/32°C. melting point.	To I.G. for fat manufacture.
From neutral oils:	50°C. melting point.	" "
From cylinder oil:	60°C. melting point.	To Cracking Plant.

In addition to the normal production of lubricating oil given under the separate plants above, 1,500 tons/month of axle grease consisting of 50% spindle and 50% furfural extract were made.

Stocks on hand at Misburg.

Materials for operation:

Acetone	16 tons
(100 tons needed to start dewaxing)	
Benzol	9 "
Furfural	180 "
(sufficient, but no plant)	
Terrana and Nordal earths	300 "
(plus a further 100/200 tons at Porta)	

Petroleum products:

Crude oil	Nil
60% topped crude	3,000 tons
Raw pressure distillate	180 "
Raw cylinders and neutrals	1,200 "
Dubbs mixture	500 "

Crude tanks destroyed, but sufficient tankage available to enable plant to run. One power house has been destroyed, but sufficient power can be made by the remaining power plant to keep what is left of the refinery in an operable condition.

(IV) NIENHAGEN FIELD.

Present:

Dr. Martin Schunck,	Vice-President of Elwerath, Deurag & Nerag.
Dr. Ing. Oscar Diecher,	Fields Technical Manager, Elwerath.

The field was bombed two days before occupation by the Allies and some tanks destroyed, but in the main the wells are in quite operable condition.

The total capacity is 350 tons/day, and the average depth of the wells 500/1,500 Metres. During the war, a hole was drilled to 3,800 M., but proved to be dry, but nevertheless afforded considerable satisfaction to the management of Elwerath in that it was the deepest well in Germany. The drilling rate throughout the war has been 80,000 M./year, and forty modern rotary drilling rigs are said to be in existence. The wells are all pumping wells, with electric pumps. At the time of the visit, no power was available, but power comes from the Uberlandwerke at Celle, some little distance away. There is a diesel generator on the field of 500 kVA, which can be got to work in two weeks. All pumps and motors are in order, and 6,000/8,000 tons of tankage are available. The gas collecting system and the propane separation plant will require ten to twelve weeks to repair.

The crude contains 2% to 50% of salt water, according to the well, and the salt content of the water is 16%. The separation is carried out on the field by heating to 60°C. and passing through centrifuges, of which there are three. They have from time to time used a demulsifier supplied by I.G., which goes under the name of "Dimulsan". The whole of this equipment, together with the necessary steam plant, is in working condition.

The loading facilities will require about a week for complete repairs, and the railway between Nienhagen and Misburg is probably in working order. There is one locomotive on the field, which operates on a propane/butane mixture, capable of pulling 10/15 trucks of 15 tons capacity each, whilst another steam locomotive exists at the salt works nearby, capable of pulling 15 trucks of the same size.

Neighbouring Oilfields.

Other fields in the neighbourhood are:-

Owner Elwerath:		
Mölme, Nr. Hildesheim.	30	tons/day
Hohenassel	100	"
Wessendorf (flowing wells, 40 kilos. N.E. of Celle).	150/200	"
Owner Wintershall, A.G.		
Nienhagen	100	"
Eicklingen (Nr. Nienhagen)	100	"
Fuhrberg (W. of Nienhagen)	150	"
Owner Deutsche Vakuum, A.G.		
Hanigsen	150	"

We were also told of a gas field owned by the Elwerath near Bentheim (Holland) at a depth of 2,200/2,300 M. It is estimated to have a capacity of 500,000/700,000 cu.M. of gas a day, but is at present worked at 250,000 cu.M. Of this, 200,000 cu.M./day are piped away for rubber manufacture, the remaining 50,000 cu.M. being used for motor fuel.

(V) UNDERGROUND HYDROGENATION PLANT IN THE BUECKEBERG MOUNTAINS.

This underground working was said to be in the village of Kleinen Bremen, south of Minden. It is not difficult to spot the quarry as one enters the village, and as one climbs the road it is possible to see where the workings begin on the left-hand side of the road.

There are considerable workings on this side of the road and there were nine quite extensive galleries. On the other hand, it was difficult to say whether it was intended for a hydrogenation plant, and unfortunately it was impossible to have a guide who knew anything of this working. I personally am inclined to think that this was not intended for hydrogenation, chiefly on the ground that there seemed to be no provision being made either outside or inside for the reaction chambers. It is my understanding that the "Schwalben" usually have their reaction chambers on the outside of the quarry.

On the other side of the road was another considerable underground working, in which there was a very impressive off-loading platform from the main railway which led into the underground working. Up to the time we were there, the only machinery which had been discharged was two large planing machines, and these were not yet installed.

However, considerable boxes of literature appear to have been thrown hurriedly into some of the chambers on this side of the road, and we removed anything that was marked "Secret" and handed it over to "T" Force, 21st Army Group. These were all of immediate operational interest, and they showed considerable detail of airfields and underground installations in conjunction with them. My own opinion is that the whole of this Bueckeberg area should be visited by a team of two or three fairly agile investigators, as the whole area appears to be honeycombed.

(VI) RUHR CHEMIE INSTALLATION AT HOLTEN.

A visit was paid to the Ruhr Chemie installation at Holten, Nr. Sterkrade. The personnel available were the Acting Manager, Herr Spanier (who was little more than a caretaker); the Manager of the "O.X.O." plant, Dr. Landgraff, who really was responsible for the plant; the Power Engineer, Herr Matz, and a Chemist, Herr Arthur Schreiber.

The plant was very badly hit, and little or nothing of technical value could be obtained by an examination of the rubble. Most of the drawings were available in the basement of the plant, although they were in such confusion that it was very difficult to get anything much in a short space of time.

The Fischer plant was conventional, with Dubbs cracking plant for preparation of olefines, and a certain amount of work was going on on polymerisation and aromatisation. The Fischer plant used the normal catalyst consisting of:-

100 parts of Cobalt,
5 " " Thorium,
8 " " Magnesium,
180/200 " " Kieselguhr.

The Kieselguhr came from the Hanover area, and the production for the whole of the Fischer plants in the Ruhr was of the order of 4 tons (in terms of Cobalt) per day. The suggested iron catalyst was to consist of:-

100 parts of Iron,
2.5/4 " " Copper,
10 " " Lime,
15 " " Kieselguhr.

The catalyst was made from the nitrates by precipitation with carbon dioxide. This catalyst was not made on a large scale up to the time of our visit, but was looked upon as being a suitable material.

There were obvious signs of considerable building activity at certain parts of the plant where they were evidently constructing cracking plants, combined probably with polymerisation, but these plants had not been completed.

A considerable amount of laboratory work was being carried out on the dehydrogenation of the paraffins for the manufacture of "O.X.O." and lubricating oils. For some time they had worked on dehydrogenation of propane and butane, using a conventional aluminium chromium catalyst, and it was alleged that the addition of 0.2% of cobalt reduced carbon formation. The dehydrogenation was being carried out at 590°C. and approximately atmospheric pressure. Their conversion was 24% per pass, and using 250 cc. of catalyst they dehydrogenated 20 litres of butane per hour. The conversion was reduced after three months to 18% per pass. None of this work was of a very high calibre.

Then they went on to the dehydrogenation of C_{16} to C_{18} cuts for use in the "O.X.O." plant. The catalyst in this case was thoria and chromia. The method alleged to have been adopted was as follows:-

Silicon carbide was treated with nitric acid to remove impurities, dried and heated to 650°C. A solution of thorium nitrate and chromium nitrate was then made up as follows. 21 grams of thoria as nitrate and 7 grams of chromia as nitrate were dissolved in water to make up a 3% solution. 200 cc. of cleaned silicon carbide, equivalent to 280 grams, were then placed in a rotating pot at 250°C., the pot being inclined at an angle of 45° to the vertical. The 3% solution mentioned above was then sprayed into the pot, and the whole took four to five hours to be sprayed. The salts were claimed to be deposited in this way on the carborundum, and the granules were heated slowly to 520°C., and lastly to 850°C., in a muffle furnace for two hours, when the catalyst was ready.

The catalyst was then transferred to a tube and at 560°C. hydrogen was passed over it at a rate of 200 litres/hour for five minutes. Then this was followed by nitrogen and the whole tube evacuated to 20 mm. of mercury. The C16 to C18 was then introduced at the rate of 150 cc. of liquid per hour. The catalyst life was said to be three months, and the yield was 15% to olefines C_{16} to C_{18}, 6% to olefines lower than C16, 2%/3% gas, and 0.5% carbon, the remainder being unchanged.

In order to improve this, barium oxide was added to the carborundum before the addition of the thoria and chromia; 12 grams of barium oxide were added to the catalyst containing 21 grams of thoria and 7 grams of chromia, but results were inconclusive.

The "O.X.O." Process.

The information on this process was given by Dr. Landgraff, and the patents in connection with the process were all taken out in the name of the Ruhr Chemie.

The work originally commenced in connection with a study to convert ethylene into aldehyde, and the raw material used was an ethylene cut containing even as low as 20% ethylene. It was stated that a dilute mixture was even better than a concentrated mixture of ethylene, because the reaction is a fairly active one.

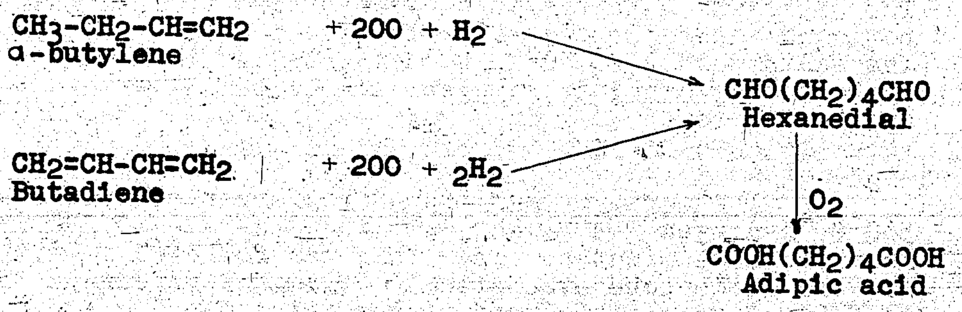
The work started in the gas phase at a pressure of 160 atmospheres, using the usual Fischer-Tropsch cobalt catalyst.

Note A: The general type of reaction to cover this synthesis, as also those using the higher olefines mentioned later, would seem to be:-

$$\underset{\text{Ethylene}}{C_2H_4} + CO + H_2 \longrightarrow \underset{\text{Propyl aldehyde}}{CH_3.CH_2.CHO}$$

Work went on then using α-butylene and butadiene, and claims were made that adipic acid could be made using this process, but no evidence was forthcoming.

Note B: Possible reactions would be:-



$$\underset{\alpha\text{-butylene}}{CH_3\text{-}CH_2\text{-}CH{=}CH_2} + 2OO + H_2 \longrightarrow \underset{\text{Hexanedial}}{CHO(CH_2)_4CHO}$$

$$\underset{\text{Butadiene}}{CH_2{=}CH\text{-}CH{=}CH_2} + 2OO + 2H_2 \longrightarrow \underset{\text{Hexanedial}}{CHO(CH_2)_4CHO}$$

$$CHO(CH_2)_4CHO \xrightarrow{O_2} \underset{\text{Adipic acid}}{COOH(CH_2)_4COOH}$$

Instead of hexanedial, cyclohexane might be formed by ring closure, splitting off water and picking up two hydrogen atoms. This gives adipic acid readily on oxidation.

The bulk of the work was carried out on two cuts of olefines ranging from C_{11} to C_{17}, which cuts for experimental purposes were sent to Holten from the Ruhr Chemie. These were further separated into four cuts as follows:- C_{11}/C_{12}, C_{13}/C_{14}, C_{15}/C_{16}, and C_{17}. These cuts, which contained about 40% olefines, the balance being paraffins, were treated separately, and it will be apparent from the process (see Note A) that a carbon atom was added to each cut so that the end products were aldehydes and alcohols from C_{12} to C_{18}. The catalyst used was, as stated, the ordinary Fischer-Tropsch catalyst, but magnesium was excluded if at all possible. The olefines were passed together with sulphur-free water gas, containing 39 parts of carbon monoxide to 45 parts of hydrogen, over the catalyst, the temperature in the first stage being 135°C. and the pressure 150 atmospheres, the temperature being controlled by means of steam pressure. The process was intermittent, each cycle taking 20 minutes.

After the material had been in contact with the catalyst it was filtered through "filter stone" and then distilled so as to remove any by-products. The aldehydes produced were distilled under vacuum at about 180°C., using a vacuum of 2 to 5 mm. of mercury absolute. The bottoms were secondary alcohols, esters, etc., and the distillation was carried out in a batch system and not continuous.

The aldehydes were then passed together with pure hydrogen over a nickel catalyst to convert them into alcohols. The temperature was 180°C. and the pressure was the same as for the first reaction.

Up to the time of our visit, it was intended to supply the finished alcohols to Henkel for soap manufacture, but we were also shown a schematic arrangement whereby they intended making sulphonic soaps. The detail of this was not gone into thoroughly at Holten, because it was already clear that details had been obtained by the Ludwigshafen party.

The curious point was that in spite of heavy bombing and the apparent failure to get the Fischer-Tropsch plant working, the whole of the plant for this "O.X.O." process had been built without any priority, and was almost in a condition to commence operation when we arrived.

Construction had commenced in 1939/40 and carried on as and when possible. This factor may have affected the economics of the process as plant costs were given as RM.11,000,000. Taking amortisation at 10%, a return of 90 pf./kilo was obtained on the alcohols. The olefines were charged at 45 pf./kilo, this being based on the olefine content (40%), the paraffins being returned. The figures were based on "O.X.O." costs, so that "O.X.O." showed a profit.

Henkel and I.G. were shareholders, and were to carry out the sulphation of the alcohols.

(VII) BOTTROP.

This plant was not examined thoroughly, as it had already been investigated by the Hydrogenation Party. It was a centre for the manufacture of aviation gasoline, and the octane number of the hydropetrol from the gas phase 700 atmosphere plant was 78.5 (I.G. Motor Method).

With .09% of T.E.L. the octane number was 89
" .115% " " " " " 91.

It was claimed by the people at Bottrop that the gasoline there was regarded as the standard for aviation gasoline for the Reich, and it was here that we were told that the main testing stations for aviation gasoline were at Spandau and Derben, Derben being the main testing station as far as Bottrop was concerned.

From records it would appear that the production of aviation gasoline at Bottrop rose to 34,000 tons in 1943, since when they had struggled steadily to keep up that production, but gave up finally in January of this year.

It was claimed that all the cresol inhibitor for the Reich was made at Bottrop.

This plant is interesting from the point of view of its having a Pett Broch system, which, however, had not worked satisfactorily on coal, and also a 700 atmosphere gas phase hydrogenation plant, both of which are being reported in detail separately.

(VIII) SCHOLWEN.

This plant was very badly damaged and was the worst seen by the writer. It was a plain straightforward hydrogenation plant manufacturing about 200,000 tons of aviation gasoline. The Manager, Dr. Jost, was the least cooperative man we found in Germany, and a recommendation was made for his imprisonment and further interrogation.

In addition to the straightforward hydrogenation plant there were also plants for the dehydrogenation of the naphthenes in the total gasoline (a report on which has been made from Ludwigshafen) and a plant for the manufacture of iso-octane, a report on which was given in connection

with the Wesseling visit. With regard to the latter, however, it should be pointed out that whereas the plant at Wesseling had not operated, the plant at Scholwen had operated, and apparently quite successfully.

The alkylation part of the equipment was a straightforward sulphuric acid alkylation plant, but the mixing and general equipment was almost of an amateurish kind. The equipment was very heavy, cumbrous, and showed signs of having been designed by a chemist rather than by a chemical engineer.

It was interesting to note that although they had in the early days polymerised their butylenes, they had in recent years gone over to alkylation of iso-butane with butylenes.

(IX) GELSENKIRCHEN.

The building of this plant commenced in 1937, and operation commenced in 1939 and ended on 13th June, 1944. The total cost of the plant was RM.300,000,000.

A considerable number of the staff were available, although the Director, Dr. Pross, was absent in the nearby town of Gelsenkirchen, which at the time of our visit was still in enemy hands.

The process was again straightforward hydrogenation for the manufacture of aviation gasoline, octane number 72, which with 1.2 cc./litre of T.E.L. gave an octane number of 89.

The annual production was 345,000 tons. The iso-butane produced was sent to Scholwen for the manufacture of alkylates, and the n-butane was sold as motor gas.

At Gelsenkirchen we heard about the new catalyst 7846, but we understood at the time that this catalyst was already known to the Ludwigshafen Party.

Further particulats of this plant are available, but as it was thoroughly investigated by the Hydrogenation Party it is not intended to repeat them in this report.

(X) THE TOPPED CRUDE POSITION IN GERMANY.

In discussing the refining situation in Germany, it was apparent throughout that the question of the disposal of topped crude had taken up a considerable amount of thought. Quite a voluminous correspondence existed in the files of Dr. Brockhaus on this question, which was becoming so acute that considerable quantities were being dumped in suitable quarry pits, and instructions had gone out to certain key power stations to turn over from coal burning to fuel burning.

The reason for this, of course, is apparent in that the production from the dispersed refineries, or "Ofens", was unbalanced. The intake capacity for crude oil was 3,000 tons/month approximately, and the yield as follows:-

15% gasoline,
35% diesel oil,
50% residue.

As very few of these had any cracking facilities attached to them, and in fact as there were no completed cracking plants as far as one could ascertain either underground or dispersed, the disposal of this 50% residue was becoming a very serious problem to the German petroleum industry.

APPENDIX I.

"OFENS"

No.	Location	Company Responsible
1/2	Boegerhof	Nerag
3/4	Bunkensen	"
5/6	Messinghausen	Dea
7/8	Muehlenbein	Nerag
9/10	Muehlental	Wintershall
11/12	Tuernitz/Treisental	Dea
13/14	Weitnegg/Donau	Donauoel
15/16	Spitz/Donau	Dea
17/18	Petzenkirchen/Erlauf	"
19/20	Alte Post b. Pirna	Gasolin
21/22	" " " "	SS u. Gasolin
23/30	Ebensee a. Traunsee Oberdonau	Dea
31/32	Statzendorf (noerdl. St. Pölten)	SS u. Vakuum
33/34	Hauskirchen (" Zistersdorf)	SS
35/36		Fanto, Ver.ung. Mineraloelwerke A.G. Ungar. Oelwerke A.G. Szöng.
37/38	Eschenbach (oestl. Gemuend)	
39/40	Grosscheinbart (suedwestl. Zistersdorf)	

"ROSTS"

No.	Location	Company Responsible
I	Mech. Weherei Hannov. Linden	Nerag.
II	Zuckerfabrik Salzwedel	Dea
III	Teutonia, Misburger Portl.-Zementwerke, Anderten	Nerag.
IV	Harpener Bergbau, Zeche Rob. Müser b. Dortmund.	
V	Ruhrchemie, Holten.	
VI	Kattowitz	

APPENDIX II.

CODE NAMES

SCHWALBE	-	Hydrogenation plant.
KUKUCK	-	Iso-octane and polymerisation plant.
DACH	-	Lubricating oil plant.
TAUBE	-	Cracking plant.
OFEN	-	Dispersed distillation plant.
ROST	-	Batch distillation plant.
JACOB	-	Primitive cracking plant in conjunction with an "OFEN".

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Report No. 293

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DACH 1
LUBRICATING OIL PLANT
PORTA, GERMANY

J. A. ORIEL
On behalf of
British Ministry of Fuel and Power
and
W. W. HORNE
J. P. JONES
on behalf of
United States Technical Industrial
Intelligence Committee

[1945]

This report has been declassified and released to the Office of the Publication Board by the War and Navy Departments.

NOTE: The Publication Board, in approving and disseminating this report, hopes that it will be of direct benefit to U. S. science and industry. Interested parties should realize that some products and processes described may also be the subject of U. S. patents. Accordingly, it is recommended that the usual patent study be made before pursuing practical applications.

8 p. diags.

TABLE OF CONTENTS

SUBJECT	PAGE NO.
Introduction	1
Personnel	1
Plant Condition	2
Plant Operating Units:	
Distillation	2
Extraction	2
Dewaxing	3
Clay Contacting	3
Clay Extraction	3
Auxiliary Equipment	3

Personnel of Team

16 April 1945

J. A. Oriel, British Ministry of Fuel and Power
J. W. Vincent, British Ministry of Fuel and Power
L. Evans, United States Petroleum Administration for War

17 and 18 May 1945

W. A. Horne, United States Petroleum Administration for War
J. P. Jones, United States Petroleum Administration for War
G. S. Bays, Jr., United States Petroleum Administration for War
B. L. MacKusick, United States Petroleum Administration for War

DACHS I (UNDERGROUND LUBRICATING OIL PLANT

PORTA (NEAR MINDEN), WESTPHALIA, GERMANY

Introduction

This is an underground plant for producing aviation lubricating oil from topped crude oil to be supplied from the Hanover area. It is located in an old sandstone quarry just back of the railroad station at Porta, near Minden, in Westphalia, Germany; Army map P. 3 (Detmold), Scale 1/100,000, coordinates 807052. The main entrance to the plant (as of 18 May 1945) is shown in Figure 1.

The existence and location of this plant were disclosed to Mr. Oriel's party in April 1945, by Dr. von Eynatten, Manager at Misburg and Freiheir Heeremann, the manager of the Lubricating Oil Plant at Misburg. Similar information was disclosed independently on 10 May 1945, to the second party who investigated the plant, by Prof. E. Terres, Director of Edeleanu Gesellschaft, during an interview at his temporary offices in Altenburg, Germany.

Personnel

Dipl. Ing. Arnold Schroder, a Hollander, was in charge of the installation of the various units. During May 1945 he was in charge of feeding the inmates of a large Displaced Persons camp in Minden on the west side of the Weser. Mr. Schroder was formerly in charge of the Refining Department of Uhde-Gesellschaft fur Hochdrucktechnik, Boevinghausen, near Dortmund (Ruhr), and was detained by the Nazis for refinery construction. He had also previously worked with Wickham and Morlander of the M. W. Kellog Co. In Fig. 1 he is to be seen in the center foreground.

Mr. Schroder was interviewed on May 17, 1945 and served as guide through the plant on May 18, 1945. He also supplied the attached sketch of the plant layout, which is substantially in agreement with a sketch supplied to Mr. Oriel by Heeremann.

Plant Condition

The office at Porta was opened and organized on August 4, 1944, to plan necessary further excavation of the old sandstone mine and construction of the plant, which was to be built under the Geilenberg plan by the Todt organization. Thus, all work was carried out under the direct supervision of the SS.

The plant was approximately 90% complete with 95% of the equipment on location; installation would have been complete in an estimated 6 weeks from the date of occupation by our troops. All records and plans were removed by the SS two days before the occupation took place and Schroder said they probably were in Hanover and that full information should be obtainable from a Dr. Weller, Manager of NERAG (Neu Erdöl Raffinerie A. G.) at Misburg, near Hanover. On the other hand, Mr. von Eynatten, who was said to be Manager at Misburg, stated to Mr. Oriel's party that all plans had been in the hands of the Todt organization at Porta itself.

Due to lack of ventilation and to the water seepage, most of the iron and steel equipment was rapidly rusting.

Plant Operating Units

The plant consisted of five units as discussed hereinafter. The in-place value has been estimated at £500,000 ($2,000,000).

Distillation

This was to be carried out in a two-stage Coubrough unit, the heaters and columns having been moved to this location from the Nerag Refinery at Misburg. The primary stage had a capacity of approximately 9000 tons/month of topped crude which was to be brought by tank car from the Hanover district. The first column was to take a naphtha cut overhead and side streams of Diesel oil and neutral oil. The bottoms were to be charged with naphtha in equal volume proportions to the second column at some reduced pressure, where a heavy cylinder oil cut was to be taken overhead, reducing the bottoms to approximately 20% of asphalt. The Diesel oil, wax, and asphalt were shipped out and, so far as known, used without further treatment. A small amount of piping was required for completion.

Extraction

The neutral and bright stocks were to be furfural extracted for the work up of aviation lubricating oils. This unit was designed by Still G.m.b.H., Rechlinghausen, near [illegible]xter-am-Weser, and apparently was of usual design. Installation of pumps, piping and insulation was incomplete.

Dewaxing

This unit is one of the most recent designs of Edeleanu Gesellschaft, using methylene dichloride and ethylene dichloride as solvents followed by a continuous band filter. The filter was a new and interesting development. There were four such filters, of the latest improved design, which were constructed by R. Wolf of Magdeburg. This unit required some insulation for completion. An earlier model of this continuous band filter had been in use at the A.G. Sächsische Werke at Espenhain. Some detailed drawings of the filters used in Dachs I were evacuated from the files of the Edeleanu Ges., at Altenburg.

Clay Contacting

Following the dewaxing and solvent recovery, the oil was to be batch clay contacted in quite conventional manner. These contactors and filters were also brought in from the NERAG refinery at Misburg. These oils were to be blended and used without further treating.

Clay Extraction

This plant was to be located outside the mountain and was simply to consist of a batch naphtha extraction of the used clay from the contacting to recover the last traces of lubricating oil. The oil, after flashing off the gasoline, was to be sold or used as motor oil without any treatment.

Auxiliary Equipment

Concrete tanks inside the mountain provided working tankage (4,000 m^3 total) whereas the feed and product storage was to be in bunkers on the mountain side.

A small steam plant had been constructed outside the mountain and was ready for operation. It was designed for 35 tons/hr. of 22 atm. steam. This plant had been constructed for use of the Philips Company which was located 50-60-m. higher in the sandstone layer. The Philips company was to manufacture radio tubes and, although it had not operated before occupation, the Military Government at Porta had directed it to start production.

Two other steam plants were to be constructed in the mountain. Although installations had not been started, all the parts were on the site and it was estimated that it would require six weeks for completion.

Water for the plant was to be supplied by four pumps at the Weser River which would deliver 400 m^3/hour each at 12 atm. pressure. The water discharge was to flow back to the Weser through an open sluice.

It was further planned to seal up the main opening (Fig. 1), through which the units had been brought, with approximately eight feet of concrete as bomb protection. In this same tunnel, the offices and control laboratories were to be built.

A single blower in the rearmost tunnel was to provide necessary ventilation. By the use of sealing doors on cross tunnels and vents, the air was to be drawn in two of the lateral tunnels and blown out the other two (see Fig. 2). This installation was incomplete.

J. A. Oriel
W. A. Horne
J. P. Jones

PORTA

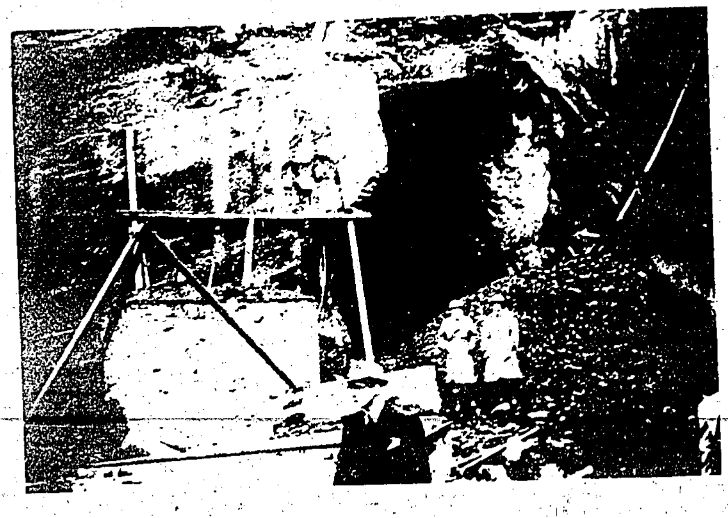

Figure 1

Dachs 1. - Main Entrance
Center Foreground - Arnold H.W.A. Schroder
Sup't. of Construction and In
Charge of Construction and In
Charge of Refining Department
for Uhde G.m.b.H.

DACHS I

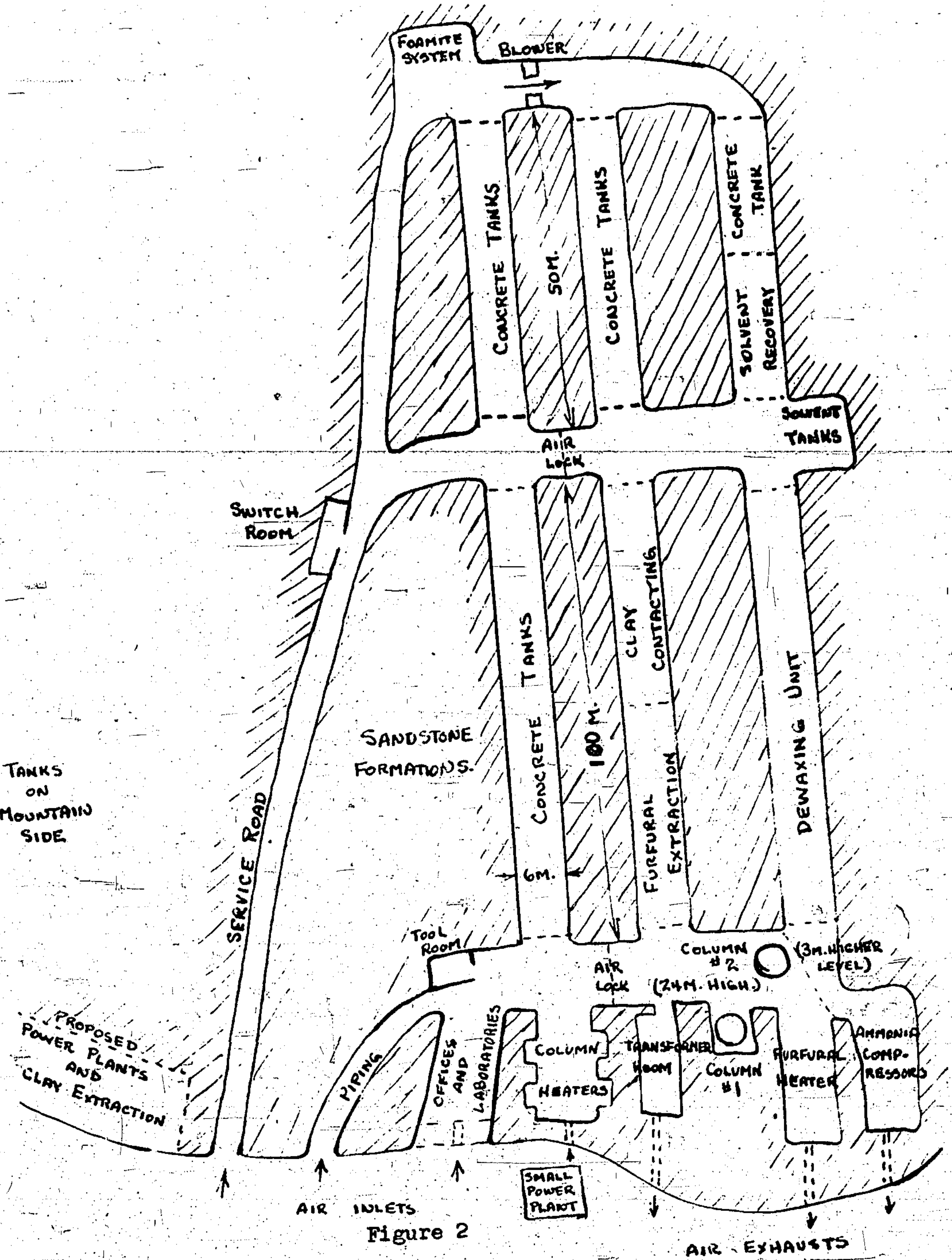

Figure 2

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by Col. E. W. Gruhn

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THE
HIGH PRESSURE HYDROGENATION PLANT ESPECIALLY FOR BROWN COALS
WESSELING

Oriel, Jones & Weir

RESTRICTED

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

XXVIII-40

RESTRICTED

REPORT OF INVESTIGATION AND INSPECTION
OF
THE HIGH-PRESSURE HYDROGENATION PLANT
ESPECIALLY FOR BROWN COALS
AT
WESSELING, NEAR COLOGNE, GERMANY

Reported by

Col. J.A. ORIEL
Mr. J.H. JONES
Mr. H.M. WEIR

On behalf of the

British Ministry of Fuel and Power
and
U.S. Technical Industrial Intelligence Committee

CIOS Target No. 30/4.10
Fuels and Lubricants

July 17, 1945

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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TABLE OF CONTENTS

SUBJECT PAGE NO.

Introduction.. II
Summary... II
The Wesseling Plant of the Union Rheinischen Braunkohle Kraftstoff, A. G........................ I
Appendix A: Discussion of DHD Plant as Proposed for Wesseling.................................. 100
Appendix B: Discussion of Alkylate (AT-244) Plant Built at Wesseling but not Operated............. 106
Appendix C: Personnel of Wesseling Plants: Some Interviewed.. 112
Appendix D: Two Schematic Drawings of the Pintsch-Hillebrand Water-Gas Generator and Associated Piping Connections.................................. 113 & 114
Appendix E: Results of Early Laboratory Tests on Rheinish Brown Coals for Determining Their Suitability for High-Pressure Hydrogenation Processes 115
Appendix F: Preliminary Report to CIOS on the Wesseling Plant. (This report, dated March 21, 1945, to the CIOS Secretariat, was written by the Investigating Team immediately after its return from Wesseling to U.K. Base and before the seized documents were available for preparation of this inclusive report).............................. 126
Appendix G: List of Documents Seized at the Wesseling Plant and Placed in Safe-keeping in Document Bag No. 1677 with MIRS after being Microfilmed for Transmittal to the Secretary of Interior of the United States.............................. 135
Appendix H: Contents of a Document of Unknown Origin found at Wesseling and Relating to the Technology of Metal Alcoholates................ 138
Appendix I: Photographs Seized or Taken at the Wesseling Plant and Showing Features of Divers Plant Units.. 142
Appendix J: Original of Report in German by Dr. Ernest Peukert of the Wesseling Plant and which formed the basis of the CIOS Report............ 150

MEMBERS OF CIOS TEAM

Col. John A. Oriel - British Ministry of Fuel and Power
Mr. Irvin H. Jones - U. S. Bureau of Mines
Mr. Horace M. Weir - U. S. Petroleum Administration for War

INTRODUCTION:

The hydrogenation plant at Wesseling, Germany, for production of motor fuels, was the first of such plants uncovered by Allied Troops invading Western Germany and the CIOS team afore-mentioned proceeded to the plant to determine: Particulars of the processes employed there and their possible use in subsequent Allied war effort especially against Japan; and methods and means employed to prepare any new and valuable products. Any incidental information which could be obtained that might aid the war effort was to be obtained in a form for immediate use, if possible.

SUMMARY:

The most timely information obtained was perhaps the exact coordinates of an underground hydrogenation plant in the course of construction and the knowledge that there were several other of these plants having the code name "Schwalben" which were proposed or perhaps under construction (See Appendix F).

The installation at Wesseling was found to have been a standard hydrogenation plant processing, before bombing attacks forced its closure, mainly Rheinish brown coal and some tar and tar-oils to Diesel oil and Benzine; later aviation benzine was principally made.

There had also been installed equipment to dehydrogenate butane that was a by-product of the hydrogenation plant with a moving catalyst bed (new and novel), and to alkylate this product with by-product iso-butane by means of sulphuric acid; however, essential parts thereof had, prior to the CIOS visit, been removed and shifted further into the interior of Germany, before actual use, for use in a possible further stand in that part of the country. Similar plants are said to be in operation at Leuna, Scholven, Pölitz, and possibly at Böhlen.

The high-pressure hydrogenation plant of the UNION RHEINISCHEN BRAUNKOHLE KRAFTSTOFF A.G. is one of the works arranged for within the scheme of the Four-Year's Plan. It is located on the west bank of the Rhine River at Wesseling (Grid Reference F 468478) about 15 kilometers due South of Cologne, Germany.

The formation of the above company was completed January 27, 1937, and in 1940 some 73% of the 90,000,000 RM of its capital stock was held by two major interests,* in the Rhineland Braunkohl Briketting industry. Additional shares were held in small blocks by some half dozen companies and one individual. Neither the Reich nor the I. G. Farbenindustrie had any stated direct financial interest. The final investment in the plant amounted to 275,500,000 RM including 50,000,000 RM for the initial "running-in," and final shut-down of the plant.

Construction of the plant started on April, 1938. A small completed portion of the plant was taken over in August, 1941, but substantial completion was only reached at the beginning of 1943. Gradual increase of production occurred during 1942 until at the beginning of 1943 the plant reached approximately its final capacity, given as:

Braunkohle as starting material, 225,000 metric tons/yr.
Oils as the starting material, 360,000 metric tons/yr.

Both these figures refer to the production of Diesel oil and benzine.

In 1943, aside from minor capacity reduction, estimated at some 5% due to bombings, the plant produced its anticipated quota of 225,000 metric tons of Diesel oil and benzine from brown coal, and in the first months of 1944 by using as starting material 75% of brown coal and 25% oils, the plant produced benzine and Diesel oil at the combined rate of 260,000 tons/yr. Production continued with only minor disturbance during the first half of 1944, but the bombing on the night of July 18-19, 1944, in which an estimated 1,000 bombs, mostly of 250 kilograms by weight, fell in the plant area, destroyed some 20% of the plant investment and a much larger proportion of its producing capacity. After 10 weeks of intensive repair work, the plant was sufficiently in order to resume operations at about 40% of its rated capacity. After having produced at this rate for scarcely more than a matter of days about sixty including 500 Kg bombs were dropped on the plant on October 3 and October 11th, the damage to the high pressure equipment, the intermediates tank-farm and the power house caused the plant to be ordered completely shut down on October 6, 1944.

On the morning of March 10, 1945, the CIOS team entered the plant and began interviews of personnel remaining in the vicinity after withdrawal of German troops to the opposite side of the Rhine. Two hours later the military T-forces of the Allied Army established temporary guard over the plant. The remaining plant personnel was found to be quite cooperative and this report is based on questionings at the plant on March 10-12, inclusive, and on seized documents which were in part dug up from individual and private caches. Appendix C of the present report gives the list of plant officials interviewed.

The basis of the following report is in part an abstract and in part a translation of one of the above seized documents which is an operational history of the Wesseling Plant including description of apparatus, practices and contemplated processes along with the details of production, capacity, operational difficulties and their practical solution.

The raw material for the Wesseling plant was primarily Rheinland brown coal from nearby mines; throughout the 2½ years of operation some 25% of the raw material was oily hydrocarbons having an origin outside the works, such as petroleum derivatives, bituminous coal-tar fractions and low-temperature brown-coal tar.

Diesel oil and automobile benzine were the characteristic products: They were produced by hydrogenation reactions performed at 700 atm. pressure in the liquid "Sumpf-phase" first step wherein coal-paste comprising powdered brown-coal in heavy-oil suspension was treated. In the second and third steps, the hydrogenation was performed in vapor-phase at 325 atm. pressure. The finished Benzine cost a total of about 400 R.M. per ton for production costs.

The employed hydrogen gas was manufactured in two ways: (1) from the gasification of brown-coal briquettes to water gas by employment of the recent Pintsch-Hillebrand process and an apparatus of which the preheaters were heated by producer gas likewise made from brown-coal briquettes; and (2) by cracking of methane and light hydrocarbons, produced in the hydrogenation process, using the well-known I. G.

**41.2% Rhemische A. G. für Braunkohlenbergbau und Brikettfabrikation Köln, and 31.8% Braunkohlen und Brikettwerke Roddergrube A. G., Brühl.

Farben steam-methane process with a nickel catalyst for producing CO and H_2 from gaseous hydrocarbons.

The pure hydrogen production was at the rate of about 78,000 Nm^3/h and was derived somewhat more than half from the water-gas. Crude hydrogen from the two sources was mixed, washed free of CO_2 at 28 atm. and of CO at 328 atm. pressure by conventional methods. About 70% of the pure hydrogen was further compressed to about 700 atm. for use in the "Sumpf-phase" converter ovens.

Compressors and pumps were driven for the most part electrically, the power being generated to the extent of about 35,000 Kw by steam turbines at the power plant which represented an investment of about 40,000,000 RM. Outside power to the extent of some 26,000 Kw was produced. Three boilers for burning powdered brown-coal generated 70 atm. steam. After passing through turbines, 185-195 tons/hr. were sent to process use in the plant at 18.5 and 2.5 atm. pressure, mainly the latter.

THE HYDROGENATION PLANT AT WESSELING

The hydrogenation plant at Wesseling operates according to the Bergius-I.G. high pressure process whereby hydrogen and carbon compounds of high molecular weight are converted into substances of low molecular weight.

The process is characterized by three primary chemical changes assisted by catalysts; i.e., the cracking of the starting material that is lean in hydrogen; the addition of hydrogen to the products of cracking; and their conversion into such hydrocarbons as are especially adapted for their intended use. There is thus produced from bituminous coal, brown coal and heavy oils of divers origins, such liquid products as Diesel Oil, Benzine, and liquified gases adapted for motor fuels.

The end-products are not produced in a single process step but ordinarily in two to three. In the first step, the raw material is converted to an oil of intermediate molecular weight and hydrogen content. This step is called the "Sumpfphase" because the conversion takes place in liquid or semi-solid condition. In a second step, oils recovered in said first step are further converted in gas-phase operation.

In most of the high-pressure hydrogenation plants, fuels for carburator motors are produced. To meet present-day requirements, these fuels must contain large proportion of iso-paraffins, aromatics and naphthenes. Up until the present time, it has not been possible to produce these hydrocarbons in two single process steps. Therefore, products obtained by operation of the first two steps are frequently treated in auxiliary plants to further conversions into more desirable products.

In Wesseling the conversion of Rheinish brown coal and heavy oils into Diesel oil and benzine is effected in two or three process steps; two steps, if the principal product is Diesel oil and three steps if auto or aviation benzine are produced. In the first step, the starting material (coal, etc.) is converted to middle oil by extensive hydrogenation; this middle oil is thereafter catalytically cracked and hydrogenated into Diesel oil and benzine. In the third step, a catalyst is employed that in addition to being a good cracking and poor hydrogenating catalyst is effective for ring-formation and isomerization. With this catalyst, benzine having satisfactory proportion of aromatics, naphthenes and isoparaffin is produced. Two other processes for the production of respectively a highly aromatic and an isoparaffinic product are described in Appendices A and B to this report. The former, commonly referred to as the DHD process, had been projected for operation at Wesseling and considerable data and correspondence relating thereto were found in the documents removed from the Wesseling plant; however, no such unit was constructed there by the time the Wesseling plant was discontinued in operation. A plant for the operation of the latter process, a so-called AT plant was nearly, if not completely finished, for the conversion of butanes into iso-octanes by dehydrogenation and alkylation with sulphuric acid. Smaller parts of this latter plant were removed to other locations by the time it was visited by the CIOS group which was told that the same had never been in operation but was nearly ready therefor.

On page 3 of Appendix J to this report and which is a copy in German of a document prepared by Dr. Ernst Peukert of the Wesseling staff describing, in greater and lesser detail, features and equipment of the process and plant, there is diagramatically shown an over-all materials balance for the processing of 17 tons of moist coal as taken from the mine,

10.0 tons are moisture
1.3 " " used for drying the coal
2.0 " " " " production of power
1.4 " " " " " of hydrogen
2.3 " " " " Hydrogenation

17.0 Tons TOTAL

The above 2.3 tons of coal upon hydrogenation with 0.2 ton of hydrogen yields, in tons:

	0.40	of water of reaction
	0.70	of gases including 0.22 tons of propane and butane
	0.02	of phenols
	0.38	of non convertibles, ash and loss
	1.00	of benzine
TOTAL	2.50	

That is, of the high-moisture coal, as mined, only about 5.88% by weight is converted to benzine; of the dry coal actually subjected to hydrogenation, about 43.5% by weight is actually converted to benzine.

A carbon balance of the hydrogenation process shows that of the carbon introduced into the process:

12% is loss
33% is gasified
55% is converted to Benzine; and a hydrogen balance shows that of the hydrogen that is inlet to the system 65% is in the form of gas and 35% is in the coal and that during the reaction there is converted

55% into benzine
35% into water
10% into other products.

The average molecular weight, the hydrogen content and number of C atoms in the original coal, and the hydrogenation products are as follows:

	Brown Coal	Middle Oil	Benzine
Av. mol. wt.	1000	220	110
No. C-atoms	100	14	8
Available H_2, percent	2	10	14

Composition of a prepared Aviation Fuel

Aromatics	8.5%
Olefins	2.5%
Naphthenes	55.1%
Paraffines	33.9%

DESCRIPTION OF APPARATUS AND THEIR USES

The plant was built primarily for the processing of brown coal and for oils from an outside source. The principal products are Diesel oil, automobile and aviation benzine, bottled gas comprising propane and butane, and also phenols.

The plant is favorably located on the Rhein and comprises the following departments:

1. Hydrogen Plant
2. Hydrogenation Plant
3. Power Plant
4. Transformer Plant for Outside Electrical Current.
5. Water-Supply Plant
6. Waste Water Purification Plant.
7. Loading and Unloading Equipment for Incoming and Outgoing Materials.

The power plant supplies the works with steam and about half of the electrical power requirements.

HYDROGEN MANUFACTURE:

The plant for the manufacture of hydrogen comprises: A Pintsch-Hillebrand water-gas plant; a cracking plant for residual gases to produce hydrogen; fuel gas plant (producers); a conversion plant; compressors; and plants for removal of CO_2 and CO from the impure hydrogen. In addition there are apparatus for supplying the works with blanketing gases and for producing nitrogen gas, including compressor and blower equipment for carbon dioxide.

In the Pintsch-Hillebrand plant, (See Appendix D for Schematic diagram), brown-coal briquettes are gasified to form water gas. Methane, ethane, and the like, of the residual gases of hydrogenation are reacted with steam in a special cracking plant to form a mixture of H_2, CO and CO_2. The water gas and the crack-gas are introduced directly into the plant for conversion of CO to CO_2 without prior treatment for removal of sulphur because the Rheinish brown coals from which they are derived are very low in their content of this element. The gases leaving the above conversion step are freed of CO_2 and CO by scrubbing them respectively with water and an ammonical copper solution yielding pure hydrogen for the hydrogenation reactions. Carbon dioxide recovered from the scrubbing water is used as a "blanketing gas" and carbon monoxide regenerated by heat and vacuum from the copper solution is returned to the above conversion step.

DESCRIPTION OF THE APPARATUS:

The Pintsch-Hillebrand Process is one of the modern developments in brown-coal gasification. The generators therefor work with a continuously operating gasification shaft. The necessary heat for the water-gas reaction is supplied by flowing a mixture of the steam to be converted along with some previously formed water-gas through highly heated regenerators which are alternately preheated by the combustion of a special producer gas.

The Wesseling plant contains 11 water-gas generators designed each for a production of 5,500 Nm^3/h.

The cracking plant for methane, ethane, and the like, operates according to the I. G. Farben Process. It comprises: A sulphur-purification plant that operates at 400°C., with an iron-oxide catalyst as the active sulphur conversion medium and Zn 0 as the absorption medium; 4 gas-heated cracking ovens operative at 750°C. each containing 66 pipes filled with nickel catalyst; waste-heat boilers; and heat-exchangers.

The heating-gas plant contains 18 rotary-grate producers each having a capacity of 3000 Nm^3/h; they employ brown-coal briquettes as fuel and combine in the same apparatus a low-temperature distillation for the removal of tar from the briquettes before they enter the combustion zone.

For the conversion of CO to CO_2 there are 14 contact ovens each having a capacity of 7000 m^3/h and 3 apparatus for saturating the inflowing gas with water and for cooling the out-going gases. The CO_2 removal takes place at 25 atm. pressure in 8 scrubbing towers each of 2 m. diameter and 20 m. high; Peltonturbines recover sufficient power to supply two-thirds of the fresh water to the scrubbers. Each scrubber can handle 15,000 Nm^3/h.

Residual CO is removed from the hydrogen gas at 325 atm. pressure; there are for this purpose seven scrubbers having a diameter of 800 mm. and a capacity of 15,000 m^3/h.

Eleven 6-stage compressors raise the hydrogen gas to 325 atm. of pressure; after the third stage gas destined for CO_2 removal is sent to a purification step therefor. The rated capacity of the compressors from the first to the third stage is 14,300 Nm^3/h. and from the fourth to the sixth stage is 9700 Nm^3/h.

Seven one-stage 700 atm. compressors are provided having a rated capacity of 10,250 Nm^3/h.

Carbon dioxide is compressed in three o.4 atm. blowers having a capacity of 3000 Nm^3/h; there are also three 8-atm. rotary compressors having each a rated capacity of 2,200 Nm^3/h.

THE HYDROGENATION

The hydrogenation comprises: a 700 atm. "Sumpf" phase and also a 325 atm. gas phase apparatus; a grinding plant for the production of coal paste -- the drying plant for the coal is at the mine; a centrifugalization plant; and apparatus for working up the hydrogenation residues; distillation apparatus for separating crude and intermediate products; an alkali-washing plant and stabilization unit for the crude benzine; a plant for the recovery of liquified propane and butane; an alkazid plant for removal of H_2S from the hydrogenation gases; a dephenolization plant for waste waters and also tank storage. A DHD (Druck-Wasserstoff Dehydrierung) and an AT (Alkylation) plant were in the course of construction or contemplated.

The hydrogenation process operates as follows:

The brown coal after having been dried at the mine is stored in the plant bunkers. After crushing and screening over the vibratory screens to the proper size, it is sent to the coal-paste mill where it is ground to a paste with the added catalyst, sulphur and grinding oil. The grinding oil is a mixture of heavy oil or components thereof previously formed in the process, along with an oil from some other source and which is to be hydrogenated. The resultant coal-paste is then passed through a vibratory screen and is delivered to the hydrogenating converters at 700 atm. pressure by reciprocating pumps. In said converters the coal is completely converted to liquid and gaseous products with the exception of the ash and non-convertible constituents which in admixture with oil are drawn off; this mixture is technically called "Abschlamm" which, after removal of the oil by centrifugalization followed by a low-temperature distillation, is discarded to waste. The liquid product from the hydrogenating converter and which is referred to as the "Abstreifer" or "hot catch-pot" product is passed to let-down vessels and passes to the A-distillation stills where it is separated into two fractions -- the A-Middle oil and Heavy oil. The latter product is admixed with that oil recovered by centrifugalization and low-temperature distillation from the "Abschlamm" and the so-joined admixture is employed as medium to form fresh quantities of coal-paste.

The A-Middle oil is then converted into finished products. It is first hydrogenated in the presence of catalysts 5058/7846 W to Diesel oil and benzine at 325 atm. By products are gaseous hydrocarbons. This is termed the preliminary hydrogenation (Vorhydrierung) and the product thereof is fractionated, after let-down, in a B-Distillation into benzine and a so-called B-Middle oil; the latter is either sold as Diesel oil or again hydrogenated in the benzine chambers (Nach-hydrierung) over catalyst 6434, the product being distilled in a C-Distillation step. Before its entrance into the 6434 chambers, the B-Middle Oil is sulphurized with H_2S in order to protect said catalyst which easily loses sulphur and thereby its activity. The distillate from the C-Distillation is a very valuable benzine. The distillation residue is added to the B-Middle oil which is the material treated in the benzine chambers. The benzine from the 5058 (Vorhydrierung) and 6434 converters is scrubbed with caustic and thereafter stabilized by removal of gaseous components and sold.

The excess hydrogen-containing gases, etc. (Hyabgasen) leaving the different above hydrogenation stages are scrubbed with water to remove NH_3, thereafter with Alkazid solution to remove H_2S, thence they pass to a converter catalyst and to a caustic wash to remove organic sulphur, and finally to means for recoverying their content of benzine, and liquifiable hydrocarbons. The residue gas is used either for hydrogen production or for heating purposes.

It was contemplated in the DHD plant to produce benzine of a 50% aromatic content from products of the coal hydrogenation by means of a dehydrogenation and ring-forming catalyst, and in the AT plant to produce isoparaffins from normal and isobutane.

DESCRIPTION OF THE APPARATUS

A complete description of the required units of apparatus and machines, their sizes and capacities, and the names of the manufacturers of the more important of them is found in Appendix J, pages 9-20, inclusive.

DETAILS OF OPERATION

Pintsch-Hillebrand Water-Gas Plant.

The characterizing features of the Pintsch-Hillebrand process are, the use of a super-heated mixture of steam and water gas as the heat-carrier for the water gas reaction, the employment of a continuously operating gasification and low-temperature distillation shaft, and periodically operative preheaters for the above mixture of steam and water gas (Wälzgas) that flows through all said features of apparatus and also the gas-cleaning equipment, and a portion of such gas is continuously withdrawn from the cycle as product. The Wälzgas is a mixture of low-temperature distillation gas and so-called Klargas that is a product of the water-gas reaction and it is delivered by a blower to a regenerator, as shown on page 23 of Appendix J, where it is preheated to 1300°C. whereupon it is delivered to the gasification shaft. The regenerators are structurally integral with the gasification and distillation shaft; they are lined with refractory material and divided into halves that are alternately heated directly with a lean fuel-gas and they give up their heat to the Wälzgas passed through them. In the hot connections between the regenerator and the gasification shaft, valve means are avoided. The regulating and the reversing valves for the Wälzgas, heating gas, air, and combustion products are all disposed in cool positions in their pipe connections and are operated from a central operating platform by a hydraulic system. See drawings of Appendix D. Cross leakage of combustion products into the Wälzgas stream, from the regenerator portion being preheated, is avoided by so regulating the heating gas and the combustion-products valves that a small amount of Wälzgas continuously flows into the combustion-products regenerator. Reversal of the regeneration takes place at 15 minute intervals

In the gasification shaft, the Wälzgas gives up its sensible heat for the conversion of its content of steam into water gas by reaction with the hot brown-coal briquettes. The gaseous mixture leaves the gasification shaft at 700°C. and is divided into two portions, one part flowing through the low-temperature distillation - zone at the upper part of the gasification shaft, thereby removing tar and moisture from the freshly charged briquettes and this portion of said gas is so apportioned that its temperature on leaving said shaft is above its dew-point; that is 90°C. This effluent gas then passes through an electrical precipitator and after the addition thereto of Klargas, by the Wälzgas blower, is returned to the regenerator and thence to the gasification chamber. The other portion of the gas leaving the gasification shaft flows through a waste-heat boiler, a dust catcher and a scrubber through which softened water is sprayed and wherein it is cooled to 70° C. while being saturated with water vapor; thereafter this said other portion is divided into two streams,

the one passing through an electrical dust-precipitator and into the Wälzgas stream and the other passing into product after being sprayed with water, cooled to 20°C. and then sent through a disintegrator. The product stream then passes to a conversion step for oxidation of CO to CO_2 and the production of hydrogen.

OPERATION DEVELOPMENTS

This gasification plant went into operation smoothly and during the subsequent three years operation ran in greater part without any disturbances. It became increasingly apparent with experience that a satisfactory throughput of briquettes could only be achieved if they had satisfactory resistance to disintegration in the generator shaft. Such briquettes are easily manufactured if substantially uniform pressures are provided for and the brown coal has the proper moisture content. The specific gravity of the briquettes is indicative of their proper pressing; it must be 1.23. The moisture content must lie between 13.5 and 14%. Even with such "ideal" briquettes, however, the guaranteed throughput could not be reached and, since the production "bottle-neck" was principally in the gasification unit, the capacity thereof was increased by also cracking Hy-abgase (from the hydrogenation ovens) in the regenerators of the Pintsch-Hillebrand generators. This use of the Hy-abgase was undertaken after the following consideration: the capacity of the generators was limited by the amount of gases that could pass through the briquettes. If the briquettes were sufficiently strong, 9,400 Nm^3/h of Wälzgas, etc., can be forced through the shaft and of this 5400 Nm^3 are withdrawn as production-gas leaving 4000 Nm^3 to be returned to the generators as Wälzgas. As shown by the sketch at the top of page 26 of Appendix J, the generators had previously been operated employing the above volumetric distribution of gas-streams. By decreasing the volume of Wälzgas to 3000 Nm^3/h and making up the difference with the Hy-abgase to be cracked it was possible to increase the output of water-gas production. The added Hy-abgase was added to the said cold Wälzgas stream before the regenerators and after cracking therein it functioned also as a heat transfer medium to the briquettes. In as much as the temperature in the regenerators was over 1200°C and sufficient steam was made available in the Wälzgas, the added Hy-abgase was easily cracked. Obviously, the increased heat necessary for the cracking process had to be made available by increased heating of the regenerators but their capacity was adequate to make possible the cracking of 400 Nm^3/h of Hy-abgase which made it possible to increase the output of each generator by 1000 Nm^3/h of water gas.

DIFFICULTIES AND COUNTER-MEASURES

Prominent amongst the difficulties encountered in operation was the inadequate characteristics of the briquettes and normal operations were always quickly restored when the proper briquettes were prepared. Considerable difficulties were encountered in the tar-recovery; these were in greater part due to the inadequate capacity of the apparatus and were solved by enlarging the equipment.

Another problem was the plant water. In the spray saturation for the Wälzgas heavy deposits of $CaCO_3$ separated because of the NH_3 absorbed from the water gas; this was solved by installing a Permutite plant for treating the plant water.

In consequence of bomb damage to the lean fuel-gas plant, water-gas was employed for heating the regenerators; in order not to damage them because of the higher heating value of the water gas - it was 2200 W.E. in contrast to 1450 W.E. for the lean gas - the regenerators were charged with only 60% of the usual capacity. This method of operation caused no difficulty and was continued through other periods of operational disturbance.

The most important Operating Figures are as follows:

1. Operating Data on a Generator at full capacity

Briquette consumption	3000 Kg/h
Steam consumption	4200 Kg/h
of which 60% is added steam	
40% by saturation of the Wälzgas	
Heating-Gas consumption	
4.4 million W.E./h =	3,000 Nm^3/h (Hu 1460 W.E.)
Water-Gas produced	5400 m^3/h
Wälzgas	4000 m^3/h
of which 70-90% L.D.gas (Low Temperature Distillation)	
30-10% Klargas	

2. Temperatures

Wälzgas before the regenerators	70°C.
" after " "	1300°C.
Klargas leaving the shaft	700°C.
" after waste-heat boiler	230°C.
" " the saturator	70°C.
Low temperature Distillation gas at outlet of the shaft	90°C.

3. Analysis of the Klar and L. D. Gas

	CO_2	Heavy Hydro-Carbons	CO	H_2	CH_4	N_2
Klargas	14%	--	28%	56%	1%	1%
L.D.gas	17.5%	0.8%	26%	48%	6.5%	1.2%

Dust in water gas 1 - 2 mg/Nm^3

C-content of the discharged ash - 40 - 50%

4. Operating Yields

	1943	1944
Water gas production	48,600 Nm^3/h	52,000 Nm^3/h
Average Prod. per generator	4,950 "	5,100 "

Relative Yields	1943	1944
Water gas/briquettes	1,938 Nm^3/h	1,900 Nm^3/h
Lean gas/water gas	828 W.E./Nm^3	825 W.E./Nm^3
Tar Recovery/briquettes	4.8%	4.9%
Outside steam/water gas	0.126 Kg/Nm^3	0.125 Kg/Nm^3
CO + H_2 in water gas	83.5%	84%

THE PRODUCER-GAS PLANT

Description of Process

In the lean-gas generators (producers) brown-coal briquettes are incompletely burned in a draft of air to a mixture of CO_2 and CO. The heat of reaction is also employed to convert steam or CO_2 with C to water-gas. There is thus obtained a production gas that is a mixture of CO_2, CO, H_2 and N_2 having a net heating value of about 1450 W.E.

These producers were built by Pintsch of Berlin and they are equipped with features for carrying on both a low-temperature distillation and a gasification step. The inner diameter of the gasification shaft is 3 meters.

Producer gas and low-temperature distillation gas are produced in similar quantities and are purified and cooled in separate equipment, and thereafter admixed and sent to the point of consumption as a so-called "lean-gas."

The air delivered to the producer is sufficiently saturated with steam, or charged with CO_2, that a preferred outlet temperature of the producer-gas portion results. This temperature amounts to:

750°C when employing steam
650°C " " CO_2.

Sufficient of the producer gas is conducted through the compartment for low-temperature distillation as properly to remove the tar from the charged briquettes. The outlet temperature of the L. T. distillation gases is 100°C and it is flowed successively through a dust-catcher, an electrical precipitator and an indirect cooler. The electrical precipitator which it was planned to install after the indirect cooler and was omitted for reasons of economy showed its necessity because the gas contained 1g/m^3 of oil after the cooler. The producer gas leaving the generator is flowed through a waste-heat boiler and a water scrubber before being admixed with the L.T. distillation.

Operating Developments

The producer plant, as built, was of too small capacity. It was originally planned to introduce thereinto the propane and butane from the Hy-abgas. The deficiency of capacity of this part of the installation gave rise to unhappy conditions because of the high back-pressure in the producer shaft. Water spray carried from the saturator by the air blower into the lower part of the shaft gave rise to crust developments in the lower part thereof.

Employment of CO_2 instead of H_2O-saturation of the Air Blast

The formation of ashy crusts was overcome by substitution of the H_2O saturation of the blast by CO_2 made available in large quantities by the regeneration of water employed in the pressure scrubbers for CO_2 removal. It was at first contemplated to divert the H_2 and CO content of the recovered carbon dioxide to useful purpose and the steam developed in the waste-heat boilers of the producer-gas plant for some application other than saturation of the air-blast. It soon became apparent, however, that this introduction of the carbon dioxide into the generators caused an immediate reduction in the fusion of the discharged ash as well as the resistance in the generator shaft making it thus possible greatly to increase the blast.

The composition of the producer gas was changed by the above addition of CO_2. Its heating value remained, however, about the same.

Analyses of the Producer Gas	Net Heating value	CO_2	Heavy hydrocarbons	CO	H_2	CH_4	N_2
By H_2O saturation of the air	1440	4.4	0.3	31.5	15.9	1.9	45.4
BY CO_2 - addition to the air	1430	6.3	0.3	38.6	8.3	1.6	45.1

The addition of carbon dioxide gives the following advantages:

The output of the producers increased about 35%; instead of 3000 Nm^3/h it became possible to produce 4000 Nm^3/h per producer and the output of the whole producer-gas installation was increased about 18000 Nm^3/h, which thus released the Hy-abgase, above referred to in the description of the water-gas plant, for use in the cracking plant and thus made possible the increase in hydrogen production up to nearly 10,000 Nm^3/h. The steam from the waste heat boilers was used in the conversion step and liberated 2.5 atm. plant steam for other uses. The quantity of steam thus made available by a producer-gas production of 60,000 Nm^3/h was 6 tons/h. The possibility of obtaining a lower producer-gas temperature and the utilization of the CO and H_2 content of the carbon dioxide led to a saving of briquettes in the producer plant. Per Kg. briquettes there was made as producer gas:

By H_2O - steam saturation	2.40 Nm^3
" employing of CO_2	2.55 Nm^3

The saving on briquettes, at a producer-gas production of 60,000 Nm^3/h, amounted to about 25 tons/day.

THE DUST AND OIL CONTENT OF THE PRODUCER GAS is quite high, amounting to 10 mg. dust/Nm^3 and 150 mg oil/Nm^3, and can lead to operating disturbances at the point of use. Noteworthy is the effect of storage in the gas-holder which for reasons of capacity can only be used for the water-gas plant.

	Inlet Gas	Outlet to Holder
Oil content	150 mg/Nm^3	20 mg/Nm^3
Dust content	10 "	2 "

This situation can be improved by a second oil precipitator and disintegrators.

The sealing aprons of the producers suffer serious abrasion. After two years of operation several of these had to be changed because at the contact surface between the sealing liquid and gas they were eaten through in consequence of corrosive influences and the erosive action of the ash. This corrosion does not seem to take place in the water seal because it is strongly alkaline and contains no CO_2. The higher content of the corrosion product in Fe_3O_4 indicates a conversion of iron with H_2O or CO_2 in the gas space. These reactions begin at above 250°C. and can easily occur when the ash is removed at too high a temperature, which should be avoided. The metal can be protected by a tempering of the sealing aprons such for example as a so-called "Izetierung," that is, a hardening of the iron by heating to 900°C and chilling and reheating to 550° and slowly cooling.

The Most Importent Operating Results

Throughput	**1943**	**1944**
Producer gas production	48,100 Nm^3/h	60,000 Nm^3/h
Average generator output	2,815 "	3,600 "
Relative Yields		
Producer gas/briquettes	2,480 Nm^3/to	2,580 Nm^3/to
Tar/briquettes	4.8%	4.8%
Net heating value	1,444 W.E./Nm^3	1,430 W.E./Nm^3

THE CRACKING PLANT

Description of Process

In the cracking plant, the hydrocarbons in the Hy-abgase (containing methane and the like from the coal-hydrogenation ovens) are converted with steam to CO_2, CO and H_2. Two processes are known for performing the conversion, the one operating at a temperature of 1000°C. without a catalyst and the other with a catalyst at 600° - 700°C. The Wesseling installation operates according to the latter process using a method developed by I. G. Farben at Ludwigshafen. The cracking catalyst

contains Ni as the effective constituent and is sensitive to sulphur, and the gases to be cracked must be extensively freed of compounds thereof. The cracking reaction is strongly endothermic and requires considerable addition of heat which is done by gas-heating accompanied by heat regeneration.

The cracking plant thus comprises a sulphur purification plant, heated catalyst ovens and heat-recovery apparatus. The Hy-abgases to be cracked are passed into heat exchange with cracked gases leaving the catalyst ovens and are thereby heated to 420°C. for converting the organic sulphur to H_2S over an Fe_2O_3 catalyst and thereafter passing, with maintenance of said temperature, the resultant mixture over ZnO as the absorbent followed by introduction into the final five purifiers containing alternate layers of Fe_2O_3 and ZnO. After the addition of about 1.2 Kg steam for each m^3 of Hygase- 0.5 Kg of steam are for the conversion and 0.7 Kg of steam are required as equilibrium steam - the so-formed mixture is lead over the cracking catalyst.

The required conversion temperature lies between 650° - 700°C. and is produced by external heating provided by combustion of a heating gas. The waste heat of the combustion products is used to produce steam and pre-heating of the air for the heating-gas burners. The cracked gas leaves the cracking catalyst normally with 1 to 2% residue CH_4 and, after heat-exchange with the incoming Hy-gase and heating gases, passes to the conversion step in which the remainder of its steam content is used for the conversion of CO to CO_2 to produce hydrogen.

Sulphur-purification of the Hy-gase

The sulphur content of the Hy-gase to be cracked must not exceed 5 mg/Nm^3. At Wesseling, this gas contained 50 - 100 mg/m^3 of H_2S and about 50 mg m^3 of organic sulphur. The sulphur purification apperatus built by Bamag comprises an oven charged with Fe_2O_3 - catalyst for the organic sulphur followed by an H_2S removal step utilizing ZnO; the plant was inadequate. The H_2S absorption step was satisfactory but the conversion of the organic sulphur was not. The final purity of the gas was from 10 to 20 mg/m^3 of sulphur. After a series of tests with various catalysts the combination of Fe_2O_3 as the conversion catalyst for organic sulphur, with ZnO for H_2S-absorption, and the Ni-containing cracking catalyst for the final fine purification step showed itself to be very effective. The operating temperature was at about 400°C. The Ni-catalyst seemed to have retained its full activity when subsequently employed as a cracking catalyst.

Steam Production in the Waste-Heat Boilers

The waste heat boilers were at first fed with water, softened by Permutite, from the power plant.

Analysis of the Softened Water

p	m	Cl	P_2O_5	$KMnO_4$	Salts Content
0.2	0.6	350	0.5	5	200-350 mg/liter

The soft water led to heavy deposit of slime in the boilers and the distributing nozzles of the evaporator pipes and could only be kept within reason by extensive exhausting of the water from the boilers; It sometimes amounted to 50% of the water introduced into the boilers. This difficulty was overcome by using de-oiled water condensated in the plant. The analysis of this water is as follows:

Sal content	p	m	Cl	CO_2	Phenols	Fe	O_2
10 mg/l	0.0	0.1	2	3.8	0.9	0.2	0.8

$KMnO_4$
consumption
10 mg/liter

The corrosiveness of the above condensate toward iron in consequence of its content of CO_2 in the presence of O_2 and phenols, was sufficiently reduced by the addition of NaOH which was done directly before the water entered the boilers. Following the above practice, the de-sliming of the boilers was reduced from 50% to 2% of the inlet water; this corresponded to an increase of steam production of from 15-20%. A further increase in steam production of more than 25% was reached by the installing of heat exchangers in the path of the combustion products. After these improvements, the 18 atm. steam production by the cracking plant was equivalent to the 3.5 atm. steam consumption.

The Dust Content of the Producer Gas

As already mentioned in the description of the producer-gas plant, this gas contained about 10 g/m^3 of dust and 150 g/m^3 of oil. These impurities led to a rather rapid accumulation of dust, in the preheaters of the Hy-gase cracking plant which could only be corrected by their dismantling. This had to be done every three months and occasioned a week's continuous shut-down of the cracking oven and a corresponding loss in production of the cracking installation. Improvement was only noted by installation of a highly effective purification of the producer gas.

Cost Comparisons Between Cracked Gas and Water Gas

The production costs for water gas and cracked gas are about equal and are about 2.82 Pfg/Nm^3 for the CO and H_2 portions of the water gas and 2.60 Pfg/Nm^3 for the same constituents in the cracked gas, if the employed Hy-gase is valued at 0.45 Pfg/1000 W.E.

	Production Costs for CO + H_2 in Water Gas	CO + H_2 in Cracked Gas
Installation Costs Incl. Energy	16.15 RM/1000Nm^3	21.74 RM/1000Nm^3
Credit for by-products	2.27	2.23
Operating costs	3.33	1.66
Capital charges	7.64	3.75
General expenses	3.11	1.07
	28.16 RM/1000Nm^3	25.99 RM/100Nm^3

The cost of the cracked gas is in the first place determined by the cost price of the installation.

The Most Important Operating Results.

	1943	1944
Cracked gas production	33,600 Nm^3/h	40,000 Nm^3/h
Average cracking oven throughput	10,800 "	12,000 "
Relative Figures		
Cracked Gas Hy-Gase	2.717	2.400
Producer gas/cracked gas	902 W.E./Nm^3	850 W.E./Nm^3
3.5 atm. Steam consump. Cracked gas	0.544 Kg/Nm^3	0.550 Kg/Nm^3
18 atm. Steam prod/cracked gas	0.320 "	0.450 "
CH_4 in cracked gas	2.7%	1.5%

CONVERSION OF CO TO CO_2

The conversion of the CO to CO_2 in the water gas and cracked gas takes place at 450°C. in the presence of steam and a catalyst containing primarily iron. It is a strongly exothermic reaction and the whole plant can be operated without additional heat. The required steam is in part added to the inlet gas (in a saturator) and in part directly to the gases passing from said saturator to the contact oven. At the same time a small quantity of aqueous condensate is sprayed into the catalyst at a mid-point to assist in the temperature control. The gaseous mixture to be converted is saturated by spraying with hot water that is preheated in a direct cooler for the hot gases leaving the conversion catalyst and is re-circulated between the said saturator and cooler. (See sketch opposite page 40 of Appendix J). This temperature of the heated water should be maintained at about 80°C. The gas pressure in the saturator is kept low so as to favor its saturation but the resistante in the catalyst oven creates a problem in this respect. The equilibrium $CO + H_2O \rightleftarrows CO_2 + H_2$ requires a lesser excess of steam to establish at low than at higher temperatures; a low temperature in the catalyst is thus economically importent. The temperature at which the Wesseling catalyst operates lies at about 420°C.

Capacity and Increase of Output

The conversion system was operative about 30% beyond the guarenteed capacity. The bottle-neck to a further increase of capacity lay in the pressure lines of the water-gas blower and in the existing size of the saturator apparatus.

When cracked Hy-gase alone was passed through one of the conversion apparatus for changing its CO to CO_2, it is interesting to note that the conversion was self-contained in respect of heat with only 12% for conversion of CO.

Short-Comings of the Sulphur Purification Installation

By reason of the low sulphur content of the water gas produced from the Rheinish brown-coal briquettes there was made no provision for sulphur purification before the CO conversion plant. The sulphur content of the water gas was not significatnly above that of the purified gases in other works, being of the order of 400 mg/Nm³ (300 mg H_2S and 100 mg of organic sulphur.)

The conversion catalyst operated with this content of sulphur quite easily and showed scarcely any reduction of activity in three years operation.

The experience of the I G. Farben showed that fluctuations in the sulphur content by its shifting of the reaction equilibrium

$$FeS + H_2O \rightleftarrows Fe_2O_3 + H_2S$$

led to disintegration of the catalyst structure. The sulphur content at Wesseling changed with every fluctuation in the ratio of cracked gas to water gas because the former was free of sulphur. Operating provisions, for separate apparatus for these two gases, eliminated these variations and the thereby occasioned danger to the catalyst.

Deposition of Solids on the Catalyst

It is essential for the protection of the conversion catalyst to employ a residue-free steam and condensate for its cooling, so that encrusting of the catalyst surface will not take place. The steam and condensate shall have less than 5 mg/l residue on evaporation and less than 3 mg/l of Cl.

These specifications were always adhered to in the plant but despite this fact, after three years of operation, removal of the catalyst showed the topmost layer to be covered with a crust. The solid constituents had their origin obviously in saturator water-spray wherein, by evaporation of said aqueous condensate that is continuously sprayed onto the catalyst for temperature control, an enrichment of solids took place in the recirculated water of the saturating and cooling system. The addition of this aqueous condensate onto the catalyst was necessary because more water is evaporated in the saturator than is condensed in the cooler for the converted gaseous mixture.

<u>By addition of a Direct Cooler for a Part of the Saturator - Cooler Circulated Water</u>, (see sketches on page 42, Appendix J), the deficiency of water in the system was obviated and a higher saturation of the gas made possible. In ordinary operation, the hot gas from

the contact oven was first pre-cooled in a cooler apparatus by means of warm saturator-water having a temperature of about 60°C, and thereafter cooled with plant water, which was then discharged from the system. The contact gas thus passed at a full 60°C. from the lower to the upper part of the cooler and carried with it such of any added aqueous condensate as represented the saturation of the gas with water; this fraction of the added aqueous condensate was therefore precipitated in the upper part of the cooler by the cold plant water and passed to waste.

This loss of added condensate was avoided by indirectly cooling a portion of said saturator water and then employing the same for spraying into the above after-cooler, following which, it joined the water recirculated between the pre-cooler and the saturator apparatus. Solid constituents thus did not increase in the saturator-cooler circulation water. This new arrangement was tried for several weeks of operation and, despite the addition of fresh condensate, an aqueous excess had to be discharged from the system and the content of the saturator liquor in solids fell below the content thereof in the aqueous plant-condensate employed.

If a portion of the circulated water is diverted to the lower part of the cooler a 2°higher saturator temperature is obtained which in consequences of resultant higher saturation of the gas effects, a saving of the direct steam required to be used.

The Most Important Operating Results

	1943	1944
Contact gas production	103,000 Nm^3/h	120,000 Nm^3/h
Average contact-oven charge (Inlet gas)	7,000 "	7,200 "
Relative Figures		
CO in Inlet Gas	24.9%	26.4%
CO in Contact Gas	4.1%	4.4%
Contact gas/Inlet gas	1.202 m^3m^3	1.205 m^3m^3
Steam added to contact gas	0.187 kg/Nm^3	0.22 Kg/Nm^3
Evaporated saturator was	0.274 "	0.250 "
Aqueous Condensate Introduced (Cooling)	0.015 "	0.020 "
	0.594 "	0.590 "

CO_2 Purification

In the CO_2 purification there is removed down to a slight residue, the CO_2 contained in the contact gas (water gas and crack gas wherein the CO has been converted to CO_2 by a catalyst): the removal is effected by a H_2O scrubbing at about 28 atm. pressure. At the same time, the total H_2S in said gas is extracted as well as H_2, CO_2, CH_4 and N_2 in amounts corresponding to their solubility and partial pressure. By expansion, all the dissolved gases are liberated from the scrubbing H_2O.

The quantity of water employed is determined by the required purity of the gas in CO_2 and the permissible content of the expanded CO_2 in combustible gases and is so determined that the CO_2 content of the crude hydrogen amounts to 1.5 to 2% and the $CO + H_2$ content of the recovered CO_2 equals a maximum of 7.5%. The scrubbing water charged with the extracted gases is expanded through turbines which operate fresh water pumps. During the expansion about 80% of the dissolved CO_2, the inert gases and the greater part of the H_2S are liberated. The content of the expanded CO_2 in H_2 and CO amounts normally to 6 - 6.5% and in H_2S about 500 mg/m^3.

The expanded scrubbing water is freed of its residual CO_2 and H_2S content by blowing air therethrough; a portion is re-used for CO_2 scrubbing and another portion is pumped to the water system for the plant.

Re-use in the CO_2 scrubbers of a portion of the scrubbing water serves to introduce a definite quantity of O_2 into the crude hydrogen and this oxygen operates to provide in the copper solution of the CO-purification liquor the preferred content in bivalent copper.

Since the water supply for the Wesseling plant comes from a well and contains 2-3 mg O_2/m^3 too little O_2 for the stated purpose, the addition of the air-treated water having about 10 mg of O_2/m^3 is necessary. The ratio of mixture of the two waters is about 1 - 1.

Capacity of the CO_2 - Purification

The H_2O regeneration apparatus as well as the scrubbers are sufficient in size for the quantity of gas produced in the gasification and cracking plants if especially the scrubbers can be maintained in proper operating condition. They are, however, subject to growth of algae on the packing which caused the throughput of some of the scrubbers to be reduced to about one-half of their rated capacity.

Spraying of the Scrubbers with Water-Containing Chlorine

In order to maintain full production of the works, it was necessary to find some answer to the above problem which after some investigation proved to reside in chlorinated water. In practice the scrubbers were filled with water which was recirculated at 25° - 30°C while introducing chlorine. Chlorine was admitted intermittently at two-hour intervals; after each addition, the chlorine content amounted to 100 to 200 mg/l and this was consumed by the time the next addition was made. 50-80 kg chlorine were consumed in each treatment which on the average lasted one to two days. The algae were completely disengaged from the packing and were removed by draining of the scrubber apparatus and flushing the same. The cleaned scrubber exhibited the same throughput capacity as ones newly constructed.

There was the possibility that the capacity of the scrubbers might be increased by using larger sizes of packing; one of them was thus experimentally filled with 80 mm instead of 60 mm clay-rings - the gas throughput by this means was increased 15% and the efficiency of CO_2 removal remained unchanged.

Most Important Operating Results

	1943	1944
Hourly Throughputs		
Converted gas	103,300 Nm^3/h	120,000 Nm^3/h
Average throughput/washer	15,700 "	17,000 "
CO_2 Removal		
CO_2 - Amount produced	23,600 "	26,000 "
CO_2 - Dissolved in H_2O	5,400 "	6,000 "
CO_2 - In Converted Gas	27.4%	26.8%
CO_2 - In CrudeHydrogen	1.8%	1.8%
$CO + H_2$ in recovered CO_2	7.1%	7.1%
Scrubbing Water		
Water consumption	4,520 Nm^3/h	4,800 Nm^3/h
Proportion Fresh Water	55%	55%
Circulated	45%	45%
Water consumption per 100 mm^3/converted gas	44 m^3	42 m^3
T° of water	9° - 18°C.	8.5° - 15°C.

CO PURIFICATION

In the CO purification, the residual CO and CO_2 is washed out of the crude hydrogen at a pressure of 325 atm. The scrubbing medium, an ammoniacal copper solution, is revivified by gentle heating at atmospheric pressure and in partial vacuum. The solution is thereafter cooled and re-used.

The removed gases containing CO and CO_2 are added to gases flowing to the conversion step for the former compound after removing their content of NH_3 in a scrubbing step. The energy of expansion of the solution is employed in an expansion machine with nearly 100% energy recovery for pumping fresh solution to the scrubber.

The Copper Solution

The copper solution contains Cu, NH_3, and CO_2. Its active component is the complex $Cu_2, (NH_3) CO_3$ which can react with CO mole for mole. Only a portion of the copper is in combination in the complex and it increases with the concentration of NH_3. As much NH_3 as possible is to be therefore maintained in the solution. Since NH_4HCO_3 easily precipitates therefrom, its content thereof must not exceed its solubility in the solution. Warming of the copper solution causes the CO to exert its reducing effect and metallic copper is easily separated therefrom. To counteract this effect, the solution must contain some bivalent copper. Since bivalent Cu absorbs no CO its content is retained low.

The copper solution best meeting the above requirements has the following composition:

Sp. gr. 20°	1.15
mono-valent CU	12 mol/10 ltr-6.6%
bivalent Cu	2 " -1.1%
NH_3	0.480 mol/100 cm^3 - 7.1%
CO_2	0.240 " 0 9.2%

About two-third of the ammonia is in the complex whereas the rest is in combination with CO_2. The complex that combines with CO is colorless; The blue color of the solution is due to bivalent copper. Theoretically the solution can dissolve 27 times its volume of CO, and in operation, at a maximum, two third of its capacity is utilized.

Production of Fresh Copper Solution

The methods for preparing fresh copper solution have been simplified. Heretofore, NH_3 water has been pumped over metallic copper while passing CO_2 and air therethrough. The solution heats up sharply causing important losses in NH_3; the process required careful attention. According to the new method, used copper solution is pumped over the metallic copper to be dissolved. Solution progresses slowly; no attention is required and there is practically no loss in weight. No NH_3 is lost.

Operational Disclosures

The CO purification plant went into and continued operation without development of difficulties. Temporary problems were created by the packing of the expansion machine and pressure pump. It was impossible to obtain chrome leather packing for them; a Buna material proved to be a satisfactory substitute material in tests of the same duration of operation.

Capacity of the CO Purification Plant

The production bottle-neck of the CO removal plant lies in the high-pressure pumps for the scrubbers or more correctly in the pipe lines wherein at greater flow-rates higher resistances are encountered. Simplification of the pipe-line circuit made it possible to circulate 280 m^3/h instead of 240 m^3. With this quantity of solution it is possible to purify 85,000 m^3/h of crude hydrogen having 5.5% of CO. By reducing the CO content of the treated gas to 4.5% as, for example, can be done by separately treating the water gas and cracked gas in their conversion steps, about 100,000 Nm^3/h of crude hydrogen can be refined in respect of its content of CO.

The Most Important Operating Results

Hourly throughput

	1943	1944
Crude hydrogen throughput	74,100 Nm^3/h	86,000 Nm^3/h
Yield of pure hydrogen	67,500 "	78,000 "
of which 325 atm H_2	23.5%	28.5%
700 atm H_2	76.5%	71.5%
Average charge per scrubber	16,800 Nm^3/h	17,500 Nm^3/h

CO Removal

	1943	1944
Yield of CO gas recovered	5,900 Nm^3/h	7,000 Nm^3/h
CO content of crude H_2	5.4%	6.0%
CO " " pure H_2	0.2%	0.2%

Composition of Recovered CO Gas

	1943	1944
CO_2	21.0%	21.0%
CO	63.1%	63.0%
H_2	13.8%	14.0%
NH_3	3 mg/m^3	2 mg/m^3

Copper Solution

	1943	1944
Recirculation of Cu solution	251 m^3/h	255 m^3/h
Solution consumption/1000 Nm^3 of pure H_2	3.7 "	3.2 "
Temperature of fresh solution	20.5°C	20.7°C.
Regeneration temp. of solution	40.6°C.	39.5°C.

Analysis of the solution

	1943	1944
Cu_2 mol/10 l solution	11.0	12.0
Cu 0 " "	2.1	2.2
NH_3 " 10 cm^3 solution	0.488	0.490
CO_2 " " "	0.248	0.250
Cu loss (% of the added quantity)	1.83%	1.3%

PRIMARY AND SECONDARY COMPRESSORS

The primary compressors have six stages. In the 1-3 stage, the crude H_2 gases leaving the CO to CO_2 converters, are pre-compressed to 28 atm. for their scrubbing to remove CO_2. In the 4 - 6 stage the crude H_2 gas after scrubbing to remove CO_2 is compressed to 325 atm. The secondary compressors are single stage and compress the pure H_2 gas for the "Sumpf" phase of hydrogenation for the coal paste from 325 atm to 700 atm.

A series of improvements were necessary for the primary compressors before they could be operated satisfactorily. Thus, the stuffing boxes of the high pressure stages were tightened - they were too loose and held only by springs. Further, means were supplied to prevent distortion of the piston rings in the cylinders of some of the compression stages. The "Presko" - (Schleppesätte) of the cylinder of the 4 - stage was exchanged for "Thermit." By these measures and expert attention an average operating time of 5000 hours for the primary compressors was obtained.

The secondary compressors occasioned no special difficulties. A temperature of 90°C was determined as the maximum permissible temperature on the pressure side; this temperature can be exceeded by establishing too high a pressure differential between the suction and pressure sides; therefore, the permitted pressure side pressure was at a maximum double that of the suction side.

Lubrication of the Machine

During the trial runs of the primary and secondary compressors with air, a hot vapor cylinder oil of 7.5°E/100°C. and a flame point of 315°C. was used in order to avoid explosion that can result with mixtures of oil and highly compressed air. In later operation a compressor oil of 14° to 20° E/50°C. was used. Cylinder and driving mechanism contained the same oil. The oil consumption was o.7 g/hp/h = 3.8 kg/h/primary compressor and 6.5 g/hp/h = 5.4 kg/h/secondary compressor. Most of the oil finds its way into the drip pots of the individual stages of the compressors and is collected by centrifuging and re-generated and re-used in admixture with fresh make up oil. The loss per machine and centrifuge amounted to about 20%. The oil consumption of the machine thus was really only about 20% of the above given values. Eight machines required about six tons of oil per month. Improved recovery, oil separation and centrifuging reduced the consumption to one half. The oil separation was carried on at 65°C with a settling period of 24 hours.

Increase of Machine Capacity

In other plants, the capacity of the primary and the secondary compressors was increased by re-boring the cylinder. This was also contemplated for the Wesseling plant. In the primary compressors, it was sufficient to alter the 4 - 6 stages whereas the 1 - 3 stages, because of the lesser CO_2 content of the gases from the CO to CO_2 convertors than was predicted, were over-dimensioned.

The increase in capacity of the primary compressors was about 20%; that of the secondary compressors about 10%. Their driving motors were able to assume the extra load.

The Most Important Operating Results

	1943	1944
Primary Compressors		
Gas throughput	103,000 Nm^3/h	120,000 Nm^3/h
Throughput of Crude H_2	74,100 "	86,000 "
Capacity per machine		
1 - 3 stage	13,300 "	14,400 "
4 - 6 "	9,400 "	10,400 "
Average operating time	4,000 hrs.	5,000 hrs.
Secondary compressors		
Quantity of 700 atm H_2	51,000 Nm^3/h	55,000 Nm^3/h
Capacity per machine	10,800 "	10,900 "

OPERATION OF THE HYDROGENATION PLANT

In taking the hydrogenation plant into operation the following sequence was observed; the heavy oil distillation, the benzine phase, and the coal phase; no noteworthy incidents occurred.

As a starting oil there was available 11,000 tons of Estonian shale oil of the following composition:

Sp. gr.	1.012/20°C	C-83.51%
Water	trace	H- 9.31%
Solids	0.12%	N-0. 55%
Ash in Fe	7.5%	S-0. 71%
Asphalt	1.0%	Cl-0.16%
Pour Point + 2		

Boiling Range

F. D.	-203°C.
Up to 250°C.	- 9%
Up to 300°C.	-28.5%
Up to 330°C.	-39%
Up to 340°C.	-44.4%

Heavy-Oil Distillation

The content in middle oil of the employed heavy shale oil was removed by distillation in the "Sumpf" phase apparatus at Wesseling. Since some coke formation had taken place in the distillation of Estonian shale oil by the I. G. at Lugwigshafen, the temperature in the preheaters were held rather low to protect them and the distillation residue was continuously recirculated. Much steam was introduced into the columns. At the end or close of said distillation, the hottest preheater tubes were inspected for coke deposits; they were quite clean. Conditions of distillation were as follows:

Throughput: 18 tons/h shale oil and 20 tons/h recirculated residue
Steam introduced: 3.6 tons/h = 200 kg/ton inlet shale oil.
Heating-gas consumption 1650 m³/h = 150,000 W.E./ton of inlet shale oil
(net heating value 1630)

Temperature:

Outlet of preheater - 340°C
Column bottom 285°C
" top 206°C

Overhead: 5 tons/h

Sp. gr.	- 0.915/20°
F. D.	185°
200°C	5%
250°C	30%
300°C	79%
325°C	98%
E. P.	330°C.

"Sumpf" Product: 19 tons/h

Sp. gr.	1,030/20°
F. D.	335°C
at 350°C -	3.5%

In this manner, the shale oil was distilled to 3000 tons middle oil and 8000 tons heavy oil. The middle oil served as starting material for gas-phase operation, the heavy oil as pasting oil. Both products proved themselves completely satisfactory for the purpose.

GAS PHASE

In the gas phase, two conversion ovens filled with 5058 catalyst was first taken into operation. Since there was only a small quantity of middle oil available, the conversion ovens were operated in the first weeks by spraying thereinto only 3 to 5 m³/h of said material.

Detailed Operating Conditions were:

Throughput 3-5 tons/h middle oil (at 15m³ catalyst space of the oven)
sp. gr. - 0.907/20°
Phenol content - 12%
API - 10.5
Distillation E.P. 328°C.

Gas Introduced 40,000 Nm³/h
H_2 content - 88%
Pressure - 300 atm.
Catalyst T° - 16.8 mv (334°C)
first oven
16.5 mv (329°C)

Yield of Product in Hot Catch Pot - 94%

Sp. gr.	0.792/20°
API	66.5
to 180°C	24.5% by weight
E.P.	280°C.

This catch-pot product was at first put in storage and later distilled to Diesel oil and benzine, the latter to be sent to the DHD conversion plant therefor at Ludwigshafen.

"Sumpf" phase Operation

One month after the gas phase was taken into operation, the first coal stall, equipped with three coal ovens, was put into operation. The raw material was Rheinish brown-coal pasted with the distillation residue of the Estonian shale oil. Prudent operating conditions were first established; they were low concentration of coal paste and residue discharge, high gas-throughput, and low temperatures.

In addition, during the first months in order to protect the stuffing boxes of the injector machines, the pressure was maintained below the permissible 700 atm. On the basis of preliminary results, sulphur was added to the coal paste and a portion of the residue (Abschlamm) was maintained in circulation.

Detailed Operating Conditions

Throughput - 18 m^3/h coal paste incl. 20% of residue (at 24 m^3 reaction space per chamber)
Solids in coal paste - 23.0%
Added Catalyst - 6% dried Bayerschlamm (on a dry basis) and 2% sulphur

Inlet Gas 26,000 Nm^3/h

H_2 content	77%
Pressure	460 atm.

Convertor-Oven T°	
Oven 1	23.5 mv
2	23.5 mv
3	23.5 mv
"Abbau" of coal-	99.1%
Solids in the Residue	13%
Asphalts in residue	17.5%

Hot Catch-Pot Product

Sp. gr.	0.920/20°C
Middle oil to 350°C.	75%
Sp. gr. Middle oil	0.912/20°C.
Phenol content of Middle Oil	11.5%
Portion to 180°C.	16.5%

The catch-pot product was fractionated and it at 340°C. The middle oil was continuously ided to the material added to the 5058 ovens ereas the distillation residue as well as the il coming from centrifuges and the low-temperaure distillation was added to the pasting oil. he conversion time from Estonian shale oil to aw material actually produced in the Wesseling lant lasted two months and the conversion was ompleted without any disturbances in operation.

RAW MATERIALS AND FINISHED PRODUCTS OF THE HYDROGENATION

As raw materials for the finished products at Wesseling, Rheinish brown coals were used as well as also oils derived from other sources, such as brown-coal tar, high-temperature tar, and petroleum products. Up to the date of this report, July 19, 1944, 78% of the products that were made had their origin in coal, the remaining 22% in oils. Coal and oil must be treated jointly, because both in low and in high-pressure equipment they cannot be treated separately. The oils having an outside origin were in part used as pasting oil and part as inlet product to the distillation step for material separated from the hot catch-pots of the coal-convertor ovens. Their boiling-range, specific gravities, water and solids content determined the points at which the oils were introduced into the process system before the distillation of the hot catch-pot product. The others were mixed with the pasting oil. A diagram on page 58 of Appendix J shows that in 1942, there was from month to month, never more than respectively 10% and about 40% of the finished products prepared from middle oil and heavy oil, the remainder being prepared from coal. In 1943, the production averaged about 10% each for the Middle Oil and Heavy Oil. Nearly half of the foreign oil was middle oil. The greatest part of this was employed as pasting oil.

Characteristics of the Rheinish Brown Coal that are of importance in hydrogenation. The employed Rheinish coals are easily and almost quantitatively altered chemically. The formed products are, however, difficult of hydrogenation; in consequence, the high pressure of 700 atm. in the "Sumpf" phase was made necessary. Visual inspection shows these coals to have a high content of lignitic substance and they give the impression of having been mixed with decayed tree stumps and plant fibres. The process difficulties which were anticipated did not, however, develop in the hydrogenation process. Proper handling of the large-sized and small-sized screens in the coal drier produced a normally operative coal for hydrogenation.

Analysis of the Coal to be Hydrogenated

Water Content	7.5%
Ash "	6.0%
Tar "	8.0% (Fischer L.T. Method)

Elementary Analysis		Ash Analysis	
C -	67.75%	SiO_2	2.9%
H -	5.1%	Fe_2O_3	22.5%
O -	25.3%	Al_2O_3	4.1%
N -	1.1%	CaO	45.1%
S -	0.75%	MgO	4.9%
		SO_3	20.1%

Of these values, those underlined are especially important for the hydrogenation.

The CaO - Content

The CaO content makes itself especially evident in a tendency to form and separate Ca CO_3 during the chemical alteration of the coal. It separates either in the form of encrustations or as spheres (called caviar) and leads to manifold difficulties.

The O_2 Content

The oxygen content is very high (25%) and there is, in consequence, a high H_2 consumption in the hydrogenation that changes about two-thirds of the oxygen to H_2O.

The Sulphur Content at 0.75% is very low in comparison with the hydrogenatable coal of middle Germany. Since the catalytic effect of sulphur on the hydrogenation cannot be denied, the addition of sulphur from an outside source is necessary.

Foreign Oils

There has been employed in the process shale oil, petroleum residues, tar products from bituminous coal such as paving tar and oils of the middle oil type, and brown coal tars; that is, oils of aliphatic, naphthenic and aromatic character. Definite fluctuations naturally resulted in the chemical composition of the products formed. These differences, however, always were so balanced by the brown coal components, which always predominated, and also by the effect of the gas-phase catalyst, that they were not practically evident in the marketed products. Operating difficulties of a serious nature did not arise. Only the continuous addition of large quantities of tar-products derived from bituminous coals gave rise to certain problems. Thus, the addition of over 5% of paving tar to the pasting oil caused trouble in the low-temperature distillation of the unconverted residues because of insufficient conversion of asphalts. Also, too large addition of middle oil direct from bituminous-coal was unfavorable. By the occasional addition for some days of more than 35% of said Bituminous coal-tar Middle Oil to the Middle Oil produced in the process itself, the effectiveness of the benzine catalyst was temporarily reduced. Apparently, to make possible continuous additions of this magnitude, the Middle Oil must be cut at 330° instead of 350°C.

Marketable Products

There have been produced:

Normal Diesel Oil

Diesel Oil for the Navy

Cold-resistant Diesel Oil for Russian campaign.

A benzine to be treated in the DHD plant at Ludwigshafen and which had passed only through the preliminary hydrogenation.

A normal aviation benzine.

A normal liquefied fuel, and

A so-called Flumo-liquefied fuel that served for running-in aviation motors.

Specifications for the Products

Normal Diesel Oil

Sp. gr. 15°C.	0.800 - 0.885
Viscosity	1.1 - 2.6°E/20°C.
Pour Point	
Summer not over	- -10°C.
Winter " "	- -20°C.
Flame point "	- 21°C.
Cetane No. if possible not under	35

Navy Diesel Oil

Sp. gr. 15°C.	0.840 - 0.885
Viscosity	1.2 - 2.6°E/20°C
Start of paraffin separation	± 0°C
Flame point not under	+ 55°C
Cetane No. not under	35

Diesel Oil (Russian Quality)

Viscosity not under	1.1°E/20°C.
Paraffin separation below	-30°C.
Pour Point "	-35°C.
Flame Point over	21°C.

Benzine for the DHD - Plant

End Point	165°

Aviation Motor Benzine

Sp. gr./15°C.	0.725 - 0.750
F.D. not below	40°C.
at least 10 vol. %	70°
" " 50 " "	100°
" " 90 " "	145°
End Point below	165°
Vapor pressure at most	0.5 at 37.8°C.
API highest	52°
Octane No. (Motor Method) at least	70
with 0.09 Vol. % tetraethyl lead	87

Supercharge

The supercharging curve of the benzine with 0.120 vol. % of tetraethyl lead must, by test in a RLM - GL/R-M II motor permitting supercharging and at least on the day tested, lie above the Eich - B4 supercharging curve in the region of the excess air index.

1 = 0.75 - 1.3

Normal Treibgas (bottled hydrocarbons)

Vapor pressure

1 Apr - 31 Aug.	at least 0.7 atm	at	+ 0°C
	at most 16.7 "	"	± 40°C.
1 Sept-31 Mar.	" least 1.5 "	"	- 15°C.
	" most 16.7 "	"	+ 40°C

H_2S not over	0.2 mg/m^3
Org. S not over	250 " "
Elemental S	none
Mercaptan (Doctor Test)	none
NH_3	"

Flumo - Treibgas

C_4	70-80% by WT
C_3	20-30% " "
Other hydrocarbons	Max. 5%
Vapor pressure, not over	2 atm/0°C.

Other specifications same as for normal Treibgas

The above specifications were met at Wesseling by all products made with the single exception of the winter quality of Diesel oil for Russia which could not be exactly met. The solidification temperature was exceeded some degrees.

STEELS FOR THE HYDROGENATION

Large scale practice of the process of high-pressure hydrogenation could not be carried out without suitable steels for the equipment. Because of the high-pressure requirements, the steels must be very strong so that the apparatus is not too heavy and unwieldy. They must be resistant to H_2, to the corrosive effects of the hydrogenated products and especially to the H_2S contained in them. Since in greater part they operate at high temperature, they must have the above characteristics in the range of operating temperatures.

These high requirements are developed only in alloy-steels which are heat treated. Especially suitable are the V_2A steels which contain high additions of Ni and Cr up to over 25% content. The deficiency in these alloying constituents during the war compelled the developments of steels having low contents of such foreign-derived metals. The I. G. Farbenindustrie deserves the credit of having provided such difficulty-preparable steels that are economical of metals but meet the requirements of high-pressure hydrogenation. By heat treatment, the steels are provided a structure that provide them with great mechanical strength and resistance to chemical influences. The treatment consists in longer annealing in the austenitic region, in chilling and short tempering. The annealing and chilling made the steels hard yet brittle. By means of the tempering with approximate retention of the hardness, the brittleness is partly removed. The so-produced steels are sufficiently tough for safe operation.

In the tables on pages 63 to 66, inclusive, of the accompanying Appendix J, there is a survey of the steels developed up to the present and the parts of apparatus for which they are suitable in use.

The summery page of the above said pages provides a review of the steels at present available and those adapted for special purposes. There is also shown the composition, mechanical strength, methods of testing and the method of heat treatment.

The first steels employed in cold parts of the apparatus when the hydrogenation process was operated at 200 atm pressure were normal carbon-steel of which some had small additions of Cr. For the hot portion of the apparatus there was developed the N 1 to NS - steels. N 1 is the standard steel for the high-pressure hollow apparatus. Of the above materials N8 is used today for the hot pipelines. The primary characteristic of this steel is its Cr content which amounts to about 3%. Other added metals are Molybdenum and Vanadium in quantities of about 0.5%. These steels have the advantage that a local tempering is sufficient after their welding whereas the steels developed later have to be treated in an annealing oven. After the introduction of the higher pressure of 700 atm., as a process feature, the mechanical strength of the S-steels was not adequate for the piping. Steels were rather employed having a Brinell Hardness of 70 to 80 instead of the former 50 - 60. These products were prepared under the name of K-steels. They differ from the S-steels by their Cr content of 1% along with a small amount of molybdenum. The N8A to N10 and some K-steels were developed for the hot apparatus parts that operate under a pressure of 700 atm; of these the K5 and K7 are recommended; all these steels contain vanadium. The somewhat higher carbon content of the K-steels is characteristic of them. The best developed steel for high temperature is the N10 which has a pressure resistance of 16 at 550°C. It is used for the heaviest duty pipelines and other parts of the 700 atm. apparatus and especially the preheater pipes.

The shortage of Tungsten and Molybdenum compelled the production of substitute steels such as N8A, N8V, NG, K1CV, and K3CV steels. In these, the additions of Vd and Mn were increased. Since also the employed Cr was also short, there has more recently been added some Si to replace the lesser amounts of Cr employed. There thus originated the 700 atm - steels called KIMS and K4Ms. All these last mentioned steels will not be employed in normal times.

In the survey (see Appendix J, page 66) of the materials employed for individual apparatus parts of the high pressure system, the encircled numbers and those tables having the title "Werkstoffuebersicht für Hochdruck 325 and 700 atü" show the steels that are available for the period of the present war.

THE STEELS EMPLOYED IN THE HIGH PRESSURE PLANT AT WESSELING

For 325 atm

High pressure hollow members	N1
Cold pipes	S2
Hot pipe	N8, N8A, N10
Gas preheater	NS, N8V, material
Electrical preheater	N8, N10
Fittings	S2, N8, N8A, N9, N10
Lenticular gaskets	S2, N5, N5A, N8
Flanges	S1, K1, K3, K3CU, K4MS
Screws	S3, K1, K3, K4MS,K5

for 700 atm

High pressure hollow members	N1
Cold pipes	K2
Hot pipes	N8, N8A, N10
Gas Preheater	N8, N8V, N9, N10
Fittings	N8, N8A, N9, N10
Lenticular gaskets	S3, N5, N5A, N5C, N8
Flages	K1, K3, K3CV, K5, K4MS
Screws	S3, K1, K3, K3CV, K4MS

A well-schooled staff is pre-supposed for control of the proper use of the steels. The smallest details must be taken into consideration because improper use of the smallest apparatus parts, such as screw-bolts and lenticular gaskets, can have unpleasant consequences.

Control means begins with the analysis. Two methods are available for the purpose; the complete quantitative analysis and a rapid method, the so-called

"Tüpfel" or spot-method. The latter comprises moistening the piece to be investigated with a mixture of sulphur + HN_3 or of sulphur and H Cl and noticing the color of the formed spot. All parts of the equipment installed at Wesseling were investigated once according to the quantitative method and twice by the above spot method. Of particular importance is the markings of the materials. All larger parts, fittings and pipes are stamped to show the type of material, the method of forming and the testing. In the case of minor parts such as the lens gaskets, screws, etc. the type of material is shown by some special designation.

The erection of the plant must be controlled by means of drawings showing the proper parts to install at any given point and after every repair job the construction must be gone over. Such care pays dividends; in the almost four years of operation of the Wesseling works not a single disturbance in operation arose because of materials failure or their incorrect use. This splendid result can be traced primarily to the engineering and technical staff's ability to arouse in all their aides a special interest for the technical materials question.

HIGH PRESSURE 700 ATM - "SUMPFPHASE"

In the "Sumpfphase" the employed oil and coal is converted by both cracking and addition of hydrogen to middle oil.

Description of Method

The coal paste produced in the grinding plant is filtered over oscillatory screens and flowed to storage. In the screening operation, there is normally produced 0.3 Kg/ton of paste-residue but this quantity can increase to 2.0 Kg/ton of paste with variations of the content of dry coal in difficultly grindable substances. The said paste residue from the screen contains about 40% oil and is sold. By means of piston pumps, the paste is pumped to the high-pressure paste-presser, with return of an excess of the paste, and thence into the coal convertor-ovens. The paste presses on their suction sides are also connected to a supply of friction oil. The switching from coal paste to friction oil can be quickly effected by means of two valves. At each starting-up and shut-down of the convertor-ovens, as well as also during the operation in order to damper sudden rises in temperature, friction oil is pumped into said convertor-ovens. The glands of the paste-presser are protected by spraying into them flushing oil that is free of solids. The coal-paste is introduced into the coal-convertor ovens along with new hydrogen also containing recirculation hydrogen-containing gas, after first passing their admixture through heat-exchangers and preheaters. The following procedure has been found to be satisfactory. The recirculated gas is introduced into the cooler of the heat-exchangers and is then divided into two equal portions. One part goes along with the paste through the hotter heat-exchanger and through the hair-pin shaped preheaters whereas the other half goes directly to the first coal-convertor ovens after passing through a separate hair-pin preheater. The pre-heaters are gas-heated; the hot combustion products are recirculated by blowers. The temperature of the recirculated gas must be below the highest permissible wall temperature of the preheater pipes. The temperature of the gas-paste mixture entering the coal-convertor ovens is kept as low as possible in order, firstly, to save heating-gas and, secondly, because at temperatures above 21.5 mv (317°C) $CaCO_3$ crusts start to form. In the convertor-ovens the coal structure is broken down with strong heat evolution. The conversion temperature is at about 25 mv (478°C) and is regulated by addition of cold recirculating hydrogen gas. In the lower part of one of the first convertor-ovens there is a discharging device called a "de-sander". By means of this apparatus, there is continuously withdrawn about 1000-1500 liters/h of product so as to prevent the formation of so-called "Caviar;" that is, spheres of $CaCO_3$ that otherwise separate out. The conversion products of the coal paste are in greater part vaporous and are carried along with the recirculated gas stream. The ashy constituents of the coal flow, in admixture with the highest-boiling oil components in the form of so-called "Abschlamm", from the final convertor-ovens over into the hot catch-pots. The solids component which comprises about 20-22% of the "Abschlamm" determines the height of the temperature employed in the convertor-ovens. The "Abschlamm" has a tendency to form coke in the hot catch-pots. This formation of coke is due to a deficiency of hydrogen and can be avoided by introducing fresh hydrogen at that point. This addition of fresh hydrogen also effects a de-gassing of the hydrocarbons dissolved in the "Abschlamm". The latter is cooled to about 150°C, expanded to atmospheric pressure by means of special erosion-resistant valves of which the exposed parts are finished with tungsten carbide, and is thence pumped to the "Abschlam" centrifuges. The gas liberated by the expansion of the "Abschlamm" contains over 90% H_2 and is flowed into admixture with make-up hydrogen at a point before the hydrogen compressor.

From the criculated gas-stream product leaving the coal-convertor-ovens, the liquefiable constituents are condensed in heat exchangers and coolers. The yield of such liquids is collected in separators disposed after coolers therefor. The temperature of these cooler-outlets must be held to 120°C because the liquid product tends to foam at lower temperatures. The product from said separators (cold catch-pots) is expanded and flowed into a special distillation apparatus. The expansion takes place in 2 stages so that the evolved gas is divided into a gas rich in hydrocarbons and one relatively lean therein.

After separation of the said liquid products therefrom the residual circulating gas is sent to a high-pressure oil-scrubbing step and with the help of circulating pumps is returned to the coal-convertor-ovens after replacing the consumed hydrogen by additions of fresh H_2.

The oil-scrubbing for the recirculated gas comprises high-pressure gas scrubbers, a wash-oil regeneration apparatus, and a pump for spraying oil into the scrubbers. The employed scrubbing oil is the product of the Benzine convertor-ovens of the process and it is employed in such quantities that the specific gravity of the circulated gas at the coal-convertor-oven inlets is 0.350 which corresponds to a 70-75% H_2 content. The wash oil is regenerated by expansion performed in two stages.

The temperature of the stream of recirculated hydrogen gas is maintained above 30°C to prevent the separation of hydrocarbons and ammonium bicarbonate.

Normal Throughput and Analytical Results of the Coal Convertor-Ovens.

Paste introduced	40 m^3/h coal paste (when the reaction space is 31m^3)
Catalyst addition	5% Red Iron ore (based on dry coal) 1.25% Sulphur

Analysis of the Coal Paste

Solids	41%	
Pure Coal	35.6%	
Ash in the Solids	13.3%	
S " " "	3.0%	
Sp. gr. 110°C.	1.175	
H_2O content	1.9%	
Screen Residue over 0.3 mm Screen	10.6%	
Amount of Inlet gas -	28,000 Nm^3/h	
of which -	13,000 Nm3/h	is paste-gas
	15,000 Nm3/h	goes direct to ovens
Amount of cold gas -	12,000 Nm^3/h	
H_2-Content (Inlet Ovens)	73%	
Pressure	650 atm.	
Temperature Coal Chamber 1	22.5 - 25.0 mV	
" 2	22.5 - 25.0 mV	
" 3	" "	
" 4	" "	
Conversion (Abbau) of the coal -	99.6%	
Middle Oil Output (new formation)	0.275 tons/m^3 reaction space.	
Quantity of hot catch-pot product based on coal paste	50%	
Quantity of "Abschlamm" based on coal paste	33.3%	
Middle Oil - 350°C in hot catch-pot	39%	
Middle Oil - 350°C in Abschlamm	11%	
Solids in Abschlamm	20%	
Asphalt " oil	16%	

Operational Developments

In the "Sumpf" phase, great operational difficulties arose which in part were completely and in part were sufficiently solved to permit smooth operation. Amongst these difficulties were the following:

1. The difficulties of separating water and the formation of foam in the catch-pot products.
2. Separation of $CaCO_3$ in the coal conversion with formation of Caviar and incrustation.
3. Poor conversion of the asphalt and the formation of coke in the hot catch-pot.
4. Erosion of the preheater-pipes.
5. Rapid reduction in the regeneration of heat.

The Catch-Pot Product

The catch-pot product formed by the treatment of Rheinish-brown-coal has a high specific gravity; it lies between 1.000 and 1.030 at 20°C. This made it impossible to perform the necessary separation of water before introduction of said product into its distillation step. An effective solution was found in dilution of the product with an oil of low specific gravity. After a temporary use of A-Middle Oil, 10 to 15% of benzine-like forerunnings, obtained by distillation of the material separated in the hot catch-pots, was added. Establishing a specific gravity of 0.970 at 50°C. permitted smooth operation.

The liquid product separated from the reaction mixture in the hot catch-pot had a tendency to foam and the foam became entrained in the circulated gas and gave rise to difficulties in its subsequent scrubbing with oil. By increasing the temperature of the separation to the extreme height of 120°C the foam-formation was avoided.

Caviar and Salt Encrustations

Inorganic products of the coal tend to separate during its conversion and they form sphere-like objects that are referred to as "Caviar"; they appeared in the Wesseling operations in a quantity previously unobserved. It not infrequently happened that all 4 convertor-ovens of a stall were within 10 days completely filled with caviar. There simulteneously appeared also encrustations that adhered solidly to the walls of the apparatus and the equipment contained therein. These two products had the same chemical composition and comprised primarily $CaCO_3$.

Ignition Residue		Analysis of the Ash							
		CaO	CO_2	MgO	S	Fe	Al_2O_3	SiO_2	C
Caviar	58.4%	46.3	31.8	2.9	1.4	1.8	3.1	0.1	7.1
Encrustation	59.4%	47.0	26.0	4.5	1.5	4.0	1.7	0.5	5.6

The caviar formation caused especial difficulty in operation. It caused rapid reduction in the effective capacity of the convertor-ovens and consequently of throughput but, above all, gave rise to mechanical problems. The caviar at first coked together and formed an obstacle to proper heat transfer into the admixture thereof with reaction product, so that the convertor-ovens were rapidly filled with a solid mass having very limited permeability and in a short operating time the convertor-ovens had to be taken apart. The removal of the coked caviar mixture, which is almost as hard as stone, is difficult and time consuming and bound up with serious abrasion of material. The cause of the caviar has been the subject of much thinking and has been much written about. In explanation of the countermeasures taken to its production, it is sufficient to say that the caviar-formation is tied up with a definite period of the change produced in the coal structure during its hydrogenation and that it takes place in a zone - assuming the apparatus is in good condition - lying at the lower part of the first convertor-oven and that the nuclei of crystallization and the later grains of caviar must linger in the zone of formation in order to grow.

Effective alleviation of the difficulty resides in removal of a product-stream from the lower part of the first convertor-oven by means of the so-called "de-sanding apparatus" by means of which any very long residence and consequent growth of the crystalline slime of the caviar is avoided. This method has been long used at Leuna. The Leuna apparatus had to be modified for Wesseling operations because its unmodified use was ineffective. On pages 76 to 80 of Appendix J are shown diagrams of different "desanding" apparatus and methods employed and they should be referred to in conjunction with the following description.

In Leuna about 100 to 200 liters of product were hourly withdrawn through the "de-sanding" apparatus; at Wesseling about 1000 to 2000 liters/h were necessary. This latter quantity of product could not be withdrawn through the Leuna apparatus because too much gas escaped at the same time. Such was the case and for the following reasons: The pressure reduction of the removed product takes place shot-wise in order to prevent deposition in the valve of the horizontal portion of the "de-sanding" pipe. For this reason the de-sanding valve was opened at spaced intervals of 1 to 2 seconds. At each opening of the valve, the outlet pipe was thus jolted open by the pressure of products in the convertor-oven and at the same time products flowed out of the oven. The products consisted primarily of gas because the high velocity of their downward flow permitted no separation of gas and liquid. During the pause before the next release, liquid insufficiently displaced the gas contained in the pipe upstream of the valve and thus during the next release both liquid and gas were expanded when the discharge valve was opened. This difficulty did not arise at Leuna because the convertor-ovens there stood on supports permitting relatively long vertical lengths of "de-sanding" pipes; this was not the case in Wesseling. In Wesseling, the construction of a sort of pocket of 6 liters capacity, below the conical bottom of the first convertor instead of the Leuna outlet-pipe of 40 mm. diameter, provided a satisfactory solution. The expansion valves for the "de-sanding" product suffer great erosion. Provision of a substitute material for the valve offered a great problem and so a valveless de-sanding device was projected. A further reason for altering the existing construction was because of the product loss caused by insufficient chemical alteration of the "de-sanding" material and because the incidental loss of gas was necessary to avoid. The last mentioned reason will be later dealt with more in detail.

The Principle of the Valveless "De-Sanding" Apparatus

The "de-sanding" product was no longer reduced in its pressure but by-passed to convertor-oven No. 2 in a series thereof. The diameter and the length of the communicating line was so dimensioned that at the almost constant 1 atmosphere drop in pressure between said convertor-ovens a preferred quantity of product was transferred therebetween. The velocity should not exceed more than 1.5 m/sec. thereby to avoid deposition therein of heavier particles of product. Caviar formation in the convertor-oven II is obviated by providing that the paste so withdrawn from the lower part of the convertor-oven I had passed through that stage at which typical caviar formation takes place in the coal conversion. To accomplish this end, the inlet point of the coal paste is displaced to the central point of the first convertor-oven by means of an internal pipe extending about half-way upwardly thereinto whereas the preheated, circulated gas is separately introduced at the bottom of the convertor-oven, as heretofore. This device was employed in two operating periods of about 50 days each. In both cases no caviar was formed in the second oven. In comparison with the caviar formation, salt encrustations cause little inconvenience despite the fact that the trial tests made at Leuna and Ludwigshafen indicated that there must arise special difficulties due to their formation. It had been attempted chemically such as by the additions of H_2SO_4 or sulphur to prevent formation of encrustations but the results were unsatisfactory. The single positive experience gleaned from the test apparatus was that the separation of salt was related to temperature. These tests showed that at 19 mV crust formation set-in and increased with increasing temperature. The lower temperature of formation in the Wesseling coal chambers happily was somewhat higher, that is at 21.5 mV. The heaviest crust formation was encountered in the zone of caviar-formation; that is, in the lower part of the first convertor-oven. In this region, salt deposition on the oven-walls, thermo-couple and cold-gas pipes became 50 mm. thick. The salt formation in the coal convertors diminished quickly from inlet to outlet. In the pipe-lines to convertor oven 2, the deposition was about 10 mm thick; in and after said convertor-oven 2, it was only a few mm thick. Very serious difficulties were caused by encrustations in the pre-heater pipes because heat-transfer from heating gas to product was interrupted which caused over-heating of the pipe walls. The relatively high limit of solids formation at 21.5 mV made it possible to avoid their separation. The first convertor-ovens, for example, have because of the high partial pressure of hydrogen at 700 atm such a high heat of reaction that the outlet temperature of the preheaters can be maintained below 21 mV. The salt separation in the first convertor-oven caused operational difficulties in that, in consequence of the war demands and operational changes to treatment of oils, these plaques or crusts tend to drop off the walls and to plug the "de-sanding" apparatus and necessitate shut-downs.

Influence of Dilution-Oil on the $CaCO_3$ Separation

The observation that salt encrustations appeared heavier in the colder, uninsulated parts of the pipe lines than in the neighbouring insulated ones, led to the assumption that a crystallization of $CaCO_3$ from supersaturated solution was being dealt with and to the attempt to obviate the separation by use of a dilution-oil spray. Thus, one of the first things tried to combat the problem of caviar-formation was the introduction into some convertor-ovens of cold oil instead of cold gas. This expedient led to a lessening of the caviar-formation, and showed itself by deferring to from 3-4 weeks instead of 3-4 days the disturbances in the temperature readings of the convertor-ovens which usually indicated the start of forming of caviar-crusts - the operating time of the convertors was nearly doubled. Dismantling of the convertor-ovens showed them to be little different from the earlier ones; that is, the oil-sprayed chambers were filled, when shut-down, with encrusted caviar. Since the oil-spraying did not exclude entirely the difficult convertor-servicing, it was discontinued because the "de-sanding" of oven 1 led to a solution of the problem.

The Injection of Oil into the Cooler Heat Exchanger

Recently a stall was again equipped for oil injection. In this way more oil of outside origin was converted and in this way an increase of production was attained, first because the preparation of the finished products from oils consumes only about one-half the hydrogen required for coal conversion and, secondly, because there was a shortage of H_2 production in the Wesseling plant.

At the same time, it was thought there would be a reduction in salt-crust formation because the dilution-oil was added directly before the first convertor-oven - see the figure on page 80 of Appendix J - and the saturation of this fresh oil with dissolved solids would require a definite time; there was thus the possibility that in the first coal convertor an unsaturated solution would exist. The conversion of the oil proceeds under a lesser heat of reaction than the coal and requires higher conversion temperatures, thus the vessels charged with oil must be more highly heated. In order to produce this condition, without at the same time exceeding the highest permissible temperature of 21.5 mV in the hair-pins of the preheaters conveying the coal paste, the oil was separately added and more highly heated. It was added to one of the independent streams of recirculated gas and in admixture passed through the colder Heat-Exchanges and thereafter through some of the preheater pipes lying in the hottest part of the combustion-products. By means of this arrangement, heat given up by that outlet-product of the coal-convertor which passes through the colder heat-exchanger, was better utilized. In addition, the preheater pipes in question could without danger be more strongly heated since no crust-forming substances could separate out of the mixed gas-and-oil stream and therefore no over-heating of the preheater pipes charged with coal-paste was to be feared because the top-temperature of the recirculated combustion-products through the preheaters for the oil-and-gas mixture was already sufficiently reduced. The mixture of oil-and-gas was so highly over-heated that their temperature, after joining with the stream of coal-paste and gas, was 22.5 mV.

The stall was operated 50 days with a paste injection of 40 to 45 m^3/h. The improvement of oil conversion conformed with expectations. There was so much more heavy oil converted than was previously the case that the Middle Oil production increased a good 1000 tons per month. The increase in heat recovered in the colder-heat-exchanger increased about 50,000 WE/h. The effect of the oil on the crust-formation was confusing. The salt-crust in the first convertor oven was only up to about 5 mm thick whereas, in that one which was not charged with oil, it was 40 to 60 mm after the same period of operation, and the crust was harder and clung faster to the walls. It appears as possible that the crust formation can be more sharply repressed or entirely avoided if larger quantities of oil are added. In order to examine this possibility it must be done at the expense of the number of coal-paste preheater tubes or to increase the size of the preheater for the mixture of gas and oil.

In conclusion it can be said, relative to the measures taken to solve the problem of $CaCO_3$ separation, that the caviar formation is with certainty impeded by the withdrawal of a small product-stream from the first oven and that it appears also likely to reduce the formation of salt encrustations in simple manner at least to such extent that they are unimportant.

Asphalt Conversion

The constituents that are precipitated out of the "Sumpf" phase product by normal benzine are designated as "asphalts". The analytical determination of the asphalts offers difficulties because their precipitation as well as their extraction is hard to complete. It was not possible with the assistance of the I. G. Ludwigshafen and Leuna methods to obtain reliable results. Consequently, the following method was worked out by the main laboratory at Wesseling.

Two grams of the solid-free oil were weighed in an Ehrlenmeyer flask of 300 cc capacity and dissolved in 10 cc of pure benzol at room temperature. 190 cc of n-Hexane (Ruhrchemie product) were added in a stream and the flask strongly agitated for about one minute. After standing one hour in a refrigerator, the contents of the flask were filtered through an extraction thimble and washed with n-Hexane. The thin asphalt coating retained on the walls of the flask was dissolved in benzol and held in reserve.

The extraction thimble was extracted in a cold extraction apparatus for one hour with n-Hexane to remove completely the oil entrained in the same. To convert this asphalt into weighable form, the air-dried thimble was extracted with hot benzol for half-hour in a Soxhlet apparatus. To this extract, the above benzol solution of asphalt held in reserve was added and the whole introduced into a tared Ehrlenmeyer flask. After evaporation of the benzol and a half-hour drying period in the drying oven at 110°C, the flask and contents were weighed on an analytical balance.

The oils recovered from the Rheinish brown coals are difficult of hydrogenation. A measure for the degree of hydrogenation is the degree of asphalt conversion. This depends on: the type of added oil of outside origin; the catalyst and sulfur added; the rate of charging and the operating conditions. The outside oils employed were easily treated with the exception of paving tar. Of this latter not more than 5% could be added to the pasting oil because the asphalt content rose sharply and difficulties arose in the low-temperature distillation of the "Abschlamm." 5% and 1.25% by weight of the dry coal in respectively catalyst and sulphur were found to be sufficient.

The following operating conditions bear special attention:

The convertor-oven temperature

The H_2S partial pressure which is primarily determined by the gas throughput.

The time the asphalts are allowed to remain in the ovens which is important because of their thickening of the "Abschlamm" which is their carrier.

There is an inter-relation between these above conditions: for example, an increase of temperature at the same concentration of "Abschlamm" requires a reduction of the quantity of recirculated gas because the degree of thickening is influenced by the temperature and the quantity of gas passed through the coal-paste. Since in the course of the Wesseling operations, the influence of temperature showed itself to be the strongest, importance is primarily placed on maintenance of a high reaction temperature. It was held at 25 to 25.3 mV as a maximum. The concentration of the "Abschlamm" was likewise kept constant: i e. at 20-22%; it thus establishes automatically the quantity of inlet gas, which was relatively low, being about 28,000 Nm^3/h and yielded an H_2 - partial pressure of about 450 atm. at the convertor-oven inlets and 300 atm. at their outlets. The asphalt content of the "Abschlamm" oil amounted to 14-16% and satisfied operating requirements.

In an extreme instance the quantity of recirculated gas in a chamber amounted to only 14,000 Nm^3/h in consequence of a short-circuit in the preheater. The chamber temperature was held at the highest permissible value of 25 to 25.3 mV. The asphalt content of the "Abschlamm" was 16%, which is not significantly above the normal amount. A low partial pressure of hydrogen can be compensated for by the operating temperature of the reaction. By reason of this experience it would be in error to depreciate the effect of the H_2 -- partial pressure. The small-scale tests that were made on the Wesseling coal at Leuna at different H_2 - partial pressures showed that its influence was very great. Temporary increase of the H_2 - partial pressure, about 100 atm. reduced the asphalt content about 1/3 of the previous value. In confirmation of this, the occasional use of fresh hydrogen instead of recirculating gas as the cooling gas and the introduction of fresh hydrogen into the hot catch-pot accounted for some percent improvement in the amount of asphalt.

Introduction of Fresh Hydrogen into the Hot Catch-Pot

In combination with the poor tendency of the asphalts to accept hydrogen, the Wesseling "Abschlamm" showed a tendency to form coke in the hot catch-pot. This tendency increased with the increasing concentration of solids therein. The solids content of 20-22% permitted in operation are the highest tolerable amounts.

As a means for eliminating the coke formation there was used the expedients of mixing asphalt-free oil with the "Abschlamm" and introduction of circulating gas in the conical bottom of the hot catch-pot. Oil injection was later abandoned since the introduction of the recirculating gas, and later fresh hydrogen gas, proved effective for the purpose.

The introduction of the fresh hydrogen provided the following advantages: By its high content of hydrogen the gaseous hydrocarbons, which were dissolved in the "Abschlamm" and tended to go along with the gas given off from the "Abschlamm" into the heating gas, were removed therefrom and returned to the convertor-ovens by the recirculating gas stream. They then were dissolved by the cold catch-pot product and in the wash oil used in scrubbing the recirculated gas; expansion of the scrubbing oil liberates them into those gaseous hydrocarbons liquefied in the Treibgas plant.

The "Abschlamm" gas contained instead of 60% over 90% H_2 and is of utility as fresh hydrogen for the hydrogenation step. A part of the heat led away in the "Abschlamm" is carried over into the recirculated gas and is utilized in the heat-exchangers of the stalls. By adding the "Abschlamm" gas to the fresh gas, the hydrocarbon content of the "de-sanding" gas that is simultaneously withdrawn is disturbed. This unsatisfactory condition was one of the reasons for re-construction of the existing "de-sanding" device. By employing the above-described Convertor Oven 1 to Convertor Oven II -- "de-sanding" method, no "de-sanding" product was produced and consequently no "de-sanding" gas.

The high H_2 -- partial pressure of the fresh gas introduced into the hot catch-pot effects, in spite of the simultaneous cooling, a hydrogenation of the "Abschlamm" oil and occasions a reduction in the consumption of used outside oil in the coal stalls because they operate with a better yield. The degree of this hydrogenation might, however, be increased if it were to take place at full convertor temperature. This thought led to the designing of a separator or catch-pot that operated as a counter-current convertor-oven having means whereby it was possible to heat the large quantities of inflowing cold hydrogen by the outflowing Abschlamm while simultaneously maintaining the desired temperature in the catch-pot. In place of the shorter catch-pot there was employed a large size convertor-oven in order to increase the reaction space. The apparatus for heat-exchange between the hot Abschlamm and inflowing gas comprised a series of baffle-plates having toothed--aprons whereby repeated horizontal flow between gas and liquid were effected. It is important that the return of previously cooled liquid production to the top of the vessel is avoided. (See Figure on page 86 of Exhibit J). The abschlamm can be so well cooled that additional apparatus for the purpose can be extensively or completely eliminated. The temperature in the upper part of the separator is controlled by inflowing cold gas. Sufficient fresh gas must be added to induce proper mixing of the contents of the catch-pot. Almost all the fresh gas requirement in a stall, that is about 15,000 M^3h, are added. The withdrawn Abschamm is thereby cooled to 150-200°C. This type of catch-pot was built into the Wesseling apparatus but was never operated; a similar device packed with rings instead of baffle-plates gave the following results with the incomplete heat exchange and with only 4000 M^3 of fresh gas:

Temperature of the Hot Separator	Solids in Abschamm	Asphalt In Oil
21 mV	19.4%	26.7%
24.5 mV	20.4%	20.6%

Abrasion in the Pre-heater Pipes

At the beginning of 1943, eroded spots of 6 mm deep in the return elbows of the preheaters were determined. Since in earlier operations with lower throughput, no abrasions were observed and in the interim the throughput of the chambers had been greatly increased, the abrasion was attributed to the increased velocity. It was assumed for the following calculation that mass and velocity nearly approached mV^2.

mV^2 in the preheater at different rates of injection of coal paste and quantities of gas is (at 420° preheater temperatures, 650 atm. pressure and 0.380 density of the gas and a specific gravity of coal paste of 1,000 at 420°C).

Gas M^3/hr	Coal Paste M^3/h	V = M/sec.	Masse Coal Paste & Inlet Gas	mV^2
27,000	35,000	6.4	12.6	516
27,000	40,000	6.6	14.0	610
27,000	45,000	6.8	15.4	712
13,000	40,000	4.1	12.5	210
13,000	45,000	4.3	13.9	257
13,000	50,000	4.5	15.3	310
10,000	40,000	3.6	12.1	157
10,000	45,000	3.8	13.5	195
10,000	50,000	4.0	14.9	139

These calculations show that it must be possible by reduction of the inlet quantity of gas to increase the former peak throughput of coal paste. In consequence, the paste throughput was maintained and of the 27,000 n M^3/h of inlet gas, 12,000 n M^3/h were led directly to Convertor-Oven I, thus bypassing the hair-pins charged with paste. By this means the erosion was stopped.

Regeneration

The operative periods of the Wesseling regenerators and especially those charged with coal paste were unsatisfactory. The reason therefor is the deposition of certain heavy solid constituents of the coal paste on the outer flow-path of the tube-banks; there arises, however, a certain amount of soiling and stoppage on the inner flow-path of tube-banks. Analyses show these deposits from the coal-paste stem primarily from the catalyst, whereas the soiling inside the pipes must come from H_2S conversion and in part to excessive erosion by the returned Abschlamm. To remedy this situation--the original flow of coal paste through both regenerators was abandoned, the colder regenerators were employed for gas and the hotter ones for coal paste. In addition, in order to make deposition more difficult by increased velocity of flow, the regenerators were provided with tube-banks having a smaller number of pipes; also, the direction of flow of the mixed gas and coal-paste was changed from upwards to downwards.

Average operating periods of the regenerator-banks at different schemes of flow:

	Total Days	Days with a K-value over 100
Charging of both regenerators with gas+paste mixture in downward flow	55	41
Charging only the hot regenerators with paste		
Reg. Banks with 199 pipes) in	95	70
" " " 150 ")upward	95	67
" " " 108 ") flow	-	61
Reg. Tube Bank with 199 pipes and Downward Flow	104	81

The separation of the two regenerators into a gas and a mixture-regenerator brought a decided improvement in operation, their reduction in size was disadvantageous. The change in flow-direction provided further improvement. Even these advances were

not satisfactory and it was attempted to solve the problem by altering the existing regenerator construction by designing a regenerator with both gas and coal paste upflow.

A Regenerator with Downward and Upward Flows Respectively of Coal-Paste and Gas

This method of operation is distinguished from prior practice in that the regenerators are filled with liquid during the flow of gas upwards, whereas by the downward flow of the gas they are empty. During downward flow of liquid, the heavier solids therein must be carried along to the bottom . By keeping the regenerator filled with liquid, the formation of zones-of-quiet in the flow-path should be avoided by reason of the agitation of the upwardly-flowing gas-the presence of H_2 is for chemical reasons also indispensible-and also any drying out of the coal paste is avoided. (See schematic diagram of regenerator on page 90 of Appendix J.)

A regenerator of this type was in operation 12 days until the plant was shut down due to the bombing attack on July 19, 1944. Any positive conclusion as to results after such a short period of operation is, of course, not justified. It can, however, be stated that there was no reduction in regenerator efficiency and that the heating-gas consumption of the associated preheaters was 12.8 million W.E./h and did not vary over the said 12 operating days and lay, at this value, greatly below the best operating previously obtained.

Protection of the tubes in the inner flow-path of the regenerators by means of an "after separator" following the hot catch-pot, is under consideration. By conversion of coal convertor No. 4 of a stall to the above described fresh gas - counter current method of operation, the hot catch-pot for the hydrogenation convertor-ovens becomes free for the purpose. Actual operation of such a converted "after separator" was made impossible by the bombing attack of July 19, 1944.

Cleansing of the Regenerator Tubes by Flushing

The cleansing of dirty regenerator tubes by flushing with an appropriate solvent is also worthy of consideration. This has been attempted in many hydrogenation plants, without satisfactory results. However, it was felt that the expensive dismantling of the tube-banks for their cleansing was unjustified, and therefore a flushing process was again considered. In order to increase the efficacy of such a procedure, CO_2 at an extremely high velocity and in counter current flow to the cleansing liquid was introduced as an agitating medium. As the solvent, pasting oil was employed. This was changed several times during a period of several days extent. In two tests, over 600 kg of solids were removed from the regenerator tubes in this manner; this is as much solids as can be removed by complete dismantling. Resistance tests showed them to be as clean as new tube-banks. Cleaning of the inside of the regenerator tubes, in the event that a steam blast is ineffective, can be achieved by cutting off the conical tube-sheet which is an operation that is relatively minor in contrast to a complete dismantling. It can be assumed that flushing with an oil-gas mixture can save much repair of the regenerators.

Machines, etc.

Despite war-time conditions, all the stuffing-boxes of all machines were operatively sufficiently satisfactory and that there was no loss in operation attributable to them.

700 atm. Circulating Pumps

The circulation pumps were built as plunger type machines and had a diameter of 170 mm. They were converted to those of the piston-type. The diameter of their cylinders was 200 mm - that of the connecting-rod 100 mm. The capacity of the machine was thus increased from 52,000 N M^3/h to 62,000 N M^3. The average operating period of a bushing was thus increased from 60 to 200 days and the oil consumption was reduced from 80 liter/machine/hour to about 30 liters.

The difficulties occasioned in early operations by entrained foam from hot catch-pot product ceased by increasing the temperatures in the let-down tanks to 120°C and installation of the oil-scrubbing systems for their outlet gases.

The 325 atm. Circulating Pumps

In contrast to the 700 atm. pumps which have 3 bearings, the 25 atm. pumps have only two bearings and are of light construction. It was necessary frequently to repair the main and the crank-bearings. The average operating time of the bushings of these machines was 160 days.

Pumps for the Injection of Oil

The pumps for the injection of middle oil at 325 atm. are provided with Gotze-Metal packing; it contained 10 to 12% of tin. At first great difficulties were experienced with the stuffing boxes. These were eliminated to about the normal number by the following measure: Careful installation and regular inspection by specially trained workmen; dropwise lubrication of the plunger by let-down tank residues to which some middle oil had been added, instead of the provided cooling by water-jet which gave rise to rusting of the plunger; and spraying of Diesel oil into the stuffing boxes.

The auxiliary 700 atm. pumps for the recirculated oil of the oil-scrubbing plant operated more satisfactorily than had been anticipated.

The Expansion Machines

The expansion machines suffered some erosion in consequence of solids that reached the wash-oil by foaming-over of the catch-pot products. Resolution of this difficulty in addition to frequent change and filtration of the wash-oil led rapidly to normal operating conditions. The control valve whereby the cylinder is converted from its working stroke to its exhausting stroke caused considerable difficulty. This control valve was compensated by tapping of the control cylinder and since then the expansion machines have operated without difficulty.

Construction, Operating Control, Measures for Avoiding Interruptions of Operations

The 700 atm. "Sumpf" phase went into operation without any untoward incident. Only isolated instances arose later to give difficulties. The operation was confined to skilled hands. Operations were good despite the fact that the workmen represented a motley crew. It is significant that there was not a single break in the coal chambers due to the development of excessive temperatures. Only once did a leak occur in the lens gasket of a line connecting the first and second ovens. A fire resulted with only small damage. On the basis of experience in other hydrogenation plants where large jet-like flames were projected from the stalls, the brick-work of their walls was extended above the top of the ovens and the windows of the paste house opposite were bricked up. Likewise, for reasons of fire protection the windows of the staff-operating floors were walled up as well as the windows above the circulating pump house. As a further precaution, particularly dangerous expansion lines were led over the roof. Particular mention should be made of the fact that all the controls of the quick-closing valves of the high-pressure system system and the switches for the coal paste pumps were installed at a single place so that the whole high-pressure apparatus could be shut down in a few seconds. This precaution was an aid during air raids and also of value for normal operation.

Aside from single exceptions, oil production depended upon the hydrogen production. Up to the solution of the above-mentioned caviar problem (October 1942) it was only possible by special effort of the technical department to keep pace with the gasification plant. The average stall-operating period was at that time only 42 days. This was increased to 66 days in 1943 and to 84 days in 1944.

The relative short operating periods are related to war-conditions. A complete failure of electric power resulted in a complete coking-up of all the stalls in operation. The many changes made in pasting oils due to bombing attacks and the attendant unavoidable drops in temperature in the ovens, led often to deposits in the de-sanding apparatus which became stopped with salt-crusts.

Under normal conditions, the average operating period of a stall was much over 100 days.

The removal of several stalls simultaneously from operations required for use of the hydrogen-producing capacity of the plant temporarily a charging of the stalls at an especially high rate - up to 50 M^3/h. This high charging rate was later abandoned because of erosion in the preheating pipes and the poor conversion of asphalt. However, after these erosion difficulties were solved by diverting a part of the gas, introduced into the ovens, around the preheater equipment for the coal-paste, it seemed feasible from experience obtained in the two rather short de-sanding ovens, actually to employ a charging rate of 50 M^3/h. In these two chambers that were charged with cold gas, the reaction with adequate conversion of asphalt was practically ended after the first two ovens. This result warranted the assumption that it might be possible, in conjunction with counter-flow hot catch-pot tanks, to achieve the same yields with ovens greatly reduced in size.

On page 97 of Appendix A, there is shown two flow sheets using the above suggestion.

In the first diagram, there is shown a stall comprising 3 units. In the first convertor oven the main body of the coal paste flows downwardly and a small quantity thereof is flowed upwardly in order to keep the oven filled. Recirculated hydrogen gas is introduced at the bottom of the first oven. In the second oven the mixed gas and coal-paste flow upwardly. The third unit is a counter-flow hot catch-pot tank which must operate especially satisfactorily because fresh hydrogen is introduced thereunto.

The second diagram shows a purely counter-current flow-sheet for the above.

The Most Important Operating Results for 1943

Degree of Efficiency of "Sumpf" chambers	84.4%
Actual Operating hours of paste pressure	75.3%
Possible " " " Recirculation Pumps	81.9%
Number of Operating Chambers (average)	3.0
Average reaction volume (sum of all chambers)	98.1 M^3
" Paste injection/chamber	37.5 M^3/h
Coal paste - throughput	1.430 T/M^3 RV/h
Middle oil formation (new)	0.265 "
Conversion of Coal (Abbau)	99.2%
Fresh Gas Consumption based on:	
New Middle Oil Products	2038.5 N M^3/T
Coal Paste	373.6 "
Circulated Gas (Oven Inlet)	575.0 "
H_2 in Circulation Gas (Oven Inlet)	71.9%
Pressure	625 atm.
Solids in Coal Paste	41.0%
Ash in Solids	13.3%
Pure Coal in Coal Paste	35.6%
Iron Ore based on TBKo/water free	5.76%
Sulphur " " " "	0.12%
Screen Residue on 400 mesh screen	10.6%
" " " Vibratory "	0.6 kg/t

Composition of Pasting Oils

Coal catch-pot residue	34.2%
Oil from centrifuges	56.7%
Oil from outside production	9.1%
Fraction to 340°C	10.6%
Solids	4.0%
Asphalt	8.85%
Let-down (based on coal paste and flushing oil)	51.0%
Abschlamm	33.8%
Middle oil to 340° in let-down	38.8%
" " " " in Abschlamm	11.2%
Solids in Abschlamm	18.5%
Asphalt " "	15.6%

Dissolved Gas

in let-down (lean)	74.9 M^3/M^3
" " " (rich)	14.2 "
In Abschlamm	122.5 "
In Wash Oil (lean)	151.3 "
In Wash oil (rich)	46.9 "

Intermediate Expansion Pressure

Let-down (Abstreifer)	38 ats
Wash oil	35

Quantities per hour

Coal paste	112.6 M^3/h
Cold oil and flushing oil	8.8 "
Water injection	6.4 "
Catch-pot product	65.5 "
Abschlamm "	41.0 "
Middle oil (new formation)	25.4 t/h
Fresh gas consumption	51,583 N M^3/h
Catch-pot gas	
Lean	4,902 N M^3/h
Rich	929 "
Scrubber gas	
Lean	7,851 "
Rich	2,274 "
Abschlamm gas	5,027 N M^3/hr
Wash oil	50.1 M^3/h

HIGH PRESSURE, 325 ATM. - GAS PHASE

In the gas phase, middle oil from the "Sumpf" phase is further converted by hydrogenation. The gas phase comprises two steps, the pre-hydrogenation (Vorhydrierung) and the benzine (Benzinierung) steps which are operated with different catalysts. The former has for its purpose, by simultaneous cracking and hydrogenation of the middle oil, a refining of the product for the benzine step. In the pre-hydrogenation step a contact catalyst is employed that effects along with a good cracking and low hydrogenation both cyclization and isomerization.

In the Wesseling plant, there was employed for this pre-hydrogenation step the contact catalysts 5058 and 7846 W, and for the benzine step catalyst 6434. The pre-hydrogenation chambers were at first constructed with 2 convertor-ovens filled with catalyst 5058; this number was later increased to 3 ovens, of which the first was filled with catalyst 5058 and the other two with 7846 W.

The benzine convertor apparatus comprised 5 ovens in series and were filled with 6434 catalyst. The apparatus and operation of the benzine convertor were quite similar to those of the coal convertor-ovens and are visible in the diagram shown on page 100 of Appendix J.

The means taken to protect the catalyst 6434 for the benzine step should be emphasized. This catalyst is sensitive to basic nitrogen compounds and to oxygen in free and combined forms. Therefore, the inlet product has to be well refined in the pre-hydrogenation step. Its content of phenols must not exceed 0.3% and 5 mg/liter in basic nitrogen. The inlet gas to the 6434 chambers is scrubbed with water to an NH_3 content of not more than 0.02 to 0.03 g/Nm^3. Since the contact catalyst must be protected against any removal of its content of sulphur, the inlet product has to be sulphurized with H_2S. Its content of H_2S amounts to 0.4 to 0.5%.

As a measure for the correct degree of refining and benzination in the so-called Bi-Convertors, the difference in specific gravity between the ovens' inlet and outlet products serves in general. In the case of the pre-hydrogenation ovens, this difference must lie between 0.100 and 0.110; in the case of the 6434 chambers, it must be about 0.075. The catalyst temperatures are adjusted to give these results. In the pre-hydrogenation in the 5058 ovens, they are equal to about 18.5 to 21 M V, and in the 7846 ovens, about 1 m V more; in the 6434 ovens the temperatures are equal to about 19 to 21.0 mV. Increasing the temperatures within the stated range is required by the slow decrease in activity of the catalyst and by the increase of the refining requirements. The slow reduction in activity of the pre-hydrogenation catalyst extends over a period of over three years and in the case of the 6434 chambers, over a 1½-year period; this is the normal life of the catalyst.

The A-middle oil introduced into the gas phase at Wesseling originates to about 75% in the brown coal and to about 25% in outside oil and fluctuates with the type of outside oil received for use. This A-middle oil usually contains a large amount of phenols -- even up to 20% but normally 15%. The high phenolic content obtaining in the starting-up period shows itself in a high heat of reaction in the ovens and permits only an average throughput of material to be treated in the 5058 chambers. Later, because the phenol content became smaller and the catalyst had lost its initial activity, the throughput could be increased.

In the early part of 1943, a 6434 chamber was put into operation for the manufacture of aviation gasoline. This required a higher purity in the 5058 "catch-pot" product. In order to meet this requirement, the amount of material treated in the pre-hydrogenation chambers had to be decreased. An average throughput through these ovens was about 0.6 kg. inlet material per liter of catalyst space and the highest was 1.0 kg/liter. The throughput for the 6434 chambers was higher and was on the average 0.75 to 0.80 kg/liter – the highest throughput was above 1.1 kg/per liter of catalyst space.

MOST IMPORTANT OPERATING RESULTS FOR 1943

Efficiency	Benzine Convertor Ovens	75.2
	Middle-Oil Injection P.	51.8
Actual Operating House) Possible " ")	Circulation Pump	53.2
Number of Operated Ovens (Average)	5058	2.08
	6434	0.08
Average Reaction Volume	5058	37.4 m^3
	6434	12.4 "
Average Injection	5058	0.824 t/M^3RV/h
	6434	1 120 "
Middle-Throughput	5058	0.764 "
	6434	1.070 "
Benzine Produced - (-180°)	5058	0.280 "
	6434	1.070 "
Fresh Gas/Injection	5058	432.0 Nm^3/t
	6434	255.0 "
Cold Gas/Injection	5058	700 Nm^3/t
	6434	262.5 "
Circulation Gas - (Ka-Inlet)	5058	4325 "
Injected	6434	1610 "
H_2 in Circulated Gas (Oven Inlet)	5058	4325 Nm^3/t
Pressure		306.5 atm.
Bi-180° in A-Middle Oil		22.8%
Phenols in A-Middle Oil		15.4%
H_2S in Injected Product	5058	-%
	6434	0.5%

MOST IMPORTANT OPERATING RESULTS FOR 1943 (cont'd)

NH_3 and organic bases in injected prod. 6434 - 50 mg/l

Benzine - 180° in catch-pot	5058	- 38.2%
	6434	- 68.0%

Dissolved Gas in catch-pot

5058	lean	24.9 M^3/M^3
	rich	11.5 "
6434	lean	58.2 "
	rich	29.3 "

Intermediate expansion pressure - 5058 - 28 atm
6434 - "

Hourly quantity	(5058	29.9 M^3/h
	(6434	12.1 "

Water Injection 5058 and 6434 6.85 M^3/h

Fresh Gas	5058	12,340 N M^3/h
	6434	3,670 "

Catch-pot gas	lean	5058	818 N M^3h
	rich		377 "
	lean	6434	1660 "
			750 "

CHARACTERISTICS OF THE INJECTION & CATCH-POT PRODUCTION FOR 1943

Pre-hydrogenation

A-Middle Oil

Specific Gravity	0.927/20°C	Aromatics	62.5%
API (phenol free prod.)	4.0	Olefins	17.2%
Phenol content	15.4%	Naphthene	9.2%
At 180°C	20.5%	Paraffin	11.1%
End Point	348°C		

5058 Catch-Pot

Specific Gravity	0.825/20°	Aromatics	15.0%
A.P.I.	52	Olefins	2%
Phenol Content	0.03%	Naphthens	29.4%
Basic Nitrogen in B-Middle Oil	5 mg/liter		
Benzine fraction to 180°C.			38%

Characteristics of Benzine to 180°C.

Specific Gravity	0.765/20°C	Aromatics	8.4%
A.P.I.	43.4	Olefins	1.9%
Fraction-100°C.	22%	Naphthenes	30.4%
Octane No. (Motor)	60.5	Paraffins	59.3%

Samples taken occasionally after the 5058 ovens had the following composition:

Sp. gr. at 20°C	0.895
A.P.I.	20.0
Phenol Content	6%
Fraction to - 180°C.	25%

Benzination Process

6434 Inlet Product	
Pre-hydrogenation middle oil	- 56%
6434 Return Oil	- 44%
Sp. Gr. at 20°C.	- 0.824
A.P.I.	- 54
Basic Nitrogen	- 5 mg/liter
End point	- 328°C.

6434 "Catch-Pot"

Sp. gr.	0.768/20°C.	Aromatics	7.2%
A.P.I.	54	Olefins	2.6%
Benzine to 150°C	49.0%	Naphthenes	58.0%
" " 180°C	68.0%	Paraffins	32.2%

Characteristics of Benzine to 150°C

Sp. gr.	-0.724/20°C	Aromatics	6.1%
A.P.I.	-53.5	Olefins	1.9%
to 100°C	-49%	Naphthenes	43.5%
Octane No. (Motor)	73.3	Paraffins	48.5%

PLANT FOR GRINDING THE COAL

Description and Operation

The dried coal and the catalyst are delivered in large enclosed cars. By means of compressed air, these materials are delivered into storage bunkers. Iron grids 50 x 50 mm are provided at the inlet to remove foreign objects from both the coal and the catalyst. The transporting apparatus comprises horizontal and vertical scraper conveyors. There are available two delivery routes for the coal and one for the catalyst. For the vertical transport of the catalyst, the Redler conveyor apparatus has to be substituted by a bucket elevator since the catalyst has a tendency to pack and to stop a vertically-disposed Redler. The relatively long distances over which the coal and the catalyst must pass requires breaks in the transport route. In total there are 6 Redlers disposed in series. In order to avoid stoppages and interruptions of these Redler apparatus, in consequence of, for example, diversion of material to incorrect apparatus or of their operative failure, each transport route is equipped with automatic electrical connections for the power motors.

All apparatus of the grinding plant are blanketed with carbon dioxide and its composition is analytically controlled.

The coal is at first subjected to a dry screening over a vibratory screen. The over and the through products of the screen each amount to about 50% of the screened coal. The coal that passes through the screen goes direct to the bunkers intermediate the same and the coal-pasting mill, whereas that portion that passes over the screen is first passed through a rotary grinder. The bunkers outlets are also provided with a bucket scraper to prevent spurting of the coal therefrom. An important feature of the apparatus of the grinding plant is the rotary coal-pasting mill with apportioning devices for the coal, catalyst, and pasting oil. The most important parts of the mill are a screw-conveyor for introducing the coal and catalyst, and a secondary and a principal chamber which is divided into 5 segments. The last two members are separated by a slit-wall. The secondary chamber contains as the grinding bodies steel balls having a diameter of 50 to 88 mm; the principal chamber is equipped with cylindrical pieces of iron that are about 25 mm long and 20 mm in diameter. The mill is rotated about 21 times per minute.

The apportionating apparatus comprises a belt-conveyor arranged for automatically weighing the coal and a bucket scraper for the catalyst. The pasting oil is delivered by means of an air-actuated automatic valve controlled by volumetric device that supplies the impulse.

In the mill, coal, catalyst, sulphur and pasting oil are ground together to form the coal-paste. The coal-paste passes to an intermediate storage and from there is pumped to the high pressure apparatus by means of piston-pumps where it is first flowed over a vibratory screen before being delivered to the 700 atm. pumps.

Operative Development, Difficulties and Their Correction

For smooth operation of the "Sumpf" phase the production of an appropriate coal-paste is the first consideration; above all sudden fluctuations in its composition must be avoided.

The coal-paste prepared from the Rheinish brown coal has an abnormally high viscosity. This peculiarity is attributable to a special swelling characteristic of the coal; in addition, the high moisture content of the dried coal plays a part as well as also the viscosity of the fractionated heavy oil which has a first drop of 350°C. It is not possible to increase the concentration of solids in the paste beyond 42%. Exceeding this value causes the paste to become unpumpable and it over-flows on the high pressure paste screens. For manufacture of a proper composition of coal paste, there is necessary: Correct apportioning of the component constituents and especially the catalyst; low moisture content of the coal and the catalyst; constant composition of the pasting oil; maintaining the solids content low. The temperature of the pasting oil must be held as high as possible (125°-130°C) in order to evaporate the water that is contained in the coal and the catalyst, and to keep low the viscosity of the paste.

Analysis of the Coal Paste (Average 1943)

Specific Gravity at 110°C.	1.175	
Water, % by WT	1.9	
Solids "	41.0	
Pure Coal	35.6	% by WT
Ash in Solids	13.3	"
Fe in Solids	3.0	"
Total Sulphur based on solids	1.6	"
Screen residue of solids on 0.3 mm Screen	10.6	"

The Coal

In general, the coal as supplied from the mines was of consistent composition and good quality. Mechanical difficulties arose if the screening apparatus of the drying plant were by-passed. The water content on the average was 7.5% with a maximum of 12%. The average value was about 3.5% above the anticipated. Despite the fact that water is undesirable in the coal because it gives rise to a difficulty with the viscosity of the coal paste and increases the heating-gas consumption in the high pressure, this high water content was maintained to reduce the danger of fires and explosions.

The dry coal had a tendency to spurt or shoot out of the storage bunkers when larger quantities of fine coal were discharged. An effective counter measure was the avoidance of any working of the coal in the scraper-conveyor so as to eliminate as much comminution thereof as possible; also, once or twice emptying each week of the storage bunkers to avoid the accumulation therein of large quantities of fine coal. The bucket wheel disposed beneath the intermediate storage bunker proved of great value in this connection.

The dust of the dry coal is both unpleasant and dangerous. In the drying plant at the mine there was a severe explosion, and also in our grinding plant a series of fires and minor puffs occurred. The interior of the apparatus was finally so well protected with CO_2 flushing that no more such difficulties arose. Considerably more trouble was caused by the dust outside the apparatus. Along with a complete sealing of the apparatus, the removal of the pressure equalizer and the de-airing lines and above all the construction of a hopper-like restriction in the discharge throat of the weighing apparatus brought a great improvement in the mill operations.

Attempts to moisten our coal with pasting oil in accordance with Leuna procedure for fighting dust, had successful results. It would have been difficult to construct such equipment at Wesseling because of lack of space.

<u>The Catalyst</u>

The employed catalyst was the so-called "Bayer Schlamm" which is the extraction residue of Bauxite treatment to remove alumina; it is pre-dried and of a grain size up to 30 m.m. and delivered by Gerdemann in Quadrat-Ichendorf and the Chem. Fabrik Hoesch, Duren. The Fe_2O_3 content of the Gerdemann product was always over 50%, the product of the Firma Hoesch about 5% less. The iron ore also contains some $CaCO_3$, alkali, (Al_2O_3) and silicate. The H_2O content of the catalyst is on the average 6 to 8% and is occasionally as high as 12%.

The high water content of especially the Gerdemann product, after construction of an exhauster for the vapour of the ore-drying plant, was avoided. The catalyst inclined to cake formation in high storage-bunkers and in result they had to be completely emptied about every three months.

<u>The Sulphur</u>

The sulphur was formerly weighed and added by hand. War conditions prevented the installation of automatic apportioning apparatus. In order to reduce the cost of sulphur and to simplify the addition of large quantities of materials through the apportioning apparatus, (when employing pure sulphur, only 100 kg/h had to be added) the use of sulphur-containing gas purification oxide was considered. The existing equipment was tested for a couple of days with such an oxide containing 40% sulphur and the apportioning apparatus operates successfully.

<u>The Pasting Oil</u>

The pasting oil is a mixture of heavy oil, from the coal catch-pot separators, of oil from the centrifuges, heavy oil from production and foreign oils. The mixture is prepared in a heavy oil plant with the assistance of a recording Eckhardt measuring device.

Composition of the pasting oil: (average 1943)

Heavy oil from coal catch-pot	34.2%
Oil from centrifuges and heavy oil	56.7%
Foreign oil	9.1%

The most important analytical data: (average for 1943)

Specific gravity at 110°C.	1,062
Solids	4.0% by wt.
Ash in solids	50.7% by wt.
Asphalt in oil	8.8% "
Frantion to - 350°C.	10.6% "

Of the above values the most important is the solids content of the oil because with constant additions of coal, the solids content of the coal paste will vary therewith.

The oil fraction obtained from the centrifuges is the carrier of the solids. It is thus of particular importance that the fraction and its proportionate addition be kept constant.

Fineness of the Coal

Screen residue on screening machine, mm	3.0	2.0	1.0	1.5	0.3	0.1	0.6
Dried Brown Coal as delivered	10.0	24.0	46.0	60.0	71.0		
Over 48%	21.0	49.5	86.1	98.0	100.0		
Through 52%	-	0.3	9.0	25.0	43.5		
Outlet to Rolls	0.8	1.8	25.2	45.7	64.8		
Inlet to Mill	0.4	1.0	16.8	34.9	53.7		
Outlet to Mill (coal paste)	-	-	-	3.5	13.0	16.7	36.0

Capacity of the Pasting Mill

The maximum attained capacity of a mill was 25 tons/hour of dried brown coal in a 41% coal paste. The bottle neck was the penetrability of the slit-wall between the secondary and primary chambers of the mill. Also, the weighing conveyor has scarcely a capacity of 25 tons/hour. The fineness of the so produced paste is not markedly different from that prepared from coal at the rate of 20 tons hour. In order to improve the fineness of the coal paste the number of grinding bodies in the chamber was increased. In the secondary chamber the weight of iron spheres was increased from 9 to 12 tons and in the primary chamber the weight of grinding bodies was increased from 55 to 60 tons. The loss in weight of the spheres was about 4% per month and about 1% per month for those in the primary chamber. The erosion of the walls, etc., which were formed of harder material than the grinding apparatus, is small and was only inconsiderable after 3 years' operation.

Most Important Operating Results

Efficiency of the mills	60.0%
Number of Operated mills	2.445
Average capacity of all mills in dried brown coal	54.0 tons/hr
Mill operating hours	21,416.0 h/month

Coal Paste

On 0.3 mm Screen	10.6%
Solids	41 %
Viscosity (500 CM^3/100°/5 MM opening)	48 - 60 sec.
Ash in solids	13.4%
Pure coal in coal paste	32.4%

In the Pasting Oil

Coal catch-pot residue	34.2%
Oil from centrifuge and low temperature distillation	56.7%
Foreign oil	9.1%
Fraction to 340°C	10.5%
Solids	4.05%
Asphalt	8.9%
H_2O in dried brown coal	7.86%
Ash " " " "	5.83%
(Roterz) Iron Oxide based on dried brown coal	5.88%
Fe in Roterz	35.5%
H_2O in "	9.3%
Sulphur based on dried brown coal	1.19%

Quantities Per Hour

Dried brown coal	48.98 t/h
Roterz (iron oxide)	2.88 "
Sulphur	0.58 "
Pasture oil	79.67 "
Coal paste production	130.57 "

CENTRIFUGING OPERATIONS

Description of Apparatus and Process

The suspension of solids emerging from the "coal conversion" chambers is separated into a high-solid-content centrifuge residue, and a low solid content centrifuge overhead oil. Prior to centrifuging, two small but important steps in handling the oil were carried out. First, the solid suspension oil was diluted to a constant solid content by overhead distillate from previous batches of oil. Second, a de-sanding process was used whereby the oil was forced upward in a conical vessel through an 8 millimetre sieve and the larger sandy constituents were drawn off at the bottom by means of a spiral conveyor.

The product so prepared was pumped to Laval centrifuges through a closed circulation line which kept solids in suspension while they were fed regularly to the centrifuges.

Laval centrifuges were used at 3200 revolutions per minute. The basket diameter of 430 millimetres produce a centrifugal force of 2500 kilograms. The centrifuges were fitted with the usual series of conical plates found in this type of machine, and the solids were removed from the walls by means of a perforated basket device revolving at differential of 15 revolutions per minute from that of the conical plates. Centrifuges were charged through the hollow spindle of the machine; separation occurred in the small distance between the conical plates and the oil rose and flowed through holes near the periphery of the spindle at the top. The solid-containing residue passed through holes in the basket and through an outlet. The basket which cut the solid residue from the walls naturally was subject to a load and to prevent same from becoming excessive and injuring the delicate differential drive, sheer bolts were employed. Later, in the course of the experiments with the centrifuges, an arrangement was made whereby feed to the machines was cut off when a certain previously arranged resistance was encountered by the basket in removing the solids from machine. This prevented sheering of the above mentioned safety bolts and effect the desired protection of the machines.

Experience showed that to effect the smooth operation of the centrifuges, the following were necessarily held constant:

1) Pressure in the circulating feed line to the centrifuges
2) Content of solids in the feed mixture
3) Temperature of the centrifuge feed mixture (140°C.)

It was, of course, necessary to choose valve and orifice diameters for the two centrifuge products, such that the highest possible solid content of the residue and the lowest possible content of the "overhead" oil was obtained. In operation, by variation of feed dilution, a solid content of 16% was maintained in the centrifuge feed though the material coming from the coal chambers varied between an 18 and 22% solids. Obviously the loading of the centrifuges could be changed and controlled by the pressure in the circulating feed system. One atmosphere gauge pressure was normally held, though in special cases this was raised to 1.2 atmospheres. Change in the throughput for a single machine was effected by changing the orifice in the feed line. A diameter of 1.8 to 2.2 millimetres was used for the residue orifices. Under these conditions the following operating figures were obtained:

Production per centrifuge 3.2 T/Hr = 100%
Oil "overhead" 2.2 T/Hr = 69%
Residue 1 T/Hr = 31%
Solids in oil overhead = 6%
Solids in residue = 39%

It was found that if one increased the throughput without changing the orifices, the solid content of both oil and residue increased, while the ratio of oil to residue increased. On the other hand, with a constant feed, if the residue orifice was increased in diameter, the solid content of both residue and oil decreased.

The Solids in the Material led to the centrifuge contain 90-92% ash, including ash from the coal and the added (spent) catalyst. The ash content of the solids in the overhead oil amounted to about 75%; that in the residue from the centrifuges 95%. A certain amount of enrichment of ash content in the residue from the centrifuge therefore took place. The asphalt content of the oil led to the centrifuges divided itself equally between the overhead and residue product and amounted to about 15%. The sand content in the solids, led to the centrifuge, amounted to about 5%; the solids taken off in the de-sanding apparatus about 12%. The residue from the de-sanding operations was combined with the residue from the centrifugal operations.

Difficulties in Operation

At first an attempt was made to operate without the de-sanding operation, but overloading of the centrifuges and the fouling of the orifices made this step imperative. It was found that if the solid content in the residue from the centrifuge operation rose above 42%, the centrifuges were overloaded.

When the coal chambers were originally put in operation or after a shutdown, the preparation of the oil for the centrifuges became especially difficult. Often orifice clogging occurred, which, however, was obviated when the de-sanding apparatus was put into operation. Furthermore, in starting up after plant was "run-in", the residue from the chambers could only be centrifuged with difficulty. Settings for normal input material were such that this first product from the chambers would cause centrifuges to fill up with solids, a circumstance which was avoided by liberal admixture of normal product during the starting up period. Simplex pumps were used exclusively to pump the product. The centrifugal pumps which were originally foreseen for pumping of the overhead product oil were replaced by Simplex pumps because no satisfactory packing for the centrifugal pumps was found. The pneumatically controlled pressure control valve for the feed circulated system was found unsatisfactory and was replaced by a hand controlled valve. Especial difficulties were encountered in measuring the pressures and in recording same, since the lines to these instruments tended to clog. This circumstance was partly avoided by filling these instrument lines with oil and employing carbon dioxide from cylinders as an intermediate or buffer medium.

Important Operation Statistics

Time utilization of centrifuges	53%
Centrifugal hours	166906 Hr/Mo
In Feed Mixture:	
Chamber product	88.51%
Dilution oil	11.49%
Solids in -	
Product from chambers	18.5%
Centrifuge input oil	16.4%
Centrifuge overhead	6.8%
Centrifuge residue	38.9%

RECOVERY OF OIL FROM CENTRIFUGE RESIDUE

Description of Apparatus

Centrifuge residue was de-oiled by coking in the presence of steam into two overhead fractions, Heavy Oil 1, and lower boiling overhead called Heavy Oil 2. Cracked gaseous constituents taken overhead in the fractionation were used as heating gas for the coking ovens themselves. Residue from the operation itself, which contained 65% solid ash and 35% of coked residue oil, was thrown away.

The recovery equipment consisted of six rotating ovens or kilns and six stationary "I.G." ovens fitted with spiral conveyors. The rotating ovens or kilns were 2.2 m. diameter and 11 m. long, and were slightly inclined for solid delivery. The drum was rotated at 9 RPM and heated with twelve gas burners from the outside. A second series of gas burners were used for the steam superheater and the product preheater which were arranged above the kiln. About three-quarters of the kiln length was taken up for the coking operation itself, and this part was filled with nine tons of grinding balls each weighing 1.8 kilograms. In the remaining one-quarter length of the drum, 24 grinding balls, each of 10 kilograms, were employed.

The kiln was fed continuously with preheated feed through a feed tube. The balls in the early part of the kiln prevented formation of coke on the hot walls and improved heat exchange at the same time. The coke product moved slowly through the last part of the oven to the delivery chamber and was dumped into a water seal. The quenched coke was carried by a continuous band conveyor out of the water seal and to the railroad cars for disposal.

Through the hollow shaft of the spiral conveyor steam was led into the interior of the kiln. Vapour delivery from the coked material was effected at the entering end of the kiln. Condensation of these vapours occurred in three coolers by direct liquid contact. The pre-cooler was showered with Heavy Oil 1, the product from itself, which was circulated through a tubular cooler and back to the pre-cooler. A part of this circulated oil was also used by injection into the vapour line between the dust catcher and the pre-cooler. In the secondary, or after cooler, Heavy Oil 2 was condensed, together with the steam blown into the kiln. Part of the water separated in the water separator was circulated oven an Imperial cooler to shower the secondary or after cooler. Both the pre-cooler and the after cooler were filled with Rashig rings. The residue gas after passing through a seal was released to atmosphere.

Spiral Conveyor Ovens

These ovens consisted of two stationary cylinders lying horizontally one above the other: length 16 m., diameter 1 m. The spiral conveyors in both cylinders turned at a rate of 1 RPM. The product was led into the upper horizontal cylinder through the hollow shaft and was moved forward, as it slowly coked, by the conveyor until it fell through conveying pipe into the lower horizontal section. At the discharge end of the lower section a vapour tight discharge pipe allowed the product to drop into a water seal. A continuous band conveyor lifted the quenched residue from the water seal and discharged it into railway cars for disposal.

Heating of the oven was effected by one burner at the underside of the bottom cylinder. Combustion gases from the combustion chamber could be led directly against the cylinders by means of a regulating valve or damper. In the second pass (around the upper chamber) a steam superheater was placed. The superheated steam produced was blasted into both the upper and bottom drum. The spiral conveyors in each drum carried, aside from the spiral plates, chains hung on rods which were supposed to prevent coke setting up on the plates. The oil gases left the oven through a riser pipe at the entrance end of the upper drum and were passed through a regenerator, thence to condensation in two steps, in a fore and after cooler. Both of these cooling stages were water cooled. A dephlegmator sat atop of the first packed tower giving direct contact cooling. The secondary cooler was a tubular one. Condensate out of the bottom of the first cooler and out of the dust catcher under the regenerator was combined and carried away as Heavy Oil 1. Heavy Oil 2 was taken from the secondary cooler to a water separator and the gases from the oil layer were released to the air after passing through a water seal. This gas could, of course, be used as a heating gas for the burners of the oven itself.

Method of Operation

The following factors were of importance in the operation of the ovens:

1. Amount of feed
2. Oven temperature
3. Amount and temperature of the steam blasted into the ovens.
4. Top temperature of the first and secondary coolers.
5. Pressure in the ovens.

Method of operation differed with the two types of ovens corresponding to their different construction. The coke product from the kilns dusted easily. To prevent carrying the dust into subsequent apparatus only about 100 kilograms of steam per ton of feed material could be used in the kilns as compared to 500 kilograms of steam per ton of feed in the spiral conveyor ovens. Consequently, in order to obtain the same coking effect, the temperature was necessarily held at about 590°C instead of the 560°C used with the spiral conveyors. The 35% organic matter in the residue from each of the two types of ovens was mostly coke in the case of the higher temperature kilns, but from the spiral conveyor ovens some 3/5 of the organic material was soluble in benzole.

Top temperatures of the pre and after coolers were respectively 220 to 240°C for the first, and 110 to 130°C for the second. These temperatures demanded close watching in order that a sharp separation of water from the Heavy Oil 2 (which approached Sp. Gr. = 1) could be made subsequently. The pressure in the coking ovens gave a measure of the degree of coking taking place. By normal operation this pressure in the kilns was 80-120 mm. of water column and 40-80-mm. water column in the spiral conveyor ovens.

Difficulties in Operation

1. Kilns. No difficulty was encountered in bringing the kilns into operation. However, at the time of air raid alarms the frequent off-and-on operation of the product preheaters caused coking difficulties. Accordingly the preheater was later cut out. For satisfactory operation under this condition a decrease in the through-

put of about 15% and an increase in the kiln temperature from 575° to 590°C was necessary. In the event a new preheater is built it is recommended that vertical tubes should be used in place of horizontal tubes and that a central burner be employed.

Steam superheating: The three parallel paths for steam without separate control of the amounts in each path led to rapid destruction of the Sichromal tubes in the original steam superheater. Much better operating time efficiencies were obtained after a single path superheater was built into the operation, even though common steel tubes were employed. As a result of the milling action of the balls in the ovens the coke residue was pastey, tended to "mud up", and had a small oil content. Because of these circumstances, transportation by means of a rubber conveyor belt failed and same was replaced by a water de-ashing device. The residue was brought out of the water seal with a cleat conveyor and delivery made over a grate. The grate served to take out the larger chunks which were led in the normal way to the original conveyor. The major part of the residue fell through the grate in fine form and was washed with much water into a collection container, from which it was pumped with a residue pump through a pipe to an ash settling tank. Loading of cars out of the settling tank was accomplished with a crane and bucket.

2. Spiral conveyor oven: The spiral-conveyor ovens were also put into operation without difficulty. Nevertheless difficulties were encountered with the heating of the oven chambers. In spite of the highest allowable temperature in the combustion chamber, the heating up of an oven required 48 hours instead of some 6 hours in the case of the kilns, simply because the heat available was insufficient. By masonry closure of the regulating canals between the combustion gas ducts, and by making the combustion chamber smaller, a small improvement was obtained. A hot gas circulation oven should be employed with the spiral ovens as in the case of the kilns,

Coking of the spiral conveyors: The spiral conveyor in the lower cylinder became filled with coke in from 17 to 20 days, that in the upper chamber after 100 to 120 days. An improvement in these time efficiencies would be obtained in the event of the introduction of a circulation fan on the combustion gases, and providing that the many operation interruptions due to war conditions could be avoided. On account of many air raid alarms and the frequent shutting down of the spiral conveyor ovens, the pre-heater tubes tended to stop up. Accordingly an experiment was made to operate one of the ovens likewise without product preheating. On doing this, heavy oil condensed in the uncooled regenerator and set up in the pipes leading away from same. After showering the regenerator with Heavy Oil 1 the operation proceeded satisfactorily without significant decrease in the throughput and with an increase in the heating gas requirement of about 10%.

On pages 123 and 124 of the report, Appendix J, a series of figures portraying the efficiency of the above coking operation are given. The reader is referred to same as they are not translated here.

PRODUCTION OF BRIQUETTE PITCH

The great need for briquetting media for French coals led to the experiment of introducing coal converter residue directly into a kiln whereby, by driving off the light oil at low temperatures, a residue which might be satisfactory for bituminous coal briquetting could conceivably be obtained.

Coincidentally with this scheme of operation the asphalt content in the circulation through the coal chambers would be reduced. As is shown in the process diagram (page 124 of Appendix J), the outlet of the kiln was altered so that liquid product would pass into a conical vessel from which it could either be put into circulation through a cooler or loaded directly into a tank car.

The experiment was carried out under the following conditions:

	Initial Charge	Residue
Solid content	16.8%	32.1%
Ash in the solids	85.3%	79.6%
Asphalt in oil	26.3%	41.0%
Specific gravity at 80°C	1.20 kg/l	1.42 kg/l
Softening point according to Kramer-Sarnow		74.5°C
Charge to the ovens		3.6 t/h
Overhead oil		3.0 "
Solid		0.6 "
Steam addition		0.21"

Cont'd	Initial Charge	Residue
Oven temperature		500°C
Products:		
Distillates 1 and 2		1.36 t/h
Residue		2.13 t/h
Residue Oil		1.44 t/h
Residue Solid		0.69 t/h

Scheme of the Topping Plant

The results of the described topping experiments makes it seem possible that processing of the total products from the coal conversion chambers in a topping plant would be possible, provided the high oil loss would indeed prove to be operable and economically OK.

Distillation experiments show that it is possible to use exclusively the sensible heat in the residue from the ovens to drive off the required amount of overhead product at about 360°C under vacuum. The development of a suitable high pressure reduction valve would be necessary. By granulation of the residue from the flash distillation vessel in air, or in a stream of water, the briquette pitch should be obtainable in a usable solid form. The apparatus might correspond to the flow scheme shown on page 125 of Appendix J. By the application of a topping plant, the apparatus of the liquid phase in the hydrogenation operation itself would be materially simplified. The centrifuging operation would be completely obviated and the coking equipment for same would be replaced by simple distillation apparatus.

A - DISTILLATION

In the A-Distillation, separation was made between the coking overhead product itself and the foreign oil added for the operation, thus producing an "A middle oil" for the gas phase and "heavy oil" for the liquid phase hydrogenation. The coking overhead, and foreign oil, were introduced as a mixture to the distillation units because only one feed tank is available for the distillation.

Arrangement of the Apparatus

As may be seen by reference to the diagram opposite page 128, Appendix J, the A-distillation equipment was conventional. It will not be described here, but the interested reader can refer to the German text, pages 128 130 inclusive. It will be noted here only that six sections of packing were used in the tower, each 4 metres in height, with distributors above each section.

Operation Difficulties

By and large, the plant ran without difficulty. At the beginning some trouble was caused by water separation from the feed material. By improvement in the apparatus and operating conditions in the hydrogenation steps themselves and by the addition of dilution oil, these difficulties were practically obviated. The columns could be loaded between 30% and 150% over the guarantee figures, while at the same time good separations and normal energy usages could be obtained.

Most Important Operation Figures

Time Efficiency	85.6%
Average throughput per apparatus (including circulation)	48.6 t/h
Production exclusive of gas cooler condensate	
A-middle oil	40.05%
Residue	59.32%
Distillation gas K	0.58%
Losses	0.05%
End point of the middle oil	342°C
First drop of the residue	349°C
Gap in distillation	7°C
Ratio, product in circulation to total throughput	14.6%
Distillate exclusive of circulation(?)	2.92%
Throughput with circulation distillate	2.50%
Hourly amounts	
Throughput, exclusive circulation	71.3 t/h
A middle oil	28.5 "
Residue	42.3 "
Distillate gas	27.8 Nm³/h
Gas cooler condensate recycling	5.92 t/h
Energy usage	
Heating gas per ton of throughput	0.125 Mill WE
Steam per ton of throughput	0.078 t

B-DISTILLATION

The overhead product of the benzine ovens is separated in the B-distillation. The apparatus is quite similar to that of the A-distillation: columns are higher and auxiliary columns are used to remove an intermediate fraction. The gas heated pre-heater operates under normal pressure.

Arrangement of apparatus and operating procedure

The products of the preliminary hydrogenation and benzine forming chambers were distilled separately.

The following conditions were chosen for the manufacture of "normal" product aviation benzine and diesel oil.

The overhead product of the prehydrogenation chambers was cut between 125 - 130°C.

Because a portion of the distillation residue was to be sold as diesel oil an intermediate fraction at 135 - 205°C was taken out.

Materials boiling above 205° formed diesel oil. The intermediate fraction after the addition of some diesel oil served as the charge material for the benzination ovens. Cutting of the intermediate fraction was, of course, stopped when the entire distillation residue was processed in the benzination ovens.

The catch-pot product from the benzination ovens was separated into a benzine with an end point of 150°F. and a distillation residue which, in mixture with the distillation residue from the prehydrogenation distillation, was led back to the benzination ovens. The benzine from both distillations was combined and, after the addition of gas benzine from the motor fuel plant, it was treated with caustic soda and stabilized.

Most Important Operating Figures (for 5058 distillate)

Time efficiency	81.8%	
Average throughput per apparatus (exclusive of circulation)	37.3	t/h
Products		
Benzine (5058)	31.70%	
Heavy Benzine (5058)	2.28%	
B Middle Oil (5058)	64.66%	
Distillation gas (5058)	1.27%	
Losses	0.09%	
End point of benzine	150°C	
Initial point of heavy benzine	136°C	
End point heavy benzine	192°C	
Initial point B middle oil (5058)	155°C	
Circulation product, ratio to total throughput	10.3%	
Distillation factor without circulation	3.26	
Throughput distillate with circulation	3.63	
Hourly amounts		
Throughput exclusive circulation	26.73	t/h
Benzine (5058)	8.21	"
Heavy Benzine	0.61	"
B Middle Oil (5058)	17.55	"
Distillation gas	180	Nm³/h
Energy usage		
Heating gas per ton of throughput	0.190	Mill WE
Steam per ton of throughput	0.139	to

(6434) Distillate

Time efficiency	79.3%	
Average throughput per apparatus, exclusive of circulation	29	t/h
Production		
Benzine (6434)	48.08%	
B Middle oil (6434)	49.5%	
Distillation gas (6434)	2.35%	
Losses	0.00%	
End point Benzine	149°C	
Initial Point Middle Oil	152°C	
Gap	3°C	
Circulation product, ratio to total throughput	8.33	t/h
Distillation factor, throughput distillate		
Without circulation	2.08	
With circulation	2.27	
Hourly amounts		
Throughput exclusive of circulation	15.99	t/h

Benzine (6434)	7.69 t/h
B middle oil (6434)	7.92 "
Distillation gas	162 Nm^3/h

Energy Usage

Heating gas per ton of throughput	0.163 Mill WE
Steam per ton of throughput	0.038 to

CAUSTIC TREATING AND STABILIZATION

In the caustic treatment crude benzine is freed of H_2S and phenols by treatment with sodium hydroxide, after which stabilization through separation of the light hydrocarbons to give the satisfactory end product vapour pressure is effected.

The stabilization unit was fitted with a plate column and laid out for an operating pressure of 16 atmospheres. It operated completely automatically after setting the charge quantity by hand. Charge benzine for this operation contained 150 to 200 mgs. of hydrogen sulfide per litre. A preliminary outgasing was not demanded. The benzine was generally stabilized to a vapour pressure of 0.5 atmospheres and to do this the entire quantity of C3s and C4s was separated out. At the beginning the liquid top product was vaporized and led to the gas plant.

Since guaranteed production figures could not meet the increase of production in the case of the cylinder gas plant, an increase in the reflux amount at the stabilization plant was effected to give a liquid top product which met the purity requirements of the commerical cylinder gas plant. By this means the cylinder gas plant was loaded up in a satisfactory way. Indeed the portion of the top of column cylinder gas production was about 30% of the total cylinder gas production.

Operating data on stabilization

Benzine throughput	21.5 t/h
Vapour pressure:	
Raw material to stabilization	0.95 ata
Product from stabilization	0,48 ata
Liquid top-of-column product	.1 t/h
Gaseous top-of-column product	0.2 t/h
Reflux ratio	1:8
Operating pressure	5 - 7 atü
Top temperature	45 - 55°
Bottom temperature	150-165°

Input 18 atmospheres steam per ton of charge	0.19 t
as compared to	0.15 t when no liquid motor gas was produced at the top

Analysis of the liquid top product

C_2	Practically free
C_3	10 Mol. %
iC_4	45 " %
nC_4	45 " %
C_5	practically free

Analysis of the top gas

C_1	2.0 Mol %
C_2	8.0 " %
C_3	28.0 " %
iC_4	28.0 " %
nC_4	25.0 " %
C_5	8.0 " %

PROCESSING OF HYGASE

i.e. Processing of Exit Gases from Hydrogenation Chamber

In the Hygase preparation the effluent gases from hydrogenation were freed from ammonia NH_3, CO_2 and H_2S. The higher hydrocarbon containing gases were worked up to power gas; those with smaller hydrocarbon content were led to the cracking plant and to the heating gas system. The apparatus comprised an Alkazid plant to separate and remove both hydrogen sulphide and carbon dioxide, an oil washer to remove the higher hydrocarbons, means to convert the organic sulphur compounds, sodium hydroxide washer to substantially completely remove hydrogen sulphide and carbon dioxide, and the power-gas plant to liquify and separate by distillation the liquid products.

During the reduction of pressure at the hydrogenation plant, the following gases were obtained:

1) Expansion gases from both Ko and BI chambers
2) Expansion gas from the circulation gas washer
3) Expansion gas from the residue of the coal conversion chambers
4) Effluent gas from the Ko and BI distillation
5) Effluent gas from stabilization
6) Cracked gas from the residue coking operation

Items 3 and 6 were either disposed to the heating gas system or consumed at the units when made. The others were further treated. Liquid products and the wash oil from the circulation gas washer at the high pressure units were freed of gas in several pressure steps, thereby yielding gases which were hydrocarbon rich, and a hydrocarbon poor -- called respectively rich and poor gas.

In particular, the following were obtained:

1) Pressure relieve to 30 or 40 atmospheres: Ko, BI, and W - Poor gas. (Translator's note: gases are respectively from coal hydrogenation chambers, both second and third stage hydrogenation chambers, and from wash oil at coal chambers, - "W" gas)

2) Pressure reduction to about one-tenth atmosphere: Ko, BI and W-Rich gas.

3) Through heating under atmospheric pressure in the stripping distillations - "A" and also "B" distillation gases.

4) Through heating under pressure in the stabilization: Stabilization gas.

In order to avoid the extremely detailed discussion of the gas handling facilities which appears in the report, Appendix J, it is recommended that the reader refer to the flow diagram on page 137 of said notes. This flow diagram is entitled "Hygase Verarbeitung" and the five gas streams just mentioned are shown at the extreme left of the diagram as incoming streams.

The flow diagram depicts strictly conventional equipment for the various steps which are plainly indicated. Perhaps the only point of particular interest is that the two "alkazide lauge" units labelled DIK use the potassium salt of dimethylglykokoll in solution for scrubbing agent whereas the one labelled M used a solution consisting principally of the potassium salt of mono methylalanin. Both have the same purposes and general property, namely the ability to absorb H_2S and CO_2 from cold gases coupled with the possibility of regenerating the solution by heating up and driving off the hydrogen sulphide and CO_2. Details of each of the systems are shown in a diagram on page 147.

On pages 146 and 150 respectively of the Appendix J, performance data of the units in the manufacture of butane-propane power gas are given; also figures on the characteristics of the alkazide units.

PLANT SEWAGE WATER

The water effluent from the entire plant was segregated into two streams, one containing oil only, the other being water having phenol content. Simple settling, withdrawal of the oil layer and subsequent blowing with carbon dioxide to remove hydrogen sulphide sufficed to give water, suitable to be delivered to the Rhine River and oil returned to hydrogenation. On the other hand, the phenol containing water was necessarily treated with commercial tricresylphosphate which was indeed a mixture of triphenyl, tricresyl and trixylenyl phosphates. Difficulties ensued which are discussed in the full report. These were mainly tied up with the fact that the solvent was higher boiling than the impurities dissolved; hence on distillation and re-use, impurities, mechanical and otherwise, tended to collect in the solvent. At times improperly treated water was necessarily thrown into the Rhine on account of the difficulties mentioned, particularly since no suitable high vacuum still was available to purify the solvent. On the average, it was necessary to renew the solvent at least once every three months. The situation finally led to the recommendation to use the Lurgi process in place of the above mentioned process. The solvent used in the Lurgi process is principally isobutylacetate with other esters, and hence when a distillation separation takes place the impurities remain in the extracted material (phenols) as residue and can be discarded by "sales" of the extract. The phenol content of the water can be reduced to 100-200 mg/M^3 by this process.

THE POWER PLANT

Just prior to page 161 of the Appendix J there are three drawings which show respectively the preparation of boiler water for the boilers, the arrangement of the boilers themselves for powdered brown coal firing, and the use of the product steam through turbines and through the steam pressure conversion chamber to produce the necessary electric power and various pressure steam lines required for plant services.

Steam was generated at 70 atmospheres pressure in five boilers, having a capacity from 64 to 80 tons of steam per hour at 80 atmospheres and 500°C. Shortly before the plant was shut down, due to bomb damage, four additional boilers had been put in. All of the boilers were of the radiant heating single drum type provided with feed water preheaters, air preheaters and steam superheaters. Brown coal of 15% water content was ground in the three mills provided for each individual boiler, hot flue gas being used in mills to dry the powdered coal below its initial 15% water content. Aside from the dried brown coal, coke dust and briquette dust was used through the mills; also excess hydrogenation residue gas was burnt. The bunkers for the brown coal and other dusts were protected from explosion by a blanket of CO_2. Forced draught augmented the natural draught of the 180-200°C flue gas in a 100 metre high chimney, which was later complemented with a second chimney when the second four boilers were installed. Ash and cinders were removed from the combustion chamber by wet ash disposal, special pumps being used to deliver the ash to a settler. Residue settled from the conveying water stream was discarded by means of power shovels to suitable discard transport.

As the power flow diagram shows the 70 to 80 atmospheres steam was put through turbines driving 6000 volt generators. From the turbines both 18 atm and 3½ atm. steam was withdrawn. Make up water for the boilers was exclusively steam condensate, returned in part from large condensate points in the plant, and in part made by steam transformers generating 2½ atm. steam from softened water by use of 3.5 atmosphere steam. The softened water was obtained by the permutit process. It may be noted that about 55% of the condensate from steam used in the plant was returned to the boiler plant for further conversion to steam. The 45% loss was occasioned at many small points. Condensate was led back by two main lines and that portion in which it was possible to have oil contaminations was subject to de-oiling over a coke bed and filter.

Though the electric power generated from time to time amounted to 40,000 K.w., so that the power house could be called a large one, nevertheless the power production for war conditions was too small. Accordingly the previously mentioned expansion of the power house was undertaken.

Energy Usage

On page 179 of the Appendix report is a chart showing the principal utility consumptions, such as that of electricity, heating gas, steam and cooling water. Each of these items is divided into the portion required for gas production or hydrogenation itself and for power production. It will be noted in particular that the estimate of heating gas usage was considerably less than the actual requirements. Several circumstances contributed to this, not the least being that the poor performance of heat exchangers at the hydrogenation units, which as previously stated tended to become clogged with solid material. Also among other things, were conditions, which necessitated discontinuance of the preheaters at the coking ovens as already described. This, of course, increased the demand for heating gas.

THE MOST IMPORTANT FIGURES OF THE PRODUCTION HISTORY

Production of Hydrogenation Plant

(Compare chart on page 181 of attached Appendix J)

Nominal production of the works was considered to be 215,000 yearly tons of diesel oil and auto-benzine, or alternatively 215,000 tons of auto-benzine or 195,000 tons of aircraft benzine. The full production was first obtained in December 1942. It fell off in the first months of the year 1943. However, in the following months the under-production was made up so that a complete year's production was obtained for 1943. From January to May of 1944, the nominal production was exceeded by about 15%. In June and July the much heavier air bombing became noticeable. On the 18th of July the work was put out of operation for a long time by a heavy attack. Up to Arpil 1943 Benzine and diesel oil was produced. Thereafter, the production of aviation benzine also was taken up. This change accompanied the expansion of the benzine chambers, which up to July 1943 were outfitted with three converters, and after August 1943 with five converters. Correspondingly, the aviation benzine production after August 1943 rose sharply. The reduction of the production at the end of 1943 can be referred back to the change in the 6434 contact catalyst, while the serious production reduction in June and July 1944 is to be attributed to several weeks' repair of chambers. Production of power gas, "cylinder gas," began in February 1943.

One can recognize the true level of production best if figures of production are converted to diesel oil and autobenzine. After constant operation conditions were attained the estimated production was exceeded from 10 to 15 percent. The red curve on the chart shows the direct and indirect effect of enemy air attacks up to July 1944. They reduced total production by 23,220 tons or 5% of the total production.

The Gas Production

The production of the entire plant was in general limited by the amount of the gas production. At the beginning only water gas was produced. In July 1943 the cracked gas plant came into operation. By November of the year the additional three cracking gas ovens came into operation. From this time on the cracked gas production depended upon the amount of charge which was available from the hydrogenation exit gas.

The Capacity of the Works

As can be seen from the second part of the diagram on page 181 of the attached report (Appendix J), there was produced in 1944 some 21,700 tons per month of diesel oil plus benzine, that is at the rate of some 260,000 yearly tons of the two materials. This production corresponds to

complete utilization of the gas making facilities and that of the high pressure installation. As raw materials, 75% brown coal and 25% oil was used. From brown coal alone some 225,000 tons of benzine and diesel oil could be produced which is exactly the originally estimated amount of production. The bottleneck of the production was the hydrogen production (i.e. water gas), but capacity of the liquid phase and gas phase equipment was increased by the use of foreign oil to the above mentioned 260,000 yearly tons of diesel oil and benzine. By using oils only, sufficient production gas was at hand, since in processing oils only half as much hydrogen is used as in coal hydrogenation. The liquid phase, after slight enlargement of the preheater, could be loaded to about 50% above its present capacity. The amount of overall production would then be determined by the capacity of the gas phase. In turn, this production could be increased about 35% if oil alone was used, because the impure oil from such raw material is in the usual case much less rich in phenols.

One can therefore state that the present capacity of the plant is as follows:

Using brown coal: 225,000 yearly tons of diesel oil and benzine

Using oils: 360,000 yearly tons of diesel oil plus benzine.

In order to manufacture aviation benzine corresponding to 360,000 yearly tons of diesel oil and benzine, it would be necessary to add a fifth HI chamber.

The Most Important Means Used to Increase the Production of the Plant

The development of plant production was hindered by three circumstances: (1) the meagre provisions in the layout of the plant, (2) operating difficulties with Rhineland brown coal, and (3) the failure of the gas producing units to meet guaranteed performance. With full cognizance of the difficulties presented by war conditions which made a further increase in the very high plant cost undesirable, the means taken to increase production were only those which could be made without substantial amounts of new apparatus.

Plant personnel succeeded by such means in increasing production capacity of the plant some 20%. The following summary gives a review of the most important means applied and how they worked out. The means consisted of:

1) Production increase in the fuel gas plant which made possible diversion of hydrogenation waste gases for the production of fresh hydrogen.

2) Many process improvements in high pressure hydrogenation stages consisting mainly in overcoming the difficulties with the unusual raw material. The liquid phase chambers were thus given increased capacity, which made further amounts of discard gases available for hydrogen production.

3) In the creation of cracking capacity for the working up of the additional amounts of available quantities of Hy-gase.

4) In removing certain miscellaneous bottlenecks in the production line.

Production Increase at the Fuel Gas Plant

Most important change consisted in employing carbon dioxide admixture in place of water saturation of the blast for the fuel gas (producer gas) generators. This has already been described as reducing the crusting of the ash bed of the generators, mainly by obivating the water droplets carried with the water-saturated blast. The gas capacity of the generator bodies was thus increased by the order of 35%. Simultaneously, of course, waste heat became available at the generators, and a not inconsiderable amount of fuel was saved.

Altogether, some 18000 Nm^3/hour more of fuel gas was produced with some fuel saving, and this corresponded to some 9000 Nm^3/hour greater production of hydrogen.

Improvements at the High Pressure Hydrogenation Units

On pages 185 to 191 inclusive of the report, given as Appendix J hereto, various other means which resulted in an overall improved capacity are described in considerable detail, such as improvements in the high pressure hydrogenation operations, in the cracking capacity of the hydrogen gas generators, and in various other improvements in gas purification, stabilization, and the like. Finally, these improvements are reduced to savings in marks per year. The details do not seem to be of other than very specific application to this plant and will not be summarized here.

OPERATING BALANCES FOR HYDROGEN

On page 192 of Appendix J are a series of diagrams showing the use of raw material and production of products. The data is all based on one ton of middle oil production or processing. Reference is made in what follows directly to these diagrams. At the upper left there is a small diagram showing "Kohleverbrauch" or consumption of brown coal to make one ton of middle oil. This diagram refers to brown coal as "mined" and the following figures can be read out of the diagram plus the small rectangle immediately below it:

	Metric Tons	
Brown coal as mixed	15.6	
Water	9.2	
True brown coal ("TB.KO")	6.4	15.6
of which		
To dry mine run of BK	1.5	
Heat equivalent to outside electricity	0.7	
Power house	1.3	
Heat gas production	0.6	
Water gas production	0.8	
Hydrogenation itself	1.5	6.4

In the text page 193 the statement is made that the table just above showing usage of 15.6 tons of brown coal, as mined, to produce one ton of middle oil, would be increased by about 10%, that is to some 17 tons of raw brown coal, if the production of motor benzine instead of middle oil was considered. The writer states that it is interesting to note that the Fischer process and the methanol synthesis operate with about the same energy consumption.

In the four other diagrams at the top of the page similar breakdown figures are given for total hydrogenation, and for the liquid phase and gas phase operations. The heat units in the gases are also given as produced and as used in the upper right hand diagram. In each of the diagrams one column is given with the word "Soll" at the base, referring to the original estimates by the I. G. of the usages for the various mentioned operations. At the bottom of the page is diagrammed the entries and the exit values in hydrogenation for the following elements, namely, carbon hydrogen, oxygen, sulphur, iron and ash. The following comments can be made:

Carbon balance: It may be noted that some 30% of the total carbon brought in originated in "foreign" oils.

Hydrogen balance: About 7.28% of the hydrogen is recorded as lost and 9.5% as being consumed to produce reaction water. The hydrogen consumed to water is unusually high on account of the high oxygen content of the Rhineland brown coal.

Oxygen balance: The important factor here is how much of the oxygen contained in the raw materials passes into CO_2 and CO, and how much to H_2O. The latter reaction consumes hydrogen, whereas the former, of course, do not. It will be noted that the oxygen balance shows that some two parts of oxygen are converted to water and one part into oxygen containing gases. This is normal and corresponds to the original estimates.

Sulphur balance: The low sulphur content of Rhineland brown coal required the addition of considerable sulphur as such. For process purposes, obviously only the sulphur in the gas is recoverable, that in the coking residue is lost.

Iron and ash balance: About 40% of the iron incoming to the plant was present in the brown coal, the other 60% was added as liquid phase contact material. In the case of the ash the reverse ratio is approximately correct.

GUARANTEED PRODUCTION VERSUS ACTUAL

All parts of the plant with the exceptions noted below produced gases or a treated product in amounts at least equal to the guarantee figures. It is interesting to note that no guarantee figures were given by the I. G. for hydrogenation itself; nevertheless, their estimates of what would be required for the Rhineland brown coal hydrogenation were approximately met.

Part of the plant which definitely failed to produce according to guarantee was the water-gas producing plant put up by Pintsch and employing the Pintsch-Hillebrand process. Guarantee figures and the actual production results at the end of 1943 were as follows:

	Guarantee	Actually Attained
$CO + H_2$ content of the water gas	84%	84%
Capacity of generators Nm^3/H	5500	5250
Water gas/units briquettes, i.e., Nm^3/ton of fuel	2030	1900
Heating gas required WE/Nm^3 of water gas	780	820
Outside steam required	-	0.12 Kg/Nm^3

Because the guarantee was not met, Pintsch paid an indemnity, size not stated.

The second failure to perform was in the case of the power gas, or "Treibgas," plant which was 40% under the guarantee figures. The company (UHDE) had agreed to deliver a second plant to make up the deficiency, but apparently this was not done before the entire plant had been bombed out of commission.

Cost of one ton of liquid product - Based on the 1st Quarter 1944

Beside page 208 of the report comprising Appendix J is a chart showing cost items entering into a ton of liquid fuel produced by the plant, exclusive of cylinder gases. On page 208 itself the figures are given as a tabulation. This may be summarized as follows:

	R.M.
Raw materials and chemicals, etc.	81.01
Maintenance	41.36
Salaries and wages	20.48
Utilities, such as electricty, steam, water, etc.	141.31
Investment write-off	56.18
Interest	32.21
Taxes	15.39
Licences	2.87
Shipping costs	7.66
Commission and miscellaneous costs	71.28
TOTAL	469.75
Credit to operations, sales, bottle gas, heating gas, generator power, phenols, miscellaneous TOTAL NET COST	403.71

The details of the cost table of which the above is an abstract are of some interest inasmuch as unit prices are shown as well as the amount of each material or service used, but they will not be further detailed here.

In the succeeding pages 208 to 215 inclusive, considerable discussion of the possibilities of lowering costs of the admittedly high priced product (s) from this plant are given. It appears that the economic future of the plant depended on lowering almost all of the costs having to do with the financial structure and substituting oil in place of brown coal for hydrogenation. By so doing the capacity of the plant would be increased in respect to the volume of product made and the cost of each unit volume would naturally be decreased.

<u>Concluding discussion:</u> In pages 216 to 222 inclusive, the author of the report (Appendix J) discusses the effect of bombing on the plant in terms of production lost which he estimates to have totalled at 23,220 tons between May 29th 1942 and July 11th 1944. The later date was, of course, shortly before the final attack which substantially completely set the plant out of operation. The above mentioned loss in production corresponded to 5.1% based on the actual production during the time mentioned, which was namely 455,356 tons.

In the final pages, the bombing attack of July 18th to 19th is graphically described. On page 220 is a plot plan of the plant showing where the stated "more than 1000 bombs" fell within the plant boundaries. This attack was nothing less than a disaster to the plant and while some 10 weeks were occupied with very vigorous efforts to repair the works, and 40% production of a provisional sort was obtained, it was finally decided to shut the plant down. On October 6, 1944, this was done.

It is interesting to note, among other things, that the plant personnel had been trained to shut the plant down in the order of two minutes' time, and this actually occurred at the time of the final bombing attack. Of the 600 workers present during the attack only three were killed - said to be because of their own negligence. Nevertheless, communication facilities between the plant and Cologne were

broken off and difficulty was had in handling the fires which resulted. Furthermore, the plant smoke-manufacturing apparatus did not work properly to cover the plant against an attack, but instead formed a ring around it, thus presenting an excellent target for the flying personnel in the attack. After the attack the smoke forming equipment continued to function and hindered the work of the fire extinguishing forces.

APPENDIX A

DHD PROCESS ("DRUCK-WASSERSTOFF-DEHYDRIERUNG")

The Wesseling plant had no DHD process in operation for converting especially naphthenes of the coal-hydrogenation into aromatics thereby improving the octane number of its motor-fuel product and increasing its desirability as a fuel for airplanes by providing them with greater overloading capacity. However, such a plant was contemplated and the following outlines of the process have been obtained from documents seized there during the C.I.O.S. inspection of the plant and interrogation of its remaining personnel.

The DHD process comprises treating with a dehydrogenating and cyclisizing catalyst, either alone or in admixture, those benzine fractions of the coal-hydrogenation process that are obtained in the steps employing catalysts 5058 and 6434; i.e. in the gaseous phase operations of the hydrogenation process. These fractions are first distilled to remove those components that boil below 85°C. (benzine and cyclohexane) this low-boiling fraction constituting about 10% of the fraction that is selected to be treated to DHD: this low-boiling fraction is then later added to the DHD product. By means of centrifugal and piston-pumps, the above benzine fraction boiling above 85°C. and to about 160°C. is injected into the DHD convertor-ovens at about 45 atm. pressure. Immediately before these convertor-ovens, the benzine liquid and a continuously recirculated stream of gas at the same pressure are admixed, the gas having its origin in previous operation of the process and naturally containing some hydrogen and gaseous hydrocarbons.

Leaving the convertor-ovens and after passing through a cooling means, the treated mixture is separated in a cold catch-pot into a gaseous and a liquid product. The gas, which during passage through the convertor-ovens (dehydrogenation) has dropped to about 25 atm. pressure, is recompressed to 45 atm. and returned to the apparatus inlet to repeat its recirculation. The excess gas formed (by dehydrogenation gas is continuously liberated) after its scrubbing with wash oil to remove heavier hydrocarbons, is then sent to the lean-gas mains. The absorbed hydrocarbons liberated by expansion of the wash oil go to the rich-gas mains. In this manner, the volume of gas recirculated through the DHD catalyst is maintained at a constant volume. The liquid product separated in the cold catch-pot is sent to a distillation step where it is separated into DHD benzine and residue. The small residue is returned to the B-distillation of the coal-hydrogenation process and the DHD benzine is stabilized.

The DHD plant resembles the coal-hydrogenation apparatus but its process operation is discontinuous rather than continuous. In addition, in contrast, the reaction is highly endothermic and requires large additives of heat which is, however, importantly supplied by the gas-evolved during the process.

The DHD apparatus comprises: Three heat-exchangers; a principal and four intermediate preheaters; five catalyst ovens; and a refining oven wherein resin-formers are hydrogenated, this latter oven being provided with a catalyst for the purpose that is operative at about 300°C. The first three contact ovens are 1.4 m. in diameter and 10 meters long whereas the final three ovens have the same diameter and are 12 meters long. A schematic flow-diagram will be found at the end of this Appendix A.

In operation of the process, the benzine-product to be subjected to DHD-reaction is, in admixture with the aforesaid recirculating gas, injected at about 45 atm. pressure into the heat-exchangers where, in counter-current contact with product leaving the catalyst ovens, it is preheated to 370°C. Thereafter, the heated mixture passes to the principal preheater that is provided with 18 hair-pin pipes and wherein the mixture is further heated to 490° C. and then passes into the top of the first catalyst oven. After flowing downwardly over the first catalyst bed, the mixture flows from the upper part of the catalyst oven through an immersion-pipe at about 430°C. into the first intermediate pre-heater equipped with 5 hair-pin pipes for reheating the mixture of gas and vapor, to 500°C. Similarly, the remaining catalyst ovens 2, 3, 4, and 5 are operated at temperatures respectively of 450°, 470°, 490° and 500°C. whereas the intermediate preheaters 2, 3 and 4 preheat the treated mixture respectively to 510°, 510°, and 520°, respectively. Since the latter catalyst ovens do not tend to form coke to such an extent as the first three ovens, the coke formation in the final two ovens is no more in each than in the former despite the fact that they are greater in length and the contact time consequently longer. For this reason the oven-inlet temperatures are increasingly higher from oven to oven and the fall in temperature in each oven is a measure for the activity of the catalyst and the conversion of product.

When in the course of operations, the activity of the catalyst markedly decreases, it must be regenerated. For this purpose, the injection of benzine is stopped and the preheater closed down, and the beds are operated in "dry" condition for 1 to 2 hours, whereupon the gas-recirculating pump is closed down and the pressure in the catalyst beds and circulating equipment is slowly released. The recirculation and catalyst beds are then flushed with an inert-gas that has its origin in a prior regeneration period and is stored in a special holder therefor. After the flushing operation, the catalyst-ovens and recirculation are filled with gas stored in the heat-exchangers and having a pressure of 25-30 atm. according to their volume. The recirculating pumps and the preheaters are then restarted. At an oven temperature of 23 mV (442°C.), about 6% by volume of air can be given to the gas entering each oven. The resultant combustion of the carbon deposited on the catalysts to CO_2 is controlled, by the temperature of preheat and the cold-gas additions, in such manner that the combustion-zone which slowly progresses from top to bottom, does not exceed 550°C - this is in order to protect the walls of the preheater pipes and the catalyst itself. The regeneration pressure is maintained at 50 atm. by expansion of the excess gas into the atmosphere after the storage-holder for the regenerations have been filled. Depending on the condition of the catalyst the lowest required "burning-off" time is about 8 hours. Complete regeneration and return to operation of the catalyst-chamber requires nearly 20 hours.

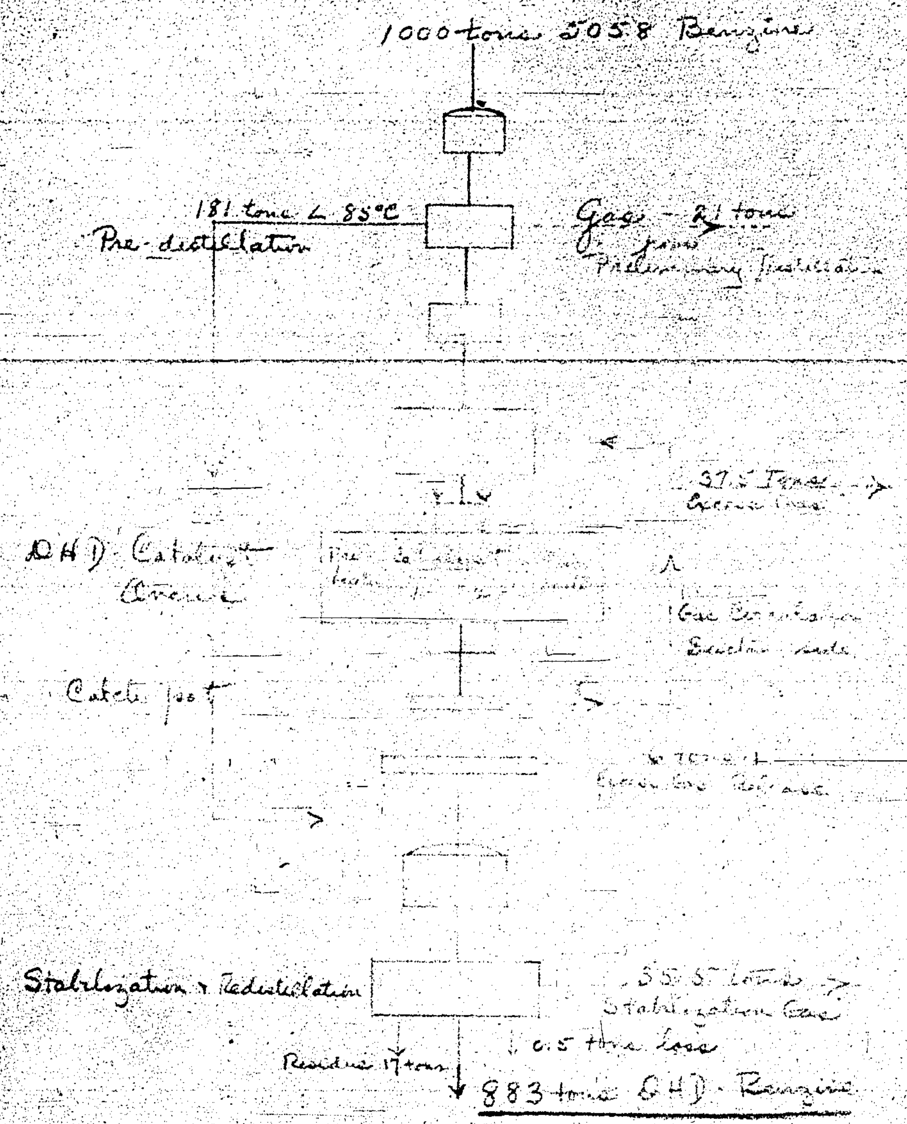
After completion of the regeneration period, the preheater apparatus is shut off as well as the recirculating pump and the catalyst beds and circulating system relieved of pressure. Without flushing, the catalyst-ovens are then filled with DHD gas stored from prior operation; it is not necessary to restore operating pressure artifically because after starting up the ovens this automatically takes place rapidly by the resultant production of gas. After starting the gas recirculating pump, the ovens are slowly re-heated to operating temperature and liquid injection re-started. By additions of cold gas, peaks of temperature can be avoided in the preheaters. The length of the dehydrogenation period, the time during which satisfactory yields are obtained, etc., depends on the nature and characteristics of the treated material. Whereas in the case of Romanian paraffin-base oils, the DHD ovens must be shut down after 40 hours for regeneration of catalyst, the naphthenic-base oils can be treated for about 80 hours. In the case of the Wesseling Vorhydrierung benzine, it is possible according to its pretreatment to operate from 200 to 400 hours. It is anticipated that a mixture of Wesseling 5059 + 6434 - Benzine can be easily dehydrogenated. The predicted catalyst life is almost one year after which it is not unuseable but can be restored satisfactorily by a screening operation. Typical of the change produced by DHD of a benzine is shown by the following table:

	AP	% Paraffines & Olefines	% Naphthenes	% Aromatics	Octane Number (Motor)	+ 0.12% Pb. (Motor)
Inlet Material	+ 42°C.	28.5	62.0	9.5	64	83
DHD Product	- 8°C.	20	15	65	80	90

Available documents at Wesseling did not disclose the constitution or preparation of the DHD-catalyst save to suggest that it had Cr_2O_3 as a constituent. For details of preparation, reports of C.I.O.S. teams that visited Leuna, Scholven, or Ludwigshafen, should be read.

The accompanying schematic diagrams of a DHD flowsheet and apparatus will assist in understanding some of the operational details.

DHD-Flowsheet



DHD - Apparatus

Preheater