U. S. GOVERNMENT TECHNICAL OIL MISSION
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(Original Designation BM-18)

The following reports written by members of technical missions to Germany have been reproduced on this reel and appear in alphabetical order by authors.

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ACTON-TAYLOR - HALLIMOND(B.I.O.S.)

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

[illegible]

ITEM No. 5
FILE No. XXI—5

COPY No. 1

SECRET

HEINKEL-HIRTH TL GAS TURBINE ENGINE

Adderley, J. W., Saddler, K., & Robinson, S.T.

SECRET

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

SECRET

HEINKEL-HIRTH TL GAS TURBINE ENGINE.

Reported By.

F/Lt. J.W. ADDERLEY (TE/G1 - M.A.P.)
Maj. K. SADDLER, U.S.A.A.F.
Lt. S.T. ROBINSON, U.S.N.R.

CIOS Target Number - 5/21

Jet Propulsion

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

SECRET

(1945)

24p diagrs

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Personnel of Team

F/Lt. J.W. ADDERLEY (TE/G1 - M.A.P.)
Maj. K. SADDLER, U.S.A.A.F.
Lt. S.T. ROBINSON, U.S.N.R.

Report on Parts of Heinkel-Hirth TL Gas Turbine Engines found at Freidrichshall, Germany.

INTRODUCTION.

On Friday, 13th April, 1945, the Friedrichshall area was cleared by American Army troops, and it was discovered that a salt mine, the Staatliche Salinen at Kochendorf near Friedrichshall, had been taken over for general dispersal of aircraft activity from the Stuttgart area. The work of preparing the salt mine for dispersal had begun in the spring of 1944 and was carried out by members of the Todt Organisation and S.S. prisoners, the installation being given the code name of Eisbak. Floor space was allocated to various firms to the total extent of 144,250 sq. meters, amongst which was Heinkel-Hirth, who were allocated 36,100 sq. meters. This firm began to move in in June or July of 1944, work probably beginning two to three months later. The work covered by Heinkel was general development and experimental work on gas turbine engines and also production of exhaust-driven turbo superchargers presumably for the BMW.801J engine.

At the time of writing this report, it was not possible to enter the mine since the lift shaft had been seriously damaged by artillery fire. However certain parts of the Heinkel-Hirth TL engine as listed in Appendix A were found above ground and from these a general idea of the construction of the engine was obtained.

GENERAL DESCRIPTION.

The parts of the engine as found were not complete, but from an examination, it was apparent that the engine could be considered to consist of five major parts.

1. Inlet ducting and accessory case.
2. Compressor assembly.
3. Combustion chamber.
4. Turbine.
5. Tail pipe.

None of the tail pipe parts are available and the only major rotating assembly found was the mixed flow first-stage impeller, but from the parts available, it can be summised that the general pattern of the design is around a single mixed flow impeller followed by three stages of axial flow compressor and annular combustion chamber and a turbine, presumably 2-stage, as indicated by inspection of parts and previous reports.

It is apparent that the engine is still in an experimental state since the parts obtained are by no means all from the same detail design of engine, although they are to the same general layout. However, a drawing has been prepared giving an idea of the general assembly of the engine as far as can be obtained from these parts, and it should be realised that this drawing only represents an assembly of components available and does not, in any way, represent any one particular build of the unit.

DETAILED DESCRIPTION.

1. Inlet ducting and accessory case.

The compressor casing mounts on an annular casting as illustrated in figs. 2 and 3 having provision for starter drive and six pads for accessories. The lower half of this casting contains the oil tank. Due to the design of the compressor casing, it is possible to mount accessories on the rear pads of this gear box without increasing the diameter of the engine. The front bearing of the main rotating assembly is supported in this casting in a housing carried by 3 streamlined struts so disposed to impart swirl to the incoming air in the direction of rotation. The accessory drive shaft is carried in the top vertical strut direct to the starter drive, this shaft being driven by bevel gears from a short stub shaft in the nose, which is connected to the principle rotating assembly by a splined quill shaft. From the starter motor drive, the drive is carried to the accessories through a series of spur gears and the accessory gear ratios and direction of rotation are shown in figs. 2 and 3.

The stub shaft protrudes from the front of the casting and has a splined surface, the purpose of which is unknown, although certainly it is not intended to drive an airscrew owing to the small size of the driving quill shaft. An impulse lubrication pump is carried on the accessory box providing oil to six lubrication points, and there is a mounting flange on the front face of the casting for the provision of inlet ducting. The front bearing housing is illustrated in fig.4. and is installed in the rear of the accessory case.

2. Compressor.

The compressor consists of a single-stage mixed flow impeller having 12 blades of aerofoil section as illustrated in figs.5, 6 and 7, followed by a row of adjustable stationary blades, followed by a 3-stage axial flow compressor of constant tip diameter. The second and third row of stationary blades are also adjustable and are of light alloy whilst the fourth-stage stationary blades are of steel and consist of two rows imparting a slight swirl to the air as it enters the combustion chamber. These are shown in fig.8. The first-stage impeller consists of 12 light alloy blades fastened to a steel hub by means of a 1.3" diameter bulb root. The blades are curved in the direction of rotation at inlet and against the direction of rotation at outlet. The inner wall of the air passage through the impeller is defined by filler blocks which are fastened to the blades. The hub terminates at the rear in flange to which are bolted succeeding stages and at the front in a stub shaft externally machined for front bearing mounting and internally splined for the quill shaft.

The axial flow compressor blades obtained are mounted on U shaped feet, the first and second stages being of aluminium alloy and the third stage of steel. They are illustrated in fig.9. No compressor discs to mount these blades were found, although the two discs illustrated in fig.10. are apparently compressor discs from their dimensions and may be to mount blades of a later or earlier design.

The casing itself (as shown in fig.11.) is an aluminium alloy casting with provision for a mechanism to adjust the angle of the first three rows of stator blades. The outer extremity of the stator blades are located axially in steel split rings keyed into the casing and there is a boss on the head of the stator blade with a slot for adjustment. The feet of the stationary blades are circular and fit into a ring supporting a labyrinth seal at the shaft through two shearplates.

A partly assembled stator ring is shown in fig.12. This assembly is made in two semi-circular sections. A cast iron disc identical in dimensions to one of the stator rings was found which may be of an alternate design.

The axial flow compressor is designed for work to be done in both moving and stationary blades and approximate blade angles are shown in fig.1. The blading is built up on an aerofoil section with a parabolic camber line.

3. Combustion Chamber. The combustion chamber is of annular type although the primary portion was not found. The outer wall of the chamber is shown in figs.13. and 14. Fuel is injected through 128 small nozzles through pads as shown in fig.13. Some secondary air is introduced at two-thirds of the length of the chamber through radial fingers as shown in fig.15, and the nozzle guide vanes are carried in the rear part of the combustion chamber which is shown in fig.16. The nozzle guide vanes are adjustable and are of hollow construction and air-cooled, air being carried to them through annular ducts on the inner and outer walls of the combustion chamber to holes at the top and bottom of these blades., the cooling air leaving through slotted trailing edges.

4. Turbine. No turbine parts are available, but it is obviously of 2-stage design, and it is clear from the general design of the engine that the rear bearing is mounted behind the turbine.

DISPOSAL OF PARTS.

The parts as listed in the Appendix have been delivered to Power Jets (Research & Development) Ltd., Pyestock, for further investigation. It is anticipated that, in the near future, it will be possible to obtain access to the foot of the mine to further investigate the Heinkel-Hirth work in this area.

Flight Lieutenant J.W.Adderley,TE/G1/MAP
Major K. Saddler, USAAF.
Lieutenant S.T. Robinson, U.S.N.R.

London.
25th April, 1945.

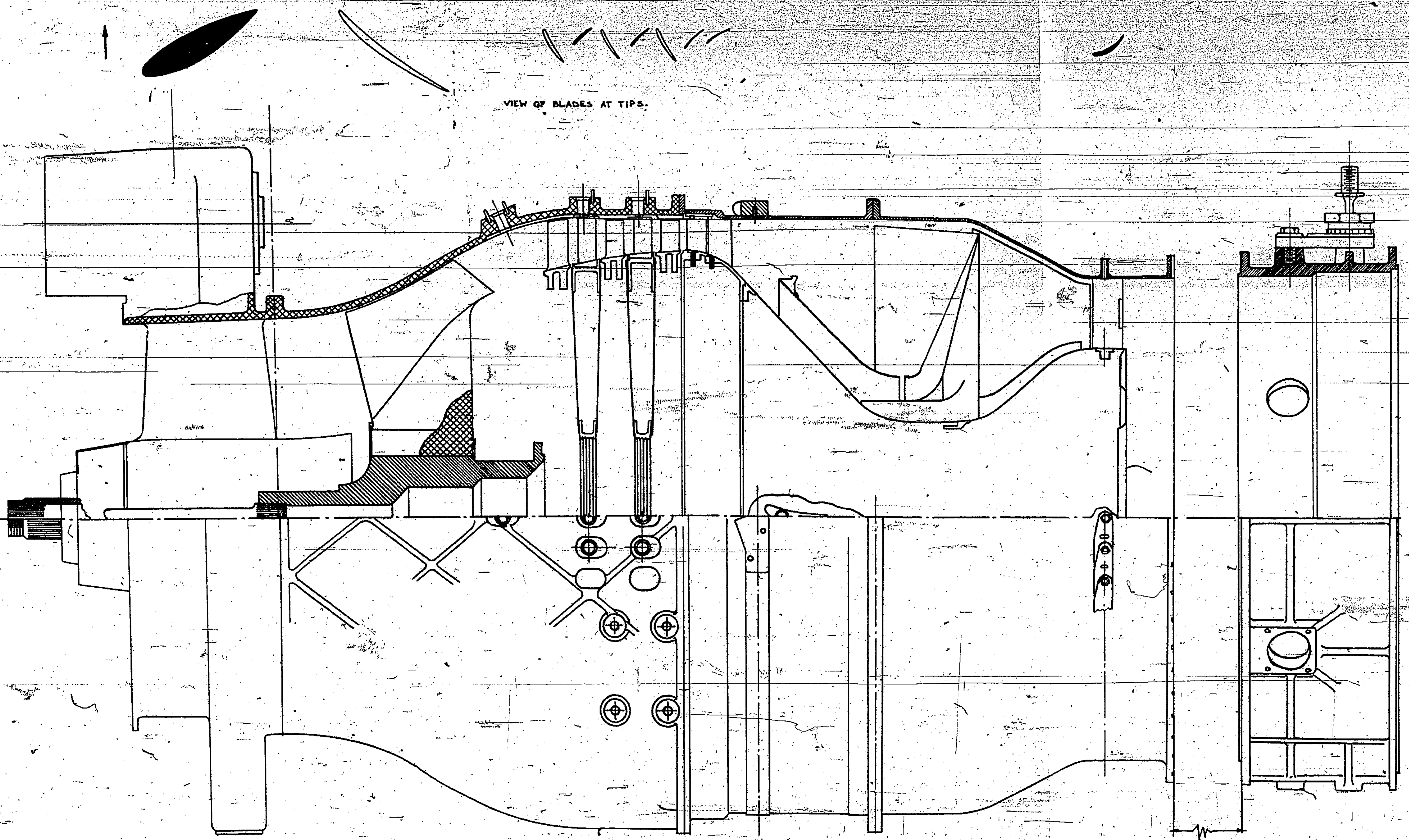
APPENDIX I.

HEINKEL-HIRTH OIL TL UNIT.

NAME OF PART.		PART NUMBER
1.	Inlet and Accessory Drive Case.	S-11-005-500-171-20
2.	Front Bearing Housing.	
3.	First Stage Impeller.	
4.	Accessory Drive Quill Shaft.	
5.	Compressor Outer Casing	W9-115726
6.	First Row Stator Retaining Rings.	
7.	First Row Spacer Ring.	
8.	Second Row Stator Retaining Ring.	
9.	Second Row Spacer Ring.	
10.	Front Half Third Row Retaining Ring.	
11.	Second Row Stator Disc.	
12.	Third Row Stator Disc.	
13.	Second Row Stator Blades	212
14.	Third Row Stator Blades	213
15.	Fourth Row Stator Ass'y	109-011-400
16.	First Row Axial Compressor Blade	(211) 109-011-100-041
17.	Second Row Axial Compressor Blades	(213) 109-011-102-043

APPENDIX I (Contd.)

NAME OF PART.	PART NUMBER.
18. Third Row Axial Compressor Blades	
19. Combustion Chamber Outer Casing	
20. Fuel Injector Block	109-011-411
21. Inner Wall and Secondary Mixing Device of Combustion Chamber	
22. Rear of Combustion Chamber including First Row Turbine Nozzle Ring	
23. Rear Combustion Chamber Forward Cooling Air Shroud	
24. Rear Combustion Chamber Aft Cooling Air Shroud	
25. Cast Steel Ring, presumably Rear Bearing Support	
26. Rear Support Pad	109-011-301-017
27. Trunion	
28. Fourth Stage Stator Blades	109-011-400
29. Turbine Bearing Hub	
30. End Cap for Front Bearing Housing	
31. Rear Bearing Oil Supply Pipe	109-011-300-707



HEINKEL HIRTH-TL- TURBO JET.

0 5 10 15 20 CM
0 2 4 6 IN.
SCALE 1/2

PHOTOGRAPHED AT REDUCTION 20-TO-1

FIG. 1.

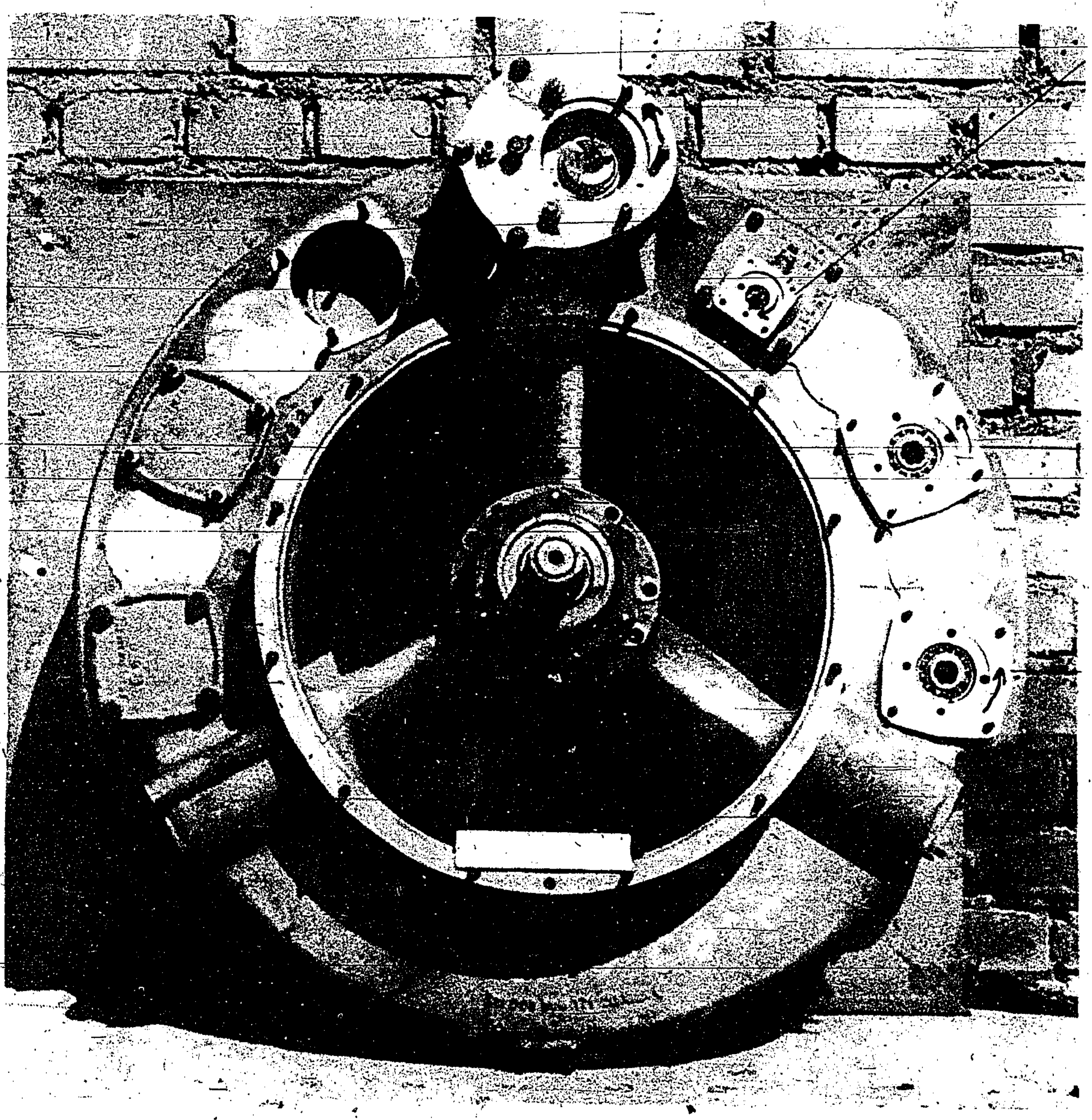


FIG. 2

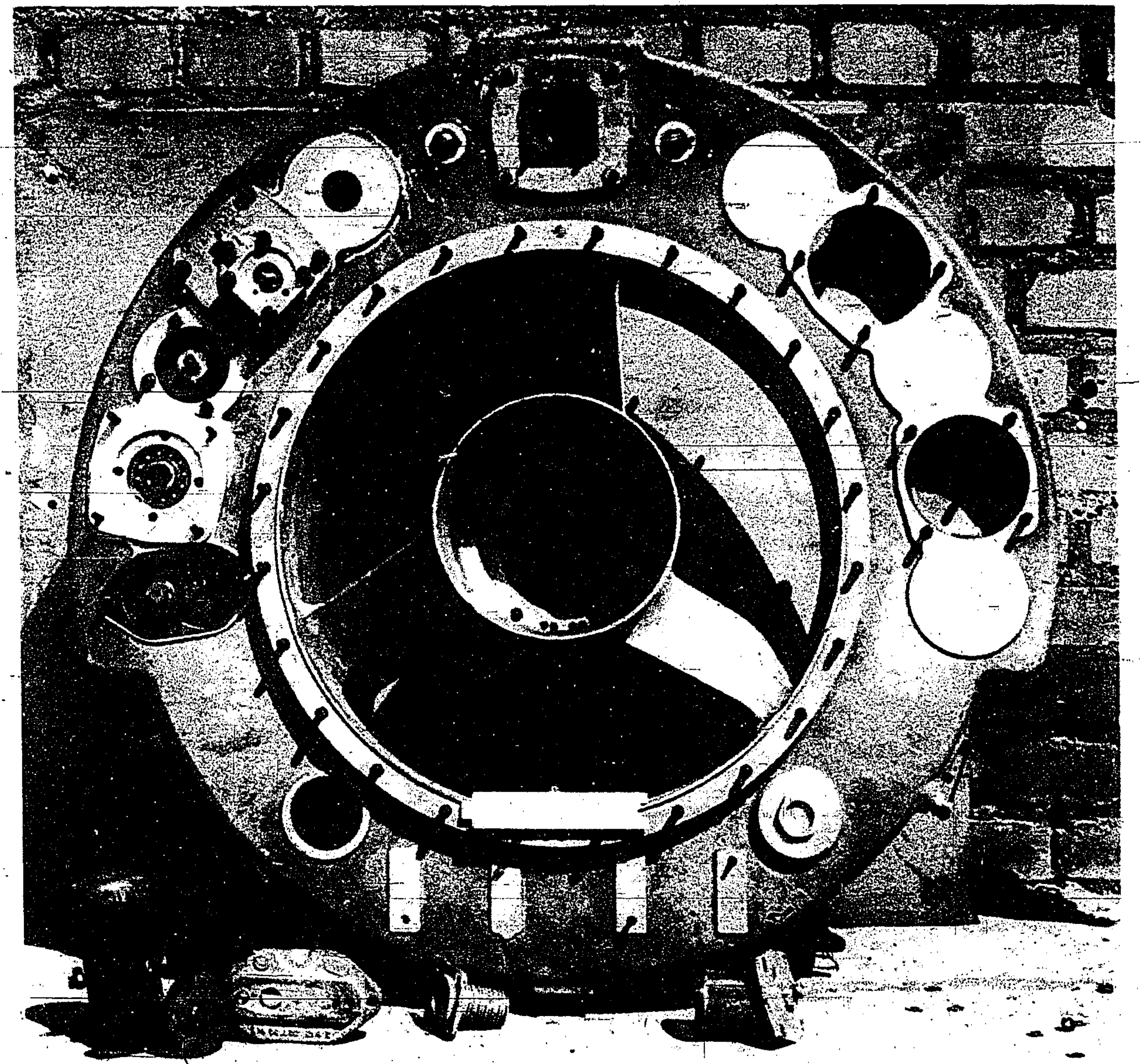


FIG. 3.

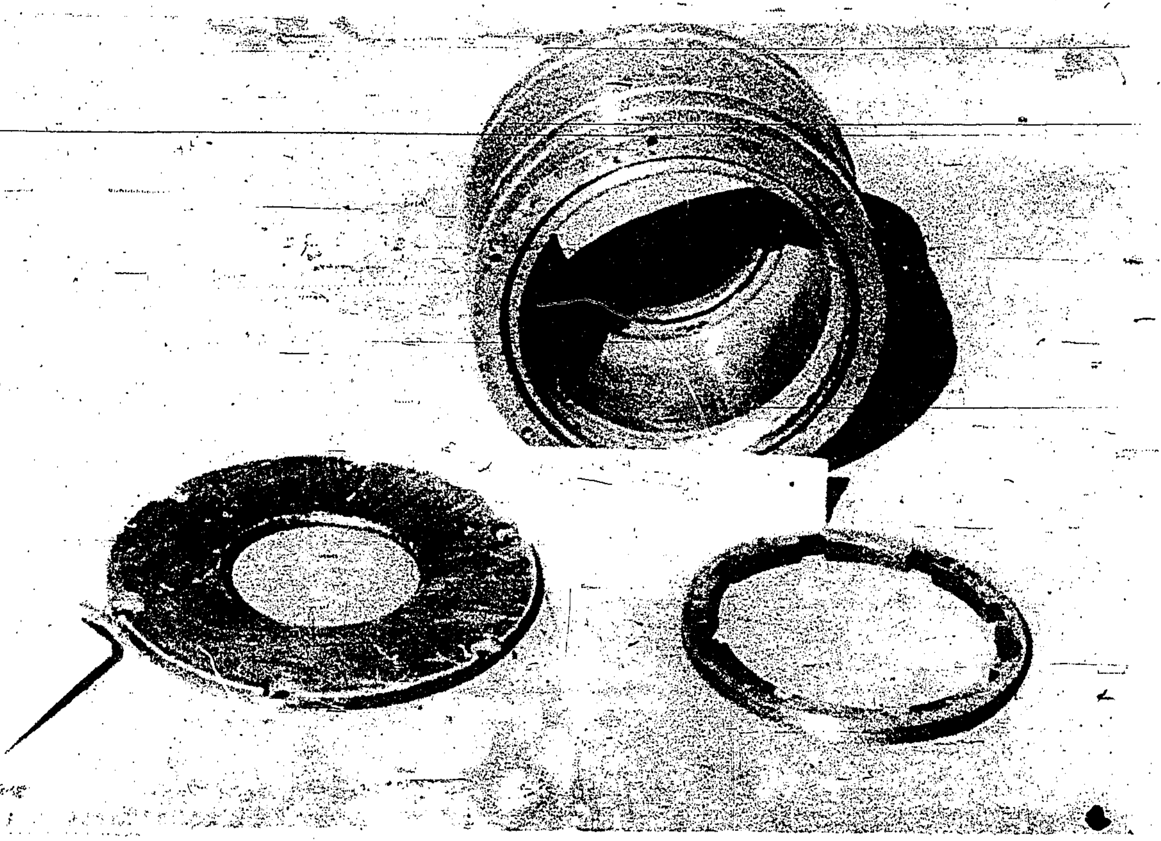
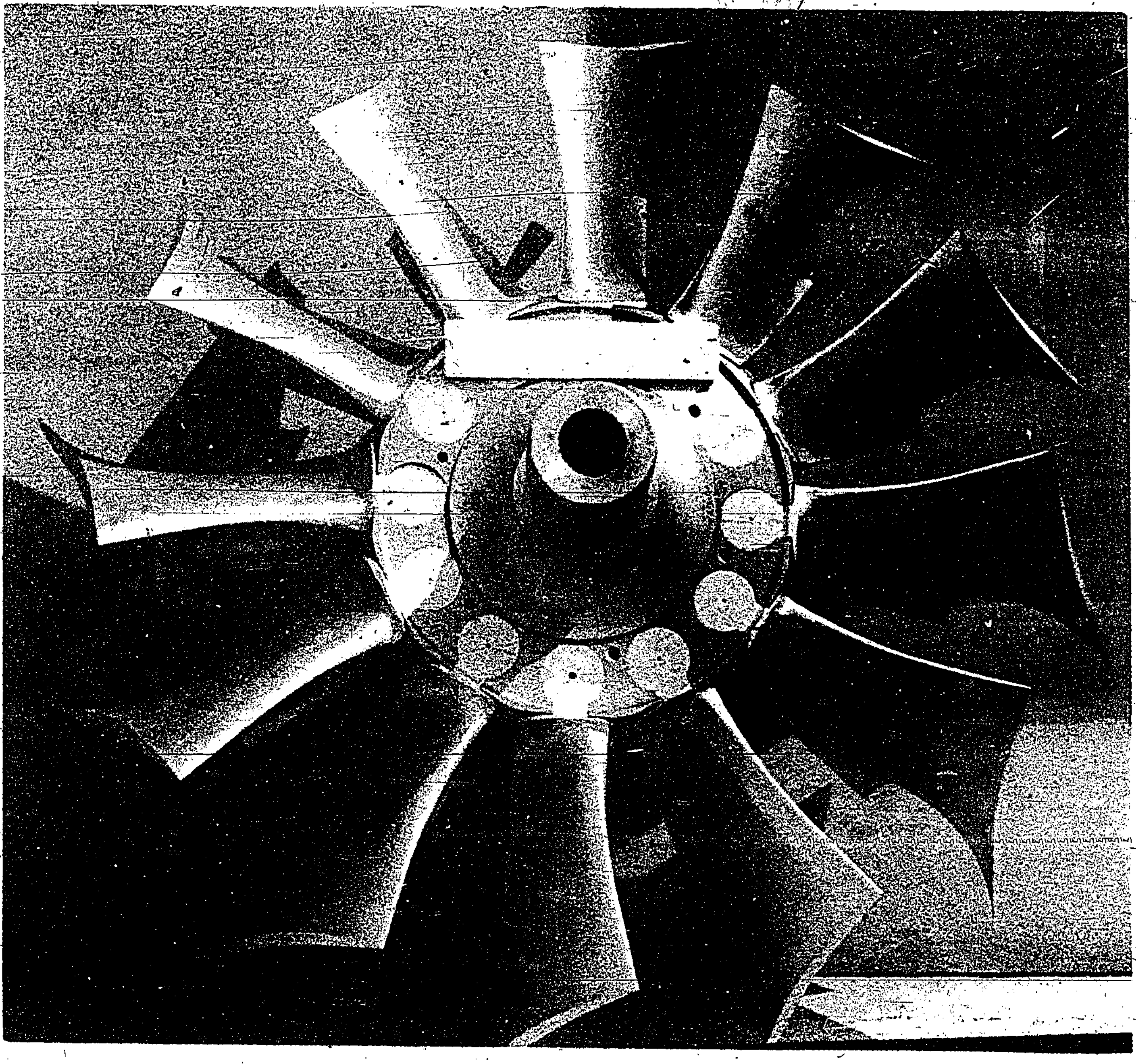


FIG. 4.



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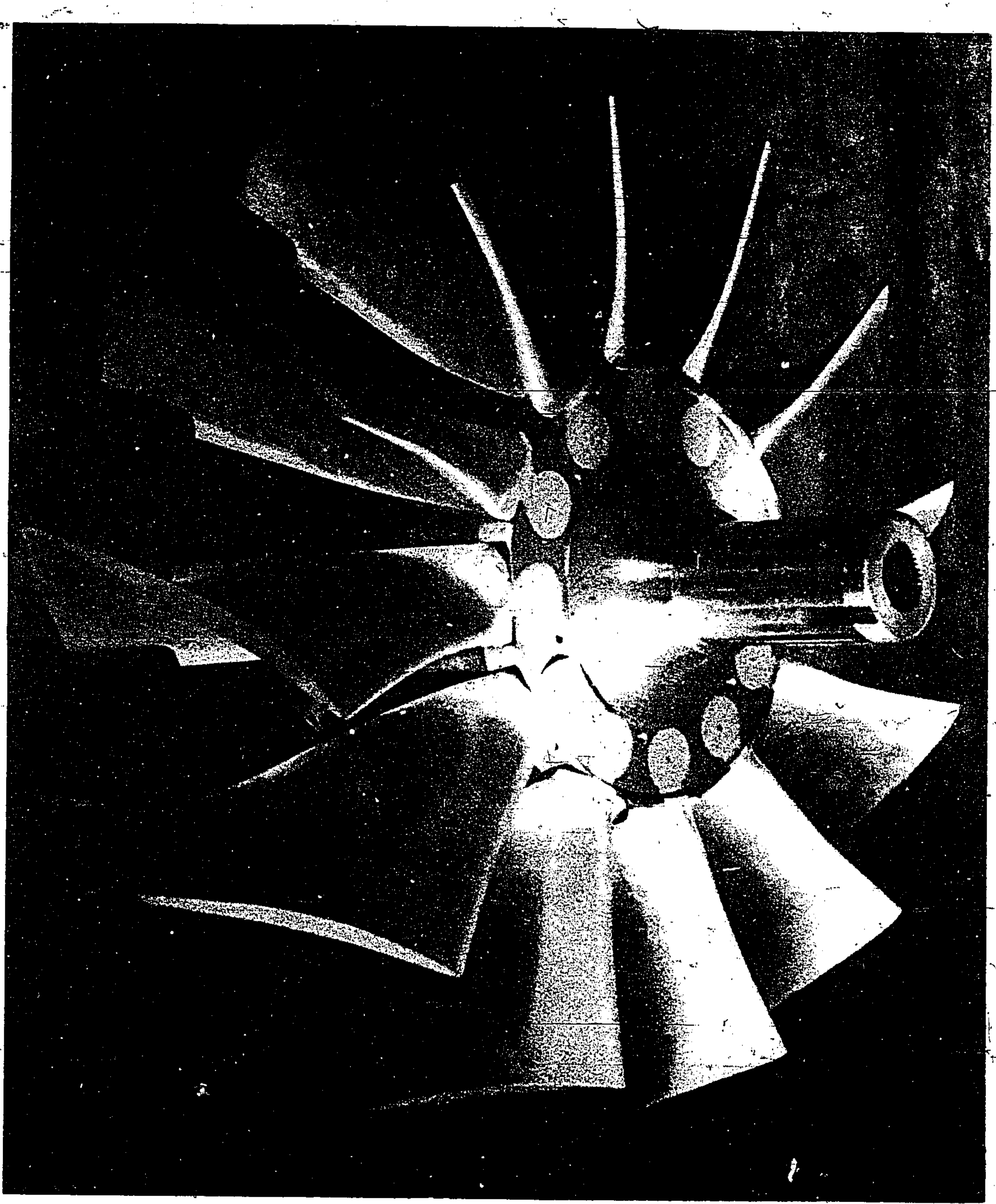
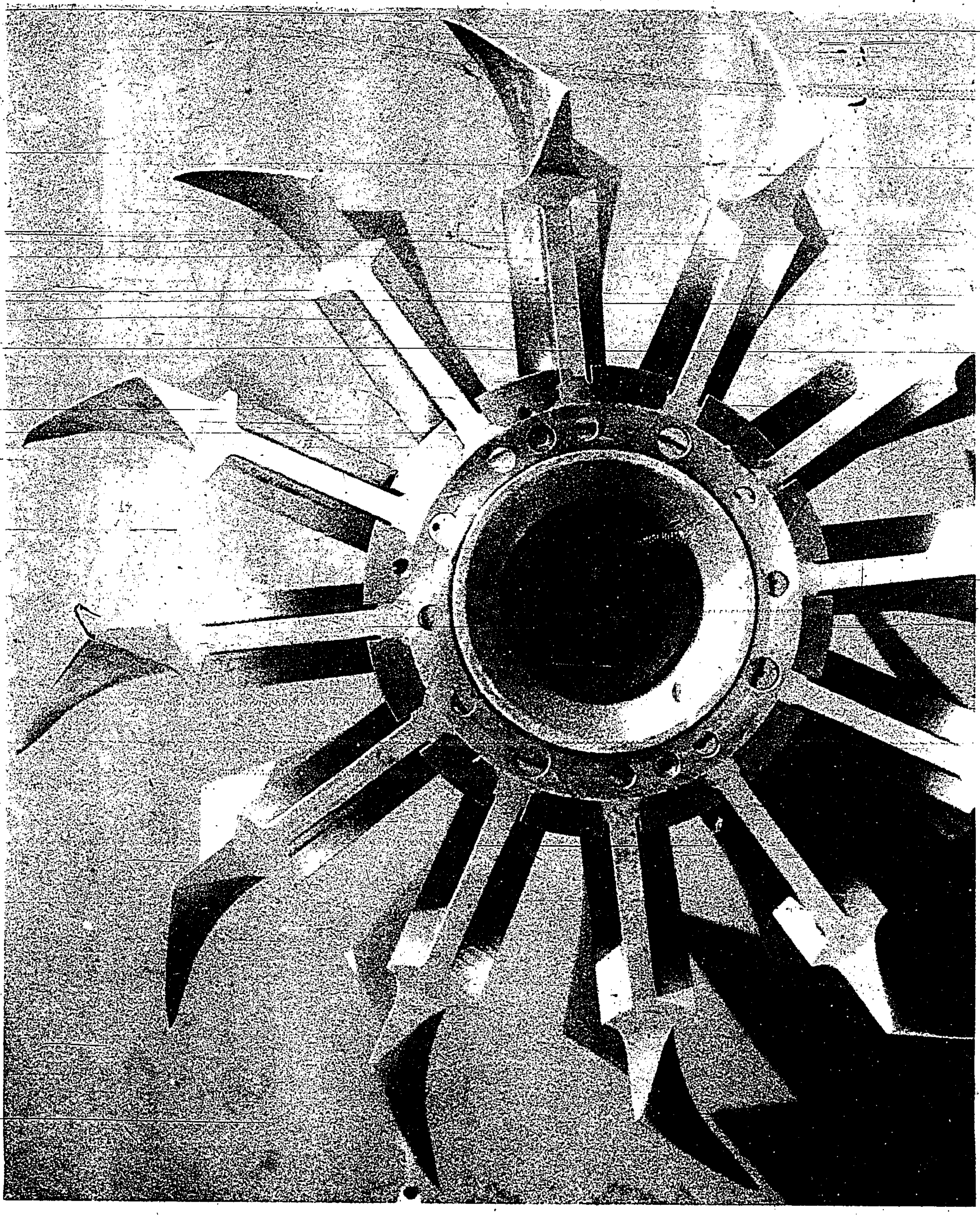


FIG. 6



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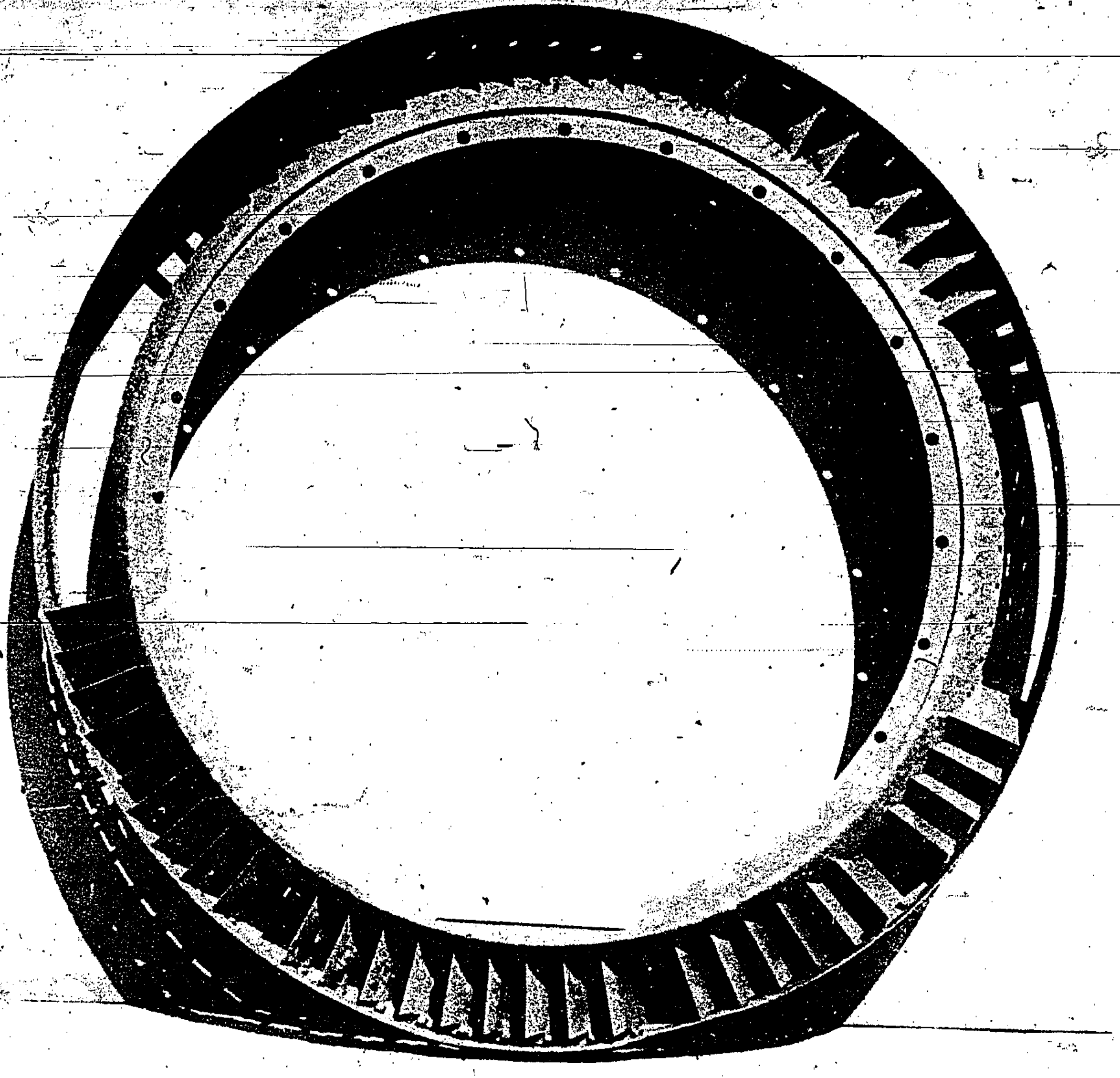


FIG. 8.

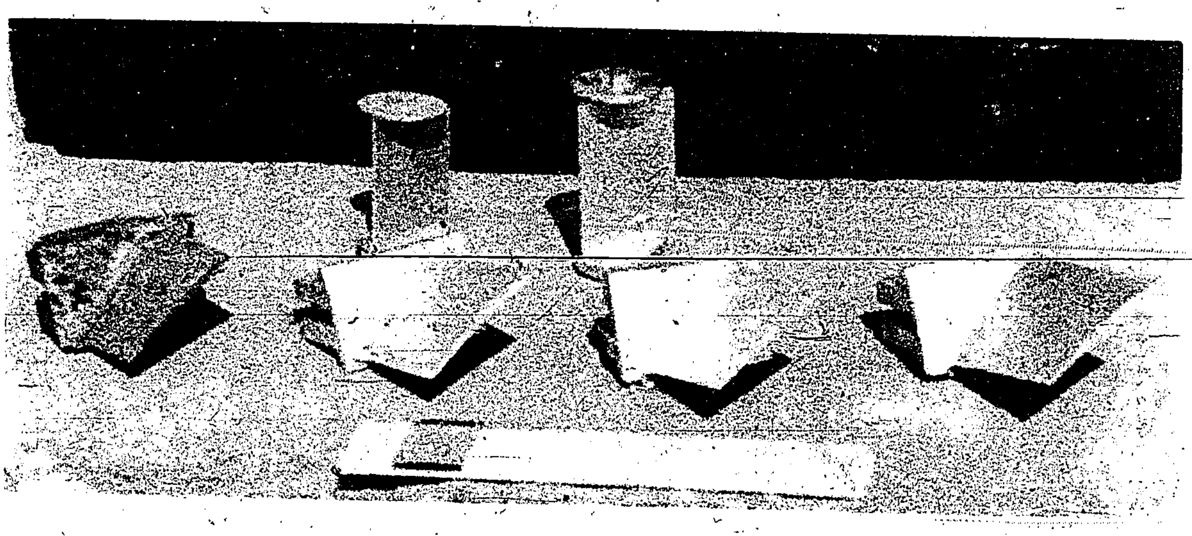


FIG. 9.

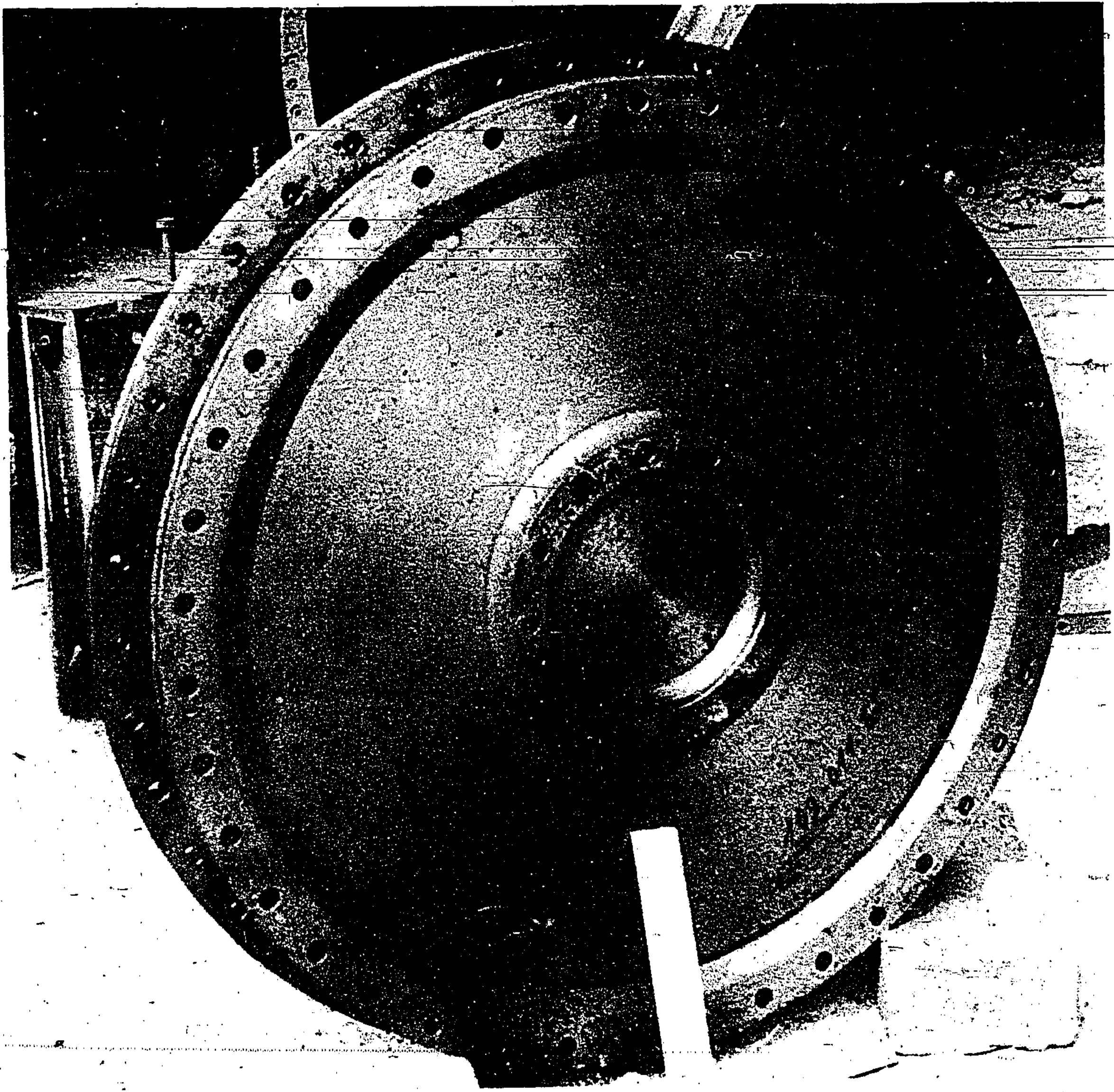


FIG. 10.

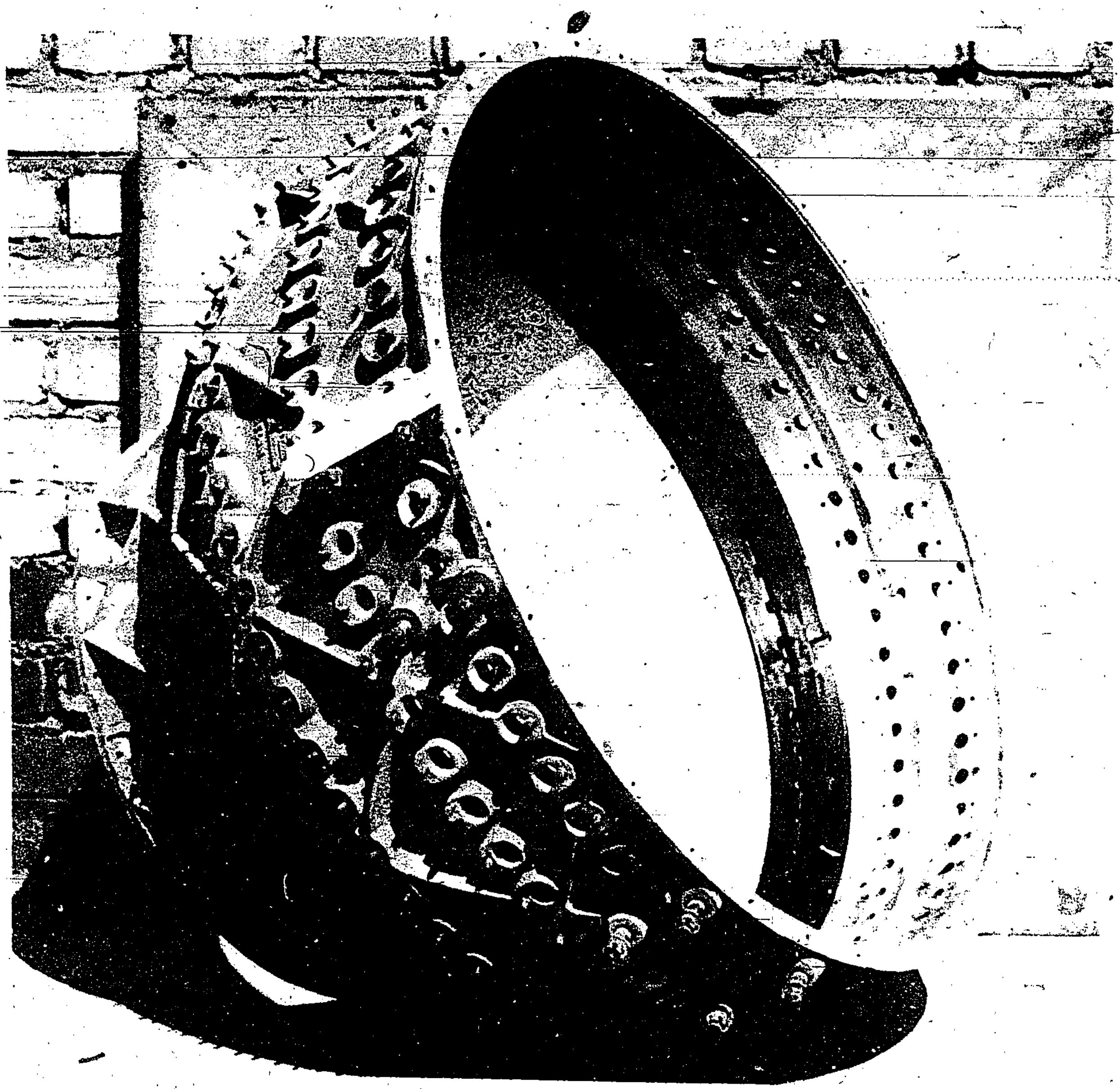
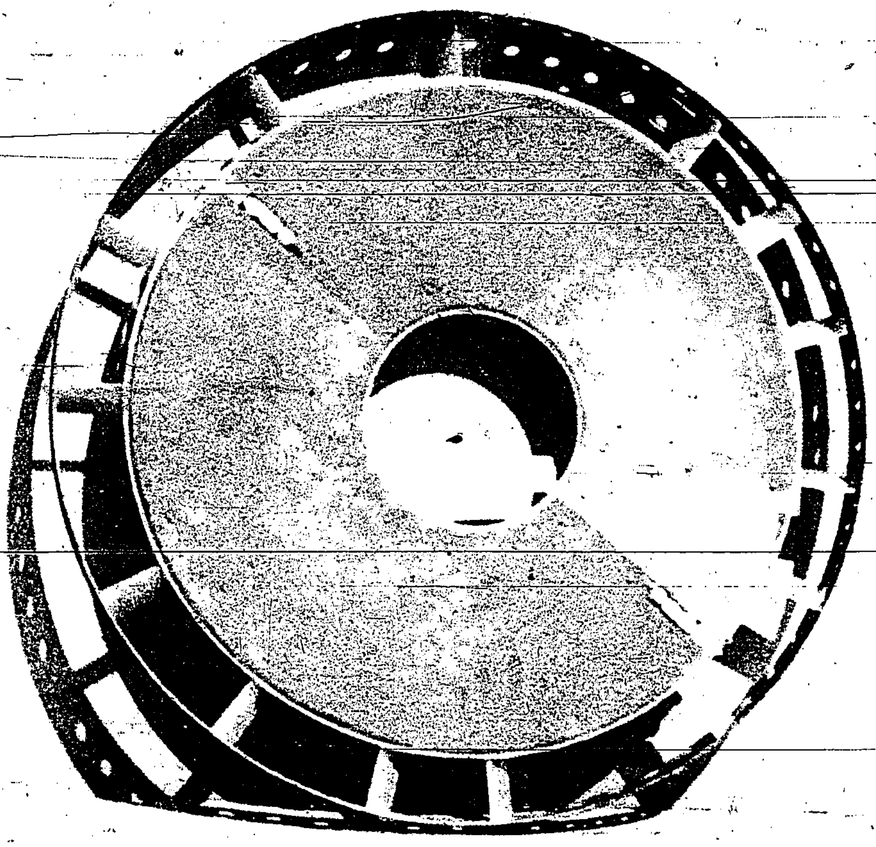


FIG. 11.



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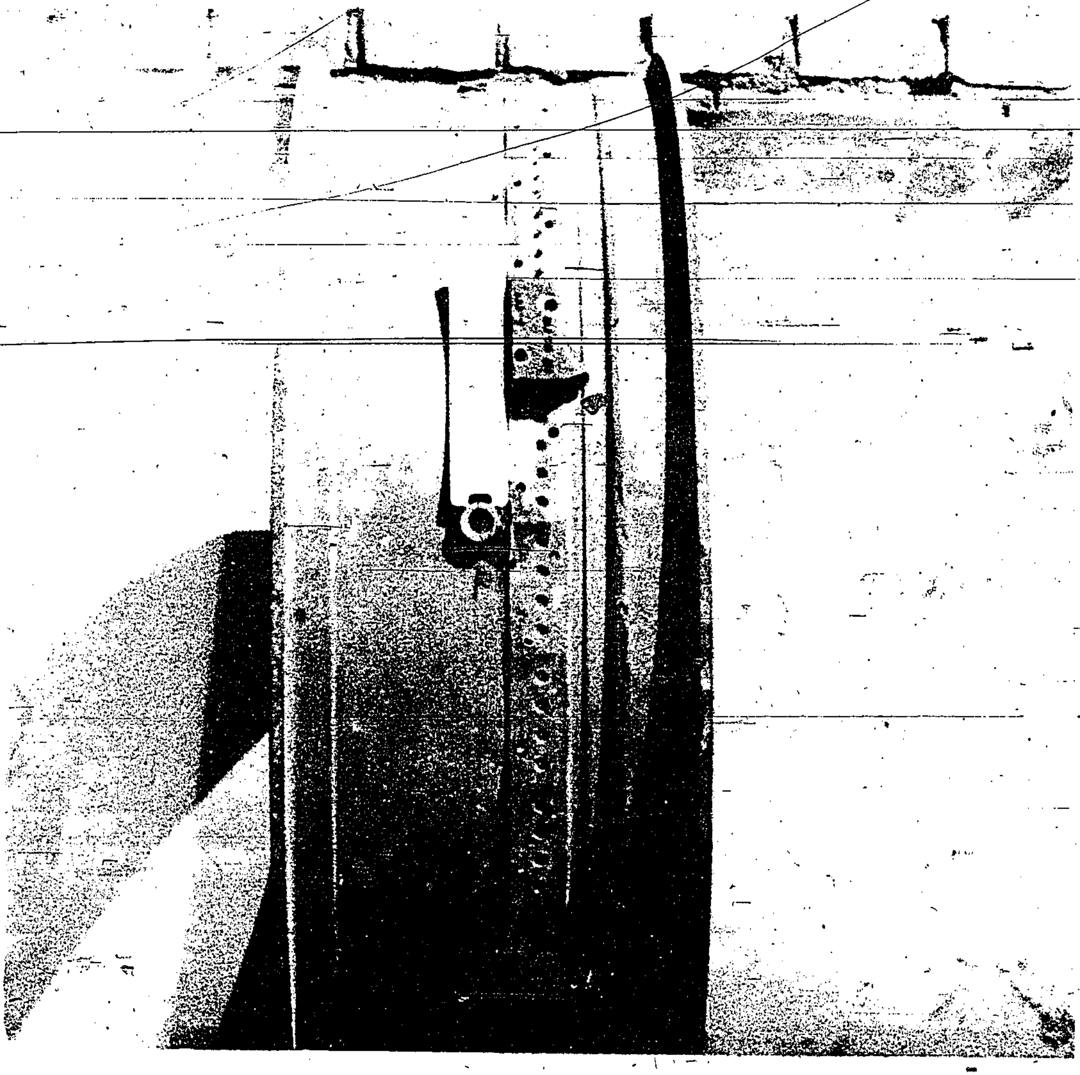

FIG. 13.

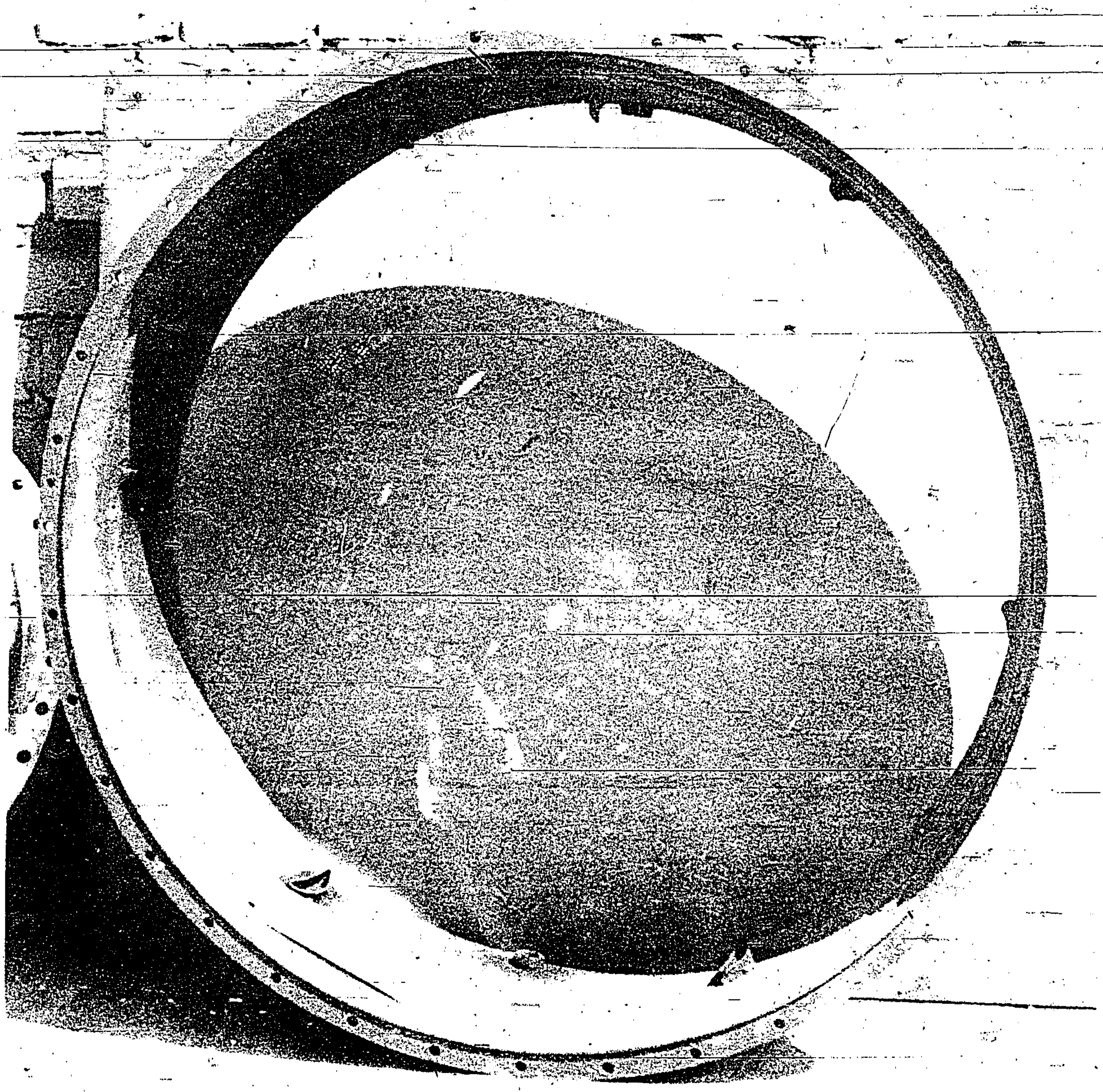

FIG. 14.

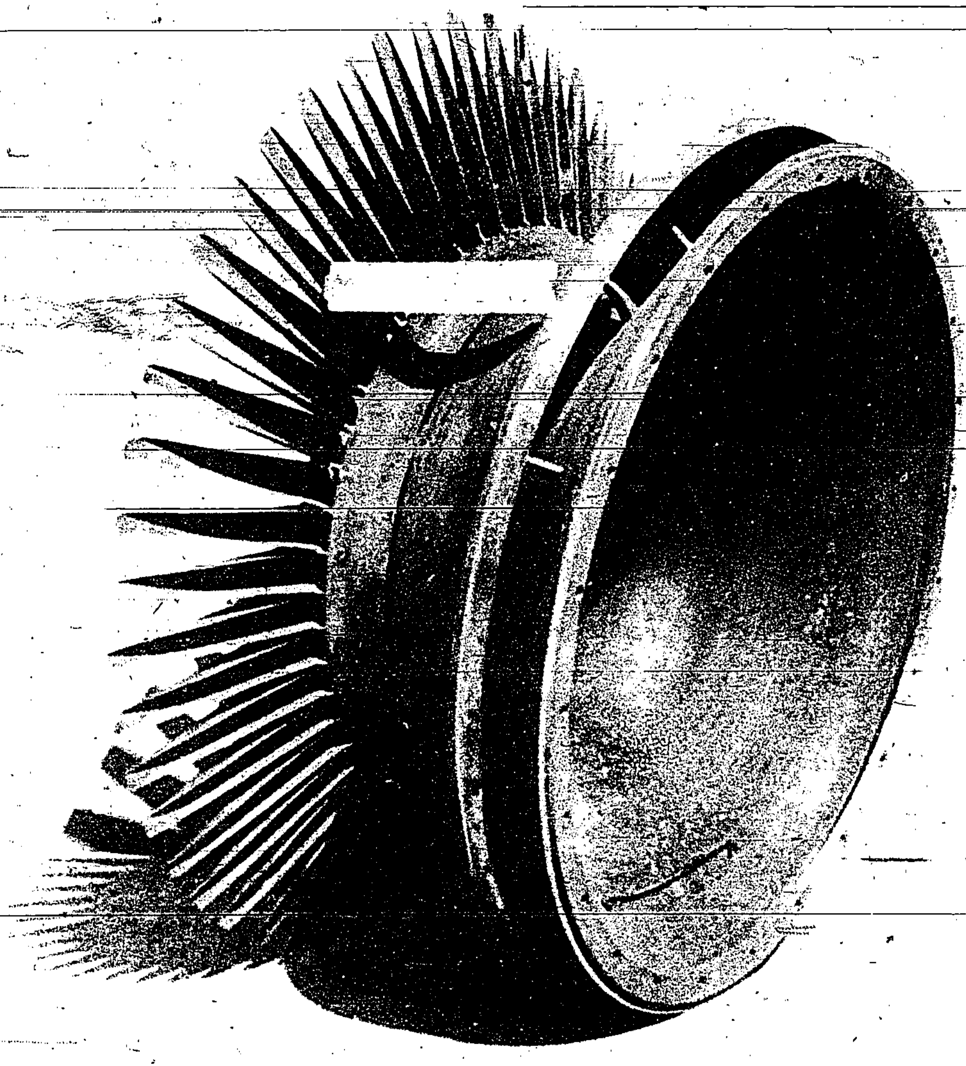

FIG. 15.

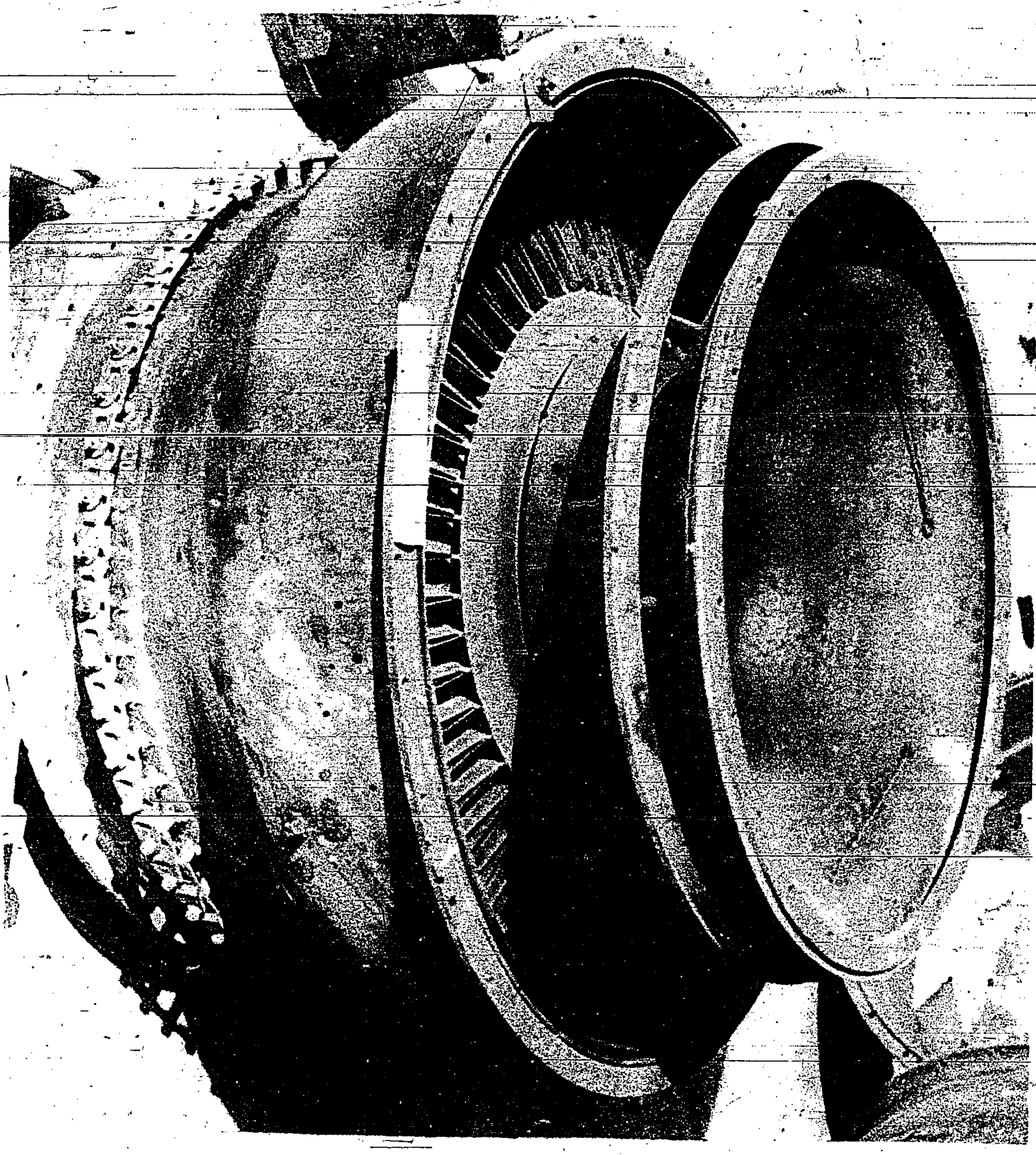

FIG. 16.

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ITEM No. 30

FILE No. XXXIII—17

COAL DRIERS

BÜTTNER-WERKE A.G. UERDINGEN - KREFELD

Bardgett, H.

This report is issued with the warning that, if the subject matter should be protected by British Patents or Patent applications, this publication cannot be held to give any protection against action for infringement.

COMBINED INTELLIGENCE OBJECTIVES

SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

REC'D. JUL 1946

TIIC L.F. & L. S-

Coal Driers

Büttner - Werke A.G.

Uerdingen-Krefeld

reported by

H. BARDGETT (British)

on behalf of

British Ministry of Fuel and Power

and

U.S. Technical Industrial Intelligence Committee

C.I.O.S. Target Nos. C30/372, C31/846

Fuels and Lubricants

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

G-2 Division, S.H.A.E.F. (Rear) A.P.O. 413

(1945)

7 p. diagr.

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Personnel of Team

H. BARDGETT, Br.	Ministry of Fuel and Power
F. H. REED, U.S.	Bureau of Mines
L. D. SCHMIDT, U.S.	Bureau of Mines

Coal Driers

Büttner - Werke A.G.

Uerdingen-Krefeld

Personnel interrogated:

Hans Giesswein, Director
Dr. Carl Glinke, Director
Ober-Ing. Gustav Zapp

Date of visit: 1st August, 1945

Object of visit: To obtain data relating to driers for coal and to inspect apparatus.

Report of visit:

This firm specializes in the design and production of several types of driers for coal, chemicals, etc. Two small-scale turbine type driers for the drying of coal and one turbine drier for chemicals were inspected.

Three pamphlets obtained from the staff describe and illustrate the various types of drier produced by the firm, and the diagram appended to this report illustrates the construction and operation of their Turbine type (Turbinen-Ringscheiben) of steam-heated drier suitable for coal. These pamphlets are listed as items I. II. and III. in the list of documents returned to London.

In this turbine type drier coal for drying, fed at the top, passes downwards on a series of horizontal metal discs mounted in a vertical cylindrical metal shell in such a manner that they can be rotated. Each disc has a series of radial slots through which the coal is transferred to the disc below, the transfer being effected by fixed scraper plates. As the discs rotate saw-toothed levelling arms spread the coal to a uniform depth on the discs. The discs are rotated at a speed of the order of 1 revolution in three minutes, but the speed can be varied depending on the moisture content of the coal. Mounted on a central vertical shaft are three centrifugal fans, or turbines, rotated at a speed of about 2 metres per second, used for circulating heated air or gases over the coal on the discs. In one type of drier heating of the air is effected by means of a series of steam pipes mounted vertically near the outer edges of the discs. In other types the heating is by the products of combustion of gas or solid fuel burned in an external combustion chamber.

The dimensions of driers of this type are approximately as follows:-

		Experimental Scale	Commercial Scale
Capacity tonnes/hr.		1½	50 - 60
Height	metres	2.4	10
Diameter of discs	"	2	7 - 8
Diameter of turbines	"	1	4½
Drying surface	sq. metres	40	1,000 - 1,100

The experimental scale drier seen at the works had thirteen metal discs about 8 inches apart, and the slots in the discs were about 1½ inches wide. The coal layers are of uniform thickness of 1½ inches.

The maximum temperature in the steam-heated plants is 120 - 130°C., and in those heated by combustion gases it is normally 220 - 250°C. The Company claim that in a drier of this type the temperature of the dried coal can be controlled to within 2°C. For example, when drying from 12 to 2% moisture the temperature of the dried coal leaving the plant may be 95°C. plus or minus 2°C.

In plants heated by gas and by solid fuel, the oxygen contents of the waste gases are about 5% and 8%, respectively.

Driers have been designed for the treatment of coal of different sizes, e.g. 40 mm. or 0 - 1 mm. The firm claim that one of the important features of this turbine type drier is that there is no loss of dust and no auxiliary de-dusting apparatus is necessary. This is due to the low velocity of drying gases through the plant. They also claim that there is no appreciable degradation of size of coal during drying. In a plant designed for use on Italian lignite of 40 mm. size, they guaranteed the product to contain less than 5% below 3 - 4 mm.

The heat requirements are 1,100 kcal. per kg. of water evaporated. The power requirements are said to be low, in the experimental model seen 1 H.P. was required.

Examples of plants designed for coal drying are as follows:-

A plant to dry 30 tonnes per hour of Upper Silesian coal of size 1 - 20 mm. from 12% to 2 - 3% moisture. The same plant would dry 50 tonnes per hour from 6% to 3% moisture. The consumption of producer gas on such a plant was 2,300 Nm³ of producer gas of calorific value 1,550 kcal. per Nm³. A plant was designed to treat coal of 0 - 1 mm. containing 50% below 0.5 mm. No de-dusting had been necessary on this plant.

The cost of an installation to treat 50 - 60 tonnes of coal per hour was said to be of the order of 12,000 - 15,000 R.M.

The Company had also designed a plant at Heide, between Hamburg and Kiel for the treatment at 300°C. of oil chalk (Ol-Kreide) for the removal of oil.

Details of drying installations for which drawings were obtained - items IV. V. and VI. in the list of documents - are as follows:-

1. Near Halle - Brown coal of size 0 - 4 mm. dried from 15% moisture to nil. Capacity 30 tonnes per hour. Drawing 81868.

2. Oelsnitz-Zwickau - Low grade bituminous coal of size 0 - 8 mm. dried from 18 to 3% moisture. Capacity 12 tonnes dried coal per hour. Guaranteed fuel consumption 2,310,000 kcal./hr. equivalent to 400 kg. coal per hour. Drawing 85518.

3. Florence - Italy -- A plant designed but never built for drying 1,860 tonnes of lignite per day from 50 to 20% moisture. Each drier 12,780 kg. dried coal per hour. Fuel consumption for each drier 4,800 kg. coal per hour of calorific value 1,600 kcal. per kg. Drawing 88860.

List of Documents obtained from
Büttner - Werke A.G.

I. Kohle-Trocknung und Kühlung

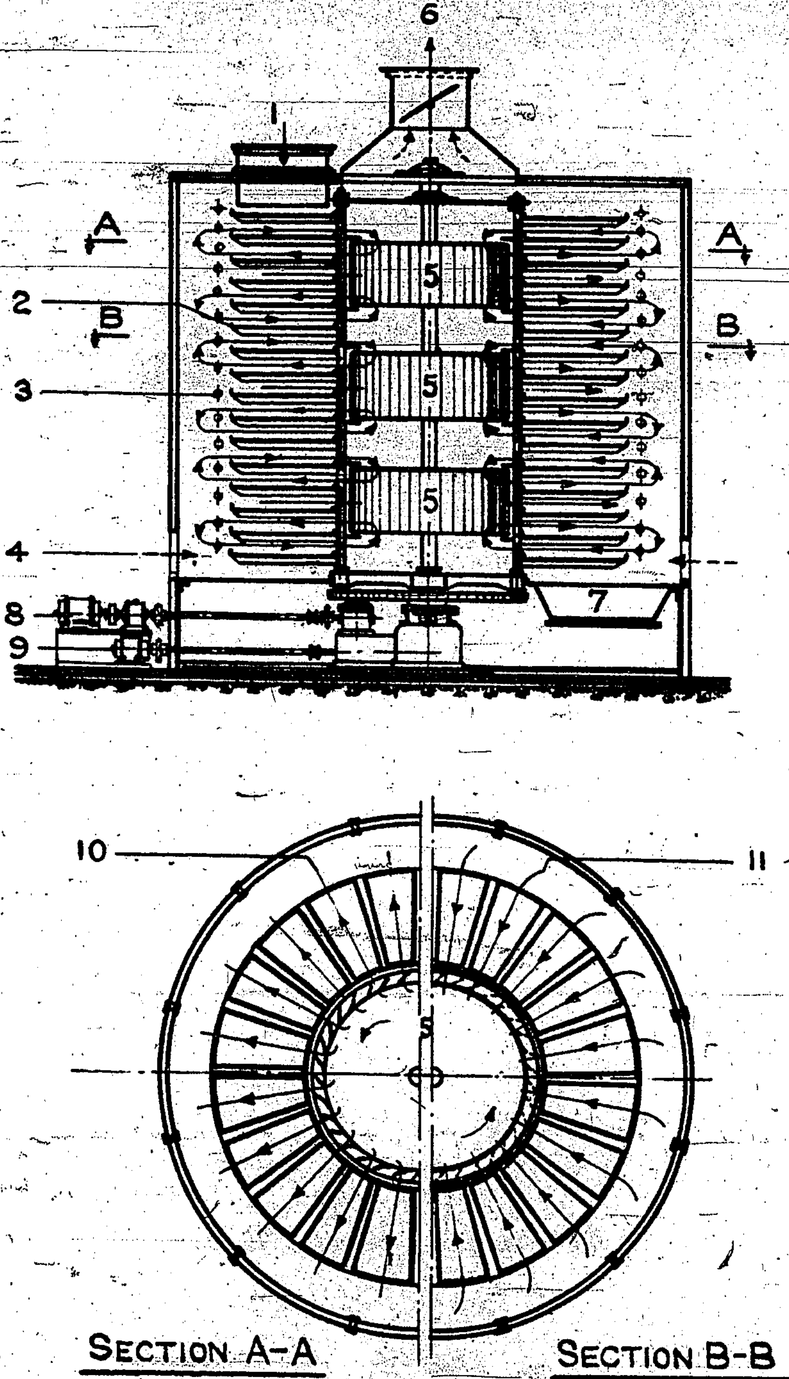
II. Der Turbinen Trockner

III. Farben-Pasten-Schlamm im Büttner Turbinen Trockner

IV. Aufstellungsplan des Büttner Vert. Turbin-Trockner Oelsnitz

V. Aufstellungsplan einer Lignit Trock. Anlage - Florence

VI. Anordung der V.B. Anlage der Grube Elisabeth - Mücheln



1: Wet coal inlet.
2. Ring-disc frame work.
3. Heating coils.
4. Air inlet.
5. Turbines.
6. Waste air outlet.
7. Dry coal outlet.
8. Ring disc drive.
9. Turbine drive.
10. Forward current.
11. Return current.

DIAGRAM OF STEAM-HEATED TURBINE-RING-DISC DRIERS.

BÜTTNER – WERKE A.G.

Copy 1

ITEM NO. 30.

FILE NO. XXXIII—30

STICKSTOFFWERK - HIBERNIA
WANNE - EICKEL - RUHR
Recovery of Hydrocarbons from Coke-Oven Gas

Bardgett, H.

This report is issued with the warning that, if the subject
matter should be protected by British Patents or Patent
applications, this publication cannot be held to give any
protection against action for infringement

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

STICKSTOFFWERK - HIBERNIA

WANNE-EICKEL - RUHR

Recovery of Hydrocarbons from Coke-Oven Gas

Reported by

H. BARDGETT (British)

on behalf of

British Ministry of Fuel and Power

and

U.S. Technical Industrial Intelligence Committee.

C.I.O.S. Target No. C30/380.

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

G - 2 Division, S.H.A.E.F. (Rear) A.P.O. 413.

(1945)

8p. diagrs

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Subject

Personnel of Team.

H. BARDGETT, British.	Ministry of Fuel & Power
F.H. REED, U.S.	Bureau of Mines.
L.D. SCHMIDT, U.S.	Bureau of Mines.

Stickstoffwerk - Hibernia

Wanne-Eickel - Ruhr

Personnel interrogated: Dr. E. Rindtorff, Director.
Dr. H. Benneissen.

Date of visit: 30th July, 1945.

Condition of target: Twenty-five per cent. destroyed and not in operation at the time of the visit.

Description of process.

The visit was made to obtain information relating to the recovery of hydrocarbons from coke-oven gas.

The Company operate a process for the recovery of hydrocarbons from coke-oven gas by liquefaction and for the production of synthetic ammonia and ammonium fertilizers. The plant was designed and erected by the firm Uhde of Dortmund, from whom it was said full details could be obtained. The plant has a capacity for treating about 800,000 Nm^3 of coke-oven gas per day, and can fix about 150 tons of nitrogen per day, including nitrogen from air separation.

A flow sheet of the process is shown in the attached diagram.

Coke-oven gas taken from the grid is passed first to a 20,000 Nm^3 holder, and is then compressed to a pressure of 12 atmospheres in 3-stage reciprocating type compressors of capacity 5300 and 8000 Nm^3 per hour. The compressed gas passes to a Linde liquefaction plant where all the constituents other than nitrogen and hydrogen are liquefied at a temperature of minus 200°C.

Air for separation is first dried by cooling with liquid ammonia to minus 35°C. at atmospheric pressure. This is accomplished by passing the air through vessels containing nests of tubes of 5 mm. internal diameter in which the water is frozen out. Particles of ice carried forward in the air are removed by means of wire gauze filters of aperture 0.001 mm., which also remove other solid material and oil. The dry air compressed to 120-150 atmospheres is separated in a series of ten Linde separators, seven of which are normally used each of capacity 10,000 Nm^3 per hour. The oxygen is bottled and sold. The separated nitrogen compressed to 150 atmospheres is fed to the coke-oven gas liquefiers and provides the additional source of cooling, and together with nitrogen and hydrogen from the coke-oven gas provides the mixture for ammonia synthesis. Residual gas from the coke-oven gas liquefiers amounting to about 40 per cent. of the total is returned for heating coke ovens.

Purification of gas

The coke oven gas is purified from CO_2 and H_2S prior to liquefaction. The H_2S is removed by an iron hydroxide process described by Gluud and Schonfelder in Stahl u.Eisen 1927, p.453, but the management considered this process to be not very satisfactory. The CO_2 is reduced from 2 to 0.25 per cent. by water washing under a pressure of 12 atmospheres, and the remaining CO_2 is then removed by washing with caustic soda lye.

Ammonia synthesis.

Ammonia is synthesised by passing the mixed nitrogen and hydrogen through a contact oven containing a catalyst of magnetite of size 2 - 12 mm., activated with traces of vanadium oxide. The magnetite was said to be obtained from Norway and to be similar to that used in America.

Products.

The products from the liquefaction process are as follows:-

1. Ruhrgasol - This is recovered at - 110°C. (12 atmospheres), is bottled at 6 - 8 atmospheres and sold as motor fuel.

2. Ethylene product - Recovered at minus 145 - 150°C. (12 atmospheres), is used for synthesis of Buna rubber. This product is piped at relatively low pressure to I.G. and Buna rubber producers.

3. Methane product - This is of somewhat similar composition to the ethylene product. It is compressed to 20 atmospheres in bottles and sold for motor fuel.

In addition to the above products the Company bottle and sell the oxygen from the separation of air, and bottle hydrogenation gases purchased from Hydinerwerke-Scholven (Hibernia). The latter gas produced by liquefaction consists approximately of 90 per cent. propane and 10 per cent. butane, and is sold as bottled gas for household purposes. The amount of gas taken from H-S, is about 150 tonnes per month.

Data for a typical working day.

Coke-oven gas received:

Quantity	Nm^3	735,000					
Density	gm./Nm^3	505					
Analysis	%	CO_2	2.0	O_2	1.0	CH_4	19.6
		H_2S	0.5	CO	6.0	C_2H_6	4.9
		C_2H_4 + C_3H_6	2.0	H_2	54.0	N_2	10.0

Products.

Ruhrgasol:

Quantity	tonnes	1.0					
Analysis	%	C_2H_6	6	C_3H_6	34	C_5H_{10}	6
		C_2H_4	14	C_4H_{10}	8	CH_4	3
		C_3H_8	11	C_4H_8	18		
Selling price		380 RM per tonne					

Ethylene product:

Quantity	tonnes	10					
Analysis	%	O_2	2.0	C_2H_6	19.5	N_2	5.0
		CO	3.0	C_2H_4	33.0		
		H_2	1.6	CH_4	35.9		
Selling price		70 RM per tonne					

Methane product:

Quantity	tonnes	13					
Analysis	%	0.2	1.8	H_2	4.4	C_2H_4	27.8
		CO	6.0	CH_4	53.4	N_2	6.6
Selling price		340 RM per tonne					

Rest Gas:

Quantity	Nm3	320,000					
Analysis	%	CO_2	3.0	CO	13.0	N_2	22.0
		C_2H_4	3.2	H_2	9.4		
		O_2	1.4	CH_4	48.0		
Density	gm./Nm^3	909					
Selling price		0.027 RM per Nm^3					

Oxygen (unpurified)

Quantity Nm^3 40,000 = 57 tonnes

Purity % O_2 92

Selling price 0.019 RM per Nm^3

Oxygen (purified)

Quantity Nm^3 1,200 = 1.7 = 1.7 tonnes

Purity % O_2 99.2

Selling price 0.37 RM per Nm^3

Power for gas compression

Coke-oven gas	93,000 KWH.
Air	40,800 "
Nitrogen	79,000 "
Mixed gas ($3H_2 + N_2$)	66,000 "
Methane for cylinders	3,400 "
Oxygen " "	310 "

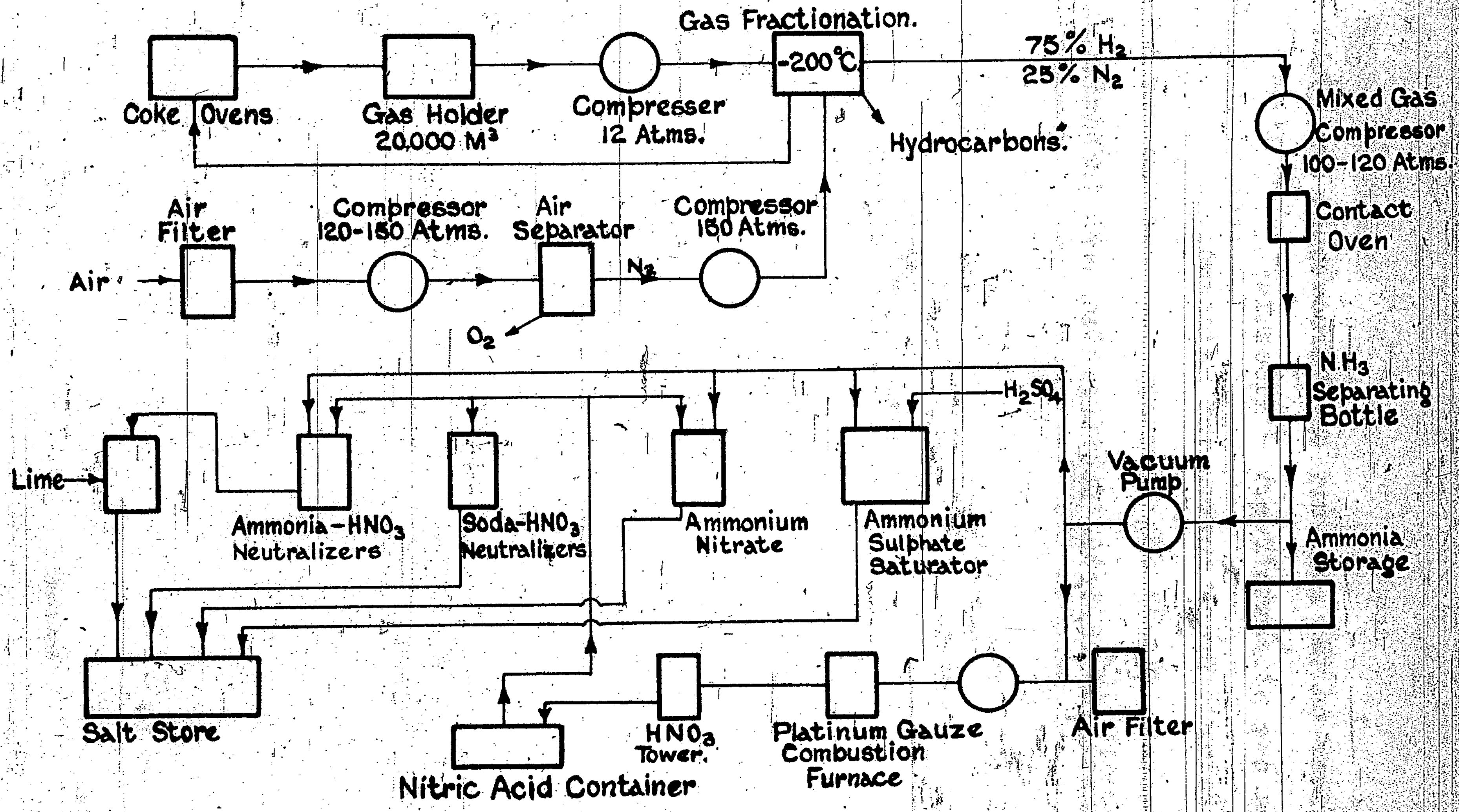
Cost of Power 0.023 RM per KWH.

Labour employed in above operations 120 men (24 hours).

Average wage 0.88 RM. per hour.

List of Documents Obtained from Stickstoffwerk - Hibernia, Wanne-Eickel.

1. Questions on the Recovery of Hydrocarbons from Coke-Oven Gas.
2. Schema des Stickstoffwerk - Hibernia.
3. Temperatur - Druck Kurven von Reingasol.
4. Fliesschema für de NH_3 Synthese aus Koksofen gas + Luft.
5. Reingasol vom 18/5/36. Destillation nach Podbielniak.



FLOW SHEET OF PROCESS-HYDROCARBON RECOVERY AND PRODUCTION OF FERTILIZERS AT STICKSTOFFWERK-HIBERNIA.

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ITEM NO. 30.

FILE NO. XXXIII—29

SULPHUR RECOVERY FROM SPENT PURIFIER OXIDE

Ruhrgas A.G.,

HERWARTH STRASSE, ESSEN

Bardgett, H.

This report is issued with the warning that, if the subject matter should be protected by British Patents or Patent applications, this publication cannot be held to give any protection against action for infringement

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

Sulphur Recovery from Spent Purifier Oxide

Ruhrgas A.G.

Herwarth Strasse, Essen.

Reported by

H. BARDGETT, (British),

on behalf of

British Ministry of Fuel and Power

and

U. S. Technical Industrial Intelligence Committee

C.I.O.S. Target No. 30/6.11.

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

G-2 Division S.H.A.E.F. (Rear) A.P.O.413

(1945)

7p. diagr.

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Personnel of Team

H. BARDGETT, British Ministry of Fuel and Power
F.H. REED, U.S. Bureau of Mines
L.D. SCHMIDT, U.S. Bureau of Mines.

Sulphur Recovery from Spent Purifier Oxide

Ruhrgas A. G. - Herwarth Strasse, Essen

Personnel Interrogated : Dr. K. Traenckner, Director
Dr. F. Summert, Commercial Director

Date of Visit : 28th July, 1945

Object of Visit : To obtain information relating to the Ruhrgas process of recovery of sulphur from spent purifier oxide.

General.

Ruhrgas A.G. is a company formed by mine-owning companies in the Ruhr and its main purpose is the distribution of gas to the grid supplying Hanover, Berlin, Magdeburg, Leipzig, etc. They do not produce gas but buy it from the various coking installations and distribute it after purification.

Dr. Traenckner stated that 70,000 tonnes per year of iron oxide were required for purification of gas in the Ruhr area and that during the war the problem of obtaining adequate supplies had been acute. As a result of this difficulty considerable attention had been devoted to the extraction of sulphur from spent oxide with the object not merely of recovering the sulphur but to recover supplies of oxide which could be used again. The process of sulphur recovery had been operated at a profit until 1933 when the American dollar was devaluated from 104 to 66 R.M. This devaluation ruined the German market for sulphur and made it unprofitable to produce and sell the sulphur from spent oxide. Nevertheless sulphur recovery was still practised in order to reduce the amount of fresh oxide which it was necessary to obtain, principally from Holland.

Process.

A brief description, supplied by the company, of the process of recovering sulphur from spent oxide by washing with CS_2 is attached together with a flow sheet. (See Appendix)

According to Traenckner the process had been developed by Dr. Jacob from Kreuznach near Bingen, whose firm makes CS_2 and has a small scale plant. The first large-scale plant was built at Gelsenkirchen Horst, and the second at Herten the respective capacities being 12,000 and 6,000 tonnes of sulphur per year.

For a plant producing 20 tonnes of sulphur per 20 hours the quantity of CS_2 in circulation was said to be 4-5 tonnes and the amount of make-up necessary was 2-3 Kg. of CS_2 per 100 kg. of sulphur The refined sulphur produced was of 99.9 per cent purity and sold at 110 RM. per tonne.

The cost of a plant of capacity 6,000 tonnes per year was said to be 500,000 RM.

Oxide for purification, containing 45 per cent water, cost 20-22 RM. per tonne. Such oxide was normally used until it contained 45 per cent sulphur at which stage it was removed and the sulphur recovered, the recovered oxide being used again. About 95 per cent of the sulphur was recovered from the spent oxide. The process could normally be repeated twice but if compounds such as $FeSO_4$ and HSCN, formed during purification, were washed out, the oxide could be used again three or four times.

A plant at the Manchester Oxide Corporation in England was said to use this process and it was said that the Sheffield Gas Company had intended to operate the process but the plant had not been erected.

Appendix

Description of the Extraction Process of the Ruhrgas A.G.

General.

The problem of supplying mass for gas purification and of disposing of the mass containing sulphur became very important with the development of long distance gas supply. The demand for fresh mass and with it the output of exhausted mass rose with great rapidity. With the strongly increased demand for mass and the deficiency of the supply of it the price for the fresh material rose and the sale price of the exhausted material containing sulphur fell to such an extent that the economy of gas supply was seriously predjudiced. The Ruhrgas A.G. was consequently obliged to work up the resulting sulphur-containing mass in its own centrally situated plant for the production of elementary sulphur and to send back the desulphurised mass when it was appropriately prepared again to the coke ovens supplying gas. These considerations gave rise to the idea of erecting a sulphur extracting plant with preparation of mass and the idea was first realised in the years of 1930-31.

Description of Process.

The process depends on the use of carbon disulphide as an extracting medium, it being a well known solvent for elementary sulphur. The toxicity of carbon disulphide and the danger of fire and explosion that are involved in operations with this solvent made special precautionary measures necessary in the construction and building of the plant. Briefly the process works as follows:

The gas purification mass containing sulphur and produced at the coke ovens during the desulphurisation of the coke oven gas is transported by rail to the centrally situated sulphur extraction plant in Horst. The course of the process can be followed by reference to the attached diagram. The sulphur containing mass is transported from the wagons 1 as they arrive by bucket conveyors and transporters to storage site 2. From here the material passes to the water extraction 3 using the said transporters; in this the mass is freed from water soluble sulphates and other admixed bodies that might disturb the process. It is then taken directly to a drying drum 4 in which it is dried to a constant water content. The maintenance of a definite water content is of special importance for the extraction which immediately follows. The material so dried comes out of the drying drum into the extractor plant 5 which consists essentially of 10 extractors. These extractors contain trays with reticulate bottoms on which the mass rests and in which carbon disulphide flows through it. The carbon disulphide passing through dissolves the elementary sulphur contained in the mass, and also removes the tar that is present at the same time.

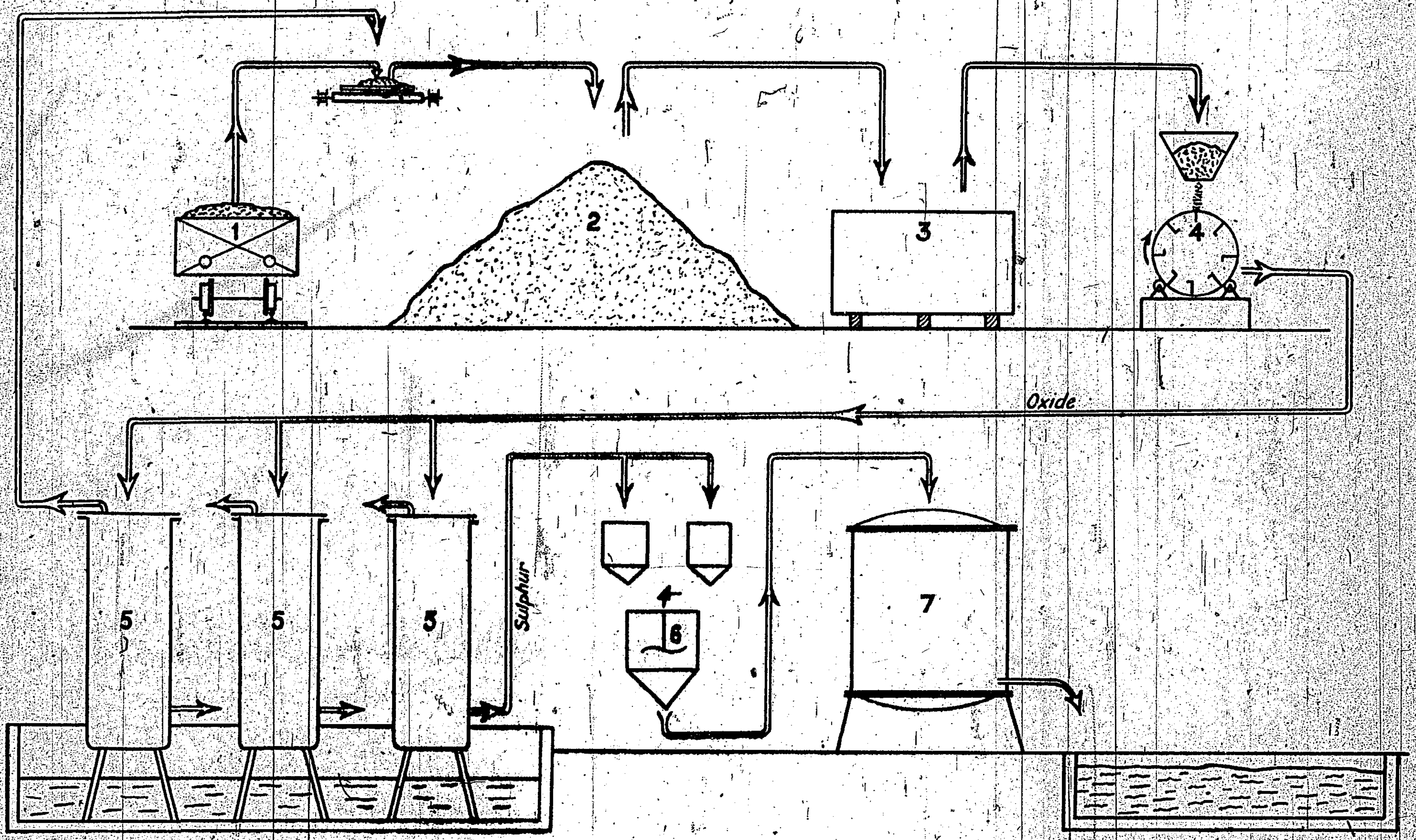
The result is a dark brown to black solution of sulphur and tarry constituents in the carbon disulphide. Details about the further treatment of this solution are attached. After the solvation process is ended the mass free from sulphur is freed from residual carbon disulphide by steam blown through it. As soon as this process is concluded the extractors 5 are emptied and the sulphur free mass passes back via the transporters already referred to to the storage site 2. After this mass has been prepared in a appropriate manner and some fresh mass consisting of Luxmasse and terrana iron-ore is added to it the material is ready for use again in gas purification and passes along the route by which it has come back to the coke ovens supplying gas.

The crude solution, as it is called, issuing from the extraction plant 5 is directly worked up for elementary sulphur. This is done by distilling off the carbon disulphide from the crude solution in the distillation plant 7 where, when the solvent is completely removed, elementary sulphur remains behind. The elementary sulphur so obtained is run off in the liquid form into sulphur vats from which it is broken out after it has cooled and loaded on rail.

During the distillation of the crude solution the crude sulphur appears with a dark grey colour as the tarry constituents remain in it.

For the production of bright yellow pure sulphur it is necessary to remove the crude solution from tar. This takes place in a refining plant 6 in which the crude solution is washed by a special process with Oleum. The solution so treated is of a bright yellow colour and after distillating in the plant 7 gives a light yellow pure sulphur with a very high degree of purity say with 99.8 - 99.9% pure sulphur content.

On account of the said dangers involved in operations with carbon disulphide it is necessary to ensure the tightness of the whole of the plant with the greatest degree of certainty and to keep the staff continually attentive to operational dangers. The buildings in which work with carbon disulphide is carried on are fitted with special spray equipment for combating any fires that may occur. Further, the extraction vessels 5 are erected in a common trench which is always kept filled with water so that any carbon disulphide that flows out is immediately caught under water.



Flow Sheet, Sulphur Recovery Process.

Ruhrgas A. G. Essen.

United States CONFIDENTIAL
equals British CONFIDENTIAL

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ITEM No. 22
FILE No. XXII—1

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Unclassified 10/15/45

CHEMISCHE WERKE HÜLS A.G.

Curtis + Fogler

RECEIVED
4 JUL 1945
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SUB-COMMITTEE

~~CONFIDENTIAL~~

REPORT ON CHEMISCHE WERKE HÜLS A.G.
HÜLS, GERMANY

19 April, 1945

Reported by

F. J. CURTIS, M. F. FOGLER
CWS, HQ ETOUSA CWS, HQ ETOUSA

14 May, 1945

CIOS Black List Item - 22
Miscellaneous Chemicals

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE
G-2 DIVISION, SHAEF (REAR) APO 413

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7 p. diag.

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PERSONNEL OF INSPECTION TEAM

Mr.Francis J.Curtis	CWS, Hq.ETOUSA
Maj.F.E.Davis	MC, Hq.ETOUSA
Cmdr. R.Cannon Eley,	MC, USNR
Cmdr. Chas.L.McCarthy	MC, USNR
Dr.Mayor F.Fogler	CWS, Hq.ETOUSA
Lt.Col.Joseph E. Smadel	MC, Hq.ETOUSA
Lt.Col.Hamilton Southworth	USPHS
Dr.Ernest H.Volwiler	CWS, Hq.ETOUSA

REPORT ON CHEMISCHE WERKE HÜLS A. G. HÜLS, GERMANY

1. GENERAL.

a. Hüls is one of the important synthetic rubber plants in Germany and has been investigated thoroughly from that standpoint. Our interest was confined to acetylene and styrene, although the following chemical products are made in this plant:-

Acetylene
Ethylene (from Acetylene)
Aldol
Acetaldehyde
Kybol
Styrene
Hexanetriol
Solvents A and AH
Acetophenone
Resin SAX (from styrene, acetophenone, xylene, hydrofluoroboric acid)
Ethylene oxide
Glycol
Glycerine D
Triglycol
Resin AP for surface coating (acetophenone, formaldehyde, methanol)
C acid (crotonic)
Plasticizer 111 (butyl crotonate, hydrogen, sulfide)

2. ACETYLENE MANUFACTURE.

The plant produces 150,000 cu meters acetylene per day by arc cracking.

a. Raw Materials

There have been three sources of raw materials:-

(1) Natural gas from Bentheim used directly

92% CH_4
3% $H_2S + CO_2$
5% N_2

(2) Coke oven gas which was first put through the Linde apparatus.

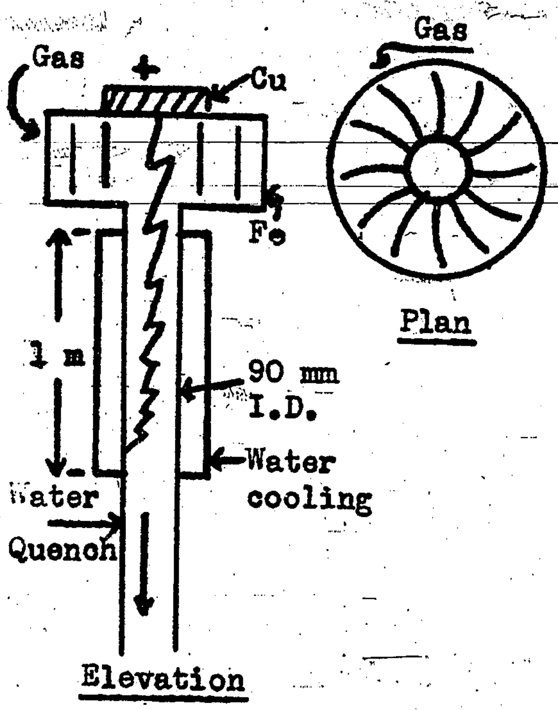
(3) Gas from Hydrierwerke containing 85% hydrocarbons averaging $C_{1.4}$ which was used directly.

b. Power

Power consumption is 8.5 kwh per kg crude acetylene and 9.5 - 10.5 kwh per kg pure acetylene.

AC at 6,000 V 50 cycles is converted in a mercury rectifier to 7,000 V DC at 7,000 kw. Each arc uses 7,000 kw and produces 4,200 cu m/hr product gas. The plant has 17 arc units.

c. Arc Apparatus. (Sketch below)



The gas at 1.5 atmospheres enters the top section at the side and is whirled by the vanes into the arc tube. The arc is not constant in position but rotates around in the tube. Immediately on leaving the arc section, the gases are quenched by water spray to 150°C.

At this point the composition is:-

H_2	55%
C_2H_2	13% (1% diacetylene)
C_2H_4	1%
CH_4 (other H.C.)	23%
CO_2, N_2, etc.	8%

d. About 3% carbon is produced based on the original hydrocarbon. The gases pass to four cyclone separators to take out water and carbon, the latter being used in the rubber industry. Further cooling with spray to 70°C takes place and then the gases go through 8 filter towers equipped with cloth bags. The temperature is then reduced to 25°C and the gases washed with tar oil to remove aromatic hydrocarbons such as naphthalene and polymerized hydrocarbons. To regenerate the tar oil it is blown with waste gases (nitrogen, CO and CH_4) from the Linde plant and a side stream distilled with high pressure steam.

Hydrocyanic acid is removed by water scrubbing in tall towers to form a 3% solution which is distilled to recover HCN. Hydrogen sulfide is then removed by passing thru boxes packed with Luxmass (hydrated iron oxide). The gas now containing 20% C_2H_2 passes to a gasometer.

The gas from the gasometer is then compressed to 18 atmospheres in compressors having 4 stages with intercoolers. There are six compressors each handling 13,000 cu m per hr. At this point the partial pressure of the acetylene is 5 atmos. Temperature is reduced to 15°C and acetylene dissolved using 750 cu m of water per 8,000 cu m of gas. Water is also sprayed into the gas lines to avoid explosions. The washed-off gas contains 0.1% C_2H_2.

The pressure of the aqueous solution is dropped in 4 steps to 1.5 and 0.1 atmospheres and to 0.15 and 0.05 atmospheres absolute. Gas from the first step at 50% C_2H_2 goes back to the compressors after mixing with arc gas causing the rise to 20% C_2H_2 in the gas holder mentioned above.

The gas from the 2nd, 3rd and 4th steps are mixed and give 97 - 98% acetylene of which 87% is C_2H_2 and 10% diacetylene and higher. The diacetylene is washed out with tar oil which is blown with Linde gas and the diacetylene sent through the system again. At this point the product gas contains 95% C_2H_2 with 3% hydrocarbon, 1% nitrogen and 1% CO_2. The last traces of diacetylene are removed with 96% H_2SO_4 and the gas neutralized with 10% NaOH and passed to the final gas holder.

The original off gas from the water solution of acetylene step containing 0.1% C_2H_2 passes at 15 atmos to the Linde plant where it is fractionated to 97% pure hydrogen, a mixture of CO and N_2, and a hydrocarbon fraction which after passing through the diacetylene oil washer is joined with the original gas feed to the arc.

3. STYRENE MANUFACTURE.

a. Ethyl Benzene

Ethylation of benzene takes place in 6 enamelled iron towers (with 2 in reserve) 11 m high x 1.4 m diameter operated continuously but not in series. Each tower handles 350 cu m/hr of 95% moist ethylene from a gasometer. They were originally 3/4 filled with Raschig rings but are not now. Ethylene goes in at the bottom through an open pipe and passes up through the benzene and aluminum chloride. Complete absorption is obtained at 95-105°C with a consumption of 3% $AlCl_3$ on ethyl benzene. No HCl is added since it forms with the moisture of the gas.

The raw ethyl benzene contains:-

35%	ethyl benzene
45%	benzene
15%	polyethylbenzene
5%	residue

It passes to a cooler and separator for the $AlCl_3$ complex which is returned to the bottom of the tower. The new $AlCl_3$ is added periodically at the top mixed with benzene. The overflow from the separator is washed with water and then with 1-2% NaOH in a stoneware lined tank. It is settled and dried with solid caustic soda.

Distillation takes place in a series of towers each with a duplicate so that, for instance, No 3 is the second step in the series, No 5 the third, etc. Towers are about 28 meters high and have 45 plates. All except the last are bubble cap plates and last is packed with Raschig rings. Reflux on the ethyl benzene tower is 1:1.

Tower No	Diameter	Temperature	Vacuum	Product
1	0.8 m	--	--	Carbon Bisulfide
3	1.0	80°C	--	Benzene
5	1.6	--	--	Intermediate
7	1.1	136°C	--	Ethyl benzene
9	0.8	130°C	20-25 mm	Polyethyl benzene
11	--	200°C	25 mm	Residue

Polyethylbenzenes are returned to the ethylator.

b. Dehydrogenation of Ethyl Benzene to Styrene.

The catalyst chambers for dehydrogenation are vertical cylinders in brick settings fired with gas on two sides. The 12 chambers are of various types some having 22 to 26 tubes of 20 cm diameter, others 90 tubes of 10 cm diameter and are 2 m diameter x 3 m high. They contain 2 cu m catalyst of 4 - 7 mm particle size made at Ludwigshafen and of the following approximate composition:-

ZnO	85%
CaO	5%
Al_2O_3	5%
K_2CrO_4	2%
K_2SO_4	2%

Each converter is first swept out with nitrogen, then fed with a 1-1.5 : 1 ratio of steam to ethyl benzene. The feed is vaporized and preheated by heat exchange with flue gases and product gas. Temperature in the converter is 630° and in the product gas 600°C. Each handles 450 kg/hr ethyl benzene with 33% conversion per pass and 90% yield.

After condensation of the crude product 0.001% hydroquinone is added. This is the only addition of inhibitor.

The crude product containing 38% styrene passes into the middle of the first distilling column, a tower 2 m diameter x 25 m high with 45 plates and operated under 20 mm vacuum. Ethyl benzene passes over the top to re-use. The bottoms go into the middle of the second column which is 2 m diameter x 15 m high with 25 plates and operated under 15 mm vacuum. The overhead containing 38% styrene and 62% ethyl benzene is returned to the first column. The crude styrene bottoms testing 102.5% on account of the residues goes into the middle of the first final distillation tower and thence to the second. These towers are 1.1 m diameter x 15 m high, some packed and some plate and run under 15 mm vacuum. 99.5 - 99.8% styrene is taken overhead. There are two sets of two towers for preliminary and the same for final distillation, one presumably for stand-by.

Capacity of the plant is 120 tons/mo per catalytic converter or 1440 tons/mo total.

F. J. CURTIS, M. F. FOGLER
CWS, HQ ETOUSA CWS, HQ ETOUSA

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ITEM No. 30
FILE XXXIII—24.

REPORT ON
INVESTIGATIONS BY FUELS AND LUBRICANTS TEAM
at the
BRABAG WORKS at TROGLITZ - ZEITZ.

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REPORT ON INVESTIGATIONS BY FUELS AND LUBRICANTS TEAM

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BRABAG WORKS at TROGLITZ - ZEITZ

Edited by

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Ministry of Fuel and Power

on behalf of

Ministry of Fuel and Power and
U.S. Technical Industrial Intelligence Committee

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ZEITZ

This target was visited during the period 9th to 20th May, 1945 by the following members of the investigating teams.

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General Manager	Dr Wille
Deputy Works Manager	Dr Daengler
Hydrogenation H.P. Manager	Dr Roth
Hydrogenation L.P. Manager	Dr Saager
Gas Group Manager	Dr Scheirer
Technical Engineer	Dr Lehmann

INTRODUCTION

Construction of the plant was started in 1937 and part was in operation by 1939. It is now under the complete direction of BRABAG.

The factory was designed to produce petrol, diesel oil, wax and lubricating oil by the hydrogenation of brown coal-tar, the necessary hydrogen being produced by the gasification of grude coke (brown coal low-temperature coke) with oxygen in Winkler generators.

Gas Measurement

Throughout this report gas quantities in M^3 are taken as measured at 15°C and 760 mms Hg, the standard used at Zeitz.

HYDROGEN PRODUCTION (G.U. Hopton and R.J. Morley)

The plant to produce and purify hydrogen was very similar to that at the BRABAG plant at BOHLEN (C.I.O.S. Target 30/4.05). Grude coke was gasified with oxygen and steam in Winkler generators, H_2S was removed by a combination of alkacid and oxide boxes, CO was converted to CO_2 with steam at atmospheric pressure and the gas was compressed to 290 ata, with CO_2 being removed by water washing at 27 ata and CO being removed by scrubbing with copper liquor at 290 ata.

Oxygen Plant

There were five Linde-Frankl units, each with a nominal capacity of 3,000 M^3/hour oxygen, normally four running, one spare or with maintenance. It was understood this plant had no novel features and it was not inspected. Typical figures for power consumption were given as 0.60 to 0.64 KWH/M^3 oxygen (99.0% O_2 on dry).

Water Gas Plant

The grude coke, i.e. low temperature coke from brown coal, was mainly obtained from the ASW plant at Espenhain, but at times it was also taken from the ASW plant at Böhlen, from Riebeck Deuben and from Offleben. A typical analysis of Espenhain grude was:

		Average 1944
C	62 to 64.5%	62.1%
H	2.1%	2.2%
N	not determined	-
S	5.75%	5.4%
Ash	27.5 to 30.5%	27.8%
Moisture	2.0%	2.5%
Net C.V.	5650 to 5850 T.cals/T.	
Ash softening point	900° to 1100°C	

The grude was passed through magnetic separators, followed by a series of roller crushers and screens to reduce the size to 3 to 4 mm diameter, although the overall size range was from 2 to 10 mms. All these operations were carried out in an atmosphere of nitrogen, obtained from the Linde-Frankl plant. The powdered grude was stored under nitrogen in two bunkers, each 7 m diameter and 8 m high, plus a conical base, and was transported in a closed conveyor to a hopper situated besides each generator.

There were three Winkler generators, normally two working. The normal production was 18,000 M^3/hour raw water gas, rising to a maximum of 22,000 M^3/hour, where the limitation was erosion of the waste heat boilers; the minimum output was 9,000 M^3/hour, below which the bed ceased to boil or dance. The design was similar to that at Böhlen, i.e. they were straight-sided brick-lined generators, about 4 to 4.5 m I.D. and 20 m overall height, with the depth of boiling bed maintained at about 1.5 m; they were provided with grates, through which passed the mixture of steam and oxygen.

Grude was introduced into the fire-bed by three water-jacketed screw-conveyors, driven mechanically, a method preferred to hydraulic oil drives. It was understood that the stationary grate was made of fire-brick, about 3" wide, assembled on edge with slits left between for passage of the oxygen and steam mixture; at Böhlen however wedge-shaped cast iron bricks were used and this may have been the case at Zeitz as well. A water-cooled scraper, of square section, 6" by 6" (although this may have been due to build-up of ash), rotated slowly just above the grate but below the grude feed; its function was to sweep the ashes towards two holes in the grate, leading to two water-jacketed screw-conveyors. The ashes collected in two small hoppers (0.1 M^3), emptied periodically by hand into small bogeys on rails. The pipes from the generator base to the screw conveyors could be rodded through as required. The ashes contained about 30% carbon, net C.V. 2090 T.cals/T but amounted to only 14% (1944 average) of the ash fed in. No trouble was experienced due to burning of the arm and Dr Schairer ascribed the difficulty in this respect at Leuna, which was one of the reasons for their evolving a grateless design, to the relatively higher alkali content and lower ash m.p. of the Leuna fuel.

90% of the oxygen-steam mixture was blown up through the grate, whilst 10% was introduced 2 m. above the fuel bed through twelve water-cooled nozzles; these ended flush with the inside wall and pointed exactly at the centre line of the generator. It was found by observation through sight-holes, that a gas velocity of 8 m. per sec. through the nozzles was the optimum; at higher velocities the flames struck and damaged the far-side wall; at lower velocities the flames licked upwards on to the lining above the nozzles; some clinker built up above the nozzles

The depth of bed was about 1.5 m, controlled by hand by altering the speed of the grude screw conveyors and measured by pressure drop. The temperature in the fuel bed, about 900° to 950°C, was kept about 20°C below the softening point of the grude ash. This point was readily controlled by observation of the ash; if this were too dusty more oxygen would be used, whilst if there were signs of clinkering more steam would be used. Thus the oxygen/steam ratio was kept as high as possible but avoiding clinkering.

Gases leaving the top of the generator at about 900° to 1,000°C were cooled to 400°C in passing through a water-tube waste heat boiler and then to 250°C in an economiser. Dust built up in the bottom of the boiler and economiser, and the resultant higher velocity prevented further build-up.

Dedusting was carried out by multicyclones, followed by direct water wash and Theisen disintegrators. It was emphasized that the successful operation of a Winkler generator depended on the successful solution of the dust problem and this is readily appreciated when it is realised that at Zeitz about 40% of the total carbon fed to the generator was blown over as dust; this percentage was appreciably greater than at Böhlen and Leuna.

Gas leaving the economiser passed through two pairs in parallel of two sets of multicyclones in series, working at about 200°C; one set was larger than the other, the object being to give a choice to suit the particular output, but in practice the hand operation of large valves was found to be a nuisance. The dust concentrations before and after the multicyclones were given as 300 and 60 kg/M^3 respectively, probably measured on wet gas, for monthly records showed about 330 kg/M^3 dry gas recovered from the cyclones. A typical analysis of this dust was

		1944 average
C	54 to 56%	54.3%
H	0.8%	0.9%
Ash	45 to 43%	43.8%
Moisture	0.0%	1.0%
Net C.V.	4800 to 5100 T.cals/T	

The dust from the multicyclones was collected in special containers; when full these were isolated by remote control, the dust roused by admission of CO_2 through a standpipe and the dust removed by an injector, working on CO_2. The dust was used as fuel in the power station. [At Magdeburg an electrostatic precipitator had been used instead of multicyclones, but due to faulty operation one day free oxygen appeared in water gas and an explosion resulted; they now also use multicyclones].

After the multicyclones the gases passed through a water seal, a safety device, which also removed some dust and cooled the gas to about 85°C. The gas was then passed into a direct contact water scrubber, which reduced the dust content to 2 g/M^3 and cooled the gas to 30°F. Finally after passage through a Theisen disintegrator the gas, now containing 3 to 4 mg/M^3 dust, passed to holder as crude water gas. Water from the scrubber and seal, besides containing dust, also contained H_2S, and both of these had to be removed before the water could be discharged to the river. The water was passed through two towers in series, (with a common spare), through the first of which CO_2 was blown to remove the bulk of the H_2S and through the second air was blown. The water leaving these towers passed into three large settling ponds, where the dust content was reduced from 100 to 20 mg/l, which was the same as the normal content of suspended matter in river water.

For starting up the generator a small auxiliary Winkler generator was used: this was 1.0 to 1.5 m. I.D, but always directly open to atmosphere through a wide open stack (0.5 to 1.0 m. I.D). A fire was started with wood and briquettes, blowing with air only, and grude run in slowly from a hopper. The glowing grude was then run by gravity into the large generator, standing full of N_2 or CO_2, with the safety valve open to atmosphere. Air was blown through the grude and the bed was built up by feeding fresh grude through the screw conveyor. The blast was then changed over to steam and oxygen, and when the gas was of sufficiently good quality the electrically-operated safety valve was closed and gas making proceeded. Dr Scheirer said this size of generator could probably be started up directly on grude without fear of explosion, but not so the larger generators at Leuna; however the Zeitz generators could not be started up on brown coal itself, since air and carbonisation gases would form an explosive mixture. The valves on the outlets from the small generator were in contact with the hot grude for only a short time, and at other times were protected by a layer of cold coke.

Raw Water Gas Analysis	%
CO_2	23.1
CO	29.6
H_2	43.8
CH_4	0.75
N_2	1.5
H_2S	1.25
Net C.V.	1978 T.cals/1000 M^3

(1944 averages)

Grude	0.752 kgs/M^3	raw	water	gas	
oxygen (100%)	0.228 M^3/M^3	"	"	"	
2.5 atg steam	0.630 kg/M^3	"	"	"	(to generator)
" "	0.2 kg/M^3	"	"	"	(to heating)
18 " "	cr.0.680 kg/M^3	"	"	"	
*Power	0.052 KWH/M^3	"	"	"	
Circulated cooling water	0.025 M^3/M^3	"	"	"	

Material Balances (1944 averages) All quantities in g/M^3 raw water gas.

	Fed to generator				Raw water gas (dry)	Cyclone dust	Ashes	Undecomposed steam (by H_2 diff.)	Unaccounted for	Total
	Grude	Oxygen	Steam	Total						
C	467.0	0	0	467.0	266.1	183.0	13.0	0	4.9	467.0
H(incl. H_2O)	18.6	0	70.0	88.6	38.3	3.3	0	47.0	0.0	88.6
Ash	209.0	0	0	209.0	0	147.5	28.2	0	33.3	209.0
O(incl. H_2O)	16.6	310.2	560.0	886.8	515.0	2.9	0	376.2	-7.3	886.8
N	-?	2.88	0	2.88+	13.4	0	0	0	-10.52-	2.88+
S	40.6	0	0	40.6	12.6	?	?	0	28.0-	40.6
Total	751.8	313.08	630.0	1694.88	845.4	336.7	41.2	423.2	48.38-	1694.88

Carbon gasified = 49.25%

Ratio steam out/steam in = 0.67

*Excludes O_2 manufacture but includes all blowers.

H_2S Removal

H_2S was removed from raw water gas in three stages. The first, by direct water wash has already been described. The second stage used alkacid and the third stage dry oxide towers. The water wash and alkacid treatment reduced the H_2S content from 14 to 16 g. S/M^3 (1.0 to 1.3% H_2S) to 5 to 6 g/M^3 (0.4 to 0.5% H_2S). Gas recovered from the alkacid plant, containing 30 to 40% H_2S, with the rest CO_2, was sent to a Claus kiln, where together with H_2S-containing gas from other alkacid plants, treating rich gas, lean gas, etc., it was treated for recovery of elemental sulphur. About half the sulphur removed in the first two stages was so recovered. Over three months, January to March 1944, 25 M^3 of fresh alkacid liquor was added, corresponding to 0.4 $M^3/10^6$ M^3 gas treated.

The dry final purification was carried out in oxide towers containing luxmasse, with addition of oxygen and steam. The H_2S content of purified water gas was 5 to 25 mg S/M^3 and the lux was worked up to 45% S.

CO Conversion

Seven converters were available, each of nominal capacity 5,500 M^3/hour. They contained brown oxide and worked at substantially atmospheric pressure. The CO content was reduced from 32% to slightly under 4.0%. The CO remaining in the catalysed gas (Kontaktgas) was later removed by copper liquor and returned to the CO conversion plant. Provision was made for the return of CO_2-containing water from the CO_2 Removal plant, to be used in the gas cooling towers and hence avoid introducing O_2 into catalysed gas, said to be a cause of corrosion in the compressors in the presence of H_2S. However records for January to March 1944 showed this system was not then in use.

2.5 atg steam = 0.33 T/1000 M^3 catalysed gas
Power = 5 KWH/ " " " "
Circulated cooling water = 4 M^3/ " " " "

The analysis of catalysed gas was

CO_2	39.9%
CO	3.9%
H_2	54.4%
$N_2 + CH_4$	1.8%

Compressors

There were eight six-stage compressors, each of capacity of 10,500 M^3/hour catalysed gas (≡ 5450 M^3/hour crude hydrogen). After the third stage CO_2 removal was effected at 27.8 atg, and the final pressure was 298 atg.

Power 403 KWH/1000 M^3 purified hydrogen
Cooling water 38 M^3/ " " " "

CO_2 Removal

There were eight scrubbing towers, each of capacity 15,000 M^3/hour catalysed gas with a water temperature of 6.5°C. Water leaving the towers was let-down through four pelton wheels, directly coupled with electric motors and pumps, and also through two pelton wheels, coupled with pumps but not with motors.

Typical performance figures were:

Water temperature	4.5°C
Wash water	36.3 M^3/1000 M^3 catalysed gas
Gas rate/tower	11,230 M^3/hour
Exit CO_2	1.7%
Power	not obtained

CO Removal

There were three copper liquor scrubbers, each of capacity 20,000 M^3/hour inlet gas, although usually all three were run when available. There were three copper-liquor let-down machines, each of capacity 100 M^3/hour, together with three injection pumps, each of capacity 20 M^3/hour.

Typical performance figures were:

Inlet gas rate/tower	8950 M^3/hour
Liquor rate	4.5 M^3/1000 M^3 inlet gas
Power	12.5 KWH/1000 M^3 inlet gas
Steam 2.5 atg	0.135 T/ " " " "

Exit gas analysis:	CO_2	0.18%
	CO	0.30%
	H_2	96.65%
	$CH_4 + N_2$	2.87%

Overall Efficiencies of Hydrogen Production

Based on 1000 M^3 100% H_2 in final hydrogen: March 1944

Crude			1.168 T
100% O_2			0.348 M^3
2.5 atg steam :	water gas plant	1.078 T	
	CO conversion	0.583 T	
	CO_2 and CO removal	0.139 T	
			1.800 T
Power :	oxygen plant	263.8 KWH	
	water gas plant	76.8 KWH	
	compression	417.3 KWH	
	CO_2 and CO removal	68.4 KWH	
			826.3 KWH

HYDROGENATION

WORKS HISTORY.

The construction of the plant was started in 1937 as a combined effort of the I.G., Brabag, and other firms interested in the production of fuel from Brown Coal, but was later taken over completely by Brabag. Part of it was in operation by the middle of 1939.

It was planned to make, from Brown Coal Tar, in one stage over catalyst 5058 by the T.T.H. process (Low Temperature Hydrogenation) Petrol, Diesel Oil, Paraffin Wax, and Lubricating Oil, with a separate 6434 Stall to split some of the 150° - 220°C fraction to petrol in order to increase the yield of petrol and its quality.

The plant started up with 4 Stalls and because the dewaxing plant was not ready, they ran the plant on the M.T.H. (Medium Temperature Hydrogenation) process, so that the residue boiling higher than 350°C in the 5058 Stall product was a minimum. Petrol and Diesel oil were the only products and the residue was sent to Böhlen. Later, the demand for petrol fell off and they ceased to use 6434 catalyst and replaced it by 5058.

Towards the latter end of 1940 and the beginning of 1941 they made a special Diesel Oil known as S.D.K. which had a low flash point and was made at the expense of the heavy ends of the petrol.

By 1941 there were 6 stalls in operation and the propane dewaxing plant was almost finished.

When the propane dewaxing plant became available they began to change over from the M.T.H. process to the planned T.T.H. process, trials of which had been run at Leuna before the war. They had considerable teething troubles with the dewaxing plant and the maximum output was not achieved until February 1944 and even then it was not the design output. During the period of maximum output they had 5 stalls on the T.T.H. process and 1 stall on the refining of Paraffin Wax

In May 1944 the Reich told them to shut down the dewaxing section and to run to produce Petrol and Diesel Oil. Because of air raid damage to Böhlen they had to deal with the greater than 350°C fraction themselves and arranged to run one of the stalls to crack this residue.

At the end of 1944 they had 7 stalls available for operation and at the time of the inspection had 10 concrete chambers ready. They were planning to fit one up as a Brown Coal Stall and another as a stall to turn the Coal stall middle oil to petrol. They were also erecting a D.H.D. plant to deal with the petrol they made in the T.T.H. process.

They had their own test engine for testing Diesel Oil, a single cylinder Daimler-Benz engine.

PRODUCTIONS.

They achieved their maximum production on the M.T.H. process during the early part of 1941 when they were making a total quantity of 215,000 Tnes/yr, the amount of petrol being changed to suit the demand by altering the F.B.P.

With an F.B.P. of 135°C they made 37% petrol with an Octane No of 68.

With an F.B.P. of 165°C they made 45% petrol with an Octane No of 57.

The corresponding Diesel Oils had Octane Nos of 47 and 57 respectively, the latter having a Setting Point of -20°C.

The residue from these operations, which was sent to Böhlen, amounted to about 5% of the feed, and the gas make about 8%. They obtained 88% of the feed as petrol and diesel oil.

When they changed to T.T.H. operation they achieved maximum output in February and March 1944 and they reached a rate of 360,000 Tnes/yr of feed to the T.T.H. stalls. They got 92% of the feed out as finished products with a gas make of about 2%. Here again they cut the petrol F.B.P. to suit the demand but when they were limited by the dewaxing plant, as was always the case, to making 45,000 Tnes/yr of Wax, they could also make 30,000 Tnes/Yr of lubricating oils and 250,000 Tnes/yr of Petrol and Diesel Oil.

With an F.B.P. of 180°C the petrol had an O.N. of 59 and with an F.B.P. of 155°C it had an O.N. of 64. According to the record sheets available the Diesel oil quality did not change with the change in end point and it had a Cetane Number of 46 with an F.B.P. of 350°C and a Flash Point of 67°C. If the petrol was cut to 155° F.B.P. it amounted to 23% of the total Petrol + Diesel Oil, and with an F.B.P. of 180°C to 34%.

The Paraffin Wax had a Gravity of 0.777 at 70°C, a melting point of 54°C, and an Oil content of 9%.

The quality of the lubricating oils depended on how they were cut. If the Spindle Oil had a visc. of 2.85° Engler at 50°C a gravity of 0.877 and a setting point of -10.6°C then the corresponding Machine Oil had a Viscosity of 4.06° Engler, a gravity of 0.882, and a Setting Point of -7.9°C, and an acid number of 0.1.

If the Spindle Oil were cut to give an oil of 2.94° Engler at 50°C with a gravity of 0.888 and a setting point of -9.3°C the corresponding Machine Oil has a Viscosity of 5.07° Engler at 50°C, a gravity of 0.892 and a Setting Point of -6.9°C. and an acid number of 0.5.

SOURCES OF RAW MATERIALS.

The Brown Coal Tar came mainly from the Lurgi Schwelereis at Böhlen and Espenhain with small amounts from other Schwelereis. They preferred the oil from Lurgi plants because of its cleanliness.

They also obtained quantities of the Schwelbenzin from various plants this oil having an F.B.P. of 250°C on the average and a gravity of 0.840 at 20°C.

The amount of Schwelbenzin amounted to 6.5% of the total feed to the stalls.

Because of the small gas make they had to get their Propane for the dewaxing plant from the Hydrogenation plant at Böhlen.

EXPORTS.

They exported electricity from the power plant to the local mines.

The petrol and diesel oil and lubricating oil were dispatched according to instructions from the State. The Wax was sent in the first three months of 1944 to Rhenania-Ossag, Hamburg, I.G. Heydebreck; I.G. Ludwigshafen; Nordd. Mineralölw. Stettin; and Fettchemie Hubbe-Farenholtz. At the first three plants it was cracked and polymerised into synthetic lubricating oils.

OUTLINE OF OPERATION.

The imported Brown Coal Tar is fugalled to remove solids and water and then filtered to remove more of the solids. The Schwelbenzin is then added together with residues from the dewaxing plant and the mixture fed to the stalls. 5 Stalls were normally working on this feed. Because they could not get the oil completely free from solid matter they had to change the first converter fairly often because the dirt in the oil was deposited on the catalyst there. The staff said that they had to change the converter every 4-6 weeks but records were seen which indicated that three stalls had run on one occasion for at least three months without a shut down.

The Cold Catchpot product from the stalls was distilled to give petrol, naptha, middle oil and residue. The naptha, middle oil and fore runnings from the lubricating oil distillation plant were mixed to give diesel oil. The residue from the stall product distillation was dewaxed with propane to get paraffin wax which was refined by distillation and a treatment in a hydrogenation stall, and the dewaxed oil treated to get lubricating oil.

Waste water from the various processes was treated with Tri-Cresyl-Phosphate to remove Phenols, which were sent to Leuna.

PREPARATION OF THE TAR.

The raw tar is stored at 60°C and fugalled to remove water and to reduce the solids content from 0.5% to 0.05%. The fugal plant consists of 26 horizontal spindle fugals supplied by Haubolt, running at 1200 RPM and having a bowl diameter of 1500 mm. They were originally designed to run discontinuously at an average rate of 2 Tne/hr. They were however being run continuously at a rate of 4-5 Tne/hr the oil being fed at a temperature of 80°C. Twice a shift the oil feed was stopped and the speed reduced to 600 RPM. The water and emulsion were then removed and sent to separators from whence the water was sent to a phenol recovery plant. The oily sludge adjacent to the rim of the bowl was then cut out. This sludge, containing 50% oil, was exported to fire-lighter manufacturers.

After this treatment the oil was sent to the filtration plant where the solids content (insolubles in Xylol) was further reduced to 0.008%. The Plant consists of 10 filter presses each having 30 plates of an area of 1 M^2 and covered with cloth and paper. The oil is fed at 100°C by means of a centrifugal pump. The feed rate at the beginning of the filtration is 10 M^3/hr. and finishes up at 3-4 M^3/hr when the pressure is limited to 5 Ats in order to prevent the fracture of the paper. The filters last approximately 48 hrs between cleaning, when it takes 2 men 8 hrs to strip the filter, clean it, and reassemble it. The cake was said to build up to a thickness of 33 mm, but on analysis this indicates that the cake had an oil content of 90% which is rather high.

INJECTION BUILDING.

The oil is injected by electric driven three-throw pumps.

CIRCULATION PLANT.

There are 5 circulators each having an output of 55,000 M^3/hr and running at 123 RPM constant speed. About 20-40,000 M^3/hr of gas is kept on by-pass as boost control and also in case of a run-away on the stall. They try to vary the amount of by-pass to suit the reactivity on the stalls, keeping more in hand when the stalls are reactive than when the stalls are relatively unreactive. The maximum boost is 30 Ats.

The purity of the circulating gas is held by the bleeding off of stalls exit gas. There is provision on the plant for injecting stall product into the inlet of the stall coolers in order to provide some washing of the stall gas. This is not operated because the pump is a constant speed pump requiring 350 KWH and because the running of the pump demands the use of extra labour to handle the pump and to give closer supervision to the levels in the stall cold catchpot, and all these mean that it is cheaper to make the extra hydrogen.

STALLS.

The seven stalls are all the same in detail each having three converters, two interchangers, an electric preheater, a cooler and a cold catchpot.

The converters are 1000 mm bore and 18 M long having a reaction volume of 8.1 M^3 each. There is only one bed of catalyst in each converter but cooling-gas rings are fitted to the converters without any provision for mixing. There are 6 rings in the first converter and 4 in the other two.

The interchangers are 600 mm bore and 18 M long.

The cold catchpot has a volume of 1.5 M^3.

Water is injected to each stall at approximately 1 M^3/hr at the inlet to the cooler, to prevent formation of Ammonium Carbonate chokes.

The catalyst used for the reaction is the I.G. catalyst 5058 but this was being replaced by 8376, a dilute form of 5058. They said that it required temperatures about 30°C higher than 5058 but no factual evidence was seen to confirm this. They were of the opinion that 8376 was a better catalyst for them because it did not break up so easily under their rather rigorous physical conditions, and said they thought it ought to be cheaper to make.

Whenever a stall became congested with the dirt in the first converter they took it out and cleaned the catalyst by sieving it and replacing it in the converter. This was done until the catalyst had had a life of 400-600 days when it was sent to Leuna for reconditioning. As was mentioned earlier the life of a converter as given at the interrogation was 4-6 weeks but examination of the monthly records show that in 1944 they had three stalls which ran more than three months without a shut-down. Each converter was left in until its pressure drop became too great for efficient operation. The change of converter took on the average 2 days.

Details of the various types of operation were obtained by interrogation and examination of the records available, and are as given below.

M.T.H. OPERATION.

5058 Catalyst. 10 mm pellets.
Stall inlet pressure 300 Ats.
Feed 12-7 M^3/hr pending on pressure drop.
Feed Gravity 0.95 at 20°C.
Feed Asphalt 2.5%
Feed 38% boiling below 325°C.
Carbon 83.6%
Hydrogen 10.65%
Sulphur 2.0%
Nitrogen 0.45%
Vapourising Gas 25-20,000 M^3/hr depending on the pressure drop.
Maximum stall pressure drop 17 - 21 Ats depending on the stall rate and the reactivity.
Cooling gas 0 to 30,000 M^3/hr depending on the catalyst activity.
Hydrogen consumption 665 M^3 per Tonne of feed.
Gravity of stall product - 0.760 0.800
Residue boiling above 350°C - 6% 17%
Gas Make 8%

New Catalyst Temperatures	1st Converter	285 - 365°C
	2nd --	355 - 370
	3rd --	360 - 370
Old Catalyst Temperatures	1st --	295 - 315
	3rd --	- 420°C

T.T.H. OPERATION

5058 Catalyst. 10 mm pellets.
Stall inlet pressure 300 Ats. Hydrogen purity at exit 90% approximately.
Feed 12 - 7 M^3/hr depending on pressure drop.
Feed Gravity 0.95 at 20°C.
Asphalt 2.5%
38% boiling below 325°C.
Other analyses as for M.T.H.
Vapourising Gas 25,000 M^3/hr.
Cooling Gas 900 M^3/tne of feed.
Product Gravity 0.830
Residue boiling higher than 350°C in Stall Product 28% of feed.

Gas Make 2%
Hydrogen consumption. 500 M^3 per Tonne Feed. (Pure H_2)

New catalyst temperatures	1st	Converter.	280 - 340°C.
	2nd	—	330 - 345
	3rd	—	330 - 345
Old catalyst temperatures	1st	—	295 - 350°C
	2nd	—	340 - 360
	3rd	—	345 - 350

M.T.H. RESIDUE OPERATION (1944 AND 1945).

5058 catalyst. 10 mm pellets.
Stall inlet pressure 300 Ats.
Feed 10 M^3/hr. Gravity 0.860.
Vapourising Gas 14,000 M^3/hr.
Product Gravity 0.74 -
Residue boiling above 350°C in Stall Product . 10%

Temperatures.	1st	Converter.	375 - 390 -	365°C.
	2nd	—	355	360
	3rd	—	355	345

The reaction was intended to be in the nature of a cracking reaction and they believed that it would have been better to have used 6434 catalyst. The preheater was a limitation to the stall and they had thought of using two converters instead of the three they had.

PARAFFIN WAX REFINING.

The only details of the operation of this stall are:-
Pressure at inlet 300 Ats.
Feed rate 6 M^3/hr of raw Wax.
Total Gas 3,200 M^3 per tonne feed.
Hydrogen consumption (100% H_2) 0.94% by weight or 100 M^3/tne feed.

TREATMENT OF T.T.H. RESIDUE

The subsequent treatment of the refined tar in the T.T.H. operation consists essentially of separation by distillation into motor benzine, diesel oil, and residual stock which is dewaxed in a propane dewaxing plant with the aid of a crystal modifier, Paraflow. This residue contains approximately 50% of crude wax which is therefore one of the main products of this type of operation. The crude wax from the first filtration is repulped with liquid propane and refiltered to give a hard wax of moderate oil content. This material is distilled to remove the crystal modifier as a residue which is then returned to charge stock for use in subsequent dewaxing operations. The distillate wax which is white, has a Melting Point of about 54°C. and an oil content of about 8%/9%, is then hydrogenated under mild conditions to give a stable wax, and topped to remove the light products produced during hydrogenation. The product is then finished with floridin in the usual manner.

Dewaxed Oil was worked up in a conventional manner to give an overhead distillate boiling below 350°C. which was incorporated with the diesel oil obtained in the first distillation, a distillate spindle oil, and a residual oil of somewhat higher viscosity classified as machine oil.

PERSONNEL REQUIREMENTS.

In the early part of 1944 there were 535 staff and 2630 wage-earners in the whole of the works presumably including those employed by the firm on construction work.

The wage-earning section were split up as follows:-

Power section	205
Oil section (inc. gas)	1050
Workshops	1050
Etc. Cooks, Laundry.	320
	2625

SPECIFIC CONSUMPTIONS.

		1941		1943	1944	
		Jan.	Feb.	Dec.	Jan.	Feb.
Solid Fuel to Boilers (Dry Br. Coal + Winkler Coke)	Tnes/mth	25,813	24,996	25,067	25,227	24,709
Electric Power for Oil Plant	KWH/tne. feed.	850	800	780	830	720
70 Ats. steam —	Tne/tne. feed.	0.37	0.213	0.083	0.082	0.083
18 Ats - —	—	?	?	0.34	0.77	0.51
2.5 Ats - —	—	3.87	3.48	3.8	4.05	3.35
Heat (Fuel Gas) —	Tne.cals/tne.feed	274	224	?	?	?
O_2 for H_2 production.	M^3/Tne. feed.	234	226	?	170	178
Filter Aid. Paraflow.	Tnes/mth.	-	-	38	112	46

CAPITAL COST OF PLANT.

The capital cost of the plant as it stood at 31.12.43 was given as follows:-

Gas Production	22,418,862	RM.
Hydrogenation High Pressure	8,428,275	" ※
Hydrogenation Low Pressure	15,069,628	" ※
Dewaxing and Lube Oil Plant	13,754,851	"
Water supplies	8,439,288	"
Steam supplies	29,048,439	"
Electricity supply	4,826,041	"
Drains and Phenol Extraction	3,527,594	"
Pipe-bridges and piping	5,371,381	"
General	19,665,923	"
	130,550,282	

※ From a detailed analysis of these costs it is believed that these two figures should be interchanged.

PRODUCTIONS

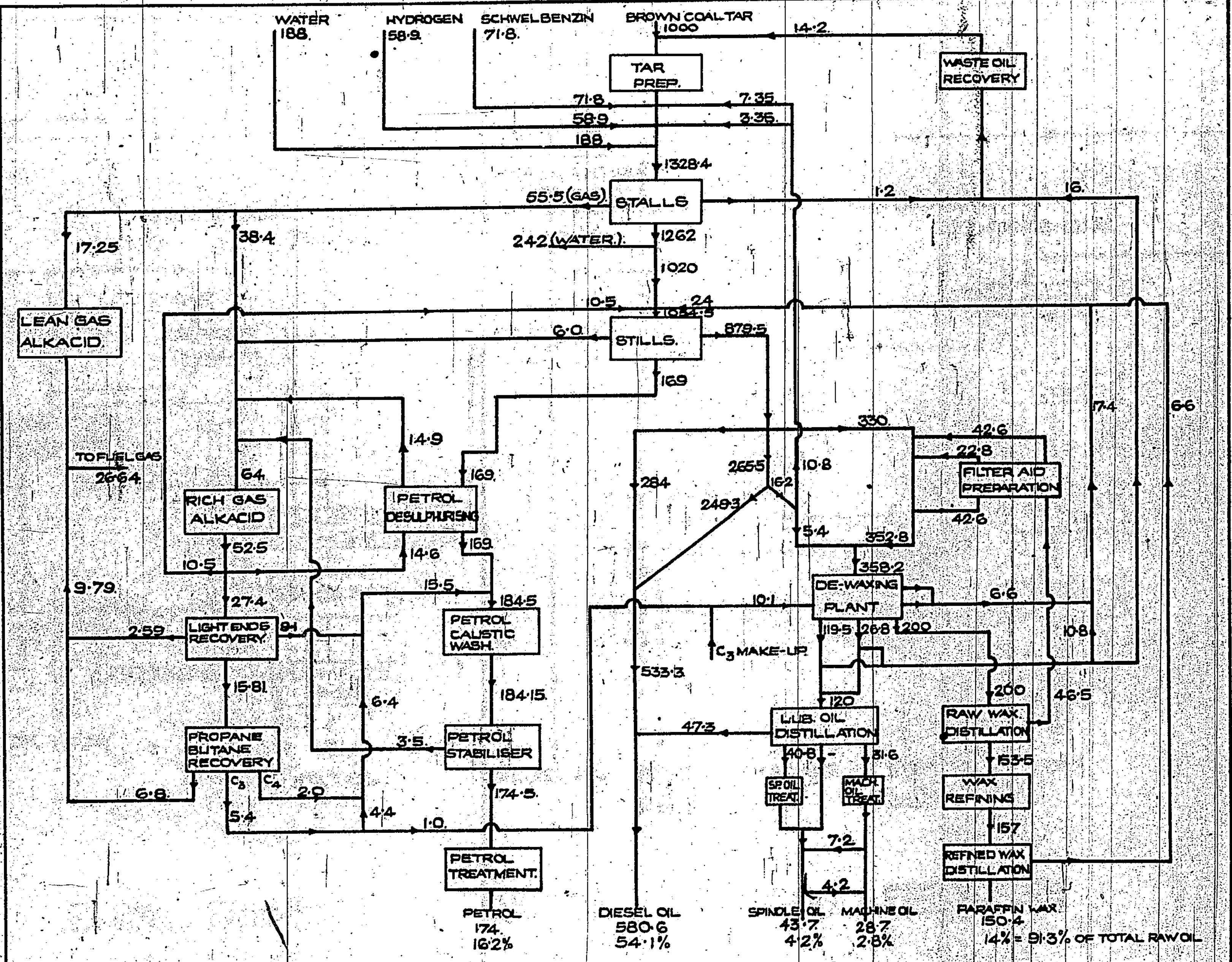
		Motor Petrol	Diesel Oil	Spindle Oil	Machine Oil	Wax	Total
1939	March	1,700					1,700
	April	3,600					3,600
	May	4,600					4,600
	June	3,500					3,500
	July	4,000					4,000
	August	8,750					8,750
	September	6,700	5,600				12,300
	October	4,750	7,650				12,400
	November	2,300	11,200				13,500
	December	3,700	11,000				14,700
	TOTAL	43,600	35,450				79,050
1940	J	3,950	10,800				14,750
	F	2,700	9,300				12,000
	M	6,200	8,200				14,400
	A	8,500	5,800				14,300
	M	11,700	8,200				19,900
	J	10,350	8,800				19,150
	J	9,450	10,400				19,850
	A	8,200	9,800				18,000
	S	6,100	7,500				13,600
	O	5,600	8,300				13,900
	N	6,950	11,200				18,150
	D	6,200	10,500				16,700
	TOTAL	85,900	108,800				194,700
1941	J	6,200	10,400				16,600
	F	6,900	10,900				17,800
	M	8,000	11,500				19,500
	A	8,850	7,800				16,650
	M	8,250	8,200				16,450
	J	9,400	6,900				16,300
	J	9,750	8,600				18,350
	A	10,600	10,650				21,250
	S	13,200	10,400				23,600
	O	7,350	14,150			1,150	22,600
	N	4,750	15,350			550	20,650
	D	8,300	15,850			850	25,000
	TOTAL	101,550	130,700			2,550	234,800

1942	J	7,700	13,900			1,400	23,000
	F	7,000	11,850			850	19,700
	M	8,250	14,200			1,600	23,050
	A	7,500	13,300			2,400	23,200
	M	3,900	12,350	150		3,200	19,600
	J	4,700	13,650	800	650	3,100	23,000
	J	5,800	13,500	-	1,150	1,550	22,000
	A	8,100	13,500	1,300	-	1,600	24,500
	S	8,300	14,250	1,100	200	2,150	26,000
	O	7,550	14,900	1,300	250	2,550	26,550
	N	8,450	12,750	1,050	450	1,450	24,150
	D	8,700	11,600	350	400	1,650	22,700
	TOTAL	85,950	159,750	6,050	3,100	23,500	278,350
1943	J	9,050	13,900	750	450	1,650	25,800
	F	3,550	11,000	300	1,100	1,800	17,750
	M	2,100	12,300	1,050	1,100	1,950	18,500
	A	3,400	11,450	350	100	1,700	17,000
	M	6,350	10,950	350	250	300	18,200
	J	9,850	9,450	350	150	-	19,800
	J	9,800	11,500	200	300	1,000	22,800
	A	8,350	11,450	700	400	1,700	22,600
	S	10,350	10,200	1,200	1,050	3,400	26,200
	O	3,800	12,100	1,650	2,450	3,800	23,800
	N	1,100	14,650	1,300	2,000	1,750	20,800
	D	8,150	10,850	1,600	1,400	3,750	25,750
	TOTAL	75,850	139,800	9,800	10,750	22,800	259,000
1944	J	2,550	15,900	1,100	800	3,900	24,250
	F	8,400	14,500	1,150	800	3,750	28,600
	M	2,350	14,550	1,250	850	3,650	22,550
	A	650	15,750	1,600	1000	2,250	21,250
	M	4,100	13,900	300	350	1,350	20,000
	J						
	J						
	A	800					800
	S	300					300
	O	6,000	5,700				11,700
	N	7,550	6,300				13,850
	D	3,250	3,400				6,650
	TOTAL	35,950	90,000	5,400	3,800	14,900	150,050
1945	J	4,550	3,800				8,350
	F						
	M	550	700				1,250
	A						
	TOTAL	5,100	4,500				9,600

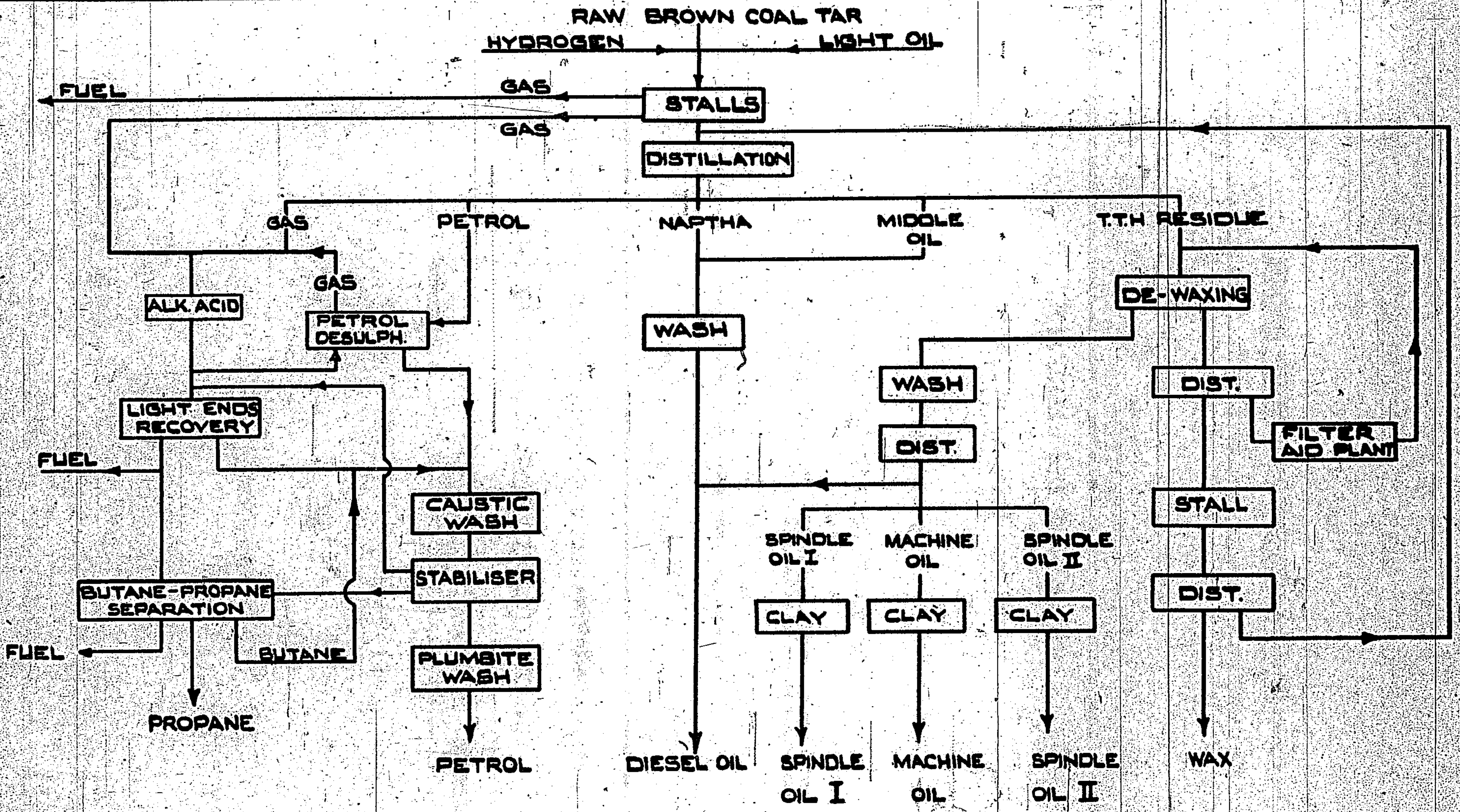
DOCUMENTS EVACUATED

The numbers of series, reel, bag and document are those given in the list of G. von Elbe, dated August 6th, 1945.

Series	Reel	Bag	Nr.	
A	33	4182	1	Production history and typical operating data for 15th March 1944.
			2	Report of operations during Jan, Feb. and March, 1944.
			2a.	Flow diagrams
			2b.	" "
			and	other unlisted and unfilmed documents, labelled "Discard".



QUANTITY FLOWSHEET FOR JANUARY 1944 FOR BRABAG WORKS AT ZEITZ.



FLOW DIAGRAM ZEITZ TTH PROCESS

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ITEM No. 30
FILE No. XXXIII—5

THE METHANISATION OF COAL GAS INFORMATION OBTAINED FROM Dr. MARTIN OF RUHRCHEMIE A.G. AND Dr. TRAENCKNER OF RHURGAS A.G.

Wilkins, E.J.

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

REC'D. JUL 1946
TIIC L.F. & L. S-C.

LONDON—H.M. STATIONERY OFFICE

The Methanisation of Coal Gas

Information obtained from Dr. Martin of Ruhrchemie A.G. and from Dr. Traenckner of Ruhrgas A.G.

Reported by

E.T. Wilkins

on behalf of

British Ministry of Fuel and Power and the

U.S. Technical Industrial Intelligence Committee.

CIOS Target Nos. 30/5.01, 30/6.11
Fuels and Lubricants.

Combined Intelligence Objectives Sub-Committee

G-2 Division S.H.A.E.F. (Rear) A.P.O. 413.

(1945)

8p. diagr.

Table of Contents

Personnel of Team

H.H. Lowry (U.S.) T.I.I.C.
H.J. Rose (U.S.) T.I.I.C.
E.T. Wilkins (British) M.F.P.

The Methanisation of Coal Gas

Information obtained from Dr. Martin of Ruhrchemie A.G. and from Dr. Traenckner of Ruhrgas A.G.

Object of Investigation. learn of recent German developments in the catalytic synthesis of methane from gases containing carbon monoxide and hydrogen.

Locations and dates of interrogations. Dr. Traenckner, director of Ruhrgas A.G., was interviewed at Ruhraller G, Essen, on 4th July, 1945, by Drs. Lowry, Rose and Wilkins.

Dr. Martin, director of Ruhrchemie A.G., and Mr. Hanisch, chemist, were interviewed on 5th July by Dr. Wilkins at the Oberhausen-Holten works. Dr. Traenckner was also present on this occasion.

Summary. A process has been developed for effecting the catalytic methanisation of coal gas, and plants for operating this process on the scale of 6,200 cu.ft./hr. have been installed at road-side filling stations. These stations supplied the processed gas compressed to 300 atmospheres for use as motor fuel.

The objects of the process were (a) to enhance the calorific value of readily available town's gas to 650 B.Th.U./cu.ft. and (b) to remove constituents which caused deterioration of the high pressure cylinders.

Details of the catalyst, the design, method of operation and performance of the plant were elicited, and some items of the equipment inspected.

The locations of six methanisation plants are given in the report. None of these was visited.

General. The process was developed by Ruhrchemie, who remained responsible for the technical side of the project and for the supply of catalyst. The plants were installed by Ruhrgas at filling stations for the purpose of 'methanising' town's gas, and charging it into cylinders at about 300 atmospheres pressure, for use as motor fuel. The A.G. der Kohlenwertstoff Verbande of Bochum, as distributors of liquid and other motor fuels, also had some interest in the commercial application of the process.

From the chemical standpoint the process had two objectives:

1. To make the town's gas into a better motor fuel by increasing its calorific value. This increase was said to be from between 450 and 500 to between 650 and 850 B.Th.U./cu.ft.

2. To remove constituents of the gas which had a corrosive effect on high-pressure storage vessels. These constituents were carbon monoxide, hydrocyanic acid, sulphur compounds and oxygen.

When the success of the process had been demonstrated in a pilot plant at Altenessen, arrangements were made for a large number of plants to be constructed. By the end of the war the following plants had been completed :

1. Altenessen (practically destroyed)
2. Dusseldorf gasworks) plants completed
3. Dresden) and used.
4. Opel works, Frankfurt-Russelheim) Plants completed
5. Carolinen Glück Colliery, Bochum) but not used.
6. Kokerei Zollverein, Gelsenkirchen - no details available.

Each of these plants was of essentially the same design which consisted of a basic unit capable of producing 180 cu.m. (6,200 cu.ft.) of gas per hour. If a greater output was required the plant was duplicated.

The plants were designed to be foolproof so that they could be operated by a non-technical filling-station attendant. They were also capable of being easily started and stopped as required by the erratic demand by road vehicles.

Details of the plant. The processing of the gas involved the following treatments :-

1. Boosting town's gas to a pressure sufficient to pass it through the plant.
2. Heating to a temperature of 400-450°C.
3. Addition of steam (in certain cases).
4. Catalytic conversion of organic sulphur compounds to hydrogen sulphide.
5. Cooling.
6. Removal of hydrogen sulphide.
7. Methanisation.
8. Cooling.
9. Compression to 350 atmospheres.

A flow diagram is given as Fig. 1.

Methane reactor: Details of the methanisation reaction vessel are given as Fig. 2. In this unit the catalyst was contained in 158 steel tubes about 28 m.m. bore x 165 cm. long. These tubes were surrounded by a water jacket which served both to take away the heat of the reaction when the plant was in operation, and, when the plant was shut down, to maintain the catalyst at a temperature at which methane synthesis would be positively started when the process gas was turned on.

A reactor which was examined at Oberhausen-Holten had been made by Gottfried Bischoff, Essen. The name plate also said that it had been tested to 30 atmospheres.

The maximum working pressure in the water jacket was said to be 20 atmospheres (215°C), with a normal temperature of 160-180°C.

Gas heating was employed to keep the reactor hot when it was not in use, and in order to equalise the temperature it was encased in fairly thick brickwork.

When the catalyst in the reactor became exhausted after about 1,000 hours of operation the whole vessel was disconnected and replaced by another containing fresh catalyst.

Sulphur reactor: The vessel in which the organic sulphur compounds were re-formed as sulphuretted hydrogen consisted of a 'churn' of approximately the same over-all dimensions as the methane reactor. It had no internal fittings apart from a grid to support the catalyst and suitable gas connections.

Various catalysts had been tried in this vessel, including one containing copper. It was now customary to use the exhausted and sulphided nickel catalyst from the methanisation stage heated to a temperature of 400-450°C.

Details of the efficiency of sulphur removal by this catalyst were not available.

Preparation of Catalyst. The catalyst containing 100 Ni + 15 MgO + 50 kieselguhr was prepared at Oberhausen-Holten as follows :-

Precipitation: The mixed nickel and magnesium nitrate solutions were precipitated at boiling temperature with a small excess of sodium carbonate solution, and the kieselguhr then added. After thorough mixing the whole was filtered and washed with water equivalent to 120 cc./gm. Ni.

Extrusion: The filter cake was extruded as rods 3 m.m. diameter by forcing it through a die by means of a gear pump. In the early stages there had been some lack of uniformity in the product because some separation of water occurred between the pump and the die, but this was overcome by putting an agitator at this point.

Drying: The drying of the rods was carried out on a gas heated belt which moved forward slowly and discharged the hardened rods from the far end.

Breaking: The dried rods were fed into a vertical cylindrical machine having an axial shaft on which turntables were fixed. The rotation of the shaft caused the rods to be flung against the inner wall of the cylinder and so ensured that all fragile material was degraded and also produced a good yield of the required size of granule.

Reduction: The granules, graded 3-5 m.m., were reduced in batches in a vessel shaped like a large covered Buchner funnel. The reducing gas consisted of 3:1 hydrogen-nitrogen mixture (obtained from the adjacent ammonia plant) preheated to 300-350°C. At the end of the reduction process the catalyst was cooled in reducing gas to 100°C. and further cooled in carbon dioxide. Catalyst prepared in this way could be handled in air for a sufficiently long time to allow filling of a reaction vessel without deterioration of the catalyst.

Kieselguhr: This was obtained from the Vereinigte Deutche Kieselguhrwerke, Hanover. It was 'burned' by the suppliers in the ordinary way to a temperature believed to be about 1000°C. The product, which was not acid washed, contained some sand and no soluble iron.

Operating Conditions.

Catalyst temperature: It was said that methanisation was carried out at a temperature of 160-180°C., and it was considered important that the temperature should not rise much higher than this if carbon deposition was to be avoided and water jacket temperatures kept within easily manageable limits.

It was recognised that during normal working the heat of reaction might take the catalyst temperature appreciably above that of the jacket. The activity of the catalyst at these low temperatures, however, appears to be confirmed because, it was stated, the plant could be reliably re-started after a shut-down when the catalyst was definitely at a temperature of 160-180°C. and the inlet gas was at a lower temperature.

It was stated that there was no appreciable loss of nickel from the catalyst under these conditions.

Space velocity: When the amount of carbon monoxide in the town's gas was less than 5 per cent. a single reactor, containing about 160 litres of catalyst space, was capable of methanising the whole 180 cu.m./hr. With higher proportions of carbon monoxide the gas throughput was maintained but two reactors were used in parallel.

Steaming: When the carbon monoxide content of the gas exceeded 10 per cent. steam was introduced into the sulphur reactor to oxidize some of the carbon monoxide to dioxide and to increase the hydrogen content.

A recommended amount of steam was that which would be required to saturate the gas at 30-40°C.

__Thermal efficiency.__ The following data relates to the plant at Altenessen.

1. The gas used for heating the reactors etc. was 6.4 per cent of the total input.

2. The maximum theoretical thermal value of the methanised gas was 94 per cent. of that of the gas processed, equivalent to 88 per cent. of the total gas used.

3. The actual thermal recoveries were 84 and 78.6 per cent. respectively.

__Design of larger plant.__ Dr. Traenckner said that although Ruhrgas hoped to use the process for other purposes and on a larger scale they had no such schemes prepared.

Attention is however drawn to the fact that during an interview with Dr. Kurt Haver of the A.G. der Kohlenwerstoff Verbande, Buchum (referred to in the report on targets 30/7.06, & 30/73 by H.H. Lowry, H.J. Rose and E.T. Wilkins) a drawing by Demag dated 8.12.42 was produced. A copy of this drawing is available in Bag 1481, K.W.V. Item 5. A.G.K.V. professed to know nothing about this drawing except that it concerned a Ruhrgas methanisation project.

The dimensions of the plant illustrated in this drawing are several times larger than those of the plants previously described. It is not yet known, however, whether this plant was an alternative design for a 180 c.m./hr. plant or whether it was intended for use with a larger gas throughput or with gas other than coal gas. In this connection it may be significant that whereas drawings of smaller plants were inscribed with the code-word "Promotor", the Demag drawing was inscribed "Permagas".

__Further information.__ Dr. Martin offered all possible assistance. Dr. Roehlen, who had been largely responsible for developing the process, was not available and according to Dr. Martin the most knowledgeable person on the subject was Dr. Rohe, a lawyer who was in charge of an establishment at Reelkirchen, near Hanover, where all records were stored.

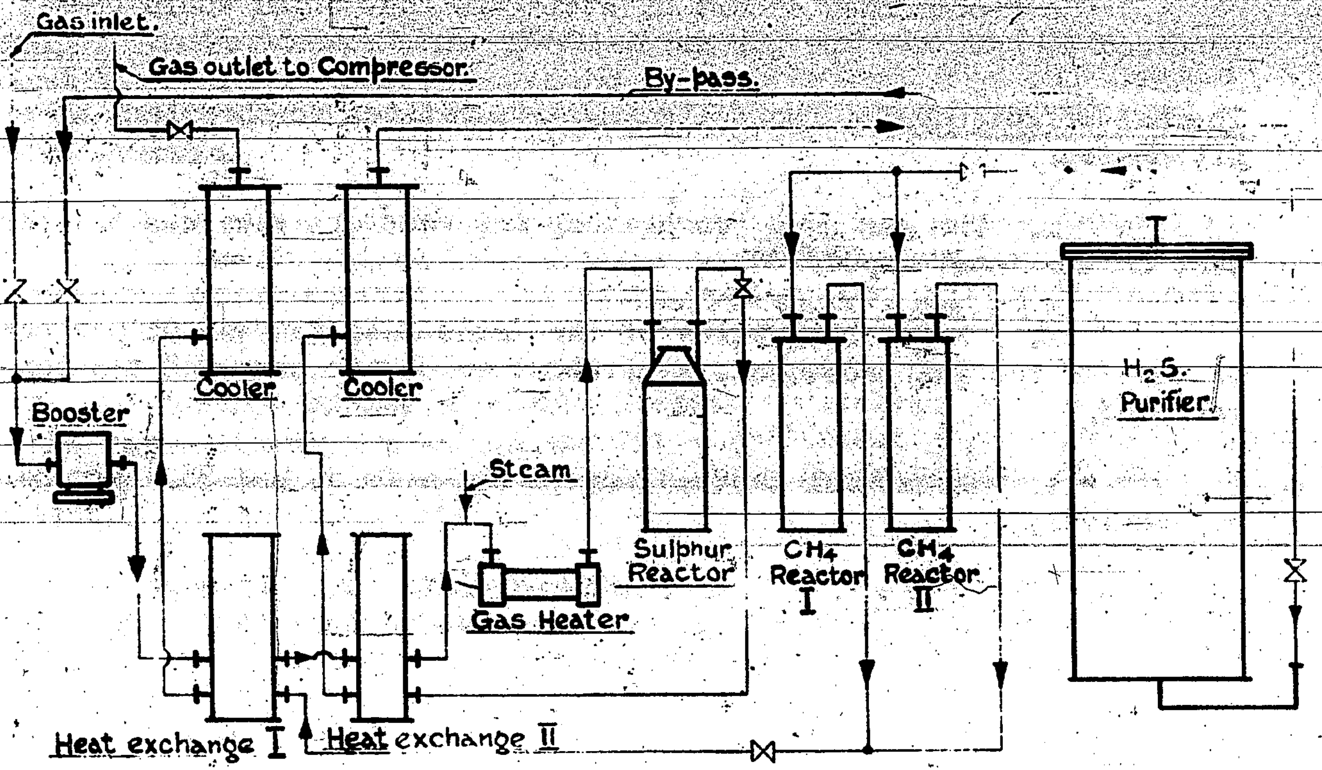

FIG. I. METHANISATION OF TOWN'S GAS.
FLOW DIAGRAM OF RUHRGAS – RUHRCHEMIE PROCESS.

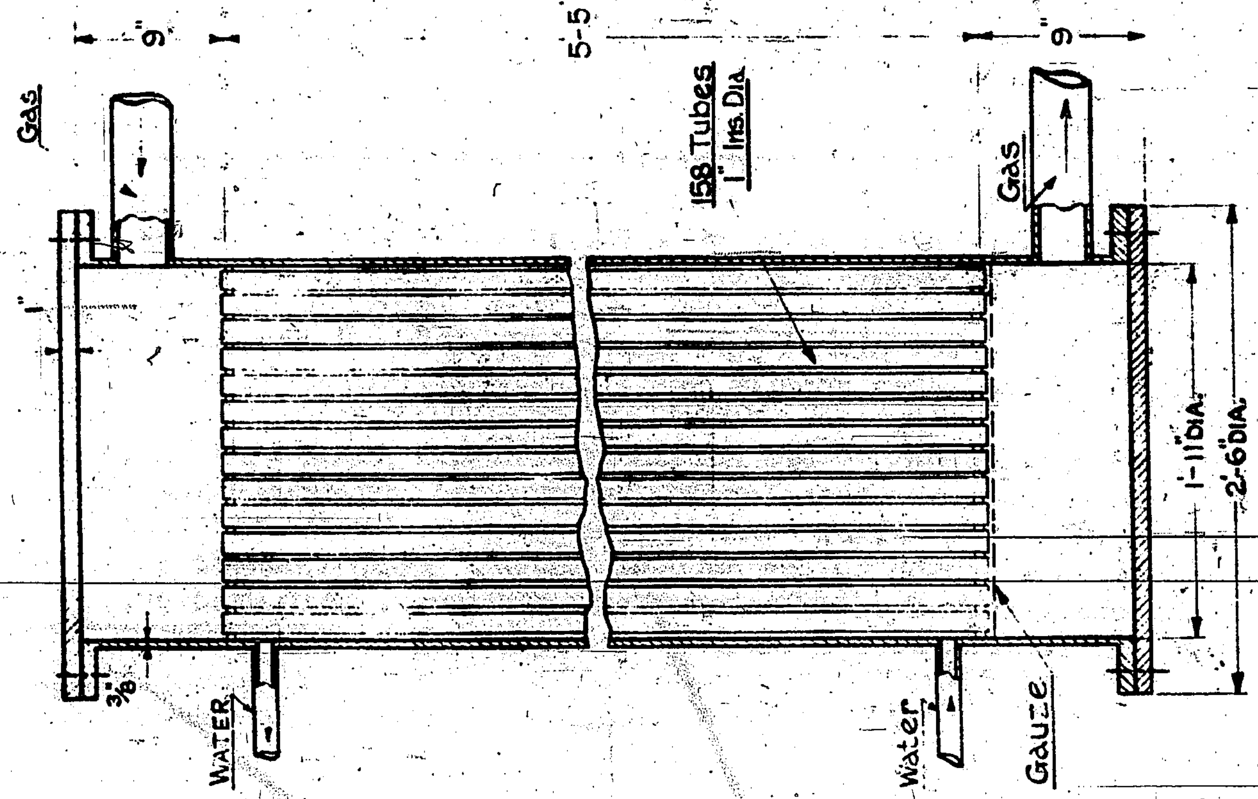

FIG. 2. METHANE REACTOR.
(Approx. to scale.)

ITEM No. 30.
FILE No. XXXIII-6

PB 18783

Copy 1

THE PREPARATION OF ULTRA-CLEAN COAL AT THE KÖNIGIN ELIZABETH COLLIERY, ESSEN-FRILLENDORF (MANNESMANNROHREN-WERKE A.G.)

Wilkins, E.T.

"This report is issued with the warning that, if the subject matter should be protected by British Patents or Patent applications, this publication cannot be held to give any protection against action for infringement."

REC'D. ....
TIIC L.F. & L. S-C.

COMBINED INTELLIGENCE OBJECTIVES

SUB-COMMITTEE

LONDON - H. M. STATIONERY OFFICE.

The Preparation of Ultra-clean Coal at the Konigin Elizabeth Colliery. Essen-Frillendorf.

(Mannesmannrohren Werke A.G.)

Reported by

E.T. Wilkins on behalf of

British Ministry of Fuel and Power

and the

U.S. Technical Industrial Intelligence Committee.

CIOS Target No. C30/344

Combined Intelligence Objectives Sub-Committee

G 2 Division, SHAEF (Rear) APO 413.

(1946)

6p.