

exhausted, it is dropped out into a pit, where it fires spontaneously. The catalyst is allowed to burn itself clean, - being turned over occasionally with a rake - and is then topped up, if necessary, with phosphoric acid and re-used. One batch had been put back four or five times.

Condensing and Separator System (9,10,11). This was stated to be make-shift. The product vapour, leaving the top of the catalyst chamber at 320°C., passes along pipe (8), through the heat exchanger (5) and so at a temperature of 40°C. to the condensing system. This consists of a receiver at the bottom, fitted with run-off, surmounted by a 0.9 m. diam. cylindrical vessel, 2½ m. high, packed with stone rings. A cooler follows, then 3 m. more of raschig rings, a further cooler and a liquid cyclone. All the parts are of iron construction.

The condensate is then run to tank storage, which consists of six 20 m<sup>3</sup> tanks. In these the product is allowed to stand at least two days for separation of water and ammonia from the adiponitrile. The crude nitrile is then subjected to distillation under reduced pressure. The aqueous ammonia contains some 2 - 3% of nitrile, which at present is not recovered.

Yield. The yield claimed is about 83%, based on adipic acid. The crude nitrile is distilled with 97% yield to give an overall figure of 80.5%. The principal by-product is 1-imino 2-cyano cyclopentane.

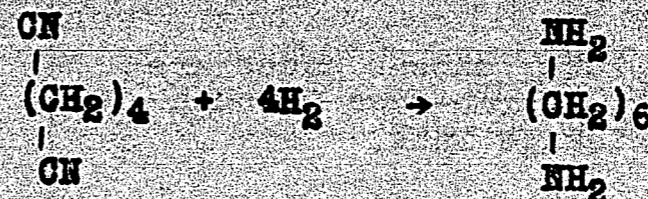
Other catalysts. Boron phosphate was said to have been tried in the laboratory. It gave no better yield and could not be regenerated.

## PART II.

### Hexamethylene Diamine from Adiponitrile.

#### General.

Adipic dinitrile can be reduced with hydrogen under pressure in the presence of nickel, cobalt and similar catalysts.



The reaction is best carried out in presence of ammonia.

#### Description of Process.

A flow-diagram is given on page 9. The apparatus required is extremely simple and consists of a large stirred autoclave fitted with the necessary valves for admission or removal of gas and liquid reactants, together with ancillary tanks for storage of reagents and products.

Pure adiponitrile is stored in a large cast-iron vessel of 1000 kgrms. capacity. A smaller vessel, fitted with a stirrer, holds a suspension of cobalt catalyst in adiponitrile.

800 kgrms. adiponitrile are run into the large autoclave, sufficient nitrile being fed from the smaller stirred vessel to give 8 kgrms. cobalt. (1% on the weight of nitrile). The autoclave, which is mounted horizontally, has a capacity of 3 m<sup>3</sup>, and is 7 m. long and 0.8 m. inside diameter. It is fitted with a stirrer and is made of alloy steel (not V2A or V4A). The autoclave has an aluminium jacket containing tubes for steam or hot or cold water.

The temperature of the autoclave having been raised to 80°C. by means of hot water or steam in the jacket, 80 - 100 kgrms. of ammonia are introduced from a cylinder, and the pressure is then raised cautiously to 50 - 80 ats. by admission of hydrogen from a high pressure source.

Maintenance of the pressure at 50 - 80 ats. is sufficient in most cases to ensure steady hydrogenation, and cold water cooling is necessary to prevent the temperature from rising above 150°-160°C. As reaction slows, the hydrogen pressure is raised to 200 ats. to finish the reaction.



When hydrogenation is complete, excess gases are blown off and the liquid product is run-down through a cooler to the crude diamine storage tank.

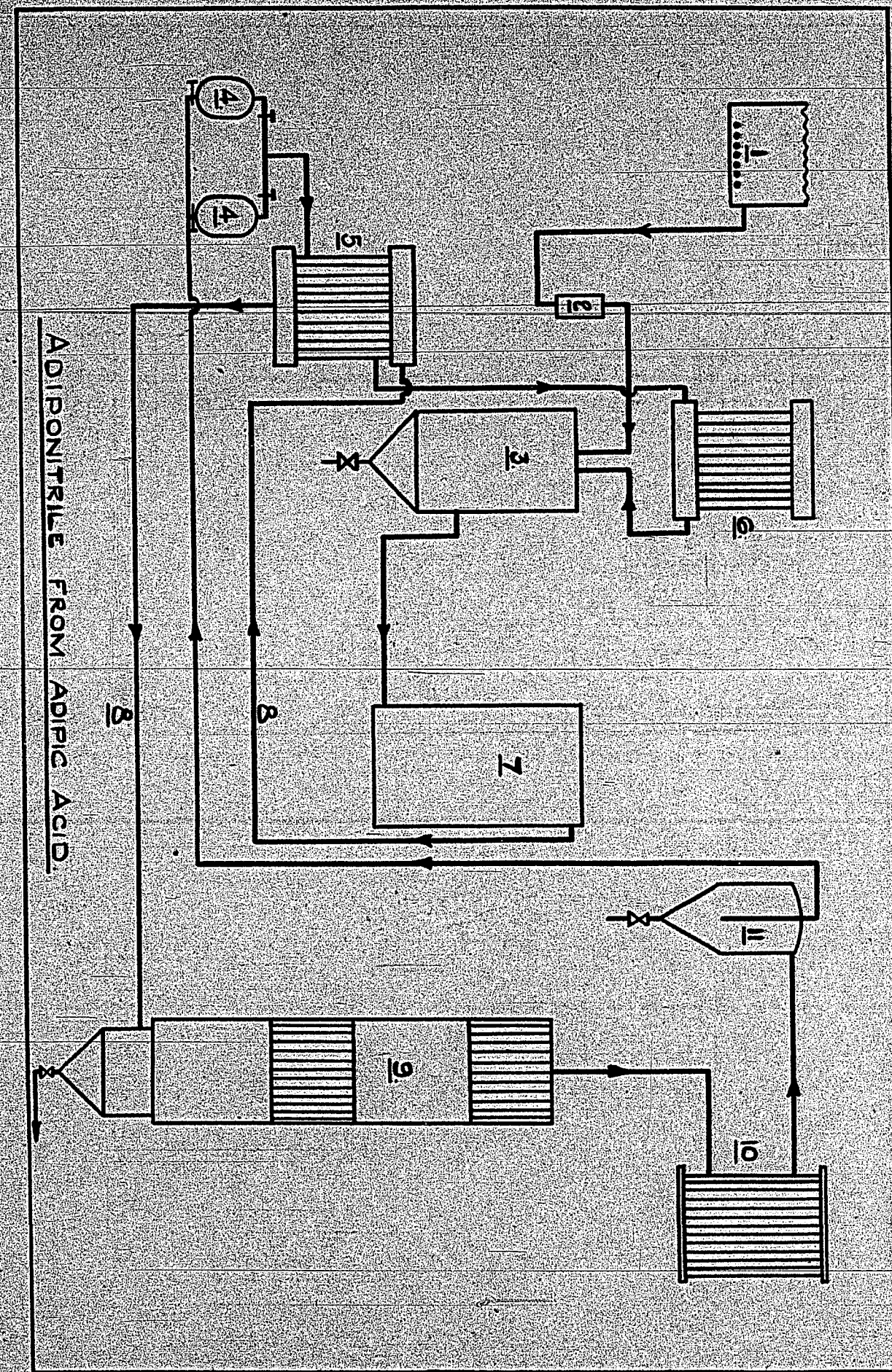
The crude product is then distilled under low pressure.

**Yield.** The yield of distilled hexamethylene diamine is claimed as 80%, based on the pure dinitrile. 7-10% of hexamethylene imine is formed.

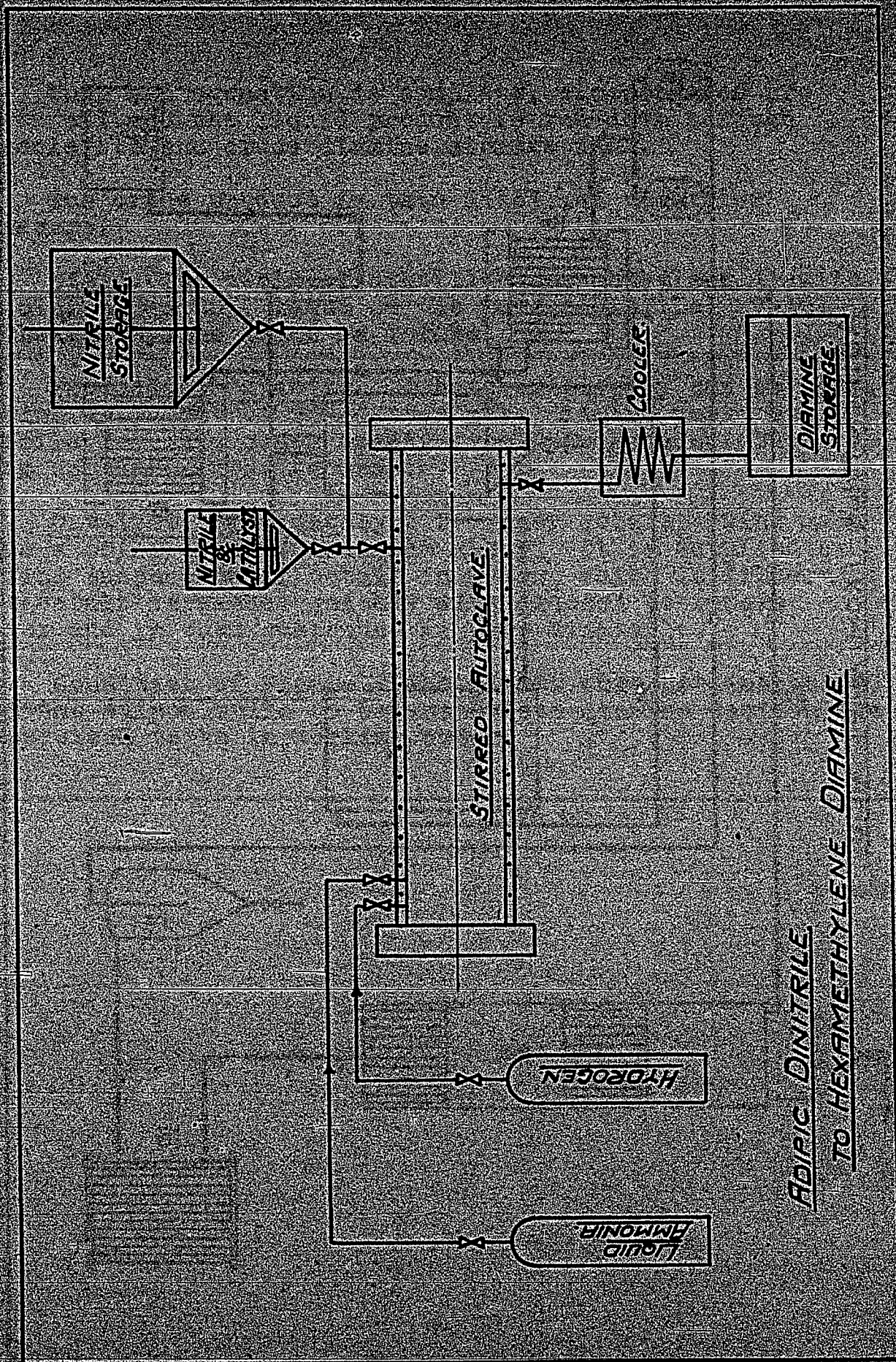
**Production Capacity.** 4 batches per day can be made, giving an output of 70-80 tons pure diamine per month. Nitrile for this comes mainly from Ludwigshafen, but some from another source is used.

**Catalyst.** The catalyst is Raney cobalt, being made from a 50/50 cobalt/aluminium alloy. It is supplied as a fine suspension of cobalt in water, which is filtered. The cobalt is washed quickly with alcohol to remove water and is then suspended in pure dinitrile.

**Condition of Plants.** The plants were not inspected, but the adiponitrile to hexamethylene diamine section was understood to be in working order.







*ADIPIC DINITRILE  
TO HEXAMETHYLENE DIAMINE*



B.I.O.S. FINAL REPORT No. 498

ITEM No. 31

*Copy 1*

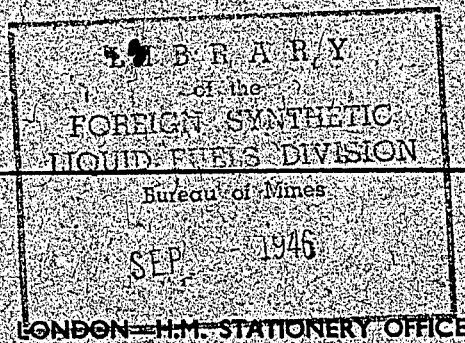
# WARTIME DEVELOPMENT IN THE DESIGN OF BOILERS AND COMBUSTION EQUIPMENT IN GERMANY

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.

*Franklin, A. (11)*

BRITISH INTELLIGENCE OBJECTIVES

SUB-COMMITTEE





BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE  
32 Bryanston Square, London W.1.  
Ambassador 2861, Ext 636

BIOS/Pbl/142/3

4 June 1946

SUBJECT: BIOS Final Report No. 498

TO : Chief, Integration and Planning Branch,  
FIAT (US)  
c/o USFET Main  
APO 757, US Army

1. Reference our BIOS/Pbl/142/3 dated 15 May 1946.

2. The following amendments should be made to BIOS final Report No. 498 entitled "Wartime Development in the Design of Boilers and Combustion Equipment in Germany" : -

Page 9, line 7 - ' (27" ) ' should read ' 31 $\frac{1}{2}$ " '   
" 17, " 15 - ' 5" 8" ' should read ' 5/8" '   
" 21, " 14 - ' Gr.No ' should read ' Cr.No. '

For the Secretariat

J.E. PARKINSON  
Captain  
Publications



WARTIME DEVELOPMENT IN THE DESIGN OF BOILERS  
AND COMBUSTION EQUIPMENT IN GERMANY.

BIOS Target Numbers

C31/1096, C31/920, C31/1896, C31/928,  
C31/1948, C31/924, C31/504, C31/1487,  
C31/770, C31/758, C31/66, C31/759,  
C31/1889, C31/747, C31/2303, C31/745,  
C31/474.

REPORT BY B.I.O.S. TEAM NO. 1381  
ON VISIT TO GERMANY DURING  
OCTOBER AND NOVEMBER 1945.

Mr. A. Franklin  
Mr. A.L. Timmins  
Mr. T. Dunsmore  
Mr. A.L. Napier

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE  
32 Bryanston Square, London, W.1.

(1946)

55 p. diagrs. (part fold.)



"A"

I N D E X

| <u>No.</u> |  | <u>Page No.</u> |
|------------|--|-----------------|
| 1.         | General Introductory Survey.   | 1 - 6           |
|            | <u>GENERAL CONCLUSIONS</u>   |                 |
| 2.         | Fuel Supplies.   | 7               |
| 3.         | Steam Generating Conditions.   | 8               |
| 4.         | Superheaters   | 9 - 10          |
| 5.         | Types of Boilers.  | 11 - 14         |
| 6.         | Economisers.   | 15              |
| 7.         | Airheaters.  | 16 - 17         |
| 8.         | Methods of Calculation.  | 18              |
| 9.         | Detail of Design.  | 19 - 21         |
| 10.        | Automatic Control.   | 22              |
| 11.        | Boiler Availability.   | 23              |
| 12.        | Pulverised Fuel Firing.  | 24 - 26         |
| 13.        | Mechanical Stokers.  | 27 - 32         |
|            | <u>DETAILED TECHNICAL REPORTS OF INDIVIDUAL PLANTS.</u>                                  |                 |
| C31/1096   | 14. La Mont Kessel Herpen & Co., Schadowstrasse<br>1a, Berlin.                           | 33 - 34         |
| C31/920    | 15. Kohlenscheidungsgesellschaft M.B.H., Gotlieb<br>Dunkelstr. 20/22, Tempelhof, Berlin. | 34 - 35         |
| C31/1896   | 16. Dr. Munzinger, c/o A.E.G. Offices,<br>Hohenzollern Damm 120, Berlin.                 | 35 - 38         |
| C31/928    | 17. Curt Von Grueber, Ruhldorferstrasse,<br>Teltow, Berlin.                              | 39              |
| C31/1948   | 18. Wagner H.D.T. Glockengieserwall 1,<br>Hamburg.                                       | 39 - 40         |
| C31/924    | 19. Deutsche Werft, Finkenwarden, Hamburg.   | 40              |
| C31/504    | 20. Deschimag, Bremen.   | 41              |
| C31/1487   | 21. Press und Walzwerk, Oberbilk, Dusseldorf<br>and Reisholz, Dusseldorf.                | 42              |
| C31/1487   | 22. Mannesmann Rohrenwerke, Rath, Dusseldorf.  | 43              |
| C31/758    | 23. Vereinigte Kesselwerke A.G. - "V.K.W."<br>Werdenerstrasse, Dusseldorf.               | 43 - 45         |
| C31/66     | 24. L. & C. Steinmuller, Gummersbach, Rhineland.   | 45 - 48         |

"A" (Contd.)

| <u>No.</u> |  | <u>Page No.</u> |
|------------|--|-----------------|
| C31/759    | 25. Walther Und Cie., A.G. Koln Delbruck<br>(Nr. Cologne).   | 48.             |
| C31/1889   | 26. Claudius Peters Wallhof, Glockengeissenwall,<br>Hamburg. | 48 - 49         |
| C31/747    | 27. Durrwerke, Ratingen, Dusseldorf.                         | 49              |
| C31/2303   | 28. Babcock-Werke, Oberhausen (Ruhr)                         | 49              |
| C31/746    | 29. Deutsche Rohrenwerke, Mulheim - Ruhr.                    | 49 - 50         |
| C31/474    | 30. Gebruder Sulzer Aktiengesellschaft,<br>Ludwigshafen.     | 50 - 51         |
|            | List of Illustrations  | 52              |



GENERAL INTRODUCTORY SURVEY

1. This report is based on investigations and enquiries during a five weeks tour of Western and Northern Germany in October and November 1945 when 95% of German industry was alleged to be inoperative. The information was acquired by interrogating technical officials of boiler design and construction concerns. Some had no manufacturing facilities and in the majority of other cases their works were shut down due to lack of power and other difficulties.

The principal problems facing boiler designers appeared to be:-

- (a) Shortage of bituminous coals, with the consequent necessity of developing boilers and combustion equipment adapted to brown coal.
- (b) Inadequate capacity for manufacturing boiler drums, due to priority demands of the synthetic oil industry.
- (c) The denial of supplies of alloys, particularly molybdenum and chrome.
- (d) There also seems to have been a shortage of iron and steel, because many boilers were constructed with brickwork settings, and mechanical stokers were of light fabricated steel construction, no cast iron being used except in the actual grate surface.

Apart from very recent experiments with Rotary boilers, little development appears to have taken place, and even such problems as bonded deposits were accepted rather than systematically investigated.

It appears that for economic reasons boilers were designed with a minimum number of drums, and were generally of a low-head type for accommodation in bomb-proof buildings. Forced circulation was adopted, and quite a number of Lamont

boilers were built for working conditions of approximately 80 Atmospheres and 500 degrees Centigrade. Owing to the bottleneck in drum manufacture and the need generally to sanction installations with a minimum raw material requirement, the Benson boiler appeared to gain favour, as in theory it required no drums, although in practice it was found that a separator drum was desirable. This was actually incorporated in practically all the latest units of the Benson type. It was also widely admitted that the Benson boiler was not flexible and had to be steamed continuously at M.C.R. to avoid furnace and superheater troubles.

At the end of the war the tendency was to revert to natural circulation boilers.

Prior to the war a standardized boiler design had been evolved by the German Water Tube Boilermakers' Association, incorporating the best features of all leading types. The intention was to develop a restricted series of evaporative capacities, working pressures, and steam temperatures, which were considered to cover all requirements. This policy was maintained to some extent throughout the war, particularly in its latter stages, but no thorough-going standardization was ever achieved. On the contrary, it was found that in many installations comprising a number of boilers, such contracts were divided among two, three, or more boiler manufacturers, one of which was nominated as responsible for details of design, the others working to the selected firm's drawings. This, of course, had as an object the spreading of work, drawing office savings etc., for example, a total of twelve boilers required for a new installation at a steel works in Gelsenkirchen was allocated as follows:-

|          |   |
|----------|---|
| Babcocks | 4 |
| V.K.W.   | 2 |



|             |   |
|-------------|---|
| Steinmuller | 2 |
| Walther     | 2 |
| Durr        | 2 |

In such cases boilers appeared, as a rule, to be designed for pulverized fuel firing and incorporated large combustion chambers, minimum convection surface, controlled superheaters specially constructed to avoid fouling, water or steam cooled superheater supports and large economisers. On boilers designed for brown coal there was as a rule no air heater. Most designs incorporated one steam and water drum with very simple baffles and one steam separating drum.

The re-heating cycle does not appear to have been employed extensively, although re-heaters were shown on a number of project drawings.

The actual Boiler Laws in Germany had not been modified for very many years, and design, scantlings, etc., were governed by standards laid down by the V.D.I. (German Engineers' Association). Contractors of importance had for many years past specified, in addition, compliance with the recommendations of the Germany Association of Large Steam Boiler Owners. Their code is readily available, but the Association of large Steam Boiler Owners had for many years past issued, in addition, confidential recommendations and comments on boiler design, construction, operation, etc., which are in part of the greatest interest and value. It is strongly recommended that the Water-Tube Boilermakers' Association should arrange through the Board of Trade to be provided with a complete set of their confidential intimations, which were issued both prior to and during the war. These printed notes had only a restricted circulation, no member of the Association being allowed to have more than one copy, and to obtain them it would presumably be necessary to requisition a set either from the German Association of Large Steam Boiler Owners or from one of its Members.

The thirty-three boiler manufacturers in Germany were organized in the German Water Tube Boilermakers' Association, which was brought into the Nazi organization and subjected to instruction on all matters by the Nazi authorities. As an Association, however, it had little or no part in framing any official regulations, and could exercise its influence only through the Large Steam Boiler Owners Association.

In regard to construction, seamless drums were used above a working pressure of 65 Atmospheres, mounting nozzles were welded but drum tube connections were expanded. Headers were fitted with welded or solid drawn stubs, to which tube and superheater elements were in turn welded, no reinforcing rings being used. Electrical welding seems to have been almost exclusively employed in wartime, except for thin shells which were gas-heated and hammered. The use of all-welded drums was confined to working pressures below 35 Atmospheres. For pressures between 35 and 65 Atmospheres, drum ends were electrically welded to seamless shells. From observation riveting work had practically been discarded.

The basis of more modern designs for large evaporations was 80 to 120 Atmospheres (very few above 100 Atmospheres) and 520 degrees centigrade final steam temperature. We were informed that stations rarely operated at full designed Superheat, but could not confirm.

Generally speaking boilers above 50 tons per hour capacity were pulverized fuel fired, using K.S.G., or a development of the old Kraemer Mill for brown coal and Loesche or Babcock Type "E" Mills for hard coals. The Anger Mill was also being developed. Small boilers were generally designed for 40 Atmospheres or less, and were usually stoker fired. The leading travelling grate appeared to be Steinmullers, and a good deal was said about their water-cooled stoker, especially for Marine Boilers. The Martin Stoker was reported to be



very successful in burning washery slurry.

It was found that little actual development has taken place with regard to the installation of slag tap furnaces although isolated plants had been built. It was noted, however, that a fair number of the most recent projects embodied this feature. The general principle when burning low-grade fuels seemed to be to grind coarsely and fit a static grate at the bottom of the furnace to collect and burn out over-size particles. This was standard with the Kraemer type mills, and extended to other pulverizers as well.

Owing to fouling troubles, boiler availability appears to have been poor as a general rule. The problem was attacked by providing the widest possible gas lanes and numerous lancing doors. Steam soot blowers; compressed air lances; gravel guns; and poking rods were employed, but water lancing was only applied in boiler furnaces to shift obstinate accumulations of slag. No cases of superheater rapping were encountered, but latest designs employed flexible superheater elements, which were expected to clear themselves by natural vibration.

Steam purity does not appear to have been seriously studied except by the Lamont Company and Wagners.

Although a large number of manufacturing works was visited, it is not considered that there was any feature in which German methods of production were in advance of this country in so far as boiler manufacture is concerned. Machine tools and other equipment were in many cases in poor condition and old. All German Boilermakers use single spindle radial drilling machines for drilling tube holes in drums and in headers.

There appeared to be very many less restrictions on manufacturing methods in Germany than are in existence in

England under British Insurance Companies' requirements and Board of Trade Inspection Regulations.



### GENERAL CONCLUSIONS

#### FUEL SUPPLIES

2. The fuel situation in Germany gradually worsened during the course of the war and had reached a very serious stage towards its conclusion, greatly accentuated of course by the heavy bombing of all means of transportation. As a result boiler plants were obliged to employ any class of fuel which could be allocated to them, in many cases the fuel actually employed differing greatly from that for which the plant was designed or being very inferior in quality. The ash content of hard coals reached 35/40%.

This state of affairs was accentuated by the German policy of reserving the bulk of good quality coal for oil distillation thus forcing power plant on to poorer grade fuel.

In addition new workings had to be opened up producing fuel of which the combustion characteristics in boiler furnaces were but little known.

It is estimated that more than half the steam generating plant throughout Germany as a whole was fired with brown coal, either by means of pulverised fuel firing or on various types of mechanical step grates, the coal containing a moisture content of 40% and more after weathering. No pre-drying of this fuel was undertaken other than, of course, drying carried out in the pulverising mills by the use of high temperature air or by connecting the mill to the furnace by means of a duct through which the hot gases were drawn.

#### STEAM GENERATING CONDITIONS.

5. In the majority of the larger sizes of boilers, as installed in Power Stations, Chemical Works, etc., M.C.R. evaporations appear to range between 75 and 130 tons/hr. and working pressures between 80 and 130 atmospheres with steam temperature generally about 500°C.

The largest sizes operating or in course of manufacture are said to be:-

Travelling stoker-fired - 80 tons/hr.

Retort stoker-fired - 120 tons/hr.

Pulverised fuel-fired - 200 tons/hr.

The maximum pressure so far designed for is 160 atmospheres and the maximum steam temperature 520°C, although it is understood that in some cases plant designed for 500/520°C. was actually being operated to restrict the maximum steam temperature to about 480°C. in order to avoid operating troubles.

Steam temperature control is generally effected by the use of interstage desuperheaters of either direct injection or surface type, gas by-passing for this purpose being rarely employed. Alternatively, a number of projects showed a combination of radiant secondary with convection primary and no desuperheater.

Standardised M.C.R. outputs, pressures and steam temperatures are laid down in standard specification DIN 2901 which contains tables covering (in stages) M.C.R. outputs of 25 up to 250 tons per hour, pressures of 0.5 to 230 atmospheres and steam temperatures of 150 up to 500°C.

Clients requiring a boiler for an intermediate output, pressure or steam temperature would be provided with a unit actually designed for the next highest conditions. It should be noted that the pressures in this specification are as measured in the steam drum and not at the superheater outlet and that normal load is to be considered as 80% of M.C.R.



### SUPERHEATERS

4. The "slot" or "channel" type of superheater is a recent development which appears to have overcome the trouble experienced with choking which occurs particularly when bad coal is used with ordinary superheaters having closely spaced tubes.

In the "slot" or "channel" type, the superheater tubes are arranged in groups widely spaced across the boiler at 800 mm. pitch (31 1/2"), each group comprising several tubes one behind the other, but each group being only one tube wide.

The groups hang vertical and the maximum amount of tube surface is thus exposed directly to radiation and is not screened from the furnace by adjacent rows of tubes.

Each group of tubes is free to move and there is a natural tendency for the tubes to vibrate and this serves to prevent ash and dust building up on the tubes.

Usually only the hottest part or final stage of the superheater is constructed in this way.

The primary superheater of convection type is placed further back in the boiler system where it is exposed to boiler gases at lower temperature but not exposed to radiation effect.

STEAM VELOCITY: K.S.G. design their superheaters with inlet steam velocity 36 ft. per second, outlet steam velocity 60/65 ft. per second..

SUPPORT OF SUPERHEATER TUBES: In many recent designs the superheater tubes, which are arranged in groups, are placed horizontal and a small number of the superheater tubes are diverted to act as supports for the remainder.

In one example, saturated steam from the boiler drum passes downwards through vertical support tubes which are threaded between the groups of tubes forming the secondary or final superheater.

Every one of the groups of tubes across the width of the superheater has its own pair of vertical support tubes which extend below the groups and are connected to a drain header below the secondary superheater.

The support tubes are provided with small side lugs, welded on, to locate and to support each of the horizontal legs of the superheater tubes in the group.

After passing through the support tube to the drain header, the steam passes through riser tubes across to the primary superheater which is located in the next pass behind the secondary superheater.

The primary superheater is built up in groups of horizontal tubes and these, in turn, are carried on another set of vertical support tubes, but in this case, these are actually the riser tubes from the economiser which is located directly underneath the primary superheater.

These vertical support tubes also have side lugs for locating and supporting the primary superheater tubes and the support tubes are threaded between the groups of primary superheater tubes and are then carried over to connect into the boiler drum and thus supply the drum with feed water from the economiser.



#### TYPES OF BOILER

5. Forced circulation boilers were largely adopted for some years, but they appeared to have given rise to serious operating difficulties, and an examination of the latest projects indicated a reversion to natural circulation designs.

It was ascertained that whereas in 1939 the proportion of natural circulation boilers to forced circulation boilers installed was 72.2%, this ratio increased in 1941 to 90.5% and has remained round 90% from then on.

Efforts were made to standardise boilers through the Associations of Large Boiler Users and Manufacturers, but except for details such as tube pitches, radius of bends, etc., nothing very definite seems to have emerged. On the other hand some authority had evidently dictated the general adoption of specific types, e.g. Lamont, and more especially, Benson.

Numerous Lamont boilers were installed for both land and marine use. They were chiefly designed for working pressures up to 80 atm. One unit was erected at I.G. Farben Works, Hoechst, for 130 atm. working pressure, but had not actually been in operation. Most circulating pumps used were by K.S.B. or Sulzer. The latest Lamont designs incorporated an internal pump fulfilling the dual purpose of circulator and steam separator. It was stated that Lamont boilers were unsatisfactory in warships owing to chokage of nozzles by rust and scale formed when standing in harbour.

Benson boilers were sponsored by the Siemens Company on the grounds of cheapness through the elimination of drums. They were assembled in sections in quite small manufacturers' works. Satisfactory operation depended on pure feed water and base load steaming. Circulation, furnace performance, and steam temperature were erratic at reduced ratings. For marine purposes Benson boilers were very expensive as all auxiliaries were designed for high pressure

and temperature. They were not adapted to manoeuvring, but performance was improved by introducing a steam separator drum coupled with Askania automatic combustion control.

Sulzer Monotube boilers did not appear to have been employed. Gebr. Sulzer's works at Ludwigshaven were controlled by Pont-a-Mousson after the occupation of France, and appear to have been engaged chiefly on pumps, Lamont boilers, and natural circulation units, before being rendered inactive. Their latest Monotube design was based on the use of a single feed pump instead of a series feeding individual Monotubes.

Schmidt boilers were not developed during the war, although about 70 units had been made by V.K.W. prior to going over to Benson and K.S.G. designs.

No Velox boilers were installed in Germany, but about 70 oil fired units of this type were manufactured for export to Switzerland and South America.

Loeffler boiler development was in the hands of M.A.N. Nuremberg. None had been made for several years. Only two Loeffler boilers were heard of outside of any activities by the proprietors at Witcovitz. Both were fired by travelling grate stokers and gave a great deal of trouble due to unequal circulation and external fouling. The example at Leverkusen had been destroyed and the unit at Hoechst was not in use.

A small Rotary boiler had been developed jointly by Vorkauf and Huettner but was still in an early experimental stage. They had also designed a gas turbine, but this had not gone beyond the drawing board. A.E.G. had also experimented with an Escher-Wyss indirectly fired gas turbine, but were in difficulties with the design of a high temperature air heater.

Although manufacture was still proceeding on Benson type boilers and Lamont were also still developing, most of the new projects examined showed natural circulation boilers.



Very few boilers of this type seemed to have been installed in Western Germany during the war, but most of the latest boiler plants were apparently erected in Eastern Germany, and these were reported to comprise a considerable proportion of natural circulation boilers. All projects examined showed pulverised fuel fired boilers adapted from a basic design which was reported to have been developed by K.S.G. The fundamental features were -

- (a) A high furnace with minimum refractories.
- (b) A water cooled ash hopper.
- (c) External unheated downcomers.
- (d) One steam and water drum screened from the furnace by a monolithic roof baffle. This was sometimes augmented by an auxiliary steam separator drum.
- (e) Minimum convection surface - usually a widely pitched slag screen formed by water wall risers or by economiser discharge tubes - which was followed by a preliminary steam dryer.
- (f) Water cooled or low temperature steam cooled economiser and superheater supports.

The superheater arrangement varied according to the class of coal. For hard coals the dryer section was followed by secondary and primary horizontal self-draining contra flow convection superheaters, or alternatively a radiant primary in the furnace side wall with contra flow self-draining convection type secondary superheater. For brown coal the secondary superheater was of the "Channel" type with a convection primary.

The reheating cycle had not been widely adopted, but several projects showed reheaters which were of the convection type with convection primary superheaters, or of the

radiant type with radiant primaries.

Some projects illustrated slag tap furnaces. These were fired by long flame burners through the front wall. The lower part of the combustion chamber was refractory built and separated from the upper water cooled section by a slag screen. The furnace floor was flat and was arranged so that molten slag flowed over a circular weir into a sump which was cooled by a supply of water independent of the boiler circulating system.

Latest marine boilers were also of the natural circulation type with unheated downcomers. War vessels were oil fired, but some merchant ships were fitted with Steinmuller water cooled stokers.

In the appendix will be found a number of typical drawings of modern boiler designs.



#### ECONOMISERS

6. The most common design of economiser appeared to be a gilled tube type comprising flanged steel gills threaded on to a steel tube and welded at the flanges. One refinement of the design involved passing an expander through the tube to ensure better contact with the gills. Steinnullers made a gilled tube economiser in special cast iron suitable for working pressures up to and above 60 atm.

#### AIRHEATERS

7. The maximum air temperature employed seemed to be 350 degrees C. on pulverised fuel fired installations. Hot air was not favoured for travelling grates.

Very few airheaters were seen during the tour, but the special light-weight airheaters constructed to the designs of Wagner of Hamburg are interesting.

The tubes for this airheater are of streamline cross section approximately  $2\frac{1}{2}$ " wide x  $\frac{1}{2}$ " thick at the nose edge, tapering to point at other edge.

These tubes are built up in banks of 6 of various lengths, the longest plate being 2.6 metres (8'6").

The ends of the tubes in each bank are welded into end plates which are rectangular, having all four edges flanged over.

The complete airheater is built up very simply by bolting together groups of banks.

The airheater tubes are made of thin alloy steel strip bent over at leading edge to streamline shape, and the two edges of the strip are welded together at the point or rear of the tube section.

The alloy steel contains 17% Chromium, and the thickness varies from 0.4 mm. to 0.5 mm. In special cases, 0.75 mm. has been used. (In English dimensions these thicknesses are respectively - 0.01575", 0.0197" and 0.0295", and it means that the thinnest metal used is approximately 1/64" thick).

Before the strips are bent over to form tubes, they are lightly corrugated with two rows of corrugations so that these will occur on both of the long sides of the tube. This corrugating has a considerable stiffening effect.

The boiler gases pass between the tubes.



Spaces between tubes approximately  $3/8"$  to  $1/2"$  at the thickest part of the tube.

Wagners said that most airheaters of their design were built by a firm in Harburg, and in one special light-weight design they used monel metal only 0.2 mm. thick (.0075").

During the war, monel metal has been unobtainable in Germany.

At Steirnullor, a cast iron airheater element was seen. Each element was  $9\frac{1}{2}"$  x  $1\frac{3}{8}"$  cross section and 10'0" long, with long edges rounded off at each end, the casting terminating in rectangular box  $9\frac{1}{2}"$  x 2", with holes for bolting to adjacent boxes.

The body of the casting had spacing lugs to register against similar lugs on adjacent castings.

The gases passed between the elements, the space for access being approximately  $1/4"$ .

The idea appeared interesting but would be a very expensive type of construction and may be of use where low temperature boiler gases might lead to corrosion of ordinary airheater sections made of mild steel.

#### METHODS OF CALCULATION

8. The methods in general use for calculating boiler plant surfaces and performance are based mainly on Dr. Münzinger's book "DAMPKRÄFT" and on the published work of Schack, Nusselt, Reiner, Rosin and Fehling, supplemented by data obtained from manufacturers' own experience.

It is agreed by German boiler designers that their methods in general are very cumbersome. The principal Boilermakers in collaboration have been trying to evolve simpler methods on a common basis but have not yet reached agreement. There seems to be little attempt to use "short cuts" or empirical methods based on actual results.



DETAIL OF DESIGN

MANHOLES

9. One of the most interesting points of design which was seen during the tour was the circular manlid designed by Wagner of Hamburg.

Copy of Wagner drawing showing details of this circular manlid was obtained.

This circular manlid can be assembled entirely from outside the drum but is really only applicable to forged drums having extra thick ends, as a portion of the manhole opening in the drum end has to be screwed to suit the dog ring of the manlid assembly.

The dog ring is screwed into the outer portion of the manhole with a very coarse Acme type thread. The manlid itself is circular and is slightly smaller than the manhole in the drum end.

Long set pins are passed through holes in the dog ring and are screwed into tapped holes in the manlid and thereby draw the manlid out towards the dog ring.

Edges of manlid and corresponding edges of dog ring are bevelled and between them is a narrow bevelled ring of soft iron, Krupps W.W.1. - nearest British equivalent is Amco iron.

The wedging action of the bevelled faces causes the joint rings to be forced tightly against the inner surface of the manhole opening in the drum end.

The joint is made actually in two places, one between the manlid and the soft iron ring; the other between the ring and the drum end.

Wagners use this type of manlid on all their boilers and they said it has been used on chemical work for pressures up to 200 atmospheres (2840 lbs).

TUBE HOLES

Usual practice is to provide one groove in the tube holes of boiler drums and headers. Most firms bore larger sizes of tube holes by first drilling a pilot hole and then removing remainder of material with a tool resembling an end milling cutter with a central guide plug which fits in the pilot hole.

All German Boilermakers use single spindle radial drilling machines for boring tube holes. These machines are of very robust construction and are fully motorised with all controls grouped for easy handling by the operator.

Practically all machines have an ammeter to show the electrical load and a tachometer showing the spindle speed. These instruments are particularly useful to the shop foreman and Works manager who can see at a glance whether the machine is being operated to the best advantage.

DRUMS

Press & Walzwerk are able to make pressed and drawn drum shells up to 39" diameter by 20' long in addition to their solid forged drums which are made up to 6' diameter with the limited ingot weight of 115 tons. The maximum dimensions attained by Krupps for solid forged drums was similar. The radial rolled drums of Thyssen were manufactured in a maximum size of 6' diameter, with a maximum finished weight of the rolled shell of 70 tons. This being the determining factor as regards length.

MOUNTING NOZZLES.

Apparently all nozzles for high working pressures were forged and were welded to the drums with screwed or plain spigots.

ALLOYS

Before the war nickel, molybdenum, chrome, and vanadium



alloys were used for high temperatures. Under wartime conditions nickel and molybdenum were generally omitted and instead silicon manganese contents were increased in carbon steels and the proportion of vanadium was increased in alloy steels. Chromium appears to have been procurable until late in the war but in the final stages it was omitted and alloy steels contained up to 1.5% manganese with 0.7% silicon. This specification applied for working temperatures up to 520° C.

Normal practice for boiler construction up to 520° C. final steam temperature was:-

|              | <u>Pre-War</u>          | <u>Wartime</u>   |
|--------------|-------------------------|------------------|
| Tubes        | Carbon steel            | Carbon steel     |
| Drums        | Carbon or Cr.Mo.<br>Ni. | 1.2% Mn.         |
| Superheaters | Chrome Molybdenum       | Chrome Vanadium. |

AUTOMATIC CONTROL

10. The only information gleaned on this subject was that Askania control was applied to Benson boilers in the German Navy with beneficial effects as regards furnace wall circulation and final steam temperature.



### BOILER AVAILABILITY

11. It was ascertained that the problem of boiler availability had proved a very serious one, accentuated as it was by the necessity to burn fuel in many instances for which the boilers were not designed. Generally speaking it was found that low boiler availability due to birds nesting resulted in boiler plants operating at from 70%-80% of the figure for which the boilers were sold. It did not prove possible to trace one single plant in which the desired availability period of 4,000 hours continuous steaming at M.C.R. had been obtained.

It was not found that the Germans had encountered any availability trouble due to back end difficulties, but this is thought to be ascribable to the fact that it is very exceptional to find a German plant with a lower outlet gas temperature than 400°F. at airheater outlet.

The question of availability has received a good deal of attention, and it appears to be becoming general that customers include in their specifications a clause, according to which the boilermaker has to demonstrate that the boilers supplied can steam at the M.C.R. for which they were sold, for a continuous period of not less than 3,500 hours with the thermal efficiency maintained throughout.

The most popular method of dealing with birds nesting appears to be to remove same by the use of the compressed air gravel gun.

There did not appear to be much development in regard to electrically operated sootblowers although power station engineers appear agreed that these are useful for keeping down birds nesting in furnace roof tubes, superheater and reheater tubes. For economisers and airheaters they were regarded as ineffective, and airheaters are usually dealt with by compressed air lances when the boiler is out of commission.

### PULVERISED FUEL FIRING

12. Pulverised fuel firing is the method generally preferred in Germany in modern plants either when employing hard coal or brown coal. The original Krämer mill has been very considerably developed under the description of Babcock HS Beater mill. This design, contrary to the original, provides means for the separation and re-circulation of coal particles. The largest mill which has been built up to the present has a capacity of 30 tons/hr. with raw lignite, the power consumption amounting to 205 KW. Considerable margin was provided on the motors, and the mill shafts calculated so that the critical speed is 100% above the operating speed. This large safety margin was found necessary for quiet running of the mills with irregular wear on the beaters.

In the later designs of Kramer type of mill a classifying device is incorporated in the upper part of the mill shaft, but this was not entirely satisfactory due to furnace radiation causing coking of particles inside the separator with resultant very short life of the separator. Accordingly the latest development was the Babcock H<sup>2</sup> Beater mill in which the mill becomes a separate unit, complete with cyclone type classifier and return system. The mill is supplied with hot air at temperature up to as high as 350°C. These mills appear to be operating satisfactorily, not only with lignite but with many categories of hard coal.

Tube mills are still finding employment, but no important developments have taken place as regards these.

Another mill which has been very largely installed and has received a very good recommendation at certain stations is the KSG Impact mill. This is a high speed mill in which pulverisation is obtained by centrifugal action and continual agitation of the coal within the mill so that the coal



particles break down by continual projection against each other. It has as an advantage a smaller space requirement than the Krämer type whilst its power consumption is also claimed as slightly less.

It would seem, however, that this mill involves fairly high maintenance cost.

For hard coals comprising 40% of all pulverised fuel installations the wellknown Loesche and Babcock "E" type mills are well introduced.

Corner firing has been further developed, and nearly all large pulverised fuel firing installations are provided with corner firing whilst a number of older plants originally installed with frontal burners (generally turbulent burners) have been converted or are in course of conversion to corner firing. It is claimed that corner firing renders possible the employment of a coarser pulverisation. The high velocities originally based on of 30 m/sec. at the burners appear to have been reduced to approximately 20 m/sec.

A certain amount of development of slag tap furnaces has taken place after an interval of 10 years since the original supply to the Mark Electricity Works in 1932. Further installations have meantime been installed at Scholven power station and the Wilhelmine Viktoria mine, in the case of the mine the fuel employed had an ash fusion point of over 1,400°C, and in the case of the seam of 1,530°C, whereas in the Scholven power station coal with an ash fusion point of 1350°C is employed. Considerable difficulties were encountered in the case of the mine which could only be overcome by the use of a mixture of coals. Slag tap furnaces are also installed at the Scholven Hydration plant, the fuel employed being residue from the hydration installation with calorific value of 5,250 calories and an ash fusion point of 1,305°C.

This plant has also worked satisfactorily as far as ashing is concerned, but gave rise to difficulty due to corrosion of the cooling wall tubes on the fire side, and clogging up in front of the burner mouths. This plant was provided with corner firing, but it is now generally considered that firing through the top of the combustion chamber only will be resorted to with slag tap furnaces in future.



### MECHANICAL STOKERS

13. The German practice with Mechanical Stokers is to make practically every part of mild steel, the fabrication usually being by welding and bolting, but in some cases, riveting plus bolting is used.

Cast iron is usually used only for the links and other parts directly exposed to heat.

Practically all mechanical stokers are of the Carrier Bar type and the universal practice with Carrier Bar Stokers in Germany is to build the carrier bars in pairs with fairly heavy cross bracing so as to build a very strong beam to span the width of the grate.

Usually, only two roller chains are used, one at either side of the grate, and the double carrier bar with bracing has to carry the weight of the grate links, etc. across the width of the grate between the chains.

In wide grates, Steinmullers introduce chains at approximately one metre pitch, but a number of fairly wide grates of other designs were seen using only two chains, one at either side.

One definite advantage of this arrangement is that the chain can be placed in a recess underneath the side wall and, therefore, is not exposed to damage by heat or by dust, as is the case when chains are placed directly beneath the working portion of a grate.

Air sealing arrangements are also simplified by the unbroken character of the carrier bar system.

Practically every German mechanical stoker is of the compartment type, which means that the forced draught is delivered directly to a series of entirely separate compartments immediately below the actual grate surface and as close as possible to the fuel bed. Individual controls are provided for precise regulation of air supply to each compartment

At Walther & Co., Dellbruck, Cologne, they have one of their own carrier bar stokers in their experimental boiler-house and this stoker has five air compartments.

The five air control levers are grouped in one control box at the front of the boiler. A 5-point draught gauge is placed alongside so that the operator can see at a glance the air conditions in all compartments and can note the effect of altering the draught controls.

The disposal of riddlings which fall through the links into the various air compartments is dealt with by various methods. In some small grates, riddlings and dust are removed by hand through side access doors. In larger grates, some form of mechanical removal is usually provided.

One method is to make each air compartment with hopper bottom and fit a 4-bladed rotary valve driven by the return chain of the grate. A constant air seal is provided by the rotary valve but the dust is allowed to fall through onto the return chain, and falling through this chain it reaches the dust hoppers below.

Another method also has each air box with hopper type bottom but fits a small screw conveyor driven by the grate.

The screw conveyors fitted to all compartments discharge the riddlings and dust into one long screw conveyor at the side of the grate and this moves the dust to a convenient place where it discharges into a dust hopper.

STEINMULLER L.ROST OR FLAT GRATE. This is a very interesting development which has already been used on a number of Marine Boilers in addition to land installations.

The grate is stationary and water cooled and fitted with a mechanical device which acts as a coal feeder and slicer. Grate bars are of cast iron and resemble usual type of fire bars, i.e. with alternate ribs and air spaces, and they are carried on a series of steel water tubes extending



for full length of the furnace. Tubes are usually connected to the boiler system and act as additional heating surface.

Forced draught is admitted beneath the fire bars and these being stationary, permit of dividing into compartments or zoning with minimum of trouble.

The flat grate is built in units each 4'3" wide, and the largest grate so far constructed has 4 units side by side, making a total width of 17' 0" and a length of 18'4".

Each unit has its own scraper bar extending the full width of 4'3" across the unit. The scraper bar is the special feature of this grate. It is made of steel 10" wide x 2" thick at the thickest part and the length is, of course, 4'3".

In cross section it has a blunt nose on the leading edge tapering back to a knife edge at the rear.

The underside of the bar rests on the grate surface, so when bar is pushed from front of grate to rear, its blunt nose pushes the fuel forward. Scraper bar returns immediately to its usual position at front of grate and the fine point or knife edge, which is now leading on the return journey, slices through the fuel bed without pushing the fuel backwards, and at the same time acts as a slice bar, scraping or slicing along the top edges of all fire bars to keep them clean by disturbing any clinker or dust which may have settled in the air spaces between the bars.

Each scraper bar is operated by a special driving chain mounted along the centre line of each grate unit.

The driving chain is continuous running over front and rear sprockets, and carries a series of flat links in groups with air spaces which move along in a slot approximately 3" wide provided in the centre of the stationary fire bars of the unit.

At one point in the chain there is a vertical lug which projects upwards through the moving links, and the scraper bar is attached to this lug by a simple clip.

The chain is motor driven through small multi-speed gearbox with a special electric control system.

By setting indicators on the electrical panel, the boiler operator can decide -

- (a) Speed of travel of scraper bar forwards.
- (b) Speed of travel of scraper bar backwards.
- (c) Distance travelled by scraper bar along grate.
- (d) Interval of time between two operations of scraper bar.

In normal operation the scraper bar is set to travel at a speed of 1 metre per second. This means that the bar will travel right along a grate 16'0" long in about 5 seconds and return immediately to the front of the stoker in another 5 seconds.

The operator can set the electrical gear so that this movement of scraper bar will take place once a minute, once every two or three minutes or at other intervals desired, or he can arrange for several movements in quick succession.

The amount of travel of scraper bar is usually arranged that it stops and reverses about 3'0" from rear end of grate and thus allows ashes to build up at rear portion of fire bars.

At the operator's discretion the travel can be increased so that the scraper bar will move right to the extreme rear end of the grate and so push the spent ashes off the grate into the rear pit or ash hopper.

This grate is claimed to be able to burn any variety of coal, but it is not too satisfactory if very much



fine coal dust is present.

Except for the periods when the scraper bar is moving forward or returning, it remains stationary at the front end of the grate buried beneath the green fuel near the point where the fuel leaves the fuel hopper.

The scraper bar is therefore only exposed to heat during the time it travels along the grate.

In the test boilerhouse, one of these grates had just come off load and was cooling down. The scraper bar had been detached and could therefore be very thoroughly examined. There were a few signs of slight burning along the pointed edge or trailing edge, but the bar was relatively in good condition, although a slight amount of warping was noticed. This bar had had a three months continuous service.

The cost of a replacement bar is relatively small and bars can be very quickly changed while the grate is in service.

#### DURR R.S. STOKER OR REPRECHT STOKER.

Durr are making a mechanical stoker in which the entire stoker frame with all bars and air boxes moves backwards and forward.

The grate is built up with stationary fire bars resting on a framework which is carried on wheels at front and rear and the entire stoker slopes at an angle of 15° from the horizontal.

The framework is divided into separate air compartments and air is supplied into these compartments from air ducts in the side wall of the boiler.

The stoker movement is provided by an electric motor working through a reduction gear box and the entire grate moves slowly upwards from rear towards front for a distance of stroke of between 2½" and 3" only.

At the completion of the upward stroke, the drive disengages and the entire grate rolls downwards from front

towards rear and comes to rest in original position.

The drive takes up again and proceeds to push the grate slowly from rear towards the front again and the sequence is repeated.

A control is fitted so that the number of strokes per minute can be varied.

The fast and slow movement together with the slope of the grate causes the fuel to move downwards gradually and also moves the ash forward until it falls over the rear end of the grate.

We understand that this idea is at present in the development stage but Durrwerke appear very confident that it would be successful as they have tested out the idea on experimental grates with very satisfactory results.

#### MARTIN STOKER

The Martin stoker has maintained its position and it appears to be generally accepted that it offers certain advantages when dealing with markedly inferior fuels. It has a rocking bar stoker and is reported to give successful results with washery slurry.

#### TAYLOR STOKER

The eight Taylor stokers installed at West Kraftwerk, Berlin gave unsatisfactory results, and the difficulties with them were only overcome after a water-cooling arrangement was improvised by Doctor Vorkauf.

#### PRE-HEATED AIR

This was avoided if possible with travelling grate stokers and Steimmüllers would only accept it with coking coals.

#### SECONDARY AIR

Stoker manufacturers use high pressure front secondary air as standard practice, but none of them favoured rear secondary air.



DETAILED TECHNICAL REPORTS OF INDIVIDUAL PLANTS

LA MONT KESSEL HERPEN & CO.

SCHADOWSTRASSE 1a, BERLIN

Dr. Vorkauf - Managing Director

Herr Vagt - Boiler Engineer

Herr Sreiber - Patent Engineer

14. This is a Technical Company concerned with preparation and development of boiler designs and allocation of licences to manufacturers in various parts of the world.

The Company has specialised in the La Mont Boiler, of which the basic principles are forced circulation and control of the circulation in the various groups of tubes by means of regulating nozzles.

Throughout the world there are now 1,800 La Mont Boilers including boilers for marine work, and some instances of re-modelling of existing boiler plant by adding forced circulation water walls, etc.

La Mont Boilers have been built for pressures as high as 130 atm. (1846 lbs), but most of their high pressure units are working on not more than 80 atm. (1136 lbs).

Circulating Pumps which are a vital part of a La Mont Boiler are usually made by K.S.B., but in some instances, Sulzer Pumps have been used.

DEVELOPMENTS. Dr. Vorkauf showed designs for a small natural circulation Water Tube Boiler for use on road wagons or lorries with original petrol engine converted to act as steam engine. A prototype is being used for actual tests.

Dr. Vorkauf is collaborating with Dr. Huttner in the development of a Rotary Boiler direct coupled to a Gas Turbine which is operated by the gases of combustion. Actual test with a working model carried out at Klingenberg Power Station showed high efficiency and proved all theoretical points.

Rotary Boiler has automatic feed regulation. No feed pump. Amount of water taken in at inlet is same as amount of steam at outlet.

KOHLENSCHIEDUNGSGESELLSCHAFT M.B.H.

GOTTLIEB-DUNKELSTR. 20/22, TEMPLEHOF, BERLIN

Mr. Cantieny - Managing Director.

Mr. Krug - Chief Engineer.

Mr. Geisecke - Commercial Manager.

15. K.S.G. were designers of boilers up to 200 tons evaporation and 150 atm. pressure for operation with pulverised fuel. Their designs were manufactured by all principal boiler makers. During the war they had concentrated on brown coal, and had developed a combination of furnace with special pulveriser for this class of fuel. They claimed to be able to deal with lignite containing up to 60% total moisture by drawing gas from the top of the furnace at 800°C. and re-firing it with the pulverised coal through tangential burners. Their system rendered preheated secondary air unnecessary. The system was being employed for hard coals, but only at the expense of high maintenance costs on the pulverisers.

K.S.G. had been closely identified with the development of the "Channel" superheater, i.e. an arrangement in which the secondary superheater comprised a series of parallel vertical walls, each constructed of closely spaced tubes. The superheater elements were suspended in the upper part of the furnace by long legs extending to headers above the top of the boiler. The elements were spaced about 80 mm. apart, and the suspensions were sufficiently flexible to permit natural vibration as an alternative to mechanical rapping.

K.S.G. had very little experience of resuperheating or of slag tap furnaces.

They claimed to have developed a basic design of



boiler, the fundamental principles of which are described earlier in this report.

DR. MUNZINGER

C/O A.E.G. OFFICES, HOHENZOLLERN DAMM 120, BERLIN

10. Dr. Munzinger is Director of Steam Dept of A.E.G. and is the recognised authority on boilers and steam plant generally throughout Germany. His book "Dampfkraft" is easily the best ever published concerning boiler plant. His methods of calculating heat transfer, evaporation, etc. are widely used by German Boiler designers.

He had completed manuscript for revised 3rd edition of "Dampfkraft" but it was destroyed by fire in the final stages of fighting in Berlin. He has a duplicate copy of manuscript elsewhere and if he is able to obtain it, he hopes to publish the 3rd edition when conditions improve.

Dr. Munzinger considers that there has not been any great development in boilers during the past five years. He thinks there is only one new feature worth mentioning and that is the "slot" or "channel" type superheater.

The war forced Germany to use very bad coal which caused great trouble with ordinary superheaters having closely-spaced tubes. The "slot" or "channel" type superheater has widely spaced single rows of tubes, hanging freely, and able to vibrate, and thus keep themselves clear of dust and ash.

Original designs had groups of tubes spaced at 1600 mm. (54") centres, but latest arrangement has groups spaced at 800 mm. (27") centres.

"Slot" or "channel" type superheaters have been in use for about two years with very satisfactory results.

FLOODING OF SUPERHEATERS. Dr. Munzinger considers that every superheater should have some arrangement for flooding during starting up and for subsequent draining after

boiler is steaming on part load and at a reasonably high pressure.

One suggestion is to endeavour to arrange starting up at a lower working pressure, proposed by using the L.P. steam in another part of the plant working at low pressure. This would keep up the steam velocities due to greater volume of steam at lower pressure.

If no low pressure system is available, then it is suggested that a small condenser be installed to condense the steam at low pressure and so return the heat to the feed system.

PULVERISED FUEL. When asked for his opinion as to comparative values of different types of pulverisers, Dr. Munzinger said -

Roller and impact mills of first class design and made by first class Makers are each good in their own way, but during the war when coal became difficult, it was eventually decided to concentrate on manufacture of simple type rotating ball mills, using them either singly or in groups.

Ball mills are relatively more extravagant in total weight of metal required for building, but they give no trouble in operation and will run for long periods without attention.

TUBE EXPANDING. Dr. Munzinger gave a brief summary of latest German practice -

- (a) Special tension clutch on expander mandrel so that expanding is done only to a certain amount.
- (b) Great care taken to ensure outer surface of tube and surface of tube hole being smooth and keen.
- (c) Definite limits are specified for the amount



of extension of tube during expanding.

- (d) Diameter of tube hole not to be increased by more than 1.4%.
- (e) Groove in tube hole, 7 mm. from inside end of hole.
- (f) In most cases, "landing" or actual depth of hole against which the tube will take a bearing is limited to  $1\frac{1}{4}$ ". If plates are thicker, then hole is counter-bored to keep "landing" about  $1\frac{1}{4}$ ".

SUPPORTS FOR TUBES. Dr. Munzinger mentioned that to avoid having supports of special heat resisting metal at high cost, some designers are using some of the tubes from the boiler or superheater system to act as supports for other groups of tubes.

STUD BOLTS FOR PIPE FLANGES. Dr. Munzinger gave an instance of one special alloy used for stud bolts for high pressure pipework -

1.2% to 1.8% CHROMIUM

0.4% to 1.2% MOLYBDENUM

0.25% to 0.28% VANADIUM

BENSON BOILER. Dr. Munzinger said that Siemens have spent between 5 million and 6 million marks in trying to develop the Benson Boiler and he thinks they have lost most of this money. The Benson Boiler is only really suitable for constant loads, and if it has to deal with fluctuating loads and operate for any length of time at low loads, then there is serious trouble.

A.E.G. found that Benson Boiler costs 10% more than a natural circulation boiler.

RE-HEATING. This was very popular in Germany some years ago, but now appears to have gone out of favour. Most

people find that its advantages are out-weighed by many troubles with maintenance, etc.

BOILER AVAILABILITY. "Availability" is the main feature of latest German designs. Dr. Munzinger considers that Boiler Makers and Boiler users are at last agreed that availability is the most important feature of a boiler. High thermal efficiency is of little value if the boiler is persistently off the line for adjustments, modifications or repairs.

Dr. Munzinger said he knew of one boiler which has run for two years without shutting down.

GAS TURBINES. Dr. Munzinger thinks there are great possibilities in the Gas Turbine. He is satisfied that smaller sizes up to 10,000 kw. are already perfected and he thinks there should be little difficulty in going to larger sizes.

Escher-Wyss have a gas turbine 2,000 kw. as an experimental set and this is run with an overall efficiency of 31.5%. Actually, this particular machine uses hot air, not gas.

Dr. Munzinger has attended many trials at Escher-Wyss and is obviously deeply impressed by the possibilities of the gas turbine.

INFORMATION FOR RUSSIANS. Dr. Munzinger revealed that he is at present working for the Russians. He has been engaged to write a memorandum on General Boiler and General Steam Practice with particular attention to war time developments. He has, in turn, engaged other engineering authorities such as Dr. Vorkauf of La Mont to write chapters on their own particular lines, and Dr. Munzinger will apparently act as Editor of the whole affair.

The Russians have also asked him to prepare a full report on all existing steam plant in the part of Germany now occupied by Russian troops.



CURT VON GRUEBER

RUHLSDORFERSTRASSE, TELTOW, BERLIN

Mr. Loesche - Chief Engineer

17. During the war the Works were confined to the manufacture of standard Loesche Mills for coal, cement, and chemical grinding, and armament manufacture. They developed no new designs. Since the Russian occupation 80% of their machine tools had been removed and were reported to be stored in the open outside Berlin. All their patterns were in a foundry in Upper Silesia. The Works were continuing to produce spares for the numerous mills installed in the Russian zone of occupation, but were receiving no raw materials and were obliged to fabricate mill bodies, gear boxes, etc., as they could not secure the return of their patterns.

WAGNER H.D.T.

GLOCKENGIESERWALL 1, HAMBURG

Mr. Braggard - Managing Director

Mr. Gebens - Technical Director

Mr. Reppel - Chief Designer

Mr. Kopke - Chief Engineer

18. This is a purely technical company concerned with the design of various kinds of boilers, primarily for marine purposes. Their marine boilers had been accepted by the German Navy, and in some cases had replaced Benson boilers in naval craft. They were also installed in the "Bismarck" and "Tirpitz".

Towards the end of the war they designed a three-drum boiler with unheated downcomers to the bottom drum and a small steam receiver, the superheater being located between two tube banks. This type was designed for a capacity of 50-60 tons at 64 atm. and 460°C., with air preheated to 350°C. It was of the single pass type with streamlined tubular air heater and no economiser.

Their most recent design was a two-drum boiler with unheated downcomers, above which were superimposed a superheater, economiser, and air heater.

They had made a special study of carry-over, as a result of which their latest design comprised a simple arrangement of feed trough with a perforated dry pipe.

Their air heaters were made by Rudolf Otto Meyer of Hamburg, and were fabricated from rolled welded plate 0.4 - 0.75 m.m. thick. In the early days corrosion was overcome by using 17% chrome steel, which also proved to be more resistant to burning.

In the latest designs of Wagner boilers it was claimed that the water content represented approximately 16% of the total weight.

DEUTCHE WERFT

FINKENWARDEN, HAMBURG

Herr Kühl - Chief Engineer

Herr Heisig - Manager of Boiler Dept.

Herr Back - Manager of Boiler Shop

19. This is the Boiler Department of a shipbuilding yard, manufacturing small La Mont and Wagner boilers for Marine use and also undertaking repairs to other boilers.

A full set of drawings and methods of calculations were obtained by a previous investigator whose report will fully cover the capabilities of this boiler shop.

Inspection of the boiler shop did not show any plant of particular interest, most of the machines being old and only suitable for small boiler units. Some Scotch type Marine boilers were seen but there were no water tube boilers in course of manufacture.



DESCHIMAG, BREMEN

Mr. Koeler - Chief Designer

Mr. Boehm - Director of Boiler Dept.

20. Deschimag was a shipyard with facilities for manufacturing marine boilers. They usually collaborated with Wagner H.D.T. Most of their records and drawings were destroyed by fire, and the remainder were held by the U.S. Authorities since Bremen was an American enclave at the time of the investigations. The Works had been almost entirely destroyed, but a number of Wagner boilers in various stages of manufacture could be identified in the wrecked boiler shop.

Their Staff were able to confirm technical information acquired at the Wagner H.D.T. Office, and also gave the following supplementary information. They had recently made marine boilers up to 90 tons capacity at 70 atm. and 450°C. The weight of such boilers filled with water amounted to approximately .7-lb per lb of steam with air heaters only, and 0.8 to 1.0 lb per lb of steam for boilers with economisers as well as air heaters.

They did not agree with Wagner's design of internal fittings and preferred a separate steam receiver drum about one-third the diameter of the main boiler drum and connected to it by numerous tubes to ensure low steam velocities. It was essential to provide drains from the receiver drum, and to carry these well below water level in the main boiler. They also favoured radial baffles to prevent excessive turbulence at riser tube discharge points.

PRESS UND WALZWERK

OBERBILK, DUSSELDORF

and

REISHOLZ, DUSSELDORF

Herr Merckens - Foreign Dept. Manager

Herr Dehner - Works Manager

Herr Goedecke - Chief Engineer } At Oberbilk

Dr. Kretz, - Metallurgist }

Herr Martin - Works Manager

Dr. Wiesmann - Chief Engineer } At Reisholz

21. At Oberbilk, the offices, laboratory and reasearch department had been destroyed by bombing and all the shops were slightly damaged. They make steel in open hearth furnaces and claim to make ingots up to 115 tons for fabrication into solid drawn boiler drums, chemical and other pressure vessels. They also manufacture railway tender and wagon wheels and tyres, their capacity per month when in full production being 400 complete sets of axles with wheels plus 700 extra spare tyres.

The works at Reisholz comprise Head Offices and Shops for manufacture of Seamless Forged Drums up to 1 metre diameter by 6½ metres long. They also press and draw tubes and headers. The ingots are made at their main Works at Oberbilk. Other products include lap-welded pipes and drums, both electrically and hammer welded, using coke oven gas. There is a large stock of completed headers up to 8 metres in length and up to 7" x 7" square section, wall thickness 2".



MANNESMANN ROHRENWERKE

RATH, DUSSELDORF

Mr. Fritsch - Works Manager.

Mr. Bungeroth - Interpreter.

22. The works, which had been seriously damaged by bombing, were entirely designed and equipped for manufacturing seamless steel tubes and rectangular section headers. Their two large Pilger machines were out of action, but they were still operating small and medium machines, and their capacity for hot drawn tubes at the time of the investigation was stated to be as follows:-

Maximum capacity - 5,000 tons per month.

Maximum diameter of tube - 521 m.m.

Minimum diameter of tube - 38 m.m.

Maximum weight of single tube - 4 tons.

All cold drawn tubes were finished at their Works at Ramscheid.

VEREINIGTE KESSELWERKE A.G. - "V.K.W."

WERDENERSTRASSE, DUSSELDORF

Dr. Wirtz - Engineering Director

Dr. Nuber - Boiler Designer

Dr. Engler - Chief Engineer

23. This Company makes Water Tube Boilers of their own design and also makes Schmidt and Benson Boilers under licence. They also make shell Boilers and Mechanical Stokers.

The Works escaped damage during the war and all buildings and plant are in first class condition.

In pre-war times there were about 700 employees; during the war a total of about 500 people were employed and the Works have been in full operation throughout the war.

BOILER DRUMS. Drums for Schmidt Boilers are solid forged or radially rolled and are obtained from Messrs. Thyssen

or from Press-und-Walzwerk. The steam drum for the Schmidt Boiler has a specially large manhole measuring 36" x 18". This is necessary for inserting the groups of evaporating tubes which are submerged in the water portion of this drum.

It was noticed that stub tubes were being expanded into tube holes in the drum and each tube was afterwards seal welded on the outer surface of drum. V.K.W. say this is their standard practice with stub tubes and they have had no trouble.

HEADERS. A number of headers for Schmidt and Benson Boilers were seen and these had been made by Borsig from solid bar by boring out the centre and machining away the outer surface to leave projecting stubs to which the tube elements could be welded.

ECONOMISERS. V.K.W. make gilled tube Economisers by fixing pressed steel discs on mild steel tubes.

Discs are pressed with an inner flange and are assembled on the tubes in pairs, each pair having the internal flanges joining together.

The tube and gills are assembled in a special grooved erection frame, and while in position, an internal expander is forced through the tube so that it expands tightly against the gills, thus giving a close metallic contact.

The tube with gills in position is then rotated slowly in a lathe while electric welding machines, which are also fitted to the lathe, are used to weld the adjoining flanges of the gills together and to weld them to the tube.

SHELL BOILERS. V.K.W. specialise in the "Holland" type boiler. This is a variety of the economic type, having one very large flue, usually corrugated, and 8'0" or 6'6" diameter, with back end of flue filled in by tube plate and fire tubes connecting from this tube plate to the back end plate of the boiler.



The tubes are arranged so that there are 3 separate passes for the boiler gases before they leave the boiler.

A "Holland" type Boiler was in process of assembly during our visit. It is interesting to note that the main boiler shell was made by Thyssen who rolled the plates to shape, did the welding and then the subsequent heat treatment. Thyssen also provided the two main flat end plates with edge flanging done.

The large corrugated flue 6'6" diameter and 11'6" long with back end plate welded in was also provided complete by Thyssen so that the only work left for V.K.W. to do was the actual fitting together, the marking off and drilling of rivet holes for connecting main shell to the main end plates and, of course, the fitting of pads for the various valve mountings.

V.K.W. have a number of large single spindle radial drilling machines which, instead of having the usual pillar, are arranged to move up and down on a vertical slide which is built into a buttress on the side wall of the building.

These machines were thus able to move the radial arm high enough to bore the tube holes in the end plate of a Shell Boiler standing on end on the shop floor.

CARRIER BAR STOKER. Several stokers were seen under construction, all being compartment type with main frame, etc. built up of mild steel. Carrier bars arranged in pairs, spaced to bridge the full grate width between the two chains, one on either side of grate.

L. & C. STEINMULLER

GUMMERSBACH, RHINELAND.

Herr. Stoltenberg-Lerche - Managing Director

Dr. Richter - Director

Herr. Strohm - Engineer

Makers of Water Tube Boilers of various types,

Mechanical Stokers and complete Boilerhouse equipment.

Employees before war - 2,000. At present - 1,000.

Machine Shops and Boiler Shop heavily damaged by air raids and not yet repaired.

Buildings 90% destroyed, but most machines have been salvaged and many are working in other shops. Temporary shops and other undamaged shops are working.

Machine tools, both light and heavy seemed in very good condition.

They build La Mont Boilers under licence.

Their latest designs are mostly bent tube Boilers having 4 drums, 3 drums, 2 drums and even one drum.

They commenced business in 1874 building header type boilers and they still build a number of this type.

Some headers staggered rather similar to B. & W. At other times they use a built-up plate header the full width of boiler with stay bolts.

Steinmullers have a very high reputation as Makers of mechanical stokers.

For all high pressure boilers they buy forged or radially rolled drums from Thyssen, and headers from Press-und-Walzwerk.

BOILERHOUSE. Steinmullers have a large boilerhouse specially built to house 3 Water Tube Boilers, the largest of which has a capacity of 3 tons per hour. They use these boilers for test purposes and are continually trying out new ideas. Different methods of arranging downcomers to various water wall headers, or different settings of Mechanical stokers.

Each of the 3 Boilers has a different kind of stoker, e.g. one with latest Carrier Bar Stoker, one with early pattern Chain Grate Stoker, and the third with latest type L.Rost or flat Grate Stoker.



The L.Rost Stoker has stationary fire bars and a moving slice bar which also acts as a feeder. This is described in greater detail elsewhere in this report.

CARRIER BAR STOKERS. Steimmullers have a very high reputation on the continent for their Carrier Bar Stoker.

The main frame and side members are all built up of steel.

Every grate is fully compartmented.

Small grates have riddlings removed from air compartments by hand through side access doors.

Larger grates have simple screw conveyor at base of each air compartment moving riddlings to one side of stoker where they are picked up by another long screw conveyor and moving so as to fall into convenient dust hopper.

These Carrier Bar Stokers have usually only two chains, one at either side of grate.

Carrier Bars are built up in pairs with cross members to make a very rigid beam to span the width of stoker.

Practically every Steimmuller stoker installation is fitted with a rear firebridge, which is a well-known speciality of Steimmuller and can be simply described as a series of small hinged flaps to seal the space between rear end of grate and the rear bridge wall above the grate.

FOUNDRY. Steimmuller have a well equipped Foundry in which they make stoker links and other parts for boilers, economisers, etc.

They have an interesting method of making stoker links without boxes but in stacks so that a number of sets can be poured at one operation.

A number of airheater elements have been made in cast iron but the construction appears expensive and does not appear to have been developed very far.

They make cast iron gilled tubes for Economisers of an extremely heavy type of construction and have claimed that these cast iron gilled tubes are suitable for high working pressures up to 1200 lbs. per sq. inch.

An interesting continuous shot blasting machine was seen in operation and all stoker links and other small castings for boilers were being shot blasted in this machine.

The castings were placed on skeleton steel trays which were carried along on a simple belt conveyor through a shot-blasting chamber which had flexible curtains at inlet and outlet.

WALTHER UND CIE., A.G.

KOLN DELERUCK (NR. COLOGNE).

Herr Ahn - Director.

Dr. Hahn - Engineer-in-Chief.

Herr Zuffe - Interpreter.

25. They build Water Tube Boilers of the header, bent tube Loeffler and Benson type also Lancashire and Cornish Boilers, and repair Locomotive boilers.

Most of their boilers are fired by pulverized fuel using Kraemer Mills. They also make travelling grate Mechanical Stokers up to 10 metres wide by 7.8 metres long and on their forced draft Stoker they claim to be able to burn coal of sizing 0-10 mm. with 50% below 1 mm., also slurry and brown coal containing up to 30% total moisture. This firm has standardised on P.I.V. gears for stoker drive.

CLAUDIUS PETERS

WALLHOF, GLOCKENGRISSENWALL, HAMBURG

Claudius Peters - Managing Director.

Herr Wolf - Chief Engineer.

Herr Grapengiesser - Commercial Manager.

26. This firm is primarily a selling agency for pulverising equipment used in manufacture of cement. They possess certain amount of technical information with respect to grinding of cement clinker, hard and soft coal, phosphate, barytes and explosive ingredients.



They also have considerable experience with Fuller-Kinyon Pumps for handling cement in powder form.

DURRWERKE

RATINGEN, DUSELDORF

Herr Piepenbrink - Chief Engineer

Herr Watzke - Technical Director

27. Old established firm making various types of Water Tube Boilers.

For past few years have specialised in manufacture of Benson high pressure Water Tube Boilers. They built four Benson Boilers for Karnap Power Station, Essen.

Some specimen sections of Benson Boilers in Shops.

Also make Mechanical Stokers, including travelling Grates, and their new R.S. Oscillating sloping Grate.

Prefabricated sections for German "U" Boats can be seen in their Shops.

50% of share capital is owned by Babcockwerk, Oberhausen.

BABCOCK-WERKE

OBERHAUSEN (RUHR)

Dr. Jantscha - Managing Director

28. Works equipped for making Water Tube Boilers of Header type and of Bent Tube type, in all details except actual Drums and Headers.

Also make Pulverising Mills and Boiler Valves of all sizes.

A small separate Works nearby, (partly damaged) makes Pipework.

DEUTSCHE ROHRENWERKE

MULHEIM - RUHR

Herr. Fiene - Chief of High Pressure Dept.

29. RADIAL ROLLING PLANT - 2 machines one original

small machine, and the other large machine -

LARGE MACHINE. Capacity. 70 Tons Finished weight rolled cylinder from hollow ingot - maximum length 18 metres.

maximum diameter 1800 mm.

maximum bore. 1600 mm.

Process usually involves several re-heats.

SMALL MACHINE max. diameter 1200 mm.

They claim to have handled 1500 Ingots since 1938 -

WELDED CYLINDERS & CORRUGATED FURNACES.

Corrugated Furnaces up to 20 feet length

Diameter up to 6'6".

Gas Welded.

Other Pipes electric welded.

GEBRUDER SULZER AKTIENGESELLSCHAFT

LUDWIGSHAFEN

Dr. Schrag - Head of Boiler Dept.

Commandant Burg - Pont-a-Mousson.

30. It was stated that a controlling interest in this Works had been acquired by the French Pont-a-Mousson concern, and that as a result that French Company had arranged to provide two senior engineers from their Works in France to repair damage and re-commence production as soon as might be found possible. The manufacturing plant had suffered severe damage by bombing, but it was ascertained that a large proportion of the machines was capable of salvage.

Repair work was actually in course. The French engineers, who were in French military uniform, explained that in view of French ownership they were under no obligation to furnish any information at all but as they knew the identity of the British Companies with whom the investigators were connected



they were prepared to answer any general questions or furnish any normal information as a matter of courtesy. It was ascertained that during the war no outstanding work had been done as regards boiler manufacture and generally speaking this Works concentrates on the production of high pressure pumps which Sulzer designed. Little of important interest.

LIST OF ILLUSTRATIONS

- Fig. 1. Pulverised Fuel Fired Boiler with two-stage Slag Tap Furnace, 'Chammel' type convection bank, unheated downcomers, separator drum, steam cooled superheater supports, controlled superheater in three stages. 125 Tons per hour at 80 atm. and 500°C.
- Fig. 2. Pulverised Fuel Fired Boiler with unheated downcomers, slag screen, three-stage controlled superheater with steam cooled supports. 80 Tons per hour at 130 atm. and 500°C.
- Fig. 3. Pulverised Fuel Fired Boiler with Radiant Primary Superheater and Radiant Reheater. 80 Tons per hour at 120 atm. and 485°C.
- Fig. 4. K.S.G. Brown Coal Mill.
- Fig. 5. Cross Section of Steinmüller Zoned Air Central Travelling Grate Steker
- Fig. 6. Details of construction of Steinmüller Steker.
- Fig. 7. Details of Circular Man-Door.



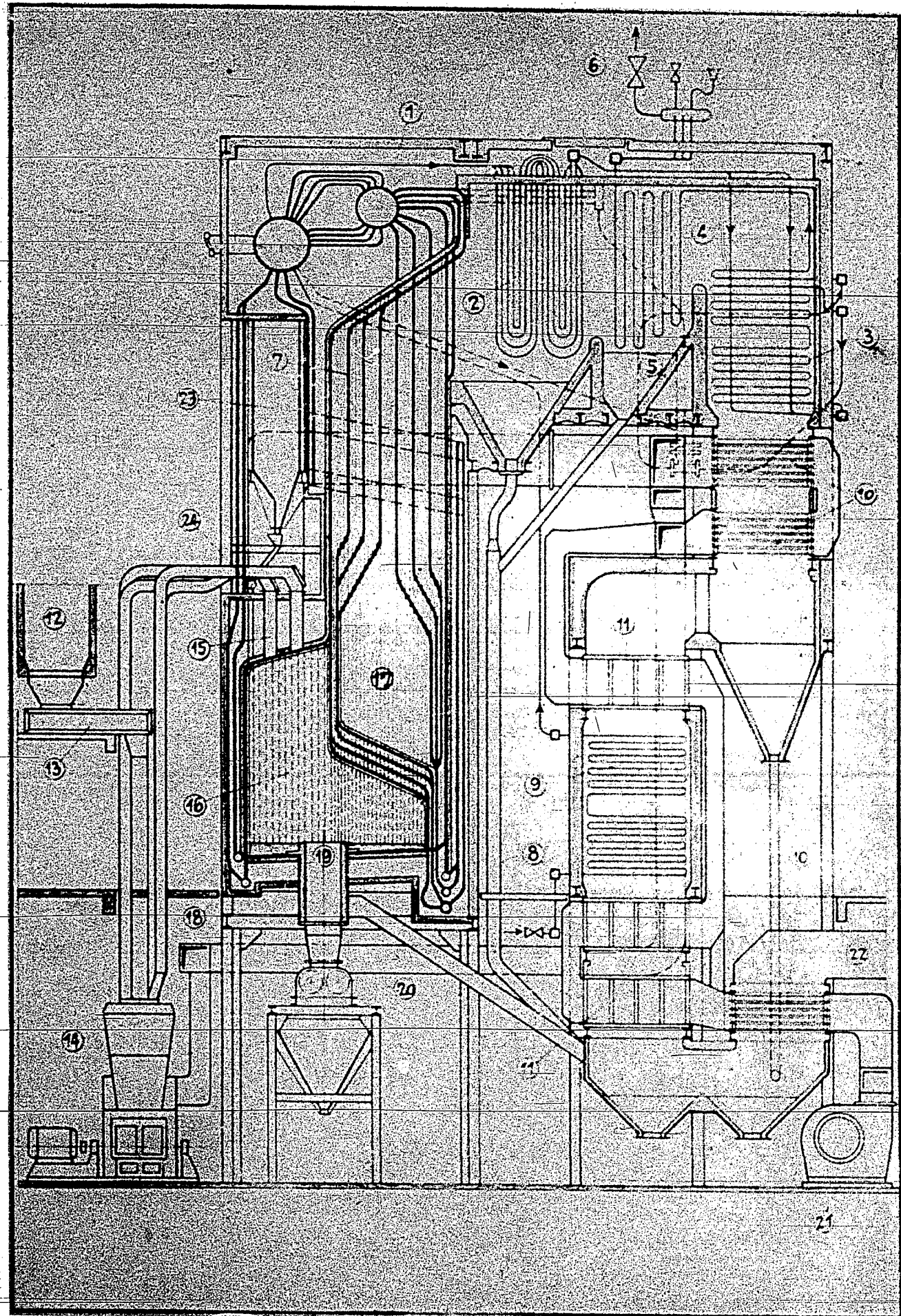


FIG. 1.



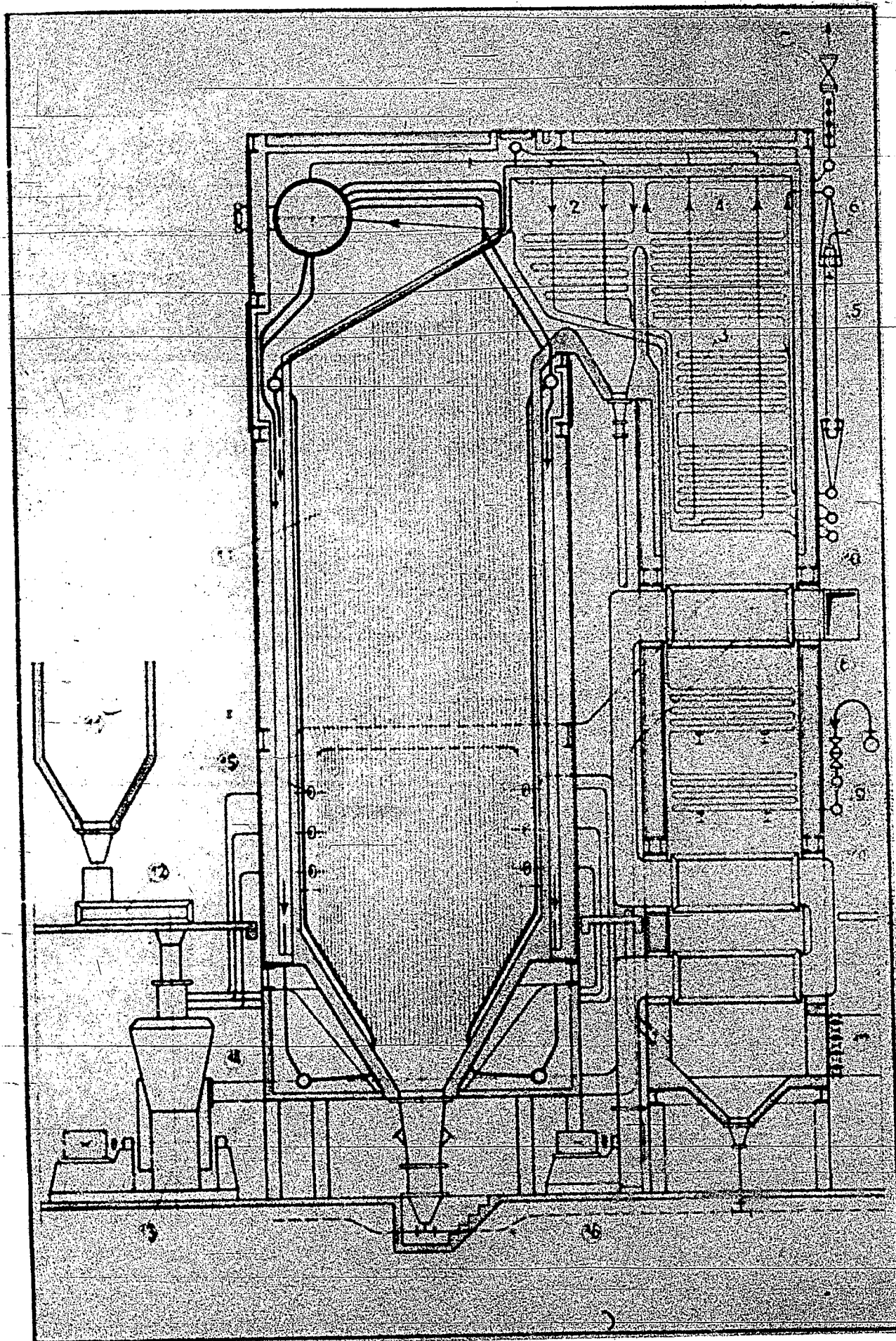


FIG. 2.

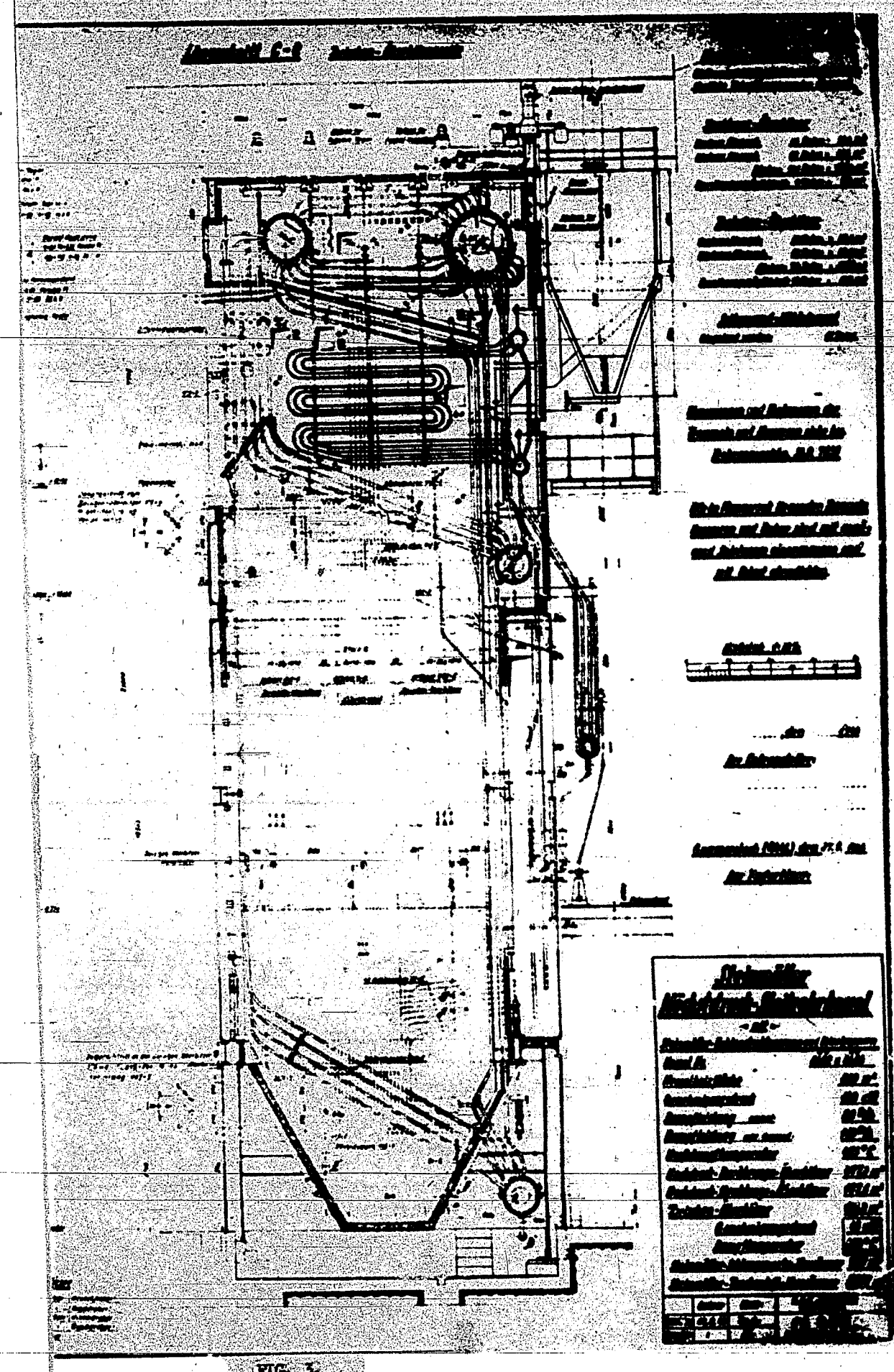


FIG. 3.

**Legend**

Material: Steel  
 Thickness: 1/2" (top), 3/4" (bottom), 1" (sides)

Support: 4" dia. x 12" high

Insulation: 2" thick

Accessories: 1/2" dia. x 12" high

Dimensions: 10' x 10' x 10'

**Material**

**Weight - Steel**

|             |      |
|-------------|------|
| Shell       | 1000 |
| Support     | 100  |
| Insulation  | 50   |
| Accessories | 100  |
| Subtotal    | 1250 |
| Material    | 1000 |
| Support     | 100  |
| Insulation  | 50   |
| Accessories | 100  |
| Subtotal    | 1250 |



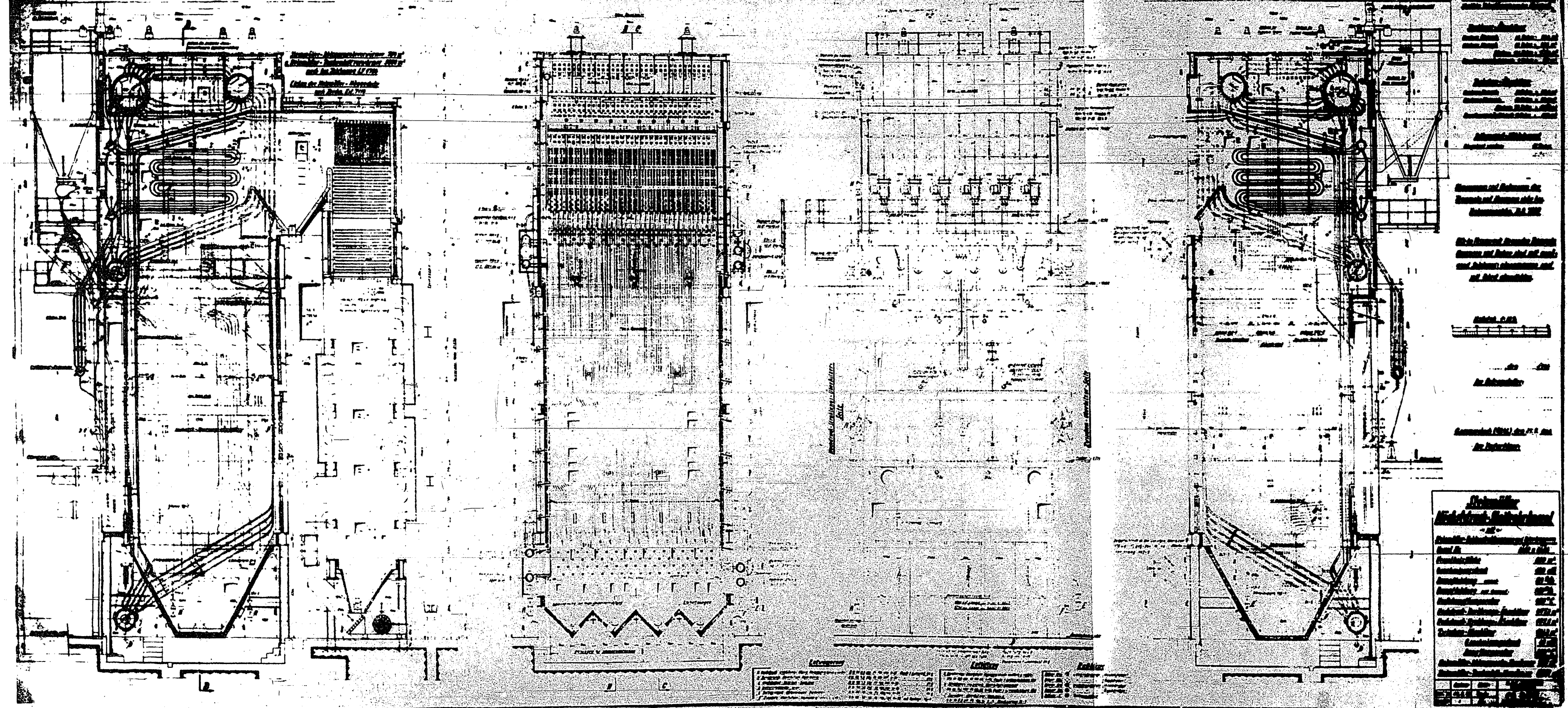
Abbildung 1-1 Vorder-Ansicht

Abbildung 1-2 Vorder-Ansicht  
Abbildung 1-3 Vorder-Ansicht

Abbildung 1-4

Abbildung 1-5

Abbildung 1-6 Vorder-Ansicht



**Legende**

Abbildung 1-1  
Abbildung 1-2  
Abbildung 1-3  
Abbildung 1-4  
Abbildung 1-5  
Abbildung 1-6

**Abbildung 1-1**

Abbildung 1-2  
Abbildung 1-3  
Abbildung 1-4  
Abbildung 1-5  
Abbildung 1-6

**Abbildung 1-2**

Abbildung 1-3  
Abbildung 1-4  
Abbildung 1-5  
Abbildung 1-6

**Abbildung 1-3**

Abbildung 1-4  
Abbildung 1-5  
Abbildung 1-6

**Abbildung 1-4**

Abbildung 1-5  
Abbildung 1-6

**Abbildung 1-5**

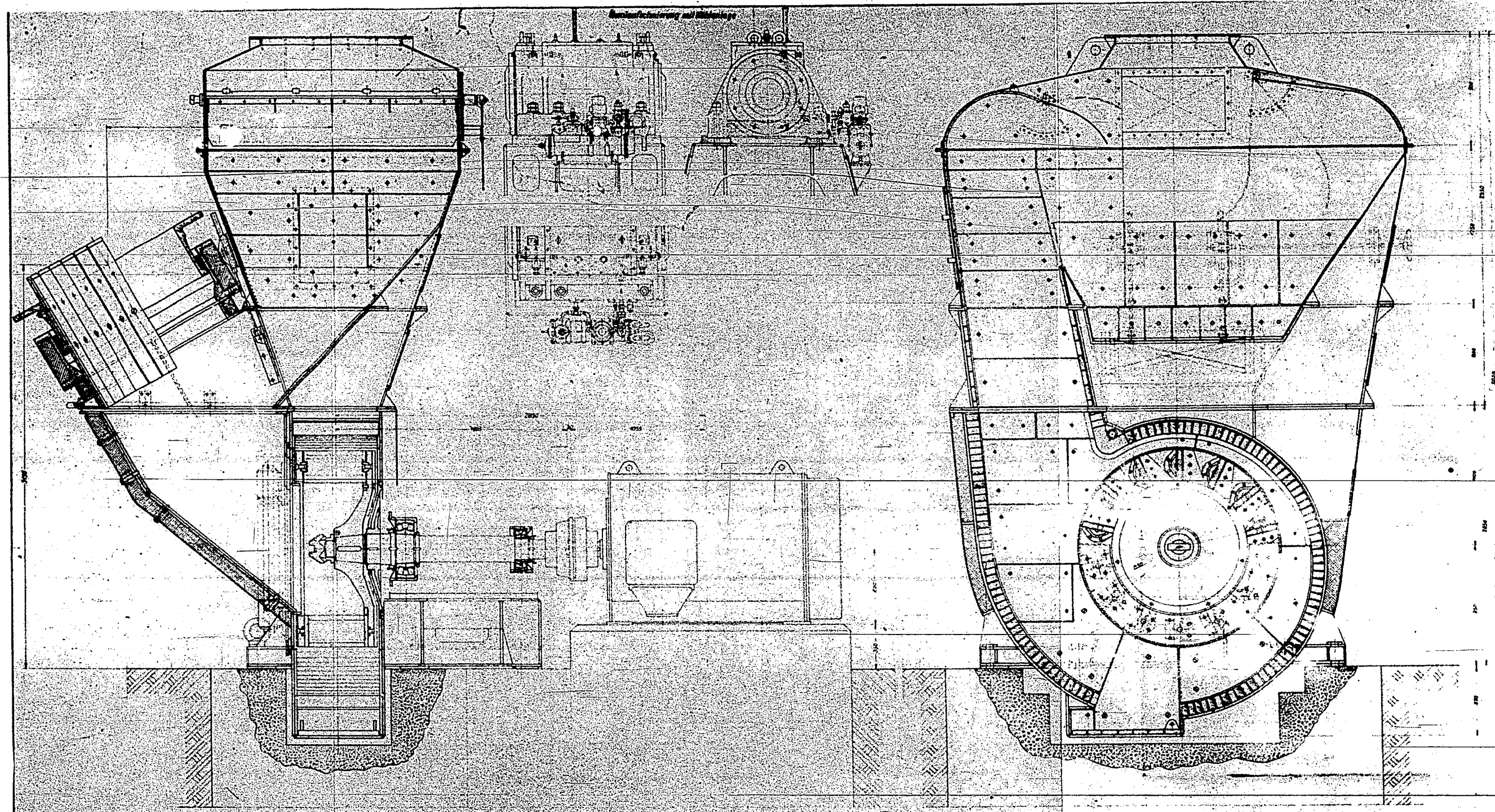
Abbildung 1-6

**Abbildung 1-6**

|                |                 |
|----------------|-----------------|
| Abbildung 1-1  | Abbildung 1-2   |
| Abbildung 1-3  | Abbildung 1-4   |
| Abbildung 1-5  | Abbildung 1-6   |
| Abbildung 1-7  | Abbildung 1-8   |
| Abbildung 1-9  | Abbildung 1-10  |
| Abbildung 1-11 | Abbildung 1-12  |
| Abbildung 1-13 | Abbildung 1-14  |
| Abbildung 1-15 | Abbildung 1-16  |
| Abbildung 1-17 | Abbildung 1-18  |
| Abbildung 1-19 | Abbildung 1-20  |
| Abbildung 1-21 | Abbildung 1-22  |
| Abbildung 1-23 | Abbildung 1-24  |
| Abbildung 1-25 | Abbildung 1-26  |
| Abbildung 1-27 | Abbildung 1-28  |
| Abbildung 1-29 | Abbildung 1-30  |
| Abbildung 1-31 | Abbildung 1-32  |
| Abbildung 1-33 | Abbildung 1-34  |
| Abbildung 1-35 | Abbildung 1-36  |
| Abbildung 1-37 | Abbildung 1-38  |
| Abbildung 1-39 | Abbildung 1-40  |
| Abbildung 1-41 | Abbildung 1-42  |
| Abbildung 1-43 | Abbildung 1-44  |
| Abbildung 1-45 | Abbildung 1-46  |
| Abbildung 1-47 | Abbildung 1-48  |
| Abbildung 1-49 | Abbildung 1-50  |
| Abbildung 1-51 | Abbildung 1-52  |
| Abbildung 1-53 | Abbildung 1-54  |
| Abbildung 1-55 | Abbildung 1-56  |
| Abbildung 1-57 | Abbildung 1-58  |
| Abbildung 1-59 | Abbildung 1-60  |
| Abbildung 1-61 | Abbildung 1-62  |
| Abbildung 1-63 | Abbildung 1-64  |
| Abbildung 1-65 | Abbildung 1-66  |
| Abbildung 1-67 | Abbildung 1-68  |
| Abbildung 1-69 | Abbildung 1-70  |
| Abbildung 1-71 | Abbildung 1-72  |
| Abbildung 1-73 | Abbildung 1-74  |
| Abbildung 1-75 | Abbildung 1-76  |
| Abbildung 1-77 | Abbildung 1-78  |
| Abbildung 1-79 | Abbildung 1-80  |
| Abbildung 1-81 | Abbildung 1-82  |
| Abbildung 1-83 | Abbildung 1-84  |
| Abbildung 1-85 | Abbildung 1-86  |
| Abbildung 1-87 | Abbildung 1-88  |
| Abbildung 1-89 | Abbildung 1-90  |
| Abbildung 1-91 | Abbildung 1-92  |
| Abbildung 1-93 | Abbildung 1-94  |
| Abbildung 1-95 | Abbildung 1-96  |
| Abbildung 1-97 | Abbildung 1-98  |
| Abbildung 1-99 | Abbildung 1-100 |

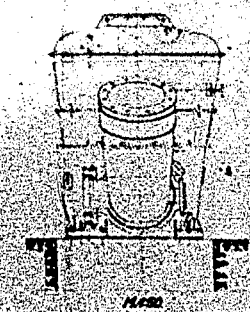
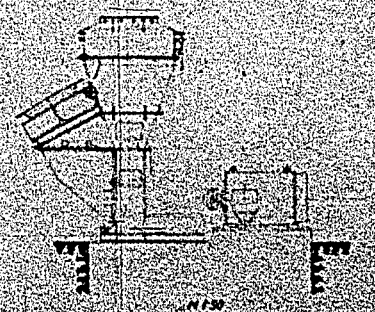
FIG. 3





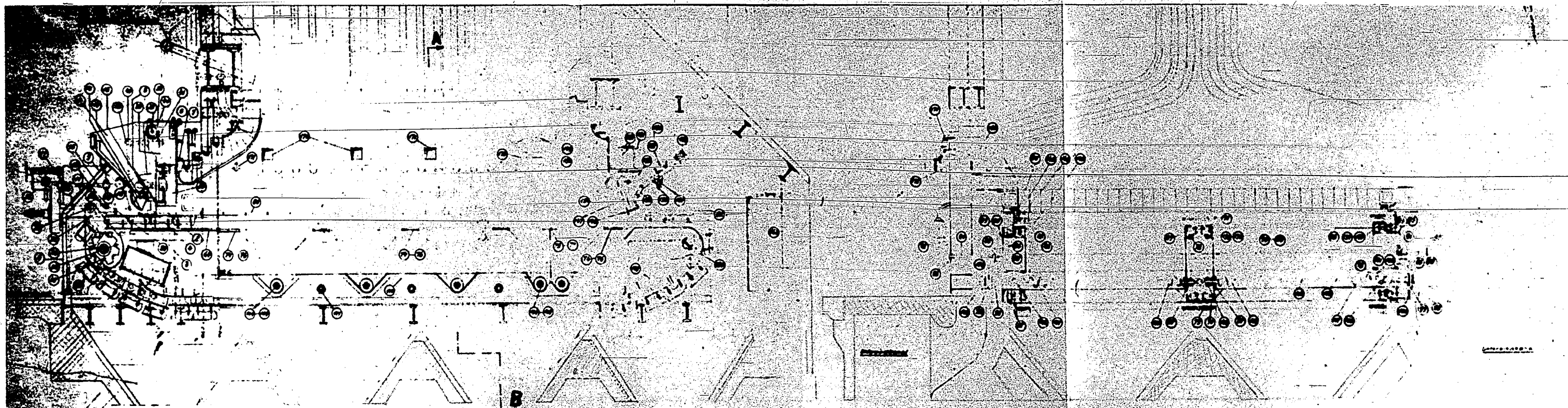
Zusammenstellungs- und Ersatzteilzeichnung  
für Naßkohlenmühle N 50

M 1:10



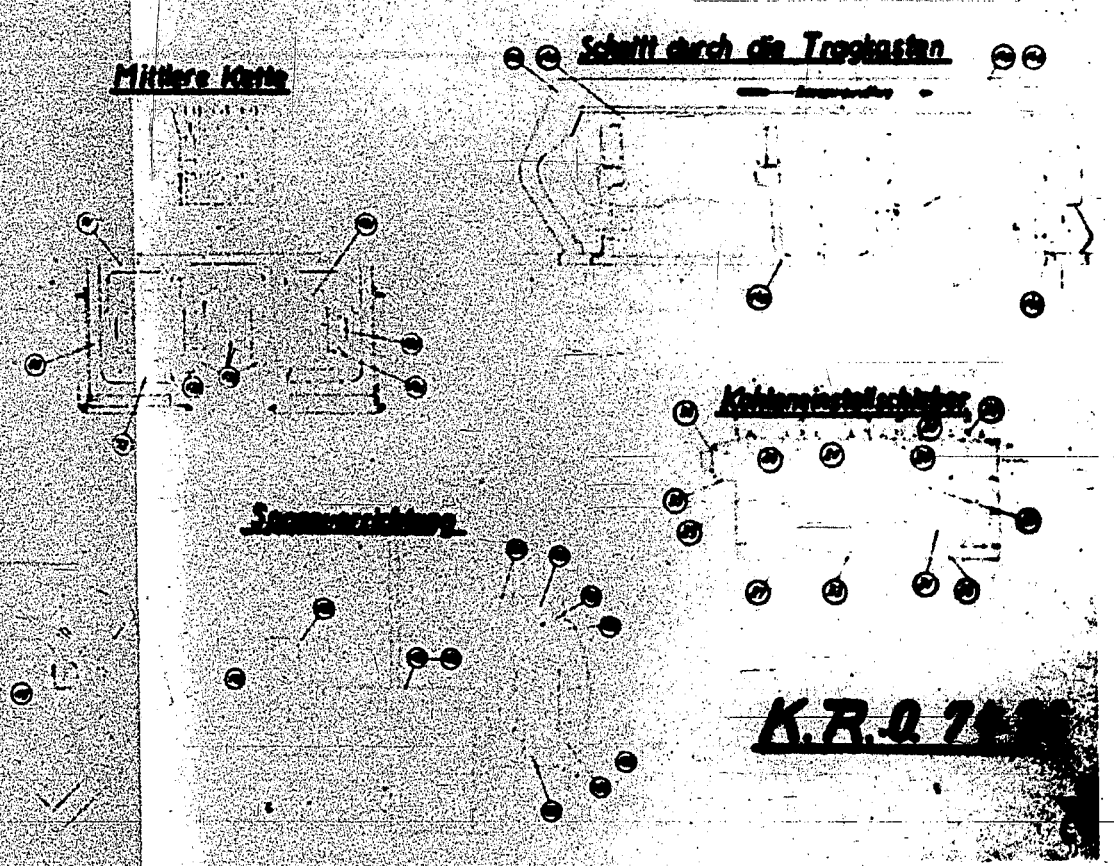
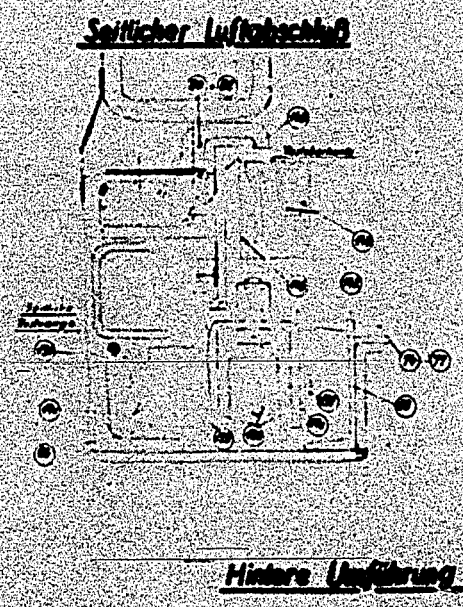
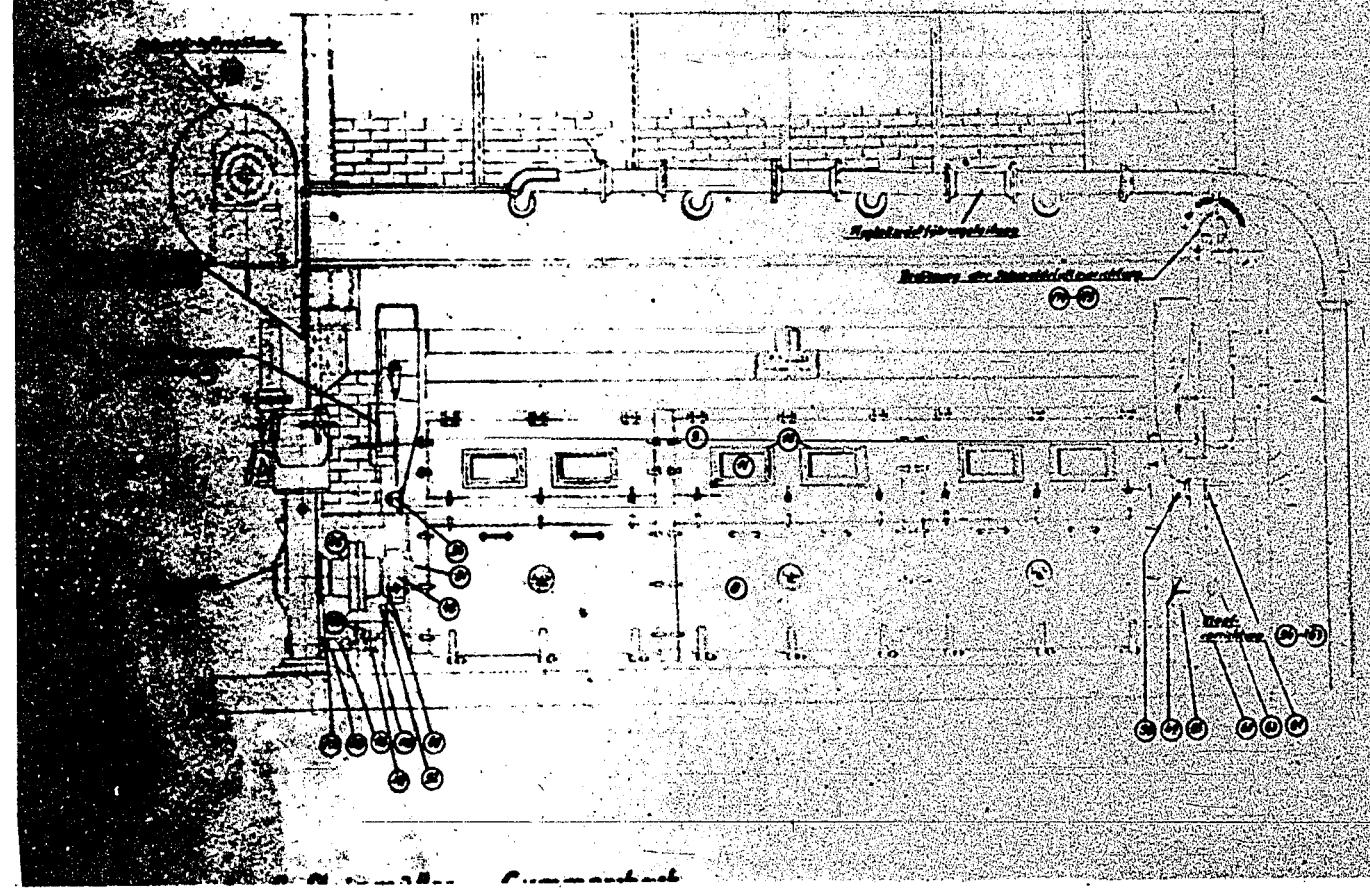
M.135382-0





**Steinmüller - Zonenwanderrost**

von 16 x 57 = 2052 m<sup>2</sup> Rest/Male



**K.R.O. 712**



Schnitt A-A

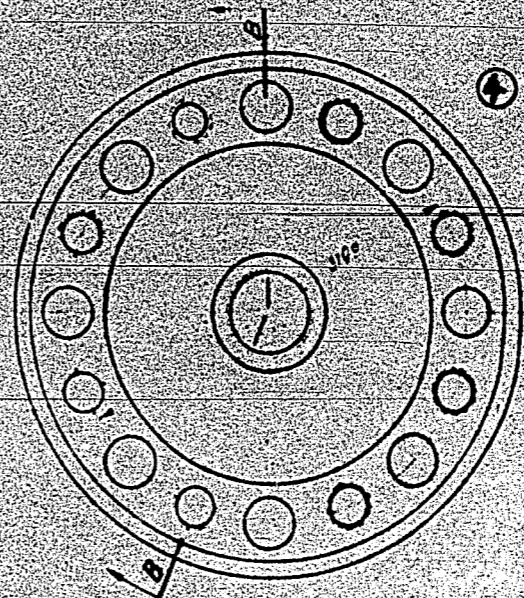
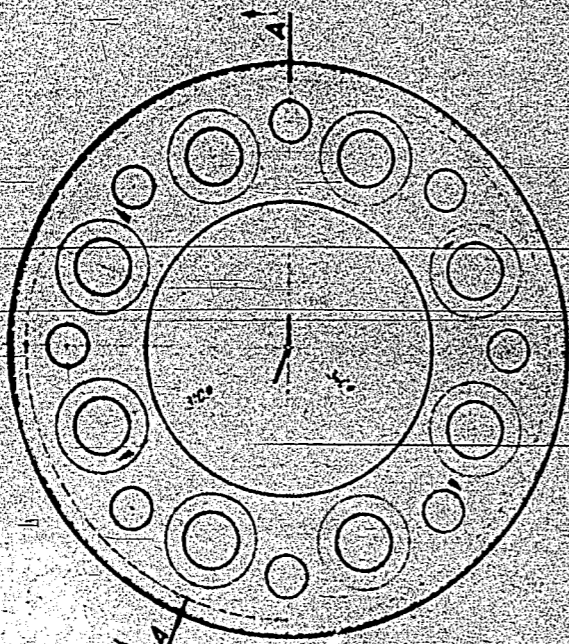
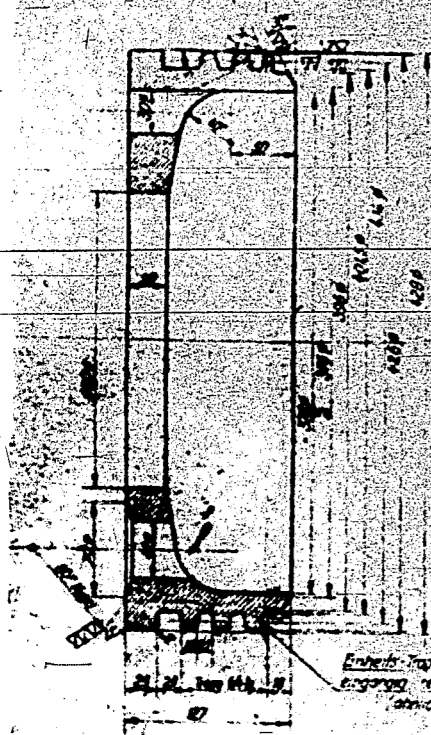
8 Löcher 32φ zum Einschrauben der Segmente

① (1)

Schnitt B-B

8 Löcher 32φ zum Einschrauben

② (w)



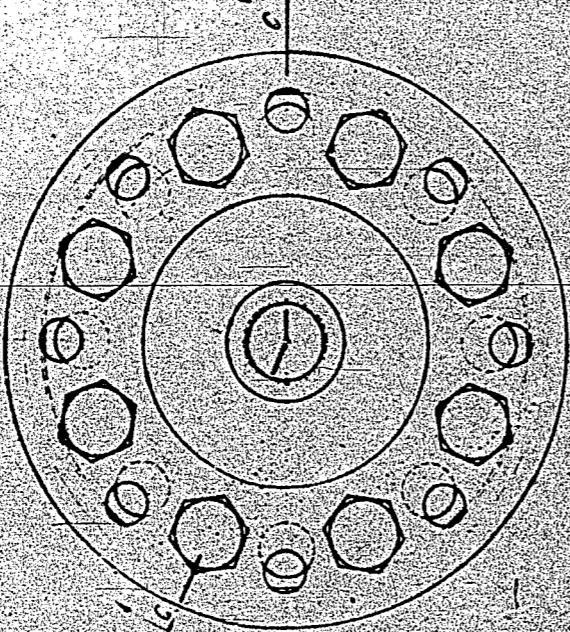
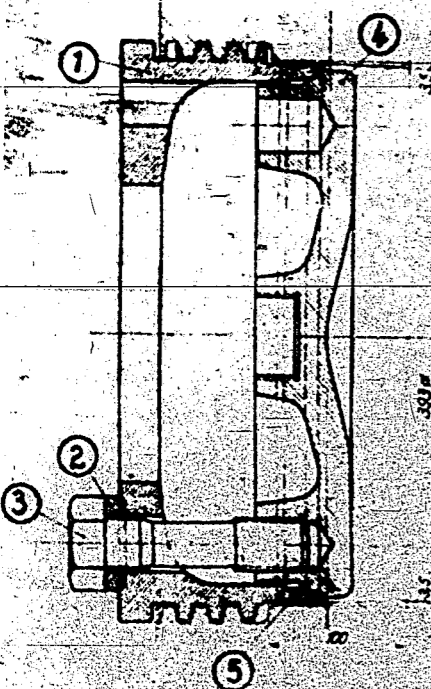
Einheits-Trapez-Segmente  
eingesetzt, nach 2 mm Steigung  
nach 1453

Schnitt C-C

8 Löcher 32φ zum Einschrauben

② (w)

③



8 Löcher 32φ zum Einschrauben

③ (w)



Gewicht eines Verschlussdeckels  
~ 94 kg

**Endgült.**

| №   | Bezeichnung | Menge | Einheit | Material  | Norm | Zeichn.-Nr. |
|-----|-------------|-------|---------|-----------|------|-------------|
| 1   | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 2   | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 3   | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 4   | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 5   | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 6   | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 7   | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 8   | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 9   | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 10  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 11  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 12  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 13  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 14  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 15  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 16  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 17  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 18  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 19  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 20  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 21  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 22  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 23  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 24  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 25  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 26  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 27  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 28  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 29  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 30  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 31  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 32  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 33  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 34  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 35  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 36  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 37  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 38  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 39  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 40  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 41  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 42  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 43  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 44  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 45  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 46  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 47  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 48  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 49  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 50  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 51  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 52  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 53  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 54  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 55  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 56  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 57  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 58  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 59  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 60  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 61  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 62  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 63  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 64  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 65  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 66  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 67  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 68  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 69  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 70  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 71  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 72  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 73  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 74  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 75  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 76  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 77  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 78  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 79  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 80  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 81  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 82  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 83  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 84  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 85  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 86  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 87  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 88  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 89  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 90  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 91  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 92  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 93  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 94  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 95  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 96  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 97  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 98  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 99  | Segmente    | 8     | Stk     | St 42-110 |      |             |
| 100 | Segmente    | 8     | Stk     | St 42-110 |      |             |

Wagner-Hochdruck-  
Dampfmaschinen-Kom-6  
Hamburg  
Anl. 55-55 / 1162-116A

Wagner-Kessel 380nt  
Kesselschüssel für die Obertrommel große  
und kleine Untertrommel

FIG. 7.



*Cofay!*

FINAL REPORT NO. 360

ITEM NO. 22

**NOTES ON  
MANUFACTURE OF ETHYLENE OXIDE**

*by I. G. FARBEN*

*Dartshore, J. F. B.*

**"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."**

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

**BRITISH INTELLIGENCE OBJECTIVES**

**SUB-COMMITTEE**

LONDON — H. M. STATIONERY OFFICE



NOTES ON MANUFACTURE OF ETHYLENE OXIDE

BY I.G. FARBEN

Reported by:-

J.F.C. Gartshore, M.O.S.

BIOS Target Nos. 22/1, 22/1(f)

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

32, Bryanston Square, W.1

(1946?)

8 p. - drafts



## CONTENTS

|   |        |
|---|--------|
| <u>Introduction</u>   | page 1 |
| <u>Details</u>  |        |
| Manufacturing record of ethylene oxide<br>in I.G., Fig.1              | 1      |
| Manufacture of glycols at Gendorf                                     | 1      |
| Development of the direct air-oxidation<br>process for ethylene oxide | 4      |
| Flowsheet of Zweckel pilot plant Fig.2                                |        |

### Personnel of team

|                        |                           |
|------------------------|---------------------------|
| J.D. Rose, Team Leader | M. O. S.                  |
| *C. J. S. Appleyard    | "                         |
| *J. W. Fisher          | "                         |
| *J. F. C. Gartshore    | "                         |
| P. W. Blaylock         | Can. Dept. Reconstruction |
| A. H. Andersen         | "                         |

\* Those marked thus took part in the investigation.

## INTRODUCTION

This is a record of some disjointed notes collected as a matter of interest in the intervals of another investigation during November, 1945. All the matter will probably be reported in full detail by others, if it has not so been already. Three sources of information are represented: a visit to Anorgana G.m.b.H. at Gendorf, an interrogation of Dr. Christ at Ludwigshafen, and two files I.G. Ludwigshafen seen in Griesheim Document Centre.

Ethylene oxide was manufactured by I.G. upon a very large scale, principally for the manufacture of glycol and polyglycols, which were used in explosives manufacture as glycerin substitutes. The route was via ethylene chlorohydrin. During the last ten years I.G. had also developed a direct air-oxidation process, and had partially erected a pilot-plant at Zweckel.

## DETAILS

### Manufacturing record of ethylene oxide in I.G.

Block diagrams showing the production and distribution of ethylene oxide month by month are contained in the I.G. Ludwigshafen file S.29/II-E-11. A specimen is given in Fig.1. There is also information in S.29/III-E-4.

### Manufacture of glycols at Gendorf

Chlorohydrin is made by passing ethylene, chlorine, and water together into the foot of a packed tower filled with water. Dilute acid chlorohydrin overflows at the top and is passed into a liming kettle, where it is mixed with lime slurry at about 100°C. Ethylene oxide distils off, is partially purified by a dephlegmator which stops most of the dichloride, and is fractionated in a bubble-plate column to remove the rest of the dichloride. The ethylene oxide is stored in mild steel tanks of about 25 cbm. capacity: they are buried in the ground as a protection against air attack. The tanks are cooled by brine coils, and a few lb./sq.in.



of nitrogen pressure is kept on them. Pipe lines are lagged and occasionally brine-traced, and are usually mild steel. Valves are Klinger piston type, and by contrast are usually stainless. The plant chemist was in favour of having stainless pipes as well as valves, as there was less trouble from rust particles collecting in valves and making them leak.

In the conversion to glycols, ethylene oxide is pumped to 22 atmos. by a 9-stage centrifugal pump at 1.5 cbm./hr. to a 750 litre mixing chamber, where it meets 9 cbm./hr. of sweet water returned from the glycol evaporators. The dilute ethylene oxide is preheated to 120-160°C. by heat exchange with the converter outflow (temperature controlled by by-pass), and passed into the foot of the converter. The converter is 11 m. working height to overflow and 0.8 m. internal diameter, of steel for 22 atmos. working pressure, filled with iron raschig rings. The temperature at the foot is 190°C., and at the top 200°C., and the contact time is 30 minutes calculated for the empty shell. There is an open steam pipe at the foot to heat up the burden at the start of a run, and a cooling coil at the top to remove heat of reaction. This coil was originally serviced with cooling water, but now acts as a boiler to boil water under pressure into a reflux condenser.

The overflow from the converter passes to the feed heat exchanger, which it leaves at 95-105°C., and is then let down to atmospheric pressure. The volatiles which blow off are acetylene (ethylene is made by hydrogenation of acetylene in this factory), acetaldehyde, and ethylene oxide (0.02% and 0.3% respectively on weight of glycol solution). The glycols solution is adjusted to pH 7 by addition of caustic soda and concentrated in a 3-stage evaporator, from which the water (containing 0.5-1.0% glycols) is returned to the conversion stage, and the concentrate containing 15% water and 85% mixed glycols is passed to a conventional 4-column still set for separation into water (returned to conversion), glycol, diglycol, triglycol, and tetraglycol foots.

The composition of the mixed glycols obtained with the feeds stated is 86% glycol, 11% diglycol, 3% triglycol. When it is desired to make diglycol as

principal product, the ethylene oxide feed to the converter is diluted with 50% glycol instead of sweet water.

#### Overall yields in Gendorf process

|                | <u>M.W.</u> | <u>Theory yields</u><br><u>kg.</u> | <u>Practical yields</u><br><u>kg.</u> | <u>Efficiency</u><br><u>%</u> |
|----------------|-------------|------------------------------------|---------------------------------------|-------------------------------|
| Ethanol 94%    | 46          | 111                                | 162.5                                 |                               |
| Ethylene       | 28          | 63.6                               | 83.7                                  | 90                            |
| Chlorohydrin   | 80.5        | 180.7                              | 192.5                                 | 80                            |
| Ethylene oxide | 44          | 100                                | 100                                   | 95                            |
| Glycol         | 62          | 140.8                              | 114.1                                 | 81                            |
| Diglycol       | 106         | 120.5                              | 10.2                                  | 8.5                           |
| Triglycol      | 150         | 113.6                              | 1.6                                   | 1.4                           |
| Chlorine       | 55.5        | 161.2                              | 204                                   | 83                            |
| Lime           | 74          | 168                                | 200                                   | 84                            |

#### Specification for explosives glycol

Only the sense of the clauses is given.

1. Water clear, etc.; completely water-soluble.
2. Specific gravity D 20/4, 1.1130-1.1132.
3. Acidity less than 0.005% as H<sub>2</sub>SO<sub>4</sub>.
4. Saponification value less than 0.3%.
5. Boiling range 196-200°C./1 atmos.
6. Polyglycols not above 3%.
7. Ash not above 0.01%.
8. Chlorine below 0.01% as Cl.

#### Analytical methods

Ethylene oxide strength is determined by conversion to chlorohydrin and back titration. Acetaldehyde in ethylene oxide is determined by adding iodine in sodium hydroxide and back titrating with thiosulphate. Acetylene in ethylene oxide is determined by the Ilosvay reagent, containing 20g. CuSO<sub>4</sub>.5H<sub>2</sub>O; 80 ml. 20% ammonia; 60 g. hydroxylamine hydrochloride, per litre.

Polyglycols in glycol are determined by a standardised distillation. In one form 200 ml. of the mixed glycol is distilled from a 1 litre Claisen flask



provided with 3 bulbs on the side-arm, and a 75 cm. condenser of which the lower 40 cm. is water jacketed. 165 ml. are distilled off at 10 mm. head pressure, and 5 ml. additional drained down from the (vertical) condenser. The density of the residue is measured:-

| <u>Density of residue D 20/4</u> | <u>% Polyglycol in original</u> |
|----------------------------------|---------------------------------|
| 1.1132                           | 0                               |
| 1.1133                           | 0.3                             |
| 1.1134                           | 0.7                             |
| 1.1135                           | 1.0                             |
| 1.1136                           | 1.4                             |
| 1.1137                           | 1.7                             |
| 1.1138                           | 2.2                             |
| 1.1139                           | 2.5                             |
| 1.1140                           | 2.8                             |
| 1.11405                          | 3.0                             |

For triglycol in diglycol, a total of 140 ml. is distilled from 200 ml. charge, and density and refractive index of the residue are measured. The following figures are quoted:

|           |        |        |        |        |
|-----------|--------|--------|--------|--------|
| Diglycol  | D 20/4 | 1.1164 | N D/20 | 1.4476 |
| Triglycol | "      | 1.1240 | -      | -      |

| <u>Density of standard residue, D 20/4</u> | <u>Refractive index of standard residue, N D/20</u> | <u>% Triglycol in original diglycol</u> |
|--|---|---|
| 1.1164                                     | 1.4476  | 0                                       |
| linear to 1.11755                          | linear to -   | 5                                       |
| linear to 1.12025                          | 1.45145   | 20                                      |

Development of the direct air-oxidation process for ethylene oxide

In the ten years or so before the war, I.G. had done a small amount of semi-technical and laboratory work on the direct air-oxidation of ethylene over silver and gold catalysts, including some work with 80% ethylene, 3% oxygen mixtures.

In 1938 they had bought from the Societe Francaise pour Catalyse Généralisée a ready-made process, which used a dilute ethylene mixture (2-3% ethylene in air) in a converter containing 3000 aluminium tubes 20 mm. bore plated on the inside with silver: this plating acted as the catalyst. The results were unsuccessful, and they put on their own most recent research process in the same converter. In this process the tubes were filled with a silver-on-pumice catalyst containing 350 g. silver per litre of catalyst. The feed gas was 3% ethylene in air, of which two thirds was consumed and one third passed through unchanged; and the off-gases contained 1% of ethylene oxide. The catalyst temperature was 220°C.

During the war a pilot plant for 100 tons/month by this process was designed, and partially built at Zweckel. Construction had been impeded by bombing and finally interrupted by the end of the war. The design flowsheet is shown in Figure 2.

There were ("to be" understood throughout) four converters, each containing 3000 tubes 3 m. long x 25 mm. bore, cooled by oil circulation. The tubes were heavily galvanised mild steel, and the working pressure on the gas side was atmospheric, plus of course any pressure drop. Each converter held 3 cbm. of catalyst.

The catalyst was made thus. Silver nitrate solution was precipitated with potassium hydroxide solution, and the silver oxide washed by decantation till the effluent had a pH of 7.5. The moist silver oxide was rolled with pumice granules 5-7 mm. mesh, and warmed with hot air until nearly, but not quite, dry. The coated granules were reduced in a separate converter with 30 mm. tubes cooled by circulated oil, at 220-240°C. with nitrogen containing 5% hydrogen, and then loaded into the ethylene oxide converters. The silver oxide could also be decomposed by heating in nitrogen at 280°C., but this did not give such a good catalyst. The catalyst contained 350 g. silver per litre of granules.

On the basis of the figures in the flowsheet, the conversion and yield in both converters were 69% and 55% respectively. The space velocity was 3500 cbm./hr.



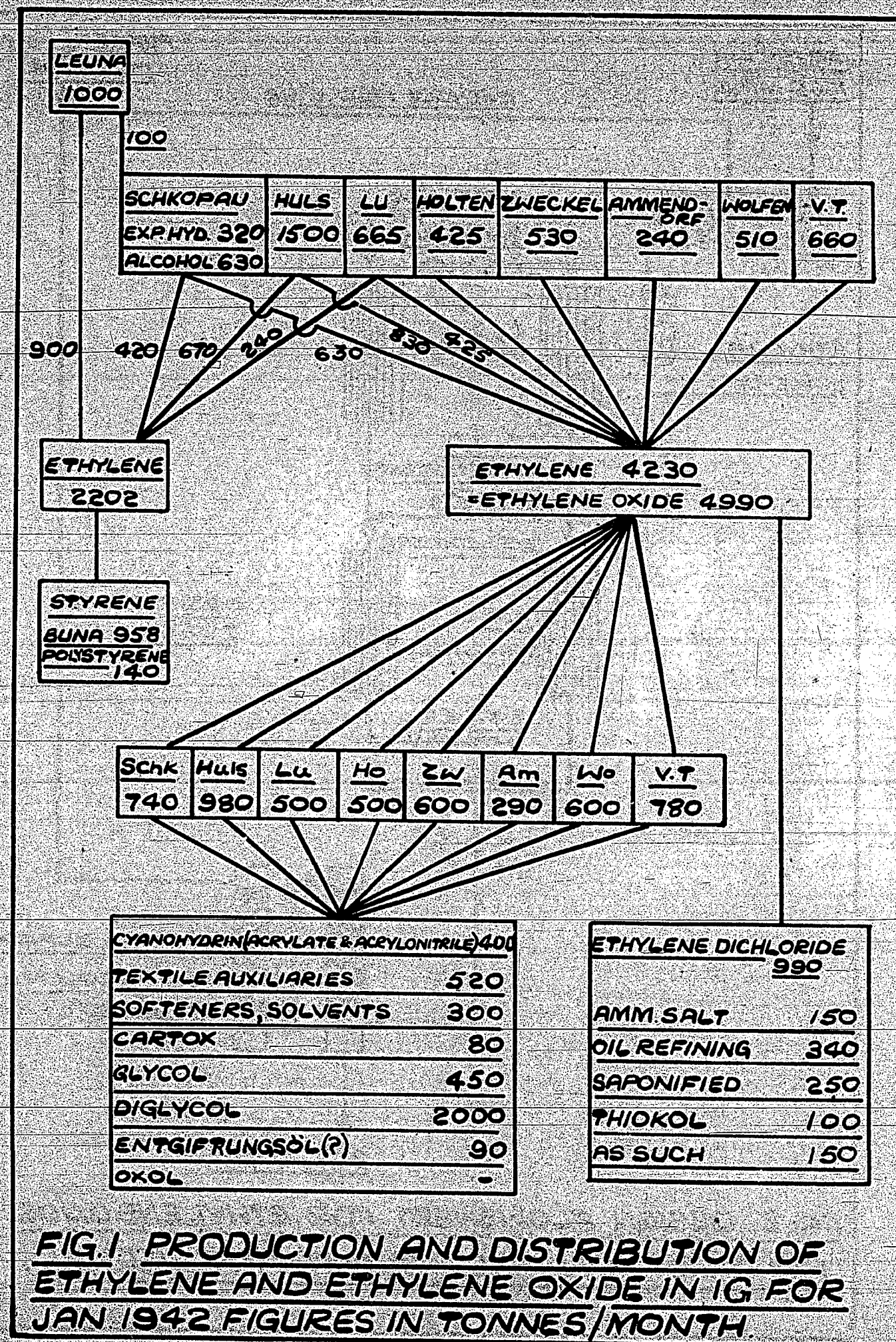
through 4 x 4.41 cbm. of catalyst space (4 x 3.0 cbm. of catalyst), or approx. 200 hr.<sup>-1</sup>. The life of catalyst with reasonably pure ethylene was expected to be about 1 yr.

At Zweckel ethylene was made from coke oven gas, and contained about 1.5% acetylene. It was purified by adding an equivalent amount of hydrogen and hydrogenating over a palladium catalyst.

It had been proposed to recover ethylene oxide as such from the converter gases by adsorption in active carbon, and recovering ethylene oxide by steaming, which could be done without appreciable hydrolysis.

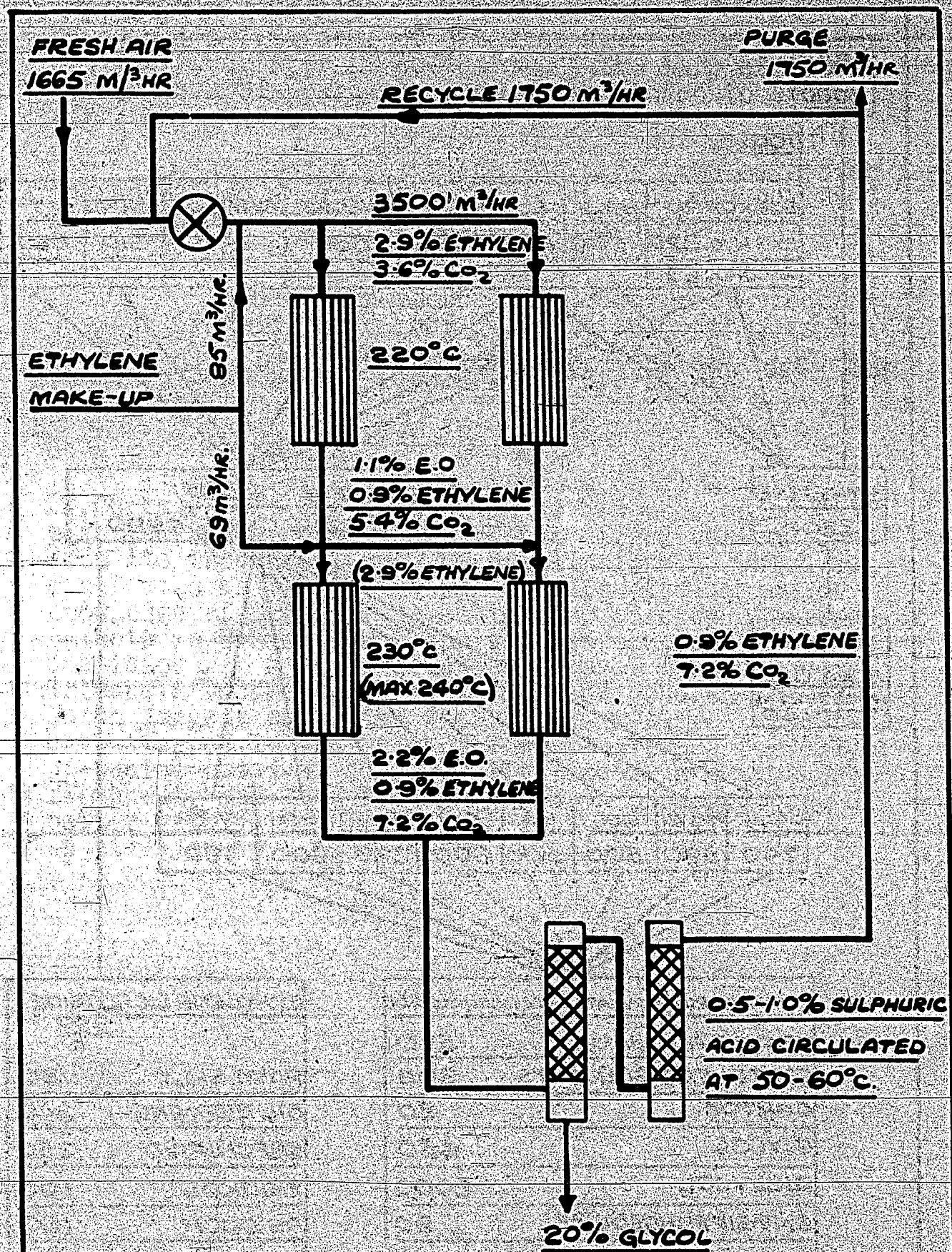
Check on above information

The above information was obtained from Dr. Christ. It was known that he had been concerned with the whole development of the air-oxidation route from its inception, but the disillusionment which he manifested as to its future, and his disclaiming of knowledge of lines of investigation already disclosed by patents in U.K. and U.S.A., were so complete as to be suspicious. A large file was however discovered in Griesheim Document Centre (I.G. Ludwigshafen S.29/IV-A-4) which largely supported him. I.G. research appears to have stopped in 1941 with the draft process for silver-on-pumice catalyst subsequently turned into the Zweckel flowsheet, and they had no prospects of developing a better catalyst. I.G. had corresponded with S.F.C.G. up till January, 1943.



**FIG. 1 PRODUCTION AND DISTRIBUTION OF ETHYLENE AND ETHYLENE OXIDE IN IG FOR JAN 1942 FIGURES IN TONNES/MONTH.**





**FIG. 2. DESIGN FLOWSHEET FOR ETHYLENE OXIDE AIR-OXIDATION PILOT PLANT AT ZWECKEL.**



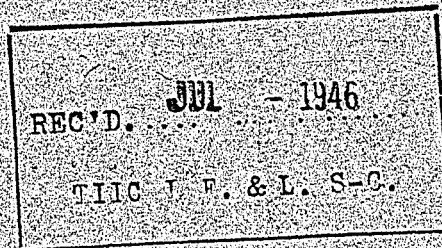
FINAL REPORT NO. 351  
ITEM NO. 22

*copy!*

# PREPARATION OF ADIPIC ACID FROM TETRAHYDROFURAN AND CARBON MONOXIDE

*Gartshore, J. F. C.*

"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."



BRITISH INTELLIGENCE OBJECTIVES  
SUB-COMMITTEE

LONDON — H. M. STATIONERY OFFICE



PREPARATION OF ADIPIIC ACID FROM  
TETRAHYDROFURAN AND CARBON MONOXIDE

Reported by:-

J. F. C. Gartshore, M. O. S.

BIOS TARGET NO. 22/1

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

32, Bryanston Square, W.1

C 1946?)

S.P. diag.



CONTENTS

Introduction

page 1

Details

page 1

Personnel of team

|                           |                           |
|---------------------------|---------------------------|
| * J. D. Rose, Team Leader | M. O. S.                  |
| C. J. S. Appleyard        | "                         |
| * J. W. Fisher            | "                         |
| * J. F. C. Garshore       | "                         |
| P. W. Blaylock            | Can. Dept. Reconstruction |
| A. H. Andersen            | "                         |

\* Those marked thus took part in the investigation.



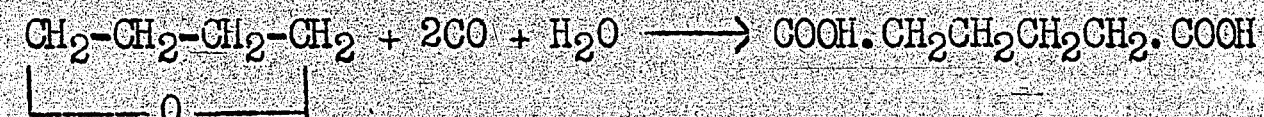
## INTRODUCTION

The information in this report was obtained from Dr. Kröper of I.G. Ludwigshafen, who was interrogated at Anorgana G.m.b.H. Gendorf on 12.11.45. There appears to be little or nothing here which has not been found by other investigators.

## DETAILS

### Principles

Carbon monoxide is caused to react with tetrahydrofuran in presence of nickel iodide as catalyst and nickel carbonyl as a carbon monoxide carrier, at 270°C./200 atmos. The yield is of the order of 80%



### Semi-technical plant

The flowsheet is given in Fig.1.

A mixture of 93% by weight of tetrahydrofuran and 7% of nickel carbonyl is pumped at 7-14 litres/day of 24 hr. to the foot of the converter, where it meets two other liquid streams; firstly the catalyst solution, a concentrated solution of nickel iodide in water supplied at a rate such that nickel iodide is fed at 1% on the tetrahydrofuran, and secondly the recycle condensate consisting of unused tetrahydrofuran and nickel carbonyl. Carbon monoxide is also pumped in at the foot of the converter.

The converter is 70 mm. bore and 3 metres high overall, constructed in steel to a working pressure of 200 atmos. at 270°C., and lined with platinum to resist corrosion. There is an overflow about 2.5 metres from the bottom, so that the liquid volume is 7 litres.

Under steady conditions the liquid overflowing at this point consists of 80% adipic acid, 5% valero-



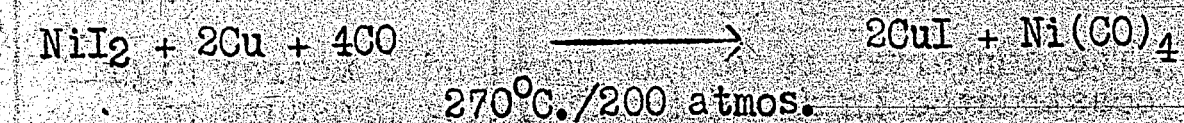
lactone, and 15% valeric acid, with a small amount of retained tetrahydrofuran; and contains all the nickel iodide catalyst. The apparatus is started up with the converter full of molten adipic acid at 270°C. The operating conditions in the converter are 270°C./200 atmos.

The vapours at the top of the converter, above the overflow, consist of carbon monoxide saturated with tetrahydrofuran, water, and nickel carbonyl. They pass through about 0.5 m. of ring packing at the head of the converter to stop entrainment, through a coil condenser, and into a receiver, where the disengaged carbon monoxide is separated off and recycled together with make-up gas into the bottom of the converter.

The condensate is recycled to the converter as liquid. (Although this point was not observed at the interrogation, this liquid cannot be recycled directly into the converter as shown dotted on the flowsheet, otherwise nickel would accumulate in the system: it is presumably worked up separately, and the nickel carbonyl and tetrahydrofuran used to make up fresh feed solution.)

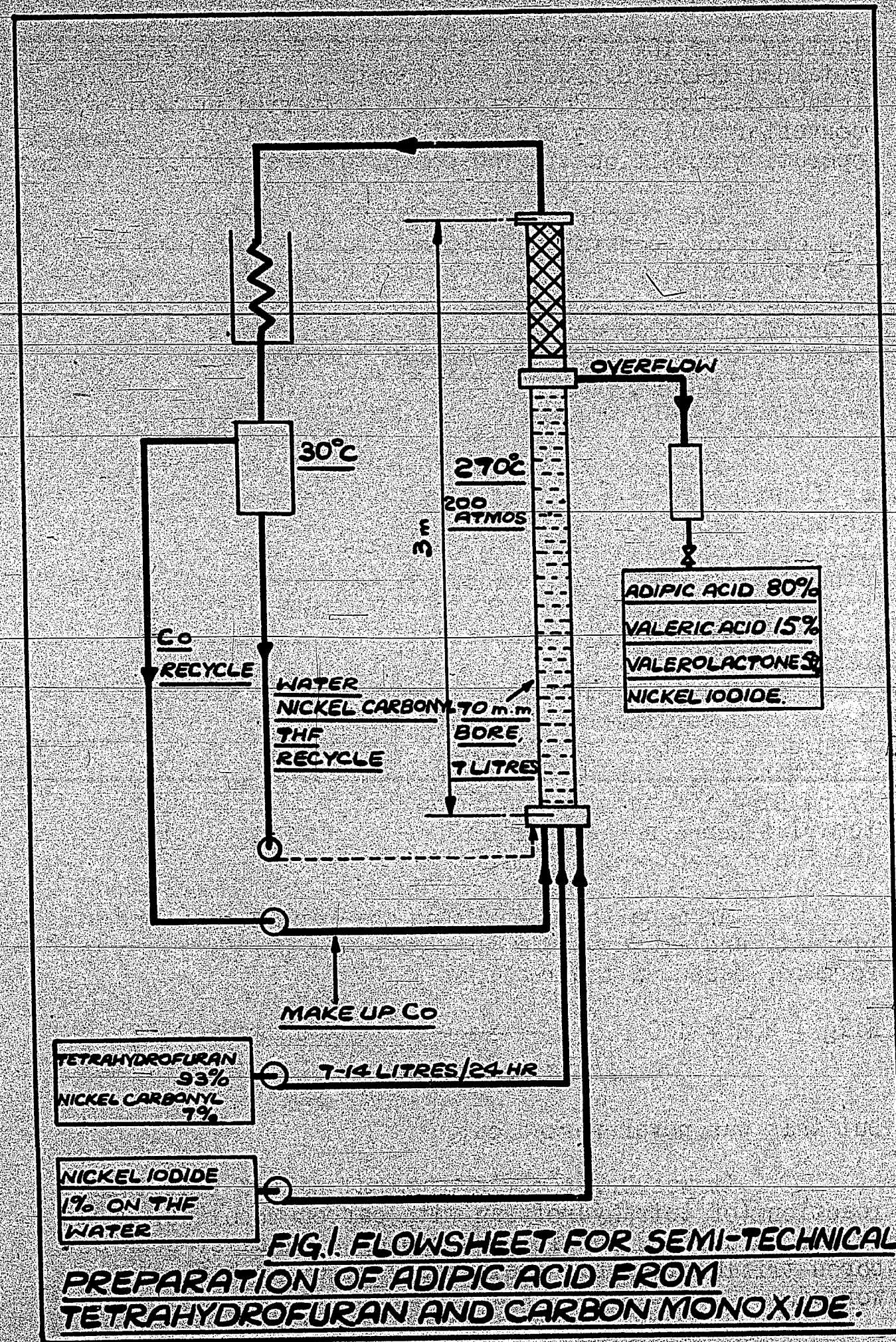
Comparatively few experiments seem to have been done in this apparatus. It was built at Ludwigshafen, and when that place was put out of commission by bombing, it was dismantled and sent to Gendorf: the platinum-lined converter however did not arrive, and the project was interrupted.

An idea for recovering the nickel from the product, and incidentally purifying the product, was put forward, based on the reaction,



but this was never worked out.

It was Dr. Kroper's personal belief that this reaction (of tetrahydrofuran with carbon monoxide) would better be carried out in presence of a catalyst such as boron trifluoride, but preliminary attempts had not worked.





FINAL REPORT NO. 358

ITEM NO. 22

*Copy 1*

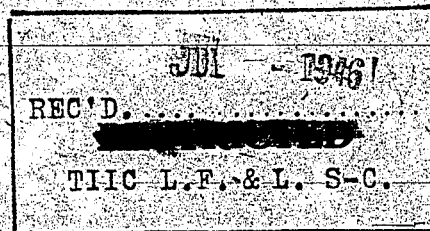
~~XXXXXXXXXX~~

## ACRYLIC ESTERS

### Synthesis from Acetylene and Nickel Carbonyl

*Dartshore, J. F. G., and Rose, J. D.*

"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."



**BRITISH INTELLIGENCE OBJECTIVES**

**SUB-COMMITTEE**

LONDON — H.M. STATIONERY OFFICE



ACRYLIC ESTERS

Synthesis from Acetylene and Nickel Carbonyl

Reported by

J.F.C. GARTSHORE AND J.D. ROSE  
M.O.S.

FIOS Target Nos. 22/1(f), 22/1

British Intelligence Objectives Sub-Committee,  
32, Bryanston Square, W.1.

(1946?)



ACRYLIC ESTERS: Synthesis from Acetylene and Nickel Carbonyl

Introduction

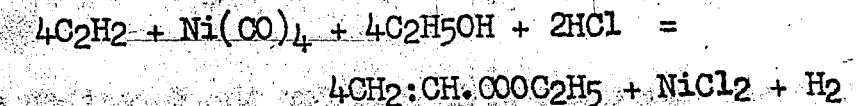
The following report is a compilation of data from various sources. The flowsheet of the laboratory apparatus for the continuous preparation of ethyl acrylate from acetylene, ethanol, hydrochloric acid and nickel carbonyl, and the outline of the process for the regeneration of nickel carbonyl from aqueous solutions of nickel salts, together with flowsheet, were obtained from Dr. Kröper of I.G. Ludwigshafen, who was interrogated at Anorgana G.m.b.H., Gendorf. Minor points were clarified by interrogation of Dr. Reppe at I.G. (Casella Works) Mainkur, Near Frankfurt, from whom the report by Dr. Keller (I.G. Ludwigshafen) was obtained. This report, a translation of which is appended, is a detailed account of a series of laboratory runs on the continuous preparation of ethyl acrylate; unfortunately the report contained no sketch or drawing of the apparatus used, and it is assumed that the report was written for an audience sufficiently familiar with the apparatus to make such drawing or description superfluous. As far as can be seen, the flowsheet obtained by interrogation of Dr. Kröper fits the experimental data of Dr. Keller's report fairly well, and the two should be considered together.

It is understood that a silver lined pilot plant for the synthesis of ethyl acrylate had been built, but in spite of efforts made, its whereabouts could not be ascertained. There appears to be some doubt as to whether or not the plant, although made, was ever assembled and erected. It is possible that this is one of the many plant items sent from Dr. Reppe's laboratory at Ludwigshafen to Anorgana at Gendorf when air bombardment made Ludwigshafen uninhabitable. No trace of it was found at Gendorf, and it may be that this was yet another item destroyed or stolen in transit.

Synthesis of Ethyl Acrylate

(a) Principles

Nickel carbonyl interacts vigorously with acetylene in aqueous alcoholic hydrochloric acid to yield ethyl acrylate according to the following equation:



The hydrogen set free reduces carbon monoxide (in part) and is partly utilised in the hydrogenation of by-products formed from vinyl acetylene and carbon monoxide.

TABLE OF CONTENTS

|   | <u>Page No.</u> |
|---|-----------------|
| Introduction  | 3               |
| Synthesis of Ethyl Acrylate   | 3               |
| Regeneration of Nickel Carbonyl from Nickel Salts   | 4               |
| Appendix I - Preparation of Acrylic Acid and Acrylic Esters. Translation of a report by Dr. Keller, Ludwigshafen, 6.7.40. | 6               |

PERSONNEL OF TEAM

|                   |  |
|-------------------|--|
| *J.D. Rose        | M.O.S. (Team Leader)                       |
| *C.J.S. Appleyard | M.O.S.                                     |
| *J.F.C. Gartshore | M.O.S.                                     |
| *J.W. Fisher      | M.O.S.                                     |
| P.W. Bloylock     | ) Canadian Department<br>of Reconstruction |
| A.H. Andersen     |  |

Those marked with an asterisk took part in the investigation



The batchwise reaction has already been sufficiently described (see, for example, "Recent Advances in the Chemistry of Carbon Monoxide" - Translation of a report by Dr. W. Reppe).

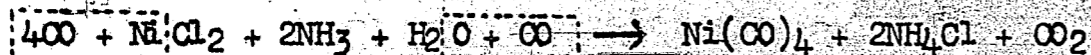
Continuous Preparation of Ethyl Acrylate See Fig. I.

Acetylene, ethanol-38% aqueous hydrochloric acid mixture, and nickel carbonyl are fed separately, in quantities calculated from the ratio  $4C_2H_2:Ni(CO)_4:4C_2H_5OH:2HCl$ , co-current through a sintered glass plate at the bottom of a column approximately 120 cm. high and 5 cm. in diameter. The column is filled either with ethanol-hydrochloric acid, or preferably with a mixture of these and ethyl acrylate. The tube is warmed to  $40^\circ$  by external electric heating on starting an experiment, but proceeds exothermically without added heat, and cooling is provided by external thermosyphons. Acetylene in very small amount is purged from the top of the column. The liquid overflow from the column, containing ethyl acrylate and minor quantities of the reactants passes via a sintered plate to the bottom of a second column (170 x 3 cm.), more acetylene being fed simultaneously. The object of this second acetylene treatment is the exhaustion by reaction of the residual nickel carbonyl; less acetylene is absorbed in this column than in the first, and the gas purge at the head is accordingly higher. The liquid overflow from Column 2 passes to a steaming-column, in which the volatiles (ethyl acrylate, alcohol) pass over as distillate, and in which polymers and aqueous nickel chloride are removed as bottoms. The alcoholic ethyl acrylate flows to a hydroselction column in which the ethyl acrylate is removed overhead and dilute ethanol as bottoms.

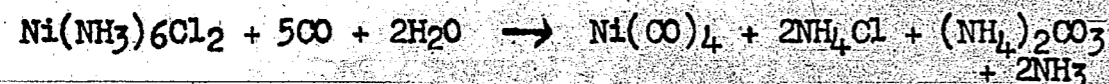
Regeneration of Nickel Carbonyl from Nickel Salts

In the acrylic ester synthesis, the nickel of the nickel carbonyl is recovered as aqueous nickel chloride, and a process was devised for recovering nickel carbonyl from such solutions.

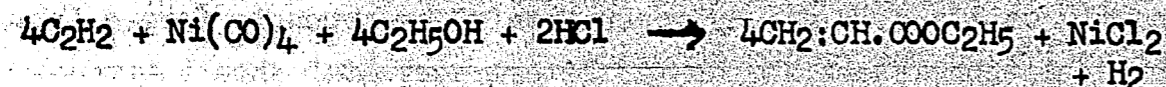
The principles of the recovery are expressed by the equation below:



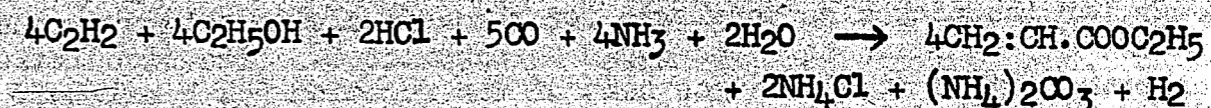
or, overall:



Combining these equations with that for the formation of ethyl acrylate, i.e.:



the overall equation, including the regeneration of nickel carbonyl, becomes:



The apparatus designed for the regeneration of nickel carbonyl is shown diagrammatically in Fig. II. Aqueous nickel chloride is treated with rather more than the amount of ammonia theoretically required for the formation of hexammino nickel chloride  $[Ni(NH_3)_6Cl_2]$  and diluted to a nickel (metal) content of 4%. This is fed by a ram pump co-current with carbon monoxide at 200 atmospheres through a tower at  $170^\circ C.$ , in which nickel carbonyl is formed, and passes over as vapour with excess carbon monoxide via a condenser to a separator. The overflow from the tower is aqueous ammonium chloride and ammonium carbonate, which is collected, and from which more ammonia is generated by liming.

The condensed nickel carbonyl is separated from entrained water and ammonia (which are passed back to the column) and via a let-down valve to a receiver cooled to  $10^\circ$ . The vapours from this receiver containing some nickel carbonyl are passed through one of two vessels arranged in parallel where, at  $170^\circ$ , decomposition to nickel and carbon monoxide occurs, the gas being recirculated into the main stream if desired. When one of these two vessels is full of nickel, the vapour is switched to the second for decomposition, whilst the first is "regenerated" by circulating carbon monoxide at 200 atmospheres which converts the metallic nickel to its carbonyl and carries it back to the main separator.



Appendix I

Preparation of Acrylic Acid and Acrylic Esters.  
Report by Dr. Keller, Ludwigshafen, 6.7.40.

The apparatus\* was so arranged that there were separate feeds for the nickel carbonyl and for the acid-alcohol mixture, and so that the reaction solution could be circulated. The circulation was primarily to dissipate the heat of the reaction (54 k cal/mol. of ethyl acrylate). Acetylene was introduced through a sinter at the bottom of the column, and the temperature was largely regulated by the acetylene flow. The reaction solution was allowed to flow back through two external thermosyphon coolers before returning to the column, the bottom 1/3rd of which was externally heated in the early stages of the reaction; this external heating was not necessary after the reaction had got under way. The temperature was measured in three places, at the bottom, at a point 1/3 from the bottom, and at the top of the column. The off gases were passed through an ice cooled condenser and then through two solid carbon dioxide cooled condensers in series, to remove nickel carbonyl, and finally were blown to exhaust via a gas meter. In larger experiments the gas could be recirculated.

Nickel carbonyl is dripped into the cooled solution at the circulation return; the acid and alcohol mixture (ethanol and HCl) were blown in at the bottom of the column, with the acetylene through the frit. This method keeps the sintered glass distributor clean and free from crystalline deposits. The volume of the column is 2.5 litres (Column 1).

The crude solution is taken from the circulation return before nickel carbonyl is added, and passed to a second column (1 litre, Column 2), where more acetylene is passed into the solution to exhaust the unused nickel carbonyl; in this column a little more acetylene is absorbed, the quantity being usually 10-20% of the quantity absorbed in Column 1. This is illustrated in Table 2.

In starting up the apparatus, Column 1 is filled with sufficient ethanol-hydrochloric acid mixture to ensure that, after the addition and reaction with acetylene of the calculated quantity of nickel carbonyl, the concentration of ethyl acrylate will be 30-35%. The "induction period" of the reaction (i.e. the time required after starting up for the concentration of the ethyl acrylate to reach 30-35%) can be appreciably decreased by adding some ethyl acrylate† to the reaction mixture.

When the optimal ester concentration (which is governed by the solubility of nickel chloride in the ester-alcohol-acid mixture) is

\*Translators' Note: The report contained no diagram or sketch of the apparatus.

† See footnote, p.9.

reached, the addition of fresh ethanol-acid is started, and during this, the continuous period of reaction, the additions of nickel carbonyl, ethanol, hydrochloric acid and acetylene must be in stoichiometric ratio.

The course of the reaction is followed by frequent analyses and Table 1 presents a representative run with the relevant data.

Table 1

| Time<br>h. | TEMPERATURE |    |    | Acid<br>Value | Sap.<br>Value | Ester<br>Value | Ester<br>Content % |          |
|------------|-------------|----|----|---------------|---------------|----------------|--------------------|----------|
|            | Column 1    |    |    |               |               |                |                    | Column 2 |
|            | T1          | T2 | T3 |               |               |                |                    |          |
| 9.30       | 32          | 43 | 40 | 48            | 79.7          | 232            | 152                | 27.1     |
| 10.30      | 25          | 38 | 35 | 37            | 109.5         | 325            | 215                | 38.4     |
| 11.30      | 35          | 44 | 43 | 40            | 95.3          | 296            | 200                | 35.8     |
| 13.15      | 35          | 43 | 42 | 43            | 91.0          | 291            | 200                | 35.6     |
| 14.15      | 33          | 42 | 42 | 42            | 87.0          | 256            | 169                | 30.1     |
| 15.45      | 33          | 44 | 44 | 42            | 93.5          | 276            | 182                | 32.5     |
| 9.00       | 33          | 44 | 43 | 46            | 101.0         | 276            | 175                | 31.2     |
| 10.15      | 32          | 44 | 43 | 42            | 98.0          | 278            | 180                | 32.1     |
| 11.30      | 32          | 42 | 42 | 41            | 97.5          | 265            | 167.5              | 29.9     |
| 13.00      | 32          | 42 | 42 | 43            | 95.0          | 264            | 169                | 30.2     |
| 14.30      | 29          | 40 | 39 | 45            | 92.0          | 273            | 181                | 32.3     |
| 16.00      | 30          | 43 | 42 | 54            | 87.0          | 268            | 181                | 32.3     |
| 9.30       | 29          | 42 | 41 | 48            | 93.0          | 266            | 173                | 30.9     |
| 10.45      | 28          | 43 | 40 | 40            | 92.4          | 269            | 176                | 31.6     |
| 12.00      | 28          | 45 | 43 | 41            | 90.8          | 267            | 176                | 31.5     |
| 13.45      | 28          | 44 | 41 | 39            | 93.7          | 273            | 179                | 31.9     |
| 15.00      | 27          | 44 | 40 | 38            | 94.0          | 272            | 178                | 31.8     |
| 16.00      | 27          | 48 | 41 | 41            | 86.5          | 270            | 174                | 31.2     |

During the continuous period of the run, Column 1 is heavily loaded by the addition of as much nickel carbonyl as can theoretically react (according to the composition of the liquor) without requiring too much acetylene in Column 2. At 40°, a space-time yield of 100 g. ester (= 1 mol.) per hour per litre (Columns 1 and 2) can be reached. Since the space-time yield is strongly dependent on the rate at which heat of reaction can be dissipated, it is expected that this yield can be increased in larger plants with more efficient cooling systems. Table 2 is a summary of the column loading and the space time yields in a series of runs.



Table 2

| Expt. No. | Ni(CO) <sub>4</sub> cc./15 min. | EtOH/HCl cc./15 min. | Space-time yield in mol. ester/l./hr. | ACETYLENE ABSORPTION |          |      |
|-----------|---------------------------------|----------------------|---------------------------------------|----------------------|----------|------|
|           |                                 |                      |                                       | Column 1             | Column 2 |      |
|           |                                 |                      |                                       | 1.                   | % of 1   |      |
| 1         | 22.1                            | 146.5                | 0.93                                  | -                    | -        | -    |
| 2         | 26.6                            | 157.5                | 1.09                                  | -                    | -        | -    |
| 3         | 19.05                           | 138                  | 0.8                                   | -                    | -        | -    |
| 4         | 31.7                            | 196                  | 1.22                                  | 1,599                | 24.7     | 15.4 |
| 5         | 20.4                            | 145                  | 0.91                                  | 1,925                | 306      | 15.9 |
| 6         | 18.4                            | 138                  | 0.84                                  | -                    | -        | -    |
| 7         | 21.2                            | 160                  | 0.94                                  | 922                  | 117      | 12.7 |
| 8         | 25.6                            | 221                  | 1.18                                  | 1,218                | 110      | 9.0  |
| 9         | 23.7                            | 174                  | 0.99                                  | 1,069                | 158      | 14.8 |
| 10        | 19.45                           | 148                  | 0.92                                  | 1,036                | 169      | 16.3 |
| 11        | 18.9                            | 146                  | 0.86                                  | 946                  | 187      | 19.7 |
| 12        | 17.8                            | 151.5                | 0.85                                  | 1,369                | 290      | 21.2 |
| 13        | 20.15                           | -                    | 0.89                                  | 1,257                | 260      | 20.7 |
| 14        | 21.2                            | -                    | 0.98                                  | 1,180                | 246      | 20.8 |

Table 3 indicates that 85-95% of the carbon monoxide in the nickel carbonyl is converted to ester - calculated on the ester content of the effluent. Acrylic acid is simultaneously formed and consumes 4-12% of the carbon monoxide, so that 94-97% of the CO is converted to the carboxyl or carboxy group. 1.0-1.5 of the nickel carbonyl is lost in the off gases. Table 3 presents an analysis of three typical runs; the times given refer to total reaction time - induction period plus continuous period, the former averaging about 3 hours during which 3 mol. of nickel carbonyl are decomposed to bring the ester content up to 35%.

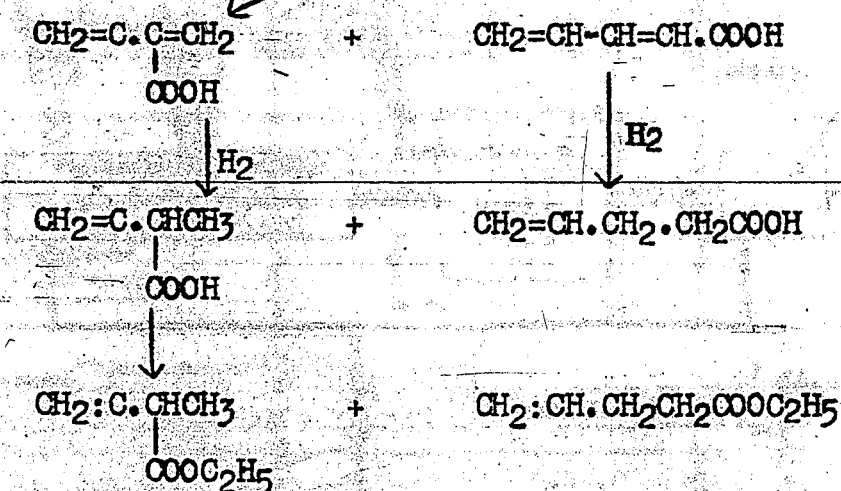
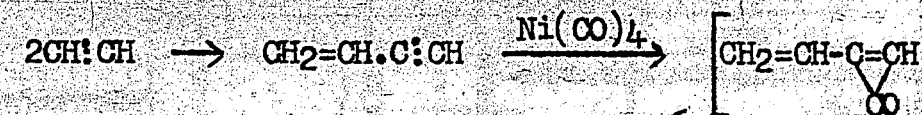
Table 3

| Number of Run                   | 1     | 2      | 3      |
|---------------------------------|-------|--------|--------|
| Duration of run - hours         | 12.5  | 18.75  | 18     |
| Temperature - °C.               | 40    | 40     | 40     |
| Nickel carbonyl used - cc.      | 1,140 | 1,570  | 1,450  |
| Nickel carbonyl used - mol.     | 8.72  | 12.00  | 11.1   |
| Acetylene used - litres         | 864   | 1,464  | 1,133  |
| Acetylene usage - calculated    | 785   | 1,080  | 999    |
| Acetylene excess usage - litres | 79    | 384    | 134    |
| Acetylene excess usage - %      | 10    | 35.6   | 11.8   |
| Effluent - Wt. in g.            | 9,600 | 12,750 | 13,100 |
| Effluent - Mol. acid (acrylic)  | 18.2  | 22.5   | 24.5   |
| Effluent - Ester content - mol. | 30.2  | 38.4   | 39.7   |
| Effluent - Ester content - %    | 31.5  | 30.1   | 30.3   |
| *Acid added at beginning - mol. | 14.0  | 22.0   | 20.0   |
| Acrylic acid formed - mol.      | 4.2   | 0.5    | 4.5    |
| Acrylic acid formed - % on CO   | 12.0  | 1.04   | 10.1   |
| Ethyl acrylate formed - mol.    | 29.2  | 37.4   | 39.7   |
| Ethyl acrylate formed - % on CO | 83.7  | 78.0   | 87.7   |

\*See footnote, p.9.

It will be observed in Table 3 that the acetylene absorption is in excess of that required by theory (based on 1 mol. acetylene being equivalent to 1/4 mol. nickel carbonyl). The excess usage decreases with increasing temperature and can be considerably reduced by dilution of the acetylene with nitrogen. Run 3 (Table 3) was carried out with a 50:50 mixture of acetylene and nitrogen.

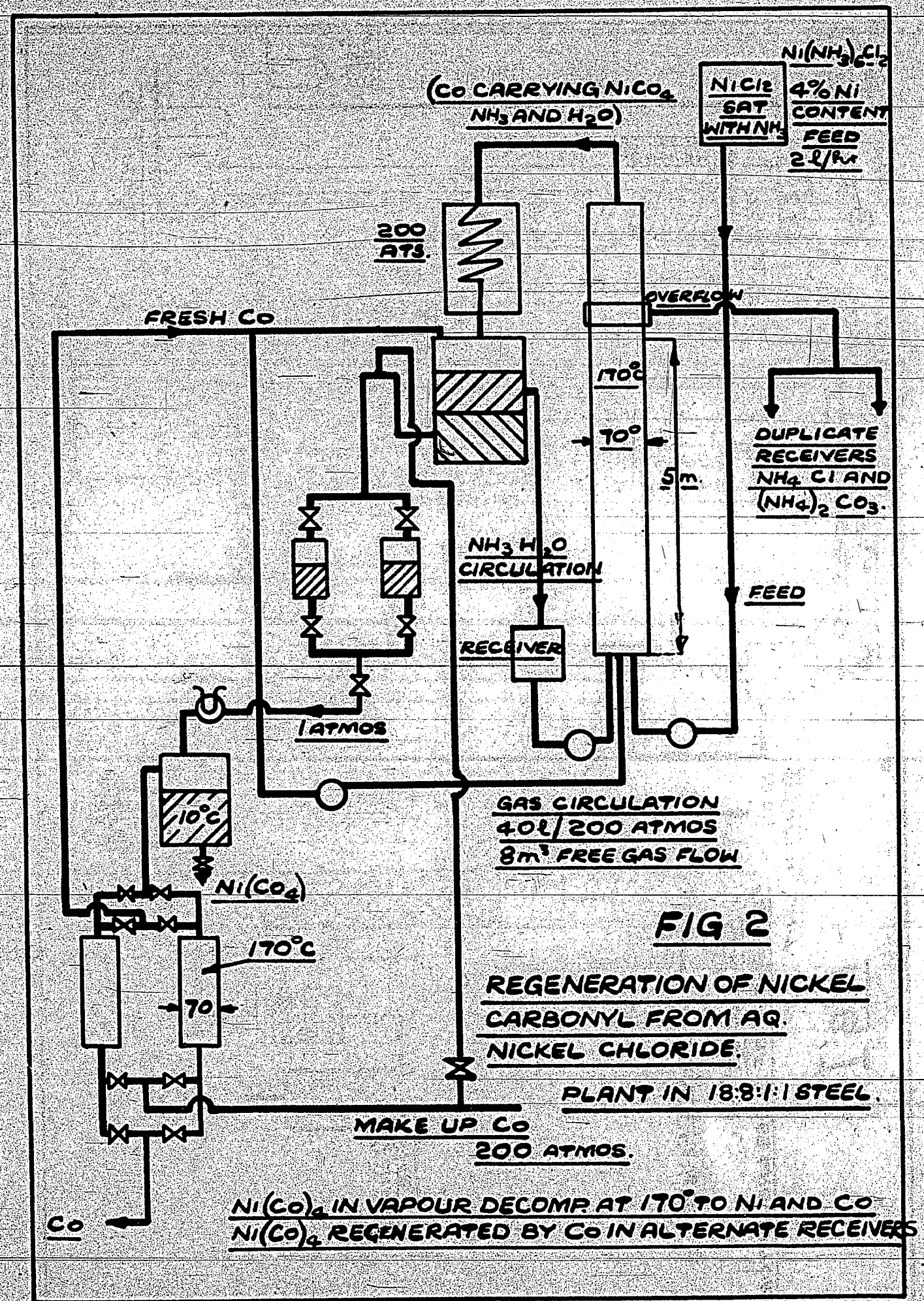
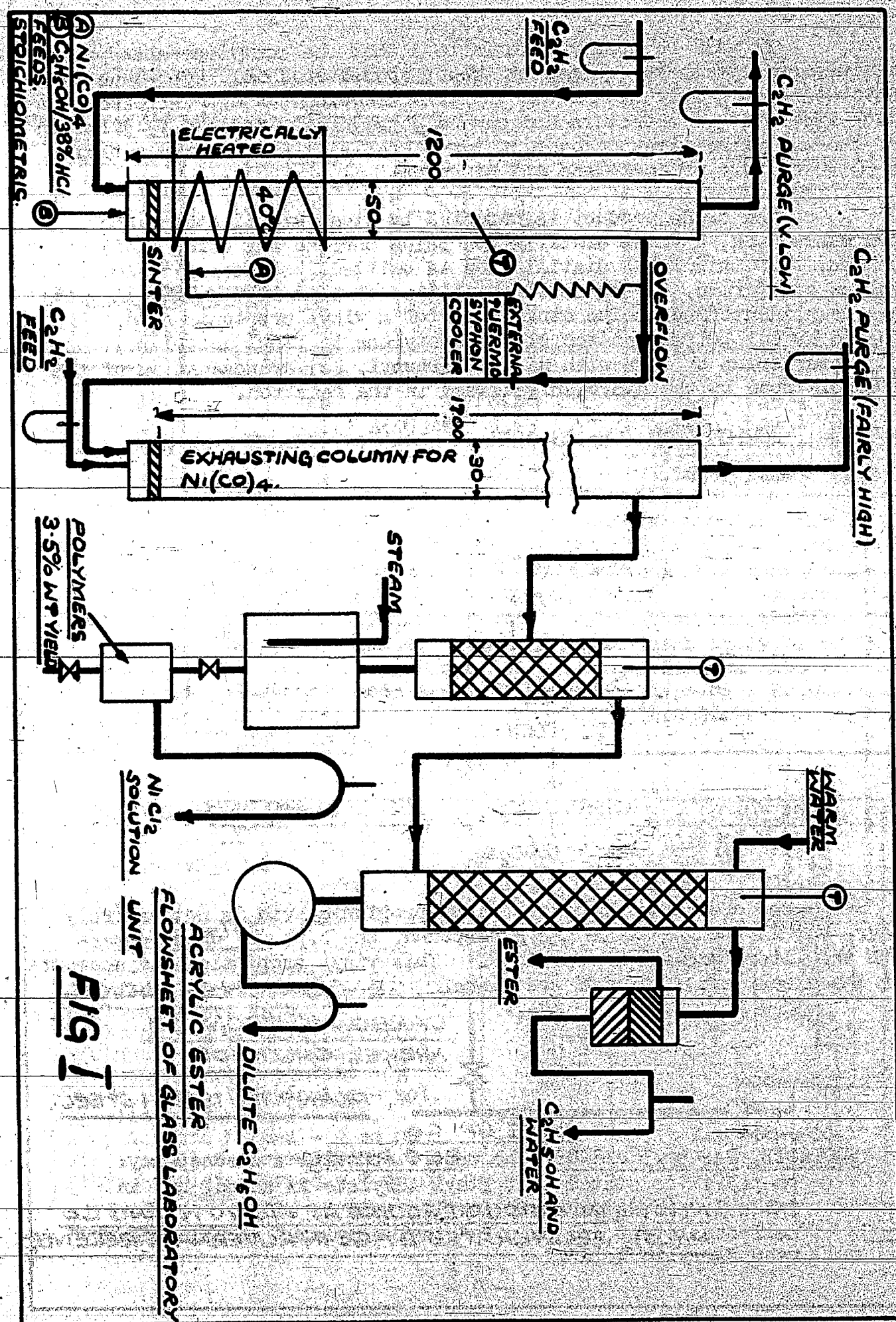
The crude product is purified by steam distillation, and hydroselction, a final distillation being carried out using a Widmer column. If this final distillation is omitted, polymers with low k values are formed, due to small quantities of impurity. This impurity has been identified as the ethyl ester of a vinyl propionic acid, formed by (a) polymerisation of the acetylene to vinyl acetylene, (b) interaction of this with nickel carbonyl, (c) hydrogenation of the a double bond by the hydrogen released in the reaction.



The constitution of the vinyl propionic acid is not entirely clear, but is thought to be the β compound, the b.pt. of which agrees with the literature value of 142.5°. This vinyl propionic acid accounts for the excess absorption of acetylene.

FOOTNOTE, See pp.3 and 8: The report contains a discrepancy: on p.3 it is stated that Ethyl Acrylate is added, but in Table 3, p.8, apparently Acrylic Acid is added. Table 3 is self-consistent and on p.3 "ethyl acrylate" should probably read "Acrylic Acid".







FINAL REPORT No. 356  
ITEM No. 22.

*Copy 1*

CHARACTERISATION OF BUTADIENE  
CATALYSTS BY X-RAY  
AND CHEMICAL ANALYSIS

*Dartshore, J. F. G.  
and  
Rose, J. D.*

"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."

BRITISH INTELLIGENCE  
SUB-COMMITTEE





CHARACTERISATION OF BUTADIENE CATALYSTS

BY X-RAY AND CHEMICAL ANALYSIS.

Reported by:-

J.F.C.Gartshore M.O.S.

J.D.Rose M.O.S.

BIOS Target No. C22/82(a)

(1946)

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

32, BRYANSTON SQUARE, W.1.

8p.



TABLE OF CONTENTS.

|  | <u>Page</u> |
|--|-------------|
| <u>Summary</u> .. .. .   | 1           |
| <u>Outline of manufacturing process for the Butadiene catalyst, as made by I.G.Ludwigshafen</u> .. .. .                                    | 1           |
| From an interrogation of Dr. Wimmer, Ludwigshafen.   |             |
| <u>Research work on characterisation of Butadiene catalysts for dehydration of 1:4 and 1:3 butylene glycol and tetrahydrofuran</u> .. .. . | 2           |
| <u>The Russian catalyst for the Lebedev process</u> .. .. .  | 5           |
| From an interrogation of Dr. von Susich, Ludwigshafen.   |             |

Personnel of team.

|                  |                           |
|------------------|---------------------------|
| *J.D.Rose        | M.O.S.                    |
| C.J.S.Appleyard  | "                         |
| *J.W.Fisher      | "                         |
| *J.F.C.Gartshore | "                         |
| P.W.Blalock      | Can. Dept. Reconstruction |
| A.H.Andersen     | " " "                     |

\* Only those marked thus took part in the interrogation.

Summary.

Dr. von Susich has been engaged upon the characterisation of butadiene catalysts for some five years, and claims to have made a correlation between the performance of catalysts on the plant and their constitution as shown by combined chemical analysis and X-ray spectrography. It is possible by this method to predict the remaining life and the yield which would be obtained from any given sample of catalyst.

A recommendation has been made in the Brief Investigation Report that this claim should be tested fully by experts in X-ray methods and butadiene processes.

Outline of manufacturing process.

Information from Dr. Wimmer, Ludwigshafen.

The catalyst used to produce butadiene from tetrahydrofuran in the Ludwigshafen Buna plant is made thus. Coke granules, roughly spherical in shape, are charged into a rotating trammel furnace heated directly by town's gas to 100-120°C. The impregnating solution is sprayed on at the ratio of 80 litres solution per 100 litres of coke (55 kg. coke approx.): the 80 litres of solution contains:-

|                             |        |
|-----------------------------|--------|
| sodium dihydrogen phosphate | 37 kg. |
| phosphoric acid             | 6.6 "  |
| butylamine                  | 3.2 "  |

The water in the solution partially evaporates from the impregnated granules during this operation, which takes from 40-50 minutes. The impregnated granules are loaded into trays 1 m. x 500 mm. x about 50 mm. deep, and the trays are stacked on trucks and sent through a brick tunnel stove heated by recirculated flue gases. The tunnel has four equal zones, controlled at 80-90°C. (entry), 130°, 180°, and 250°C. (exit), and the trucks pass through at a uniform speed, taking 72-80 hours for the whole passage. The catalyst is cooled, screened from fines, and sent to the butadiene plant.



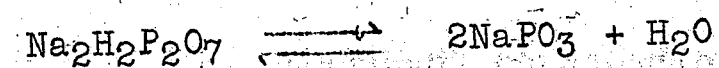
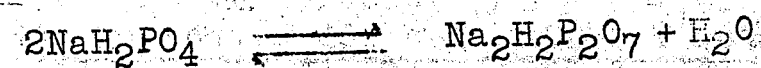
Spent catalyst returned from the plant is leached with water to recover the phosphates.

Research work on characterisation.

Investigation of the catalysts used for producing butadiene from 1:3 and 1:4 butanediol and from tetrahydrofuran has been Dr. von Susich's main interest for some 5 years; and up till the time of this interrogation he had not been questioned upon it. The authors do not feel themselves competent to set a true value upon his work.

The catalysts used by I.G. consist of coke granules (graphite was used at first, but was too expensive) coated with an equal weight of sodium phosphate: free phosphoric acid must also be present.

The constitution of sodium phosphate in presence of about 10% of free phosphoric acid, and of water vapour in concentrations occurring during the formation of butadiene by dehydration of any of the starting materials, at about 280°C., depends on the two equilibria:-



"NaPO<sub>3</sub>" can exist in four forms (cf. work of Pascal 1920-1935, and Bouille 1938):-

A - (NaPO<sub>3</sub>)<sub>3</sub> sodium metaphosphate

B - once known as "Maddrell's salt"  
(Liebig's Annalen 1848)

C - (NaPO<sub>3</sub>)<sub>6</sub> stable above 600°C.

D - now known as Maddrell's salt.

Maddrell's salt is insoluble in water, and

colloidal in particle size; it is present to a large extent in any of the butadiene phosphate catalysts, since it is stable at around 280°C.

There are three butadiene processes operated by I.G., each with a phosphate-coke catalyst manufactured in a different way. The general characterisation is:-

| <u>Works</u> | <u>Starting material</u> | <u>Principal component of catalyst</u>                       |
|--------------|--------------------------|--|
| Schkopau     | 1:3 butylono glycol      | Na <sub>2</sub> H <sub>2</sub> P <sub>2</sub> O <sub>7</sub> |
| Ludwigshafen | 1:4 butanediol           | Maddrell's salt  |
| Huls         | ?                        | Trimetaphosphate + free phosphoric acid.                     |

Specimens of sodium hydrogen pyrophosphate, Maddrell's salt and sodium trimetaphosphate, all free from phosphoric acid had been prepared, and all had been shown in the laboratory to be inactive as catalysts for butadiene. Works experience had shown that there was a fairly well-defined optimum concentration of free phosphoric acid at 12%.

A correlation had been made between performance of catalysts on the plant and their constitution, as shown by combined X-ray spectrography and chemical analysis. The outcome of the work was that it was possible to characterise a butadiene catalyst in the laboratory in about 1 day, whereas a pilot plant test - the only previous reliable method - took about a month. It was possible to predict the remaining life and the yield which would be obtained from any given sample of catalyst.

The causes of catalyst deterioration were:-

1. Disappearance of free phosphoric acid, by vaporisation along with the water vapour in the gas stream.



This is believed to be the most important single factor.

In the catalyst as made at Ludwigshafen a small amount of butylamine is incorporated, enough to react with 2/3 the free phosphoric acid charged. The intention of this had been to form butylamine phosphate which would act as a reservoir for phosphoric acid. But in fact the heating at 280°C. decomposed the butylamine phosphate and cracked the butylamine, giving a deposit of carbon black within the sodium phosphate layer. This accounts for the black colour of the catalyst. The carbon black is now believed to act as an additional spreader or carrier for the phosphoric acid. All but 5% of the butylamine is thus cracked, and the remainder distils away from the catalyst during the first few days operation.

2. Accumulation of resin upon the surface of the granules. This is not as important as phosphoric acid concentration in its effect upon catalyst activity, but as the resin tends to bind the granules together into blocks, it becomes very important when the catalyst has to be dropped from the converters.

3. Increase of crystal size. All the crystal sizes of Maddrell's salt are sub-microscopic, and the growth of crystals is demonstrated only by the splitting up of the lines of a Debye-Scherrer X-ray diagram into the dotted lines typical of a Laue diagram. (The components of the crystal mixture are identified by comparing the composite spectrograph for the catalyst, before and after extracting with water, with the spectrographs for the support and for the individual pure hypothetical components.) The picture of the catalyst is a coke core carrying an outside layer of optimum concentration -

|   |   |     |
|---|---|-----|
| Maddrell's salt                             | - | 70% |
| $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$ | - | 30% |
| $(\text{Na PO}_3)_3$                        | - | Nil |

whose fine crystals act as a porous support for a film of free phosphoric acid. Large crystals provide a smaller extending surface, and also allow phosphoric acid to be carried away more easily.

Incorporation of protective colloids to delay crystallisation, and additives such as calcium phosphate, had been tried without success. Presence of iron in the catalyst was definitely harmful; indeed a new iron catalyst tube would not give results until the inner surface had become coated with a layer of iron phosphate.

The upper layers of catalyst, where the feed entered, always deteriorate most rapidly. This had been met at Huls by using two alternate guard converters containing phosphate catalyst, which were changed frequently, before the main bed.

Plant results had been correlated against laboratory X-ray and chemical analysis from January, 1944, to about July, 1944. The correlation between the predicted and the actual performances was good (in the authors' view, only reasonably so), allowing for the fact that frequent plant shut-downs - air-raids - had caused the catalysts to deteriorate prematurely.

#### The "Russian" catalyst.

A Russian synthetic rubber plant, operating the Lobodev process, at Jphremov had been in German hands for about 3 weeks in 1942, and a sample of the catalyst had been removed and analysed. The plant consisted of an alcohol distillery, the rubber plant (butadiene synthesis and polymerisation), and a brick works which used the off-gases from the other plants as fuel. The design capacity was 10,000 tons/yr., and the actual capacity was 30,000; this was largely due to a 65% yield being achieved instead of the 30% expected. The butadiene converters each hold 12 tons of catalyst.



The catalyst was in the form of pellets 3-5 mm. x 2 mm. diameter, and analysed as:-

|                                |        |                  |        |
|--------------------------------|--------|------------------|--------|
| MgO                            | 40-50% | SiO <sub>2</sub> | 10-12% |
| Al <sub>2</sub> O <sub>3</sub> | 2-3    | CO <sub>2</sub>  | 3-10   |
| Fe <sub>2</sub> O <sub>3</sub> | tr.    | SO <sub>3</sub>  | tr.    |
| TiO <sub>2</sub>               | tr.    | water            | 9-12   |
| ZnO                            | tr.    | ignition loss    | 10-20  |

By X-ray analysis before and after leaching with dilute acids, and similar means, the catalyst was shown to consist principally of Mg(OH)<sub>2</sub> in a very fine crystalline form, together with free silica as quartz, calcium carbonate, and kaolin (Al<sub>2</sub>O<sub>3</sub>.2SiO<sub>2</sub>.2H<sub>2</sub>O).

In German eyes the advantages of the Lebedev process over their own routes from acetylene were:-

- (a) It was a single-stage process from ethanol to butadiene, and the plasticisers could be made from the by-products instead of requiring styrene as a subsidiary manufacture;
- (b) The space-time yield of the catalyst was high;
- (c) The catalyst life was 2-3 years;
- (d) There was no deposition of resin on the catalyst.

49182/1350/18.3.46/ P.S.C./27.59.2



Copy 1

FINAL REPORT No. 216

ITEM Nos. 22, 30

- (1) THE TECHNICAL HIGH SCHOOL, DARMSTADT
- (2) C. F. BOEHRINGER UND SÖHNE, MANNHEIM-WALDHOF
- (3) Dr. FREUDENBERG, HEIDELBERG UNIVERSITY
- (4) Dr. WOLMAN, WOOD PRESERVATION SPECIALIST, BAD KESSINGEN
- (5) IMBERT GAS PRODUCER PLANT, COLOGNE

*Draves, C.*

"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."

BRITISH INTELLIGENCE OBJECTIVES  
SUB-COMMITTEE

LONDON: H.M. STATIONERY OFFICE

TTC L.F. & L. S-2.



REPORT ON VISITS TO

- (1) The Technical High School, Darmstadt 22/257
- (2) C.F. Boehringer and Söhne, Mannheim-Waldhof C22/1161
- (3) Dr. Freudenberg, Heidelberg University C22/1148
- (4) Dr. Wolman, Wood preservation specialist, C22/2350  
Bad Kissingen
- (5) Imbert Gas Producer Plant, Cologne C30282

Reported by

C. Greaves - Forest Products Laboratories,  
Ottawa, Canada.

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE  
32 Bryanston Square, W.1



TABLE OF CONTENTS

| <u>Subject</u>  | <u>Page No</u> |
|---|----------------|
| 1. The Technical High School, Darmstadt.....            | 1 22/257       |
| (1) Lignin.....   | 2              |
| (ii) Poplar.....  | 2              |
| 2. C.F. Boehringer and Söhne, Mannheim-Waldhof..        | 2 022/1461     |
| 3. Dr. Freudenberg of Heidelberg University.....        | 3 022/1448     |
| 4. Dr. Wolman, on Wood Preservation, Bad Kessingen..... | 5 022/2350     |
| (1) Introduction.....                                   | 5              |
| (ii) Wood Preservation during the War.....              | 6              |
| (iii) Use of Old Ties.....                              | 6              |
| 5. Imbert Gas-Producer Plant, Cologne.....              | 6 030/282      |
| (1) Introduction.....                                   | 6              |
| (ii) Types of Gas-Producers Manufactured...             | 7              |
| (iii) Details of Gas-Producers.....                     | 7              |
| (iv) Wood Used.....                                     | 7              |
| (v) Types of Engines using Producer-Gas...              | 8              |
| Imbert Gas-Producer (Sketch).....                       | 8              |
| (vi) Power Developed, Fuel Cost, Weight....             | 9              |
| (vii) General Remarks.....                              | 9              |

Personnel of Team

C. Greaves - Forest Products Laboratories

1. Technical High School, Darmstadt

Darmstadt was visited on 5th and 9th Oct, 1945. The chief object of this visit was to interview Dr. Jayme, director of the Institute Cellulose Chemistry. On arrival on Oct 5th, it was found that Dr. Jayme was absent so a visit was made to the Institute building in which he and his students worked. The building had been considerably damaged by bombs but is still for the most part usable. No work is being carried out at present but it is hoped some work will be started in the near future.

An interview was had with Dr. Brecht, who was in charge of the design and testing of pulp and paper machinery. The building in which the work was carried out, known as the Institute for Pulp and Paper Machinery, was totally destroyed and all the equipment contained therein. As other investigators were studying Pulp and Paper processes Dr. Brecht was not questioned on these matters. However, worthy of mention perhaps are his remarks on mechanical barkers for pulp logs.

The type of barker in general use in Germany is the knife barker and the loss of wood with this type is from 8 to 10%. Dr. Brecht had carried out tests on a new type of hydraulic barker, known as the Schongau, developed within the last 3 years. This machine consisted of a stationary cylinder, 1 m long by 0.8 m in diameter. It is half filled with logs (of 1 m length) which are sprayed from longitudinally placed nozzles with water at 400C and 10 atm pressure. The water strikes the logs tangentially causing them to rotate and the friction forces developed remove the bark in 35 seconds. Before barking the logs are placed in water at 400C for 4 hours. The loss of wood is about 1%. These barkers were in use at Schongau and Augsburg. They were manufactured at Heindenheim by J.M. Voith.

Dr. Brecht exhibited a sample of wallboard made from 50% flax straw and 50% wood pulp. This was not of equal quality to wallboard made from 100% wood fibres, but during the war all possible attempts were made to save valuable raw materials. He had also experimented on the manufacture of wallboard from potato stems and wood pulp, but this was of very poor quality.

Dr. Jayme was interviewed on the morning of Oct 9th. He reported that an outstanding development in Germany during the past few years was the almost universal utilization



of the sugars in waste liquor at all sulphite mills to produce alcohol or yeast. Before the war the main species used for sulphite pulp was spruce and the sugars contained in the waste liquor are composed mainly of hexoses, about 70%, the remainder being pentoses. The hexoses can readily be fermented to ethyl alcohol. During the war the demand for pulp for staple fibre could not be met by the available supplies of spruce, so the German pulp mills turned to beech. However, the waste liquor from beech contains very small amounts of hexoses, the sugars being mainly pentoses. It was found that "torula utilis" strain of yeast or "monilia candida" will grow on pentoses, hexoses, acetic acid and aldehydes, everything but lignin. The yield of yeast is about 50% of the sugars and the yeast contains about 50% albumins. Dr. Jayme advised that complete information on this subject could be obtained by visiting a plant at Kostheim or Mannheim.

Dr. Jayme reported the following points which seemed of interest:-

(i) Lignin

No satisfactory utilization of lignin has been accomplished. Experimental work indicates that a use for some lignin may result from the fact that iron can be removed from water by base interchange using lignin. No success has been achieved by attempts to make a suitable plastic material from lignin. All plastics developed from lignin so far are poor in quality as regards waterproofness.

(ii) Poplar

Dr. Jayme is very interested in the growing of hybrid poplars. A hybrid poplar has been developed which grows to a diameter of 8 inches in 10 years. Stands of this hybrid have been planted and Dr. Jayme was absent inspecting these poplars on Oct 5th. It is expected that these poplars can be used to supply pulp, veneers and match sticks.

2. C.F. Boehringer and Söhne, Mannheim-Waldhof

The plant was visited on the afternoon of Oct 16th, 1945. Persons interviewed were Dr. Sturm, Manager & Vice-President, and Dr. Stein, chief chemist. The writer was interested in the production of vanillin from waste sulphite liquor.

It was found that this firm had produced no vanillin from this source. Dr. Stein reported that it was proposed to form a new company to be known as Ligrowa G.m.b.H. which would be owned by the three firms, I.G. Farbenindustrie, A.G., C.F. Boehringer & Söhne and F. Hoffman - La Roche & Co. It was proposed that this new company would use 10% of the waste sulphite liquor, of the Zellstoffabrik Waldhof Company, after the sugars had been fermented to alcohol and the alcohol removed. Hence only the waste liquor from spruce pulp would be used. No details of the proposed process for the production of vanillin from waste sulphite liquor were available.

3. Dr. Freudenberg of Heidelberg University

Dr. K. Freudenberg was visited at this office on Oct 20th. He is director of the chemical institute of the University which gives courses in general chemistry, organic and inorganic, to Medical and Arts Students. Dr. Freudenberg, when not engaged in his duties as teacher, has carried out chemical research for many years, first on tannins and sugars and for the past several years on lignin.

Up to the present he sees no use for large quantities of lignin. Present solution of the problem of finding uses for lignin seems to be the use of small quantities in several different fields. He considered that a promising field of utilization seems to be the treatment of clay soils with lignin to render the soil more friable and suggested that the influence of lignin on soils was worthy of intensive study.

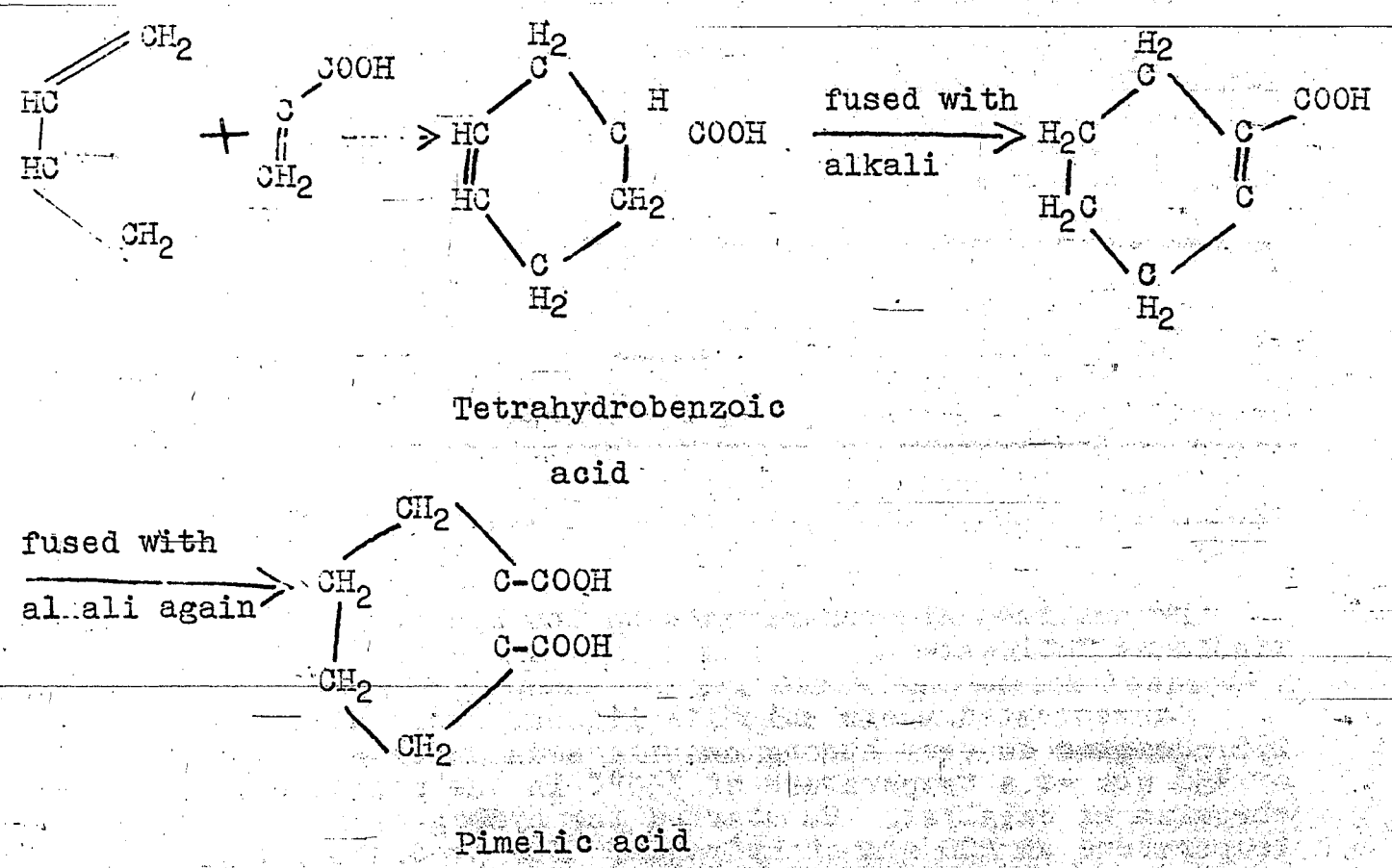
He outlines his experiments on the hydrogenation of lignin as follows:-

Concentrated waste sulphite liquor, freed of CaO was hydrogenated in very strong caustic soda under a pressure of 450 atm at a temperature of 350°C in the presence of chromium as catalyst. He started the hydrogenation at a low temperature at 100 atm of hydrogen. The products obtained were alcohols of the cyclohexylpropanol class, the same as obtained by Homer Atkins in America. About 25% of these alcohols are of low enough molecular weights to be distilled. The residue consists of alcohols of high molecular weights which cannot be distilled; they are oily and behave like



resins. They might be used as plasticizers or softeners for artificial resins. The sulphur in the waste liquid is re-obtained as NaSH in the water solution. If ethanol is added in small amounts during the hydrogenation a better product is obtained. Perhaps the good effect of the ethanol is due to the fact that when an alkaline solution of alcohol is heated under pressure it gives off hydrogen and forms acetates. The hydrogen probably assists in the hydrogenation.

Dr. Freudenberg mentioned that an interesting process had been recently worked out, by Pistor and Plieninger at the I.G. Farbenindustrie at Ludwigshaven, for producing pimelic acid from butadiene. He outlined the method as follows:-



Pimelic acid could be used for the production of nylon instead of adipic acid, and makes a superior quality of nylon.

4. Dr. Wolman, on Wood Preservation, Bad Kessingen

(1) Introduction

Attempts at locating targets on wood preservation were not very successful. The Avenarius wood preserving company at Stuttgart was completely destroyed by bombs and no trace of its present whereabouts was discovered. The Technical High School at Stuttgart was also very much damaged but the office of Prof. Egner, who is an authority on fire proofing treatments for wood, was located only to find that Prof. Egner was sick and not likely to be available for several days. Dr. Baseler of the Reichbahndirection is now located at Pasing, a suburb of Munich; he is one of the foremost German authorities on wood preservation, but unfortunately he was absent when the writer called at his office. A special trip was made to Bad Kessingen on Oct 31st 1945 to interview Dr. Wolman, who is also a well-known German authority on wood preservation and is in close contact with Dr. Baseler.

Dr. Wolman stated that the wood preservation situation in Germany before the war was as follows:-

The principal wood preservatives were creosote and Wolman Salts. The only wood treated by the Osmose process was poles, never ties, and only a very small percentage of poles. No zinc chloride had been used for 40 years because of its action on iron.

Ties - Creosote was used to treat 75% of ties and 25% were treated with Wolman salts. The absorption of creosote is about 63 kilograms per cbm using the Rueping process. The absorption of Wolman salts is 200 kg of 2% solution per cbm. 25 to 35% of the ties used were beech and the remainder pine. Oak was often used for cross-track ties.

Poles - About 50% of poles were treated with creosote and 50% with Wolman salts. Only pressure treatments were used for creosote. Poles were treated with Wolman salts either by the Boucherie Process or the open tank process, about the same amount being treated by either process. Poles are either of spruce or pine, butt treatments are very seldom applied to poles.

Mining Timbers - About 95% of mining timbers were treated with Wolman salts.



(2) Wood Preservation during the War

Germany was obliged to export Wolman salts in order to obtain foreign currency and creosote was converted to a substitute for diesel oil. Hence the only preservative used in Germany during the war was zinc chloride. Full cell pressure treatments were used, the absorption of zinc chloride being 16 kilos of dry salt per cbm.

Dr. Wolman is of the opinion that creosote will never be used to any appreciable extent in Germany as a wood preservative in future years, because the price of creosote is 0.05 marks per kilo and the diesel oil obtained from creosote sells at 0.18 marks per kilo. He considers that the important wood preservative for Germany will be Wolman salts.

(3) Use of Old Ties

Dr. Wolman reported that Dr. Baseler had carried out some interesting work on the salvage of old ties. From partly rotted ties sound portions are removed and glued together with tego-film, Kaurit or other synthetic water-proof adhesive. The glue is set by the heat generated by passing an electric current through wires embedded in the joint. The join is made as per sketch.



The glued joint can be more easily carried out for ties treated with Wolman salts than for creosoted ties. No commercial use has been made of ties salvaged in this manner.

5. Imbert Gas-Producer Plant, Cologne

(1) Introduction

The plant was visited on Nov 5th 1945. Persons interviewed were Director Dr. Mahringer, Technical Engineer Dr. Krieg and Designing Engineer Mr. Trost. The plant has been completely destroyed. Some of the machinery has been removed to a nearby plant, Schmiddingen in Cologne, where production of gas-producers was just starting. It is expected that 50 generators will be produced in November, 200 in December and from then on 500 per month. Military Government has given permission for the manufacture of gas-producers. The Cologne plant used to produce 4000 machines per month. Imbert Gas genatoren G.m.b.H. is the name of the firm and the firm operates another plant at Attendorn, near

Dortmund, which is not damaged and where 2000 machines were produced per month.

(2) Types of Gas-Producers Manufactured

There are two types of gas-producers manufactured. Type I is for general use, mostly trucks and uses wood as fuel. Type II is for private automobiles and the fuel can be either wood or brown coal briquettes. Several different sizes are made for each type; 27 different sizes are produced. In Germany the power of an engine is measured by the cylinder capacity. Type I is made in different sizes for engines from 1.7 to 18 litres cylinder capacity; Type II for engines of 1 to 4 litres cylinder capacity.

(3) Details of Gas-Producers

The gas-producers are of the down draft class and can best be described by reference to the figure following which is of Type I. Air enters through 5 to 7 tuyeres and passes downwards through the firepot where the combustible gases, CO, H<sub>2</sub> and CH<sub>4</sub>, are produced. These gases then pass upwards between the wood container and the outer shell of the producer, then in succession, through the water scrubber, the gas cooler, the purifier, the gas-air mixer, to the engine cylinders. The firepot and tuyeres are made of 3% chromium steel, the remainder of ordinary steel. The firepot and tuyeres are castings made by other companies and purchased by Imbert; until chromium became scarce during the war they were made of steel containing 30% chromium. Due to the lack of chromium the percentage had to be reduced to 3% and the 3% has been found to be just as efficient as the 30%. There is a shaker for the grate (not shown in the figure). Type II is similar to Type I except that the gas passes through the grate before passing up the annular space between the wood container and the outer shell. Further, Type II has an arrangement whereby the grate can be shaken from the driver's seat. Type II is called PUK, P for "private automobile", U for "under the grate" and K for "grate shaken from driver's seat". Type II was developed during the war.

(4) Wood Used

Dr. Mahringer stated that at present there is used any species of wood, hardwood or softwood, that can be obtained and of any moisture content up to 40 to 50%. The best results are obtained with dry beech. The size of



pieces is roughly 5 x 5 x 7 cubic centimetres. No separation of bark is made. The wood is cut by machines.

(5) Types of Engines using Producer Gas.

In Germany gas-producers are used with gasoline engines or Diesel engines. No special engines built to use producer-gas. The percentage of gasoline engines equipped with gas-producers is about the same as the percentage for Diesel engines but there are many more gasoline engines than Diesel engines. There are many other types of gas-producers besides Imbert but there are many other types of gas-producers besides Imbert but about 95% of all gas-producers in Germany are of Imbert make.

The best compression ratio for producer-gas is from 9 : 1 to 11 : 1. In the case of gasoline engines if the compression ratio is 6 : 1 or less, it is raised by altering the engine head or the piston. In the case of Diesel engines the pump is removed, spark plugs are fitted and the compression ratio is reduced by altering the engine head or by reducing the piston.

(6) Power Developed, Fuel Cost, Weight

A gasoline engine, converted to use producer-gas, develops 70 to 75% of the power obtained from the original engine using gasoline. The corresponding figure for a Diesel engine is 80%.

A ton of gasoline in Germany costs 500 marks, the equivalent in wood costs 280 marks, and the equivalent in lignite briquettes costs 22 marks.

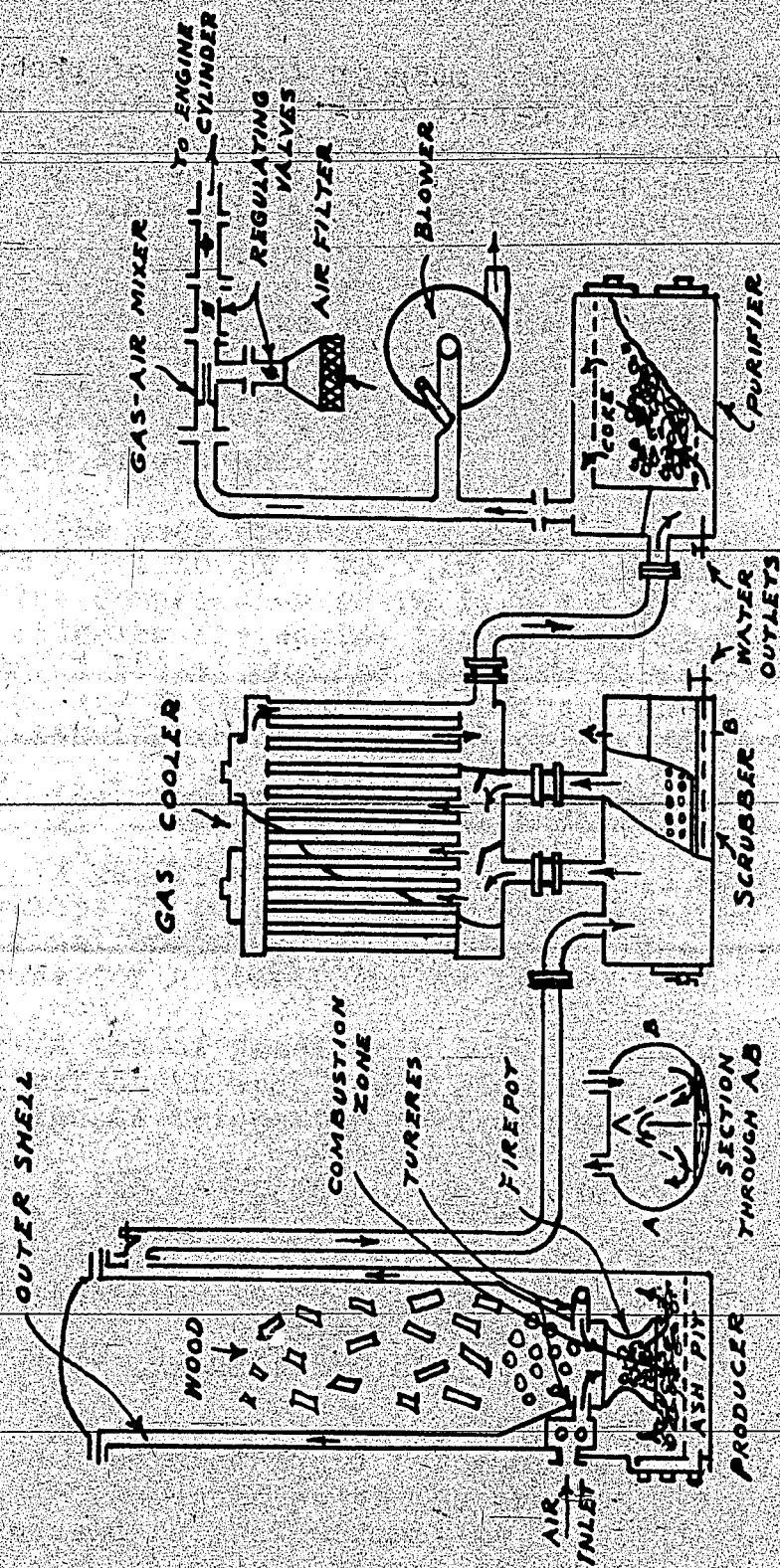
A PUK gas-producer weighs 120 kg. a gas-producer for a large truck weighs 300 kg. On one charge of beech wood, weighing 25 to 30 kg, a PUK gas-producer can travel 60 to 90 kilometres.

(7) General Remarks

Dr. Mahringer gave as his opinion that gasoline driven vehicles could not compete economically with wood-gas driven vehicles at the present prices of these fuels. He considers that Germany is likely to use gas-producers for a considerable time in the future because of the lack of foreign currency and cheapness. He considered

that great improvements can be made in the use of producer-gas as soon as research is directed in this field. No research on gas-producers has been carried out in Germany since the start of the war, the Imbert machine being essentially the same as that used before the war.





IMBERT GAS PRODUCER



*Copy 1*

FINAL REPORT NO. 128  
ITEM NO. 22

# WOOD DISTILLATION PLANT AT BRILON-WALD

*Greaves, C.*

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.

LIBRARY  
of the  
FOREIGN & METRIC  
LIQUID FUELS DIVISION  
Bureau of Mines  
SEP 1945

**BRITISH INTELLIGENCE OBJECTIVES  
SUB-COMMITTEE**

LONDON — H.M. STATIONERY OFFICE



WOOD DISTILLATION PLANT AT BRILON-WAID

REPORTED BY

C. GREAVES

FOREST PRODUCTS LABORATORIES

OTTAWA

CIOS Item 22

**BIOS Target No. C 22/2288**

British Objectives Sub-Committee  
32 Bryanstone Square, London, W.1

9 p. diag.



## TABLE OF CONTENTS

| <u>Subject</u>            | <u>Page</u> |
|---------------------------|-------------|
| Introduction              | 1           |
| Plant Details             | 1           |
| Wood Used                 | 1           |
| Yields                    | 1           |
| Other Plants              | 1           |
| Carbonizing Process       | 2           |
| Production of Alcohol     | 4           |
| Production of Acetic Acid | 5           |
| Charcoal Briquettes       | 5           |
| Activated Charcoal        | 6           |

### Personnel of Team

C. Greaves

- 1 -

## WOOD DISTILLATION PLANT AT BRILON-WALD

### INTRODUCTION

1. The plant was visited on the 6th and 7th of November 1945, and the persons interviewed were plant-director Dr Hans Schürle and Mr Fischer. The latter speaks a little English.

### PLANT DETAILS

2. The capacity of the plant is 4000 solid cbm of wood per month. One solid cubic metre of wood has a volume of 1000 litres and is equal approximately to 1.5 cbm of stacked logs. The plant is undamaged. When working at full capacity it employs 180 to 200 workers, it is now working at 40% of full capacity.

### WOOD USED

3. At least 99% of the wood carbonized is beech and is obtained from districts surrounding the plant up to 100 kilometres. The logs as purchased are 1 metre long and vary in diameter from 4 to 12 cm. After air-seasoning for about one year the moisture content is about 20%. The logs are then sawn to 25 cm lengths and the sawdust used as fuel. The average cost of a solid cbm delivered at the plant is 30 marks. No figure could be given on the value of the products from one solid cbm. The cost of processing one solid cbm varies from 8 to 15 marks. The best quality of beech is used for lumber and only the poor quality is used for carbonizing.

### YIELDS

4. From one solid cbm of beech, which contains 560 kg of dry wood, there is obtained charcoal 180 to 190 kg, acetic acid 30 to 33 kg, crude alcohol 10 to 12 kg, wood tar 50 to 60 kg, and about 100 kg of uncondensable gases.

### OTHER PLANTS

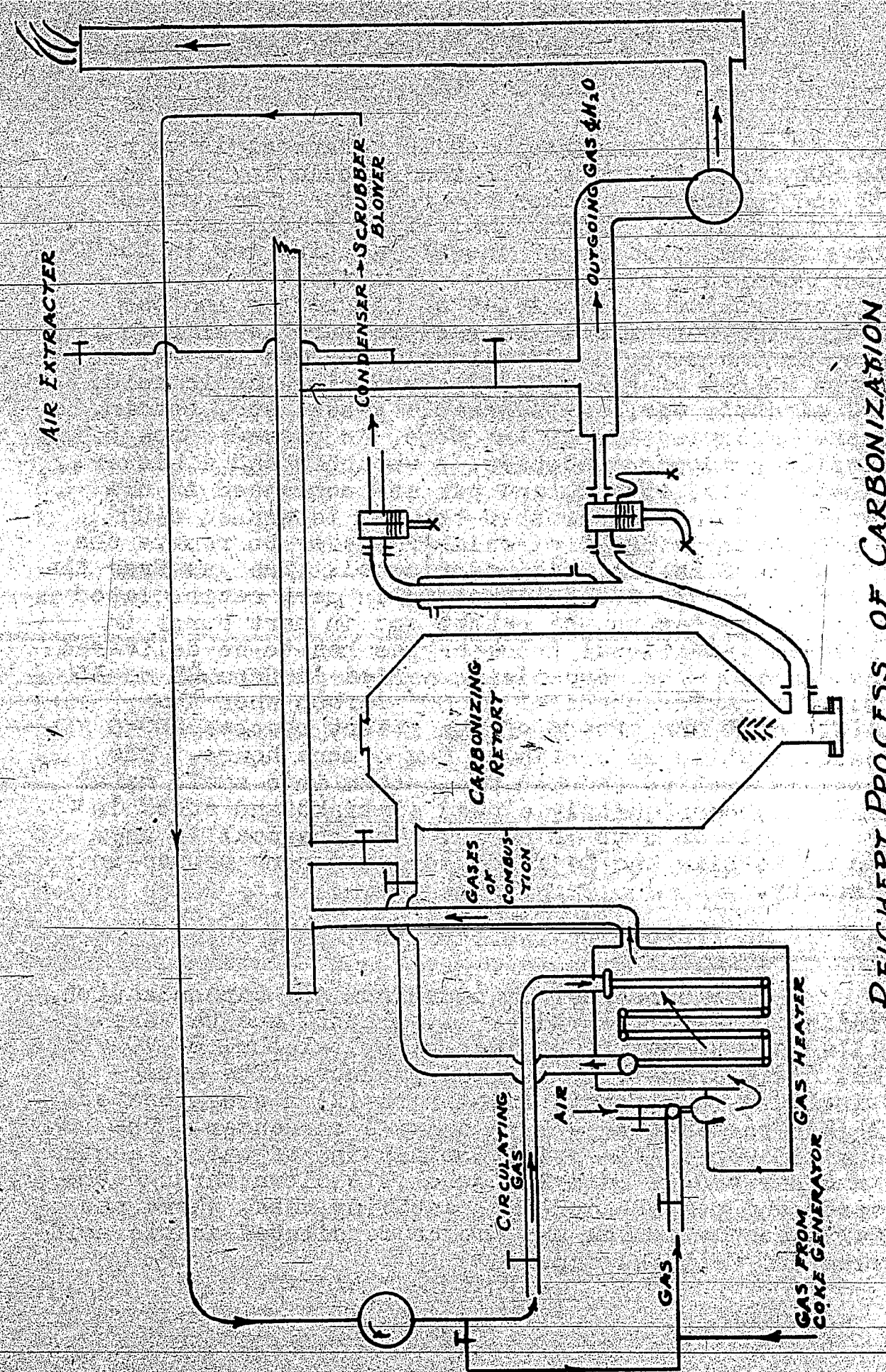
5. The company owning the plant at Brilon-Wald is called Degussa and is the main producer of wood distillation products in Germany, operating 9 plants as follows:-



| Location of plant                     | Capacity in solid cbm per month | Zone of Allied Occupation | % of total capacity by Zones | % of wood obtained in Zones |
|---------------------------------------|---------------------------------|---------------------------|------------------------------|-----------------------------|
| Lorch/Rhein                           | 2000 to 2500                    | U.S.A.                    | 8                            | 34                          |
| Bodenfelde/Weser                      | 6500                            | British                   | 80                           | 52                          |
| Brilon-Wald/Westfalen                 | 4000                            | "                         |                              |                             |
| Züschen/Westfalen                     | 3000                            | "                         |                              |                             |
| Oeventrop District Arnsberg/Westfalen | 3500                            | "                         |                              |                             |
| Bruchhausen/Westfalen                 | 3000                            | "                         |                              |                             |
| Kredenbach/Westfalen                  | 2500                            | "                         |                              |                             |
| Schleiden/Eifel                       | 2000                            | "                         |                              |                             |
| Brucken near Birkenfeld/Nahe          | 3500 to 4000                    | French                    | 12                           | 14                          |

**CARBONIZING PROCESS**

6. The process is known as the Reichert. An attempt is made to briefly describe the process with the assistance of the diagram following. The wood, cut to 25 cm lengths is charged into the carbonizing retort, a large vertical cylinder of an interior volume of 100 cbm. There are 6 of these retorts at Brilon-Wald. The retort holds 40 solid cbm of wood. The incoming wood has a moisture content of about 20% and is first dried by passing through the retort hot gases of combustion at a temperature of 130 to 200°C for 10 to 15 hours; after passing through the retort these gases are discharged to the air. After drying the wood is carbonized with circulating gas which is continuously heated in the heater to a temperature which varies with the kind of charcoal to be proc-





used. If the carbon content of the charcoal is to be 78 to 80%, the temperature of the circulating gas is 300°C and is passed through the retort at the rate of 1500 cbm per hour. If charcoal of 88 to 90% carbon is desired the temperature of the circulating gas is 550°C and the rate 1300 cbm per hour. The rate of flow of gas is controlled at the inlet of the heater and the outlet of the condenser. No gas pressure is allowed to develop in the retort. The circulating gas on emerging from the bottom of the retort, containing acetic acid, methyl alcohol, tar, uncondensable gases, etc, obtained from the carbonization of the wood, is passed through in succession a copper condenser, a scrubber and a blower. The acetic acid, alcohol and tar are condensed in the condenser and in the scrubber the gas is washed with 3000 litres per hour of circulating water to remove the last traces of acetic acid and alcohol. The gas from the scrubber by means of the blower is in part recirculated through the heater to the retort and in part burnt in the heater. Additional producer gas has to be delivered to the heater. The carbonizing period is from 25 to 35 hours. For making charcoal of 80% carbon when the temperature at the bottom of the retort reaches 300°C the carbonization is continued for 5 more hours. The charcoal is emptied through the bottom into small metal coolers which are quickly closed airtight and stood in water to cool for 8 to 10 hours. The charcoal is then graded as to size and is ready for transport or use in the factory.

#### PRODUCTION OF ALCOHOL

7. The condensed liquids from the carbonization, consisting of water, acetic acid, alcohol and tar are distilled and there is obtained methyl alcohol of 70% by weight. This alcohol is a final product. The equipment includes (1) storage containers (2) a steam heated still (3) fractionating column and (4) condenser. The residual liquor in the still, consisting of water, acetic acid and tar and no methyl alcohol, is blown through the condenser and pumped cool into settling vats, where the tar falls to the bottom and the crude diluted acetic acid is drawn off from the top.

#### PRODUCTION OF ACETIC ACID

8. The crude acetic acid solution, obtained after the tar has settled, contains from 7 to 10% acetic acid and some dissolved tar. It is washed in extraction towers with ethyl acetate in a counter-current stream in the proportion of 1 of crude acetic acid solution to 2½ of ethyl acetate. The acetic acid solution enters at the top of the tower and the ethyl acetate at the bottom. The two liquids do not mix but as the acetic acid solution is heavier than ethyl acetate, which has a sp. gr. of 0.9, the acid solution falls through the ethyl acetate and while this is happening the ethyl acetate dissolves the acetic acid and the tar so that the acid solution reaching the bottom of the extracting tower contains only 0.1 to 0.2% of acetic acid and is discarded. The ethyl acetate containing the acetic acid and some tar is drawn off from the top of the extraction tower and the ethyl acetate is separated by distillation from a steam heated still, and returned to the extraction tower.

The residue in the still, consisting of acetic acid tar and impurities, is distilled in a vacuum still with fractionating column and condenser whereby the acetic acid is separated at a concentration of about 95% and is ready for sale.

The equipment used includes (1) storage containers (2) extraction towers (3) steam heated still with fractionating column and condenser (4) vacuum steam heated still with vacuum fractionating column and condenser.

#### CHARCOAL BRIQUETTES

9. Briquettes are made with two different bonding materials, starch and wood tar. Starch briquettes are composed of charcoal fines and starch in the proportion of 4 kg of starch per 100 kg of finished briquettes. The starch solution is made by dissolving 55 kg of starch in 900 litres of water, heating with steam to dissolve the starch. The charcoal is then mixed with the starch solution and fed into a press. The briquettes from the press are stacked on trays and dried in a tunnel drier by hot air at 80°C.



For briquettes made with tar, 100 kg of finished briquettes require 40 kg of thick tar and a little water. This tar is of a quality that yields about 15 kg of coke per 40 kg of tar, so about 85 kg of charcoal fines are required with 40 kg of tar to yield 100 kg of briquettes. After mixing and pressing the tar briquettes are placed on metal trays and carbonized at a temperature of 450°C and cooled for 8 hours before being ready for sale.

ACTIVATED CHARCOAL

10. The plant capacity for making activated charcoal (Act.C.) is 75 tons (metric) per month. During the war considerable gas-mask Act.C. was produced. Ordinarily several different types of Act. C. are produced. The process is briefly as follows:-

Charcoal is ground until not more than 2% is retained on a screen having 6400 holes per sq cm. This fine charcoal is mixed for 20 minutes with wood tar in the proportions of 120 kg to 95 kg of tar. The viscosity of the tar at 60°C is 25 degrees Englen, and the coke residue is 20 to 30%.

When gas masks Act.C. is made there is also added to the mixing machine, besides 120 kg charcoal and 95 kg tar, 8 litres of 50% KOH (Sp gr 1.45), 4 litres of copper sulphate solution (sp.gr.1.2) and 100 grams of copper oxide.

The viscous mass is next pressed at 250 to 350 atm. in high pressure presses into macaroni-shaped pellets, the diameter of the pieces being:-

- 1. For industrial Act.C            4.2 - 4.4 m.m.
- 2. For water-cleaning Act.C    2.5 - 3.0 m.m.
- 3. For gas-mask Act.C            1.4 - 1.6 m.m.

The pellets are carbonized in a revolving drum by hot gas, the pellets entering at one end of the drum and the hot gas at the opposite end. The diameter of drum 1 m., length 10 m, R.P.M. 6, starting temperature 600°C and finishing temperature 250 to 300°C.

After 30 minutes the carbonized product passes through a cooling pipe to storage and from thence to the activation furnace. Length of furnace 15.5 m, inside diameter 1.35m, revolutions per hour 45, temperature at entrance 950°C and at outlet 650°C. Through the activation furnace there is passed per hour 250 cbm gas, 150 cbm of compressed air and 80 kg of steam. The charcoal remains about 28 hours in the furnace. The gas and the charcoal pass through the furnace in the same direction. The Act.C. falls through a cooling pipe into a waste-gutter and is pumped with water into the washing system.

For industrial and water-purifying purposes the Act.C is washed until the alkalinity, calculated as K<sub>2</sub>CO<sub>3</sub>, is less than 0.5%; for gas-mask Act.C. the alkalinity is 2.5%. After washing the Act.C is dried in a rotating drum, 6 m in length and diameter 1 m, by hot gas entering at 250 to 300°C and leaving at about 140°C. The industrial and water-purifying Act. Charcoals are blown into mixing tanks and after screening are ready for sale. The gas-mask Act.C. passes through a rotating spray-drum where it is sprayed with 0.2% silver nitrate solution. Copper sulphate solution is also sprayed on until 1% copper oxide is added - Finally 2% pyridine is added.

Decolourising Act.C. for the sugar industry is made either exactly as in the case of industrial Act.C and then ground to the desired fineness, or fine charcoal is activated directly and then ground.

Act.C. for industrial purposes is used mainly to absorb gasolene in the Fischer-Tropesh process and for the extraction of benzol from coal gas.



Copy 1

FINAL REPORT NO. 197.  
ITEM NO. 30.

Reichsinstitut Fur  
Erdolforschung Technische  
Hochschule, Hannover.

*Gunther, A. E.*

"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."

BRITISH INTELLIGENCE OBJECTIVES  
SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE



**REICHSINSTITUT FÜR ERDOLFORSCHUNG  
TECHNISCHE HOCHSCHULE, HANNOVER**

**Reported by**

**Major A. E. Gunther**

**on behalf of**

**British Ministry of Fuel & Power**

**C. I. O. S. Black List Item 30  
Target No. C 30/306  
Fuels and Lubricants**

**4th September, 1945**

**BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE**

**32, Bryanston Square, W.1.**



REICHSINSTITUT FÜR ERDÖLFORSCHUNG  
Technischen Hochschule, Hannover

TABLE OF CONTENTS

| <u>Subject</u>                            | <u>Page No.</u> |
|---|-----------------|
| Object of Visit                           | 1               |
| Summary                                   | 1               |
| Staff of the Reichsinstitut               | 1               |
| Origins of the Reichsinstitut             | 2               |
| Work of the Reichsinstitut                | 3               |
| Conclusions                               | 4               |
| Appendix. Work in Progress<br>at Institut | 6               |

Personnel of Team

Major A. E. Gunther, M. F. P.

Object of Visit.

To obtain an account of the work and aims of the above Reichsinstitut which was founded in 1943 to provide the background of research which before the war had been available from foreign sources.

Summary.

It was found that fundamental research into problems of oil development was most advanced in geology and less in other sciences for reasons that are given.

While always in favour in principle of fundamental research up to the limit of an industry's means, we do not believe that the continuation of the Reichsinstitut is justified at the present time and under the present conditions. It has been suggested to Control Commission that the building of the Reichsinstitut in Hannover should be taken over by the reconstituted Reichsamt für Bodenforschung and the work of the Institut merged into that body.

Staff of the Institut.

The organisation and staff of the Institut is shown in the attached table.

Reichsinstitut für Erdölforschung.

Direktor: Professor Dr. G. R. S c h u l t z e

| Abteilung<br>Geologie         | Abteilung<br>Bohr- und<br>Förder-<br>technik | Abteilung<br>Chemie               | Abteilung<br>Physik | Abteilung<br>Raffinations-<br>Technik |
|-------------------------------|--|-----------------------------------|---------------------|---------------------------------------|
| Honorar<br>Prof.<br>Dr. Bentz | Honorar<br>Prof.<br>Dr. Deicher              | o. Prof.<br>Dr. G. R.<br>Schultze | Dr. Schmidt         | Dr. Schneider                         |
| a. o. Prof.<br>Dr. Frebald    | Honorar<br>Prof.<br>Dr. Becker               | Dr. Göttner                       |                     | Dr. Moos                              |
| Frl.<br>Dr. Prange            | Dipl. Ing.<br>Lechler                        | Dr. Feichtinger                   |                     | Dipl. Chemiker<br>Grunwald            |
|                               |  | Dr. Zinnecker                     |                     |                                       |
|                               |  | Dr. Haas                          |                     |                                       |



Origins of Reichsinstitut

It has been the fortune of large British, Dutch and American oil groups to have the means to support large scale research. Of the German industry this has not been the case. Until 1934, when the Reich amended the law and stepped in with subsidies, oil had been a back-yard industry run on conservative lines mainly by German capital and often by concerns as a secondary investment to mining, manufacturing or industry. It was not until the Reich programme led to the large productions of 1939 - 1940 with the nation at war and cut off from its normal imports, that the industry became a vital factor in the national set-up.

It is necessary here to mention the sciences which the oil industry draws into its net. The standard of German geology has always been high. The State had encouraged research on a more considerable scale than has been given to our Geological Survey. Thus with the development of the oil industry in the late '30's, there existed a national organisation equipped to direct the geological side. So little was known of underground problems in North Germany that the prospect of deep drilling, of regional geophysical surveys, of obtaining solutions to the great intricacy of salt dome tectonics were inducements to geological investigation and research. The difficulties of the problems and the detail required for their solution were very suited to the German mind; only the most skilled application of known methods could bring results.

The need for the crude oil industry to develop the other sciences, chemistry, physics, metallurgy or refining were not as great (at any rate up to 1940) because developments in the U.S. and elsewhere were available in literature or could be imported in a way that geology could not. There had been difficulties in the way of wholesale modernization on the material side before the war on account of exchange restrictions and during it for obvious reasons. But the entry of foreign companies in the North German industry had contributed a good deal to operational methods, certainly in geophysics, drilling and production equipment and refining.

Because after 1940 Germany was cut off from technical advances in other countries the need was felt of having some centre of research. It was in this way (rather than has been reported by the sweep of Field Marshall Goring's baton) that the Reichsinstitut für Erdölforschung came to be founded. Whether its creation in a period of war, involving the deflection of the activities of a group of scientists into subsidiary channels, was justified is another matter.

Work of Reichsinstitut

There has long been a Technical High School at Hannover. With the development of the oil fields in the Hannover Basin, bringing an increase in the number of geologists required, it was decided to attach to the High School an "Institut für Erdölforschung" (Oil Geology) in Hannover as the most central locality. The Institute was concerned only with the geological sciences.

The "Reichsinstitut für Erdölforschung" was founded in 1943 around the nucleus of the Institute, but it was expanded into several departments to embrace all scientific subjects related to the oil industry. The aim of the "Reichsinstitut" is primarily fundamental research. But apart from this it sets out to advise the industry on all questions concerning oil. Its aim is also educational. It serves to give higher education or research facilities to various branches of the industry, geologists, chemists, physicists, engineers etc. For purposes of teaching it maintains close relations with the Hannover High School. As a Reichsinstitut it draws its funds from public sources, and therefore enjoyed independence.

The Reichsinstitut comprises the following departments and scientific workers:-

| Department                | Workers<br>March 1945 | Planned for<br>post-war |
|---------------------------|-----------------------|-------------------------|
| I Geology                 | 2                     | 2                       |
| II Drilling and Transport | 7                     | 3                       |
| III Chemistry             | 23                    | 5                       |
| IV Physics                | 2                     | 1                       |
| V Refinery                | 1                     | 1                       |

All the above are graduates to whom must be added clerks, laboratory assistants, glass blowers, library staff etc. The reduction in the number of scientific workers planned for the post war is due to finance.

An institut founded but two years ago cannot be expected to have accomplished a large amount of research particularly in the middle of a war; the wonder is, like much else in this German war industry, that so much was allowed at all. A list of the number of subjects on which work was in progress is appended. The following indicates the type of work under way: -



A. Fundamental Research

- a) Geology            Fundamental work on oil-containing rocks
- b) Technique of Drilling and Transport    Development of instruments for measuring constants in underground reservoirs, and for drilling control
- c) Chemistry        Development of new tests on hydrocarbons; new synthesis for lubricating oils; new methods of deasphaltizing; methods of dewaxing in liquid phase; oxidation of liquid hydrocarbons
- d) Physics          Methods and instruments for measurement in the oil industry
- e) Refining         Synthesis of new solvents, dewaxing, deasphaltizing  
Development of fractional distillation columns

B. Examination of refinery problems which cannot be undertaken locally on account of lack of experience or equipment.

C. Instructing and assisting students from Technical High School towards an engineers or doctors degree.

D. Testing mineral and fatty oil for the industry.

The building of the Reichsinstitut received a direct hit from a bomb in one of the final raids. Half of the centre of the building was destroyed, leaving five sixth surprisingly intact. Much of the building can be used as soon as labour and facilities are available. The destroyed part of the building can be made weather-proof at small expense; more extensive repairs would cost some RM 30,000. The replacement of total damage some RM 60,000.

Conclusions

The Reichsinstitut für Erdölforschung finds an essential place in the broad educational scheme set within the German oil industry. At the highest level there is the Reichsamt für Bodenforschung with headquarters in Berlin and with regional offices throughout the country. It is through the Reichsamt that the Government exercised its authority.

At the lowest level there is the Bohrmeister Schule at Celle which aims to train the artizan from workman to foreman in drilling, production or oil mining.

On the intermediate level, and on the level of fundamental research comes the Reichsinstitut für Erdölforschung which undertakes both to give the High School graduate higher education, to direct fundamental research and (by way of augmenting its funds) to undertake routine testing on behalf of commercial interests.

It is upon this tripod of controlled education and research that the petroleum industry relies for its scientific background.



APPENDIX

Work in Progress at Institut

DURCHGEFUHRTE ARBEITEN

Benzinabreistemperatur

Gesättigte Kohlenwasserstoffe

Cyclohexane

Isovalen

Kritischer Druck

Schwelgase

Erdölaraffination

Entparaffinierung

Herstellung von Hochdruck-Schmiermitteln  
unter Benutzung von Ultraschall

Ausarbeitung eines Schneidöles für die  
Herstellung von Spezialwerkzeugen

Herstellung eines Schmierfettes unschädlich  
für reflexmindernde Schichten auf optischen Gläsern

Gewinnung der C 7 - Fraktion aus Benzinen durch  
Feinfraktionierung

Rollenmeissel

Rotary-Ketten

Spezialzemente

Thixotropie

Lumineszenzanalyse

Olmessung

Lagerstättenkonstanten

Elektrisches Heizelement

Emulsionsschmierung von Dampfmaschinen

Flüssigkeiten mit geringer Temperaturabschlägigkeit der  
Viskosität

Heizöl Stockpunkterniedrigung

Aufarbeitung Schmieröl

Bohrturbine

Schwefelentfernung

Druckflüssigkeit

Emulsion für Verbrennungsmotore

Rohöl-Diesel

Hochdruckautoklav

Orientierungsgerät

Perforation

Olschiefer

Gerät zur drahtlosen Fernübertragung von Messergebnissen  
aus Sonden

Versuche zur einfachen Aufarbeitung von Rohölen