

FIG. III

LIQUID PHASE FLOWSHEET - 1943
ALL FIGURES IN TONNES.

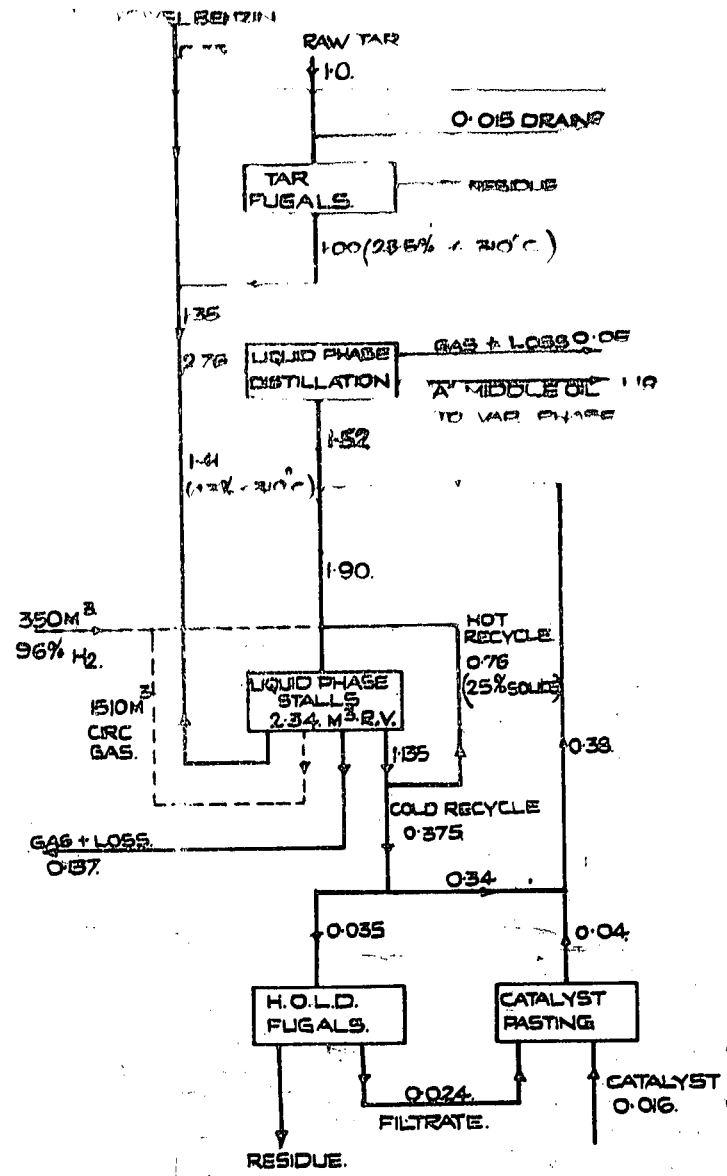


FIG. IV

VAPOUR PHASE HYDROGENATION - 1943
ALL FIGURES IN TONNES.

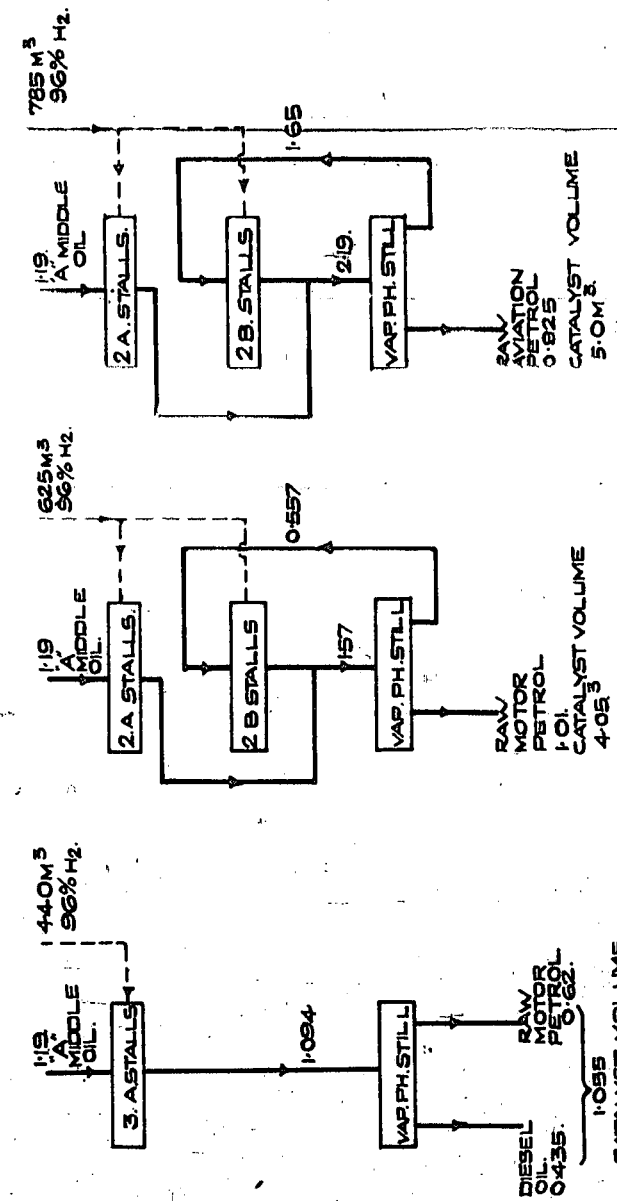


FIGURE I

ROUGH MAIN SITE PLAN
BRABAG - WERK BÖHLEN

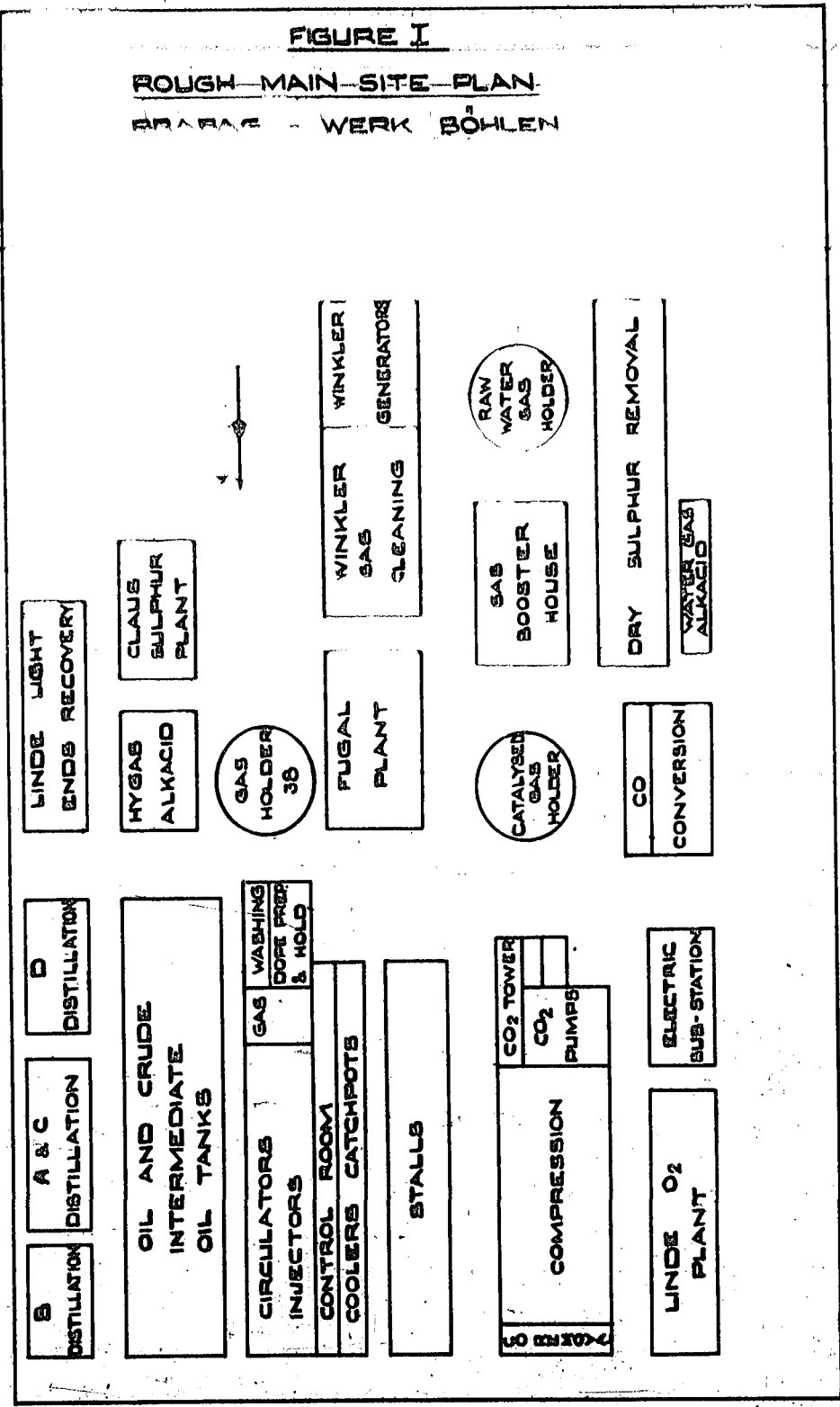
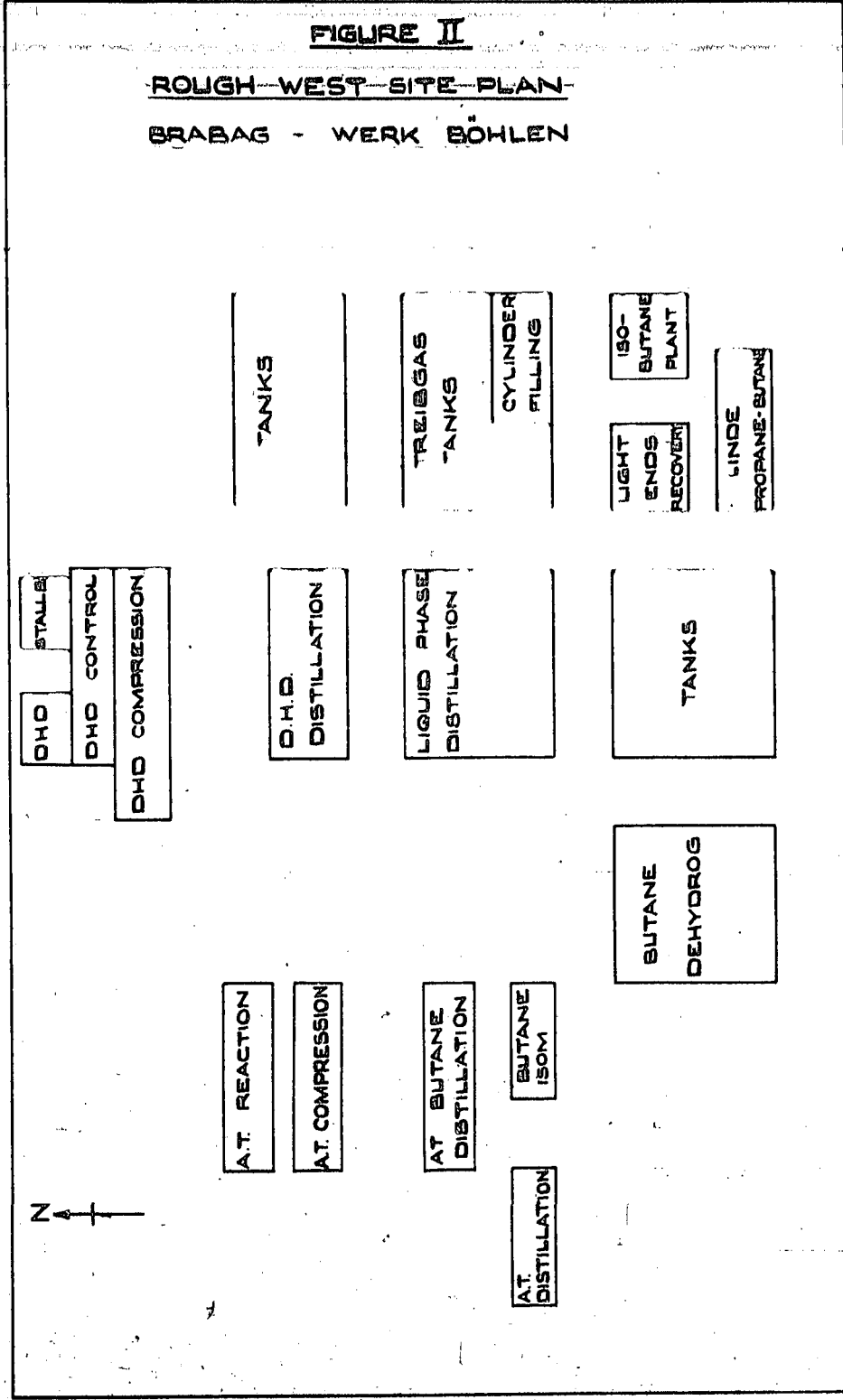


FIGURE II

ROUGH WEST SITE PLAN
BRABAG - WERK BÖHLEN



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| | MECHANICAL SECTION | CIVIL SECTION | TOTAL |
|--|-----------------------|------------------|------------|
| Brought forward | 35 750 000 | 7 500 000 | 43 250 000 |
| Hydrogenation and Sulfur General | 300 000 | 100 000 | 400 000 |
| Claus Plant | 200 000 | 50 000 | 250 000 |
| H ₂ SO ₄ Plant | 300 000 | 100 000 | 400 000 |
| IV. Total by-products. | 800 000 | 250 000 | 1 050 000 |
| Phenol Removal | 600 000 | 200 000 | 800 000 |
| Drain Water Treatment | 300 000 | 800 000 | 1 100 000 |
| V. Total Waste Water Treatment | 900 000 | 1 000 000 | 1 900 000 |
| Pipe Bridges with Pipes | 4 000 000 | 2 000 000 | 6 000 000 |
| Underground Lines | 1 500 000 | 600 000 | 2 100 000 |
| Cooling Towers | 300 000 | 300 000 | 600 000 |
| VI. Total Piping | 5 800 000 | 2 900 000 | 8 700 000 |
| Electric Cable Network | 700 000 | 150 000 | 850 000 |
| Substations | 650 000 | 200 000 | 850 000 |
| Alarm & Signalling Installations | 200 000 | 000 000 | 200 000 |
| VII. Total Electrics. | 1 550 000 | 350 000 | 1 900 000 |
| Railways & Equipment | 300 000 | 700 000 | 1 000 000 |
| Grid | 400 000 | 000 000 | 400 000 |
| Liquefied Gas Cylinders | 1 000 000 | 000 000 | 1 000 000 |
| Roads | 000 000 | 1 200 000 | 1 200 000 |
| Sewage & Drains | 000 000 | 300 000 | 300 000 |
| Workshops & Stores | 3 000 000 | 500 000 | 3 500 000 |
| Offices, Laboratories etc. | 000 000 | 600 000 | 600 000 |
| Land | 000 000 | 300 000 | 300 000 |
| Site Development | 000 000 | 400 000 | 400 000 |
| Fire Services | 200 000 | 100 000 | 300 000 |
| Foam Station | 150 000 | 50 000 | 200 000 |
| Running materials & first catalyst charge | 3 000 000 | 000 000 | 3 000 000 |
| Etc. | 5 000 000 | 000 000 | 5 000 000 |
| VIII. Total - General | 11 050 000 | 4 150 000 | 15 200 000 |
| GRAND TOTAL | 55 850 000 | 16 150 000 | 72 000 000 |

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For estimation purposes on the basis of 1935 costs he used 1000 RM per tonne of steel in the finished plant on the assumption that the plant did not include power generation, steam production, water supplies. For erection purposes he said that one man-hour would be required for every 5.5 RM of the estimated total capital cost.

CONSTRUCTION OF PIPE BRIDGES.

At Böhlen the use of concrete for pipe bridges instead of the normal steel was noticed. A photograph of one of these was taken and copies are attached. At other factories signs had been seen of the substitution of brick columns for steel but the spans themselves had always been made of steel. On the new West site the whole of the supporting structure was made up of concrete in the form of parabolic arches to which were attached the concrete booms and bearers. The only comment the engineer had to make about them in view of their short experience with them was to the effect that they would not stand up to the effect of bombs. If a bomb fell near by the shock, as one would expect, jolted the concrete and cracked it with the usual result that any bearers which cracked bent and let the pipes on them sag. See Fig. V. for photograph.

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W. 1011 The consumptions of materials were also given
for the plant.

| | Motor Diesel Oil. | Motor Petrol. | Aviation Petrol. |
|--------------------------------|-------------------|----------------------------------|------------------|
| Pressure, Atmospheres. | 0.2 to 0.65 | Depending mainly on the weather. | |
| Steam, 1.5 Ats pressure, Tonne | 1.8 to 3.7 | Depending on the outside temp. | |
| Fuel Gas, Tpc Gals | 100 to 200 | | |
| Electricity Kwh | 830 | 1125 | 1460 |
| Hydrogen M ³ | 595 | 720 | 840 |
| Fresh Water M ³ | 40 to 60 | Depending on the weather. | |
| Yield - Petrol | 78% | 75% | 68% |

CONSTRUCTION COSTS

Herr Lackner gave information concerning the capital cost of the plant based on 1935 prices and this is given in the following table.

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CAPITAL COSTS FOR NOBLEM PLANT OF BRABAG.

1935 Basis

| SYNTHETIC PLANT | MECHANICAL SECTION | CIVIL SECTION | TOTAL |
|--|--------------------|------------------|-------------------|
| Oxygen Production | 2 800 000 | 600 000 | 3 400 000 |
| 3 Winkler Generators | 2 700 000 | 500 000 | 3 200 000 |
| Alkali | 400 000 | 100 000 | 500 000 |
| Oxide Boxes | 1 000 000 | 300 000 | 1 300 000 |
| CO Conversion | 1 600 000 | 200 000 | 1 800 000 |
| 7 Compressors | 3 000 000 | 400 000 | 3 400 000 |
| CO ₂ Removal | 1 000 000 | 200 000 | 1 200 000 |
| CO Removal | 800 000 | 200 000 | 1 000 000 |
| Gasometer | 700 000 | 100 000 | 800 000 |
| I. Total Hydrogen Production | 14 000 000 | 2 400 000 | 16 400 000 |
| 3 Liquid Phase Stalls | 4 900 000 | 500 000 | 5 400 000 |
| 4 Vapour Phase Stalls | 7 000 000 | 800 000 | 7 800 000 |
| Catalyst Pasting A. H. O. I. D. | 350 000 | 100 000 | 450 000 |
| Circulation | 600 000 | 1 000 000 | 1 600 000 |
| Injection | 700 000 | | 700 000 |
| Gas Washing | 400 000 | 50 000 | 450 000 |
| Rückgas Compressors | 450 000 | 250 000 | 700 000 |
| II. Total Conversion | 14 400 000 | 2 700 000 | 17 100 000 |
| Tar Fugals | 650 000 | 250 000 | 900 000 |
| Intermediate Oil Storage | 550 000 | 200 000 | 750 000 |
| 3 Liquid Phase Stills | 2 200 000 | 600 000 | 2 800 000 |
| 2 Vapour Phase Stills | 1 900 000 | 500 000 | 2 400 000 |
| Stabilisation and Light-ends Recovery. | 250 000 | 50 000 | 300 000 |
| Petrol Wash | 150 000 | 50 000 | 200 000 |
| Propane Butane Separation | 700 000 | 200 000 | 900 000 |
| Filling Station | 100 000 | 200 000 | 300 000 |
| Finished product Storage | 850 000 | 350 000 | 1 200 000 |
| III Total Refinery | 7 350 000 | 2 400 000 | 9 750 000 |
| Carried forward | 35 750 000 | 7 500 000 | 43 250 000 |

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usual convection preheater where the feed to the stall was heated up to reaction temperature and the heat of reaction put in between each of the first five converters.

The injection pumps each had a capacity of 33 M³/hr maximum.

The catalyst to be used was the normal I.G. D.H.D. catalyst except that the last converter was filled in the last half with a refining catalyst to help to hydrogenate the olefines which were made in the other converters.

The reason for the two different sizes of converter was said to be because the reaction per converter with equal sized converters was not equal and they wanted to achieve equal work per converter and thus equalise the load on the various sections of the preheater.

The plant was to be fed on the 100° to 170°C fraction of the petrol made in the main hydrogenation plant. To get this feed they had a single column distillation plant to make the separation.

They also had a plant to distill the stall product to remove the heavy fractions formed in the process.

The aromatic content of the feed to the stall was 15% and there was 4.5 to 50% aromatics in the stall product, with a gas make of 25% in the stall. These figures compare favourable with the make leistung and gas make at Leuna, if the average feed to the Böhlen stalls is 20 M³/hr, which feed rate is necessary for the D.H.D. units to aromatise the whole of the aviation petrol make of the Böhlen plant.

EXTRACTION OF TAR AND H.O.L.D. FUGAL SLUDGE.

Early in the war experiments were made with a small plant to extract the oil from the solid residue they were getting in the tar plant fugals. The plant operated for a period but was destroyed by bombing.

The plant consisted of a number of tanks, pumps, and two filter presses of the usual design.

The fugal residue, containing 50% oil 48% solids and 2% water, was mixed with an equal weight of Schwelbenzin, 130°C F.B.P., and the mixture stirred. 5 tons of this mixture were then heated to 80-90°C and pumped into the filter press. The filling of the press lasted

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about $\frac{3}{4}$ hr. The press was then flushed out with 20-25 Tnes of Schwelbenzin over a period of two hours, the wash oil having been heated to 80-90°C. After this washing the press was steamed with 150 lbs/sq. in. steam for about 30 minutes, the amount of steam being about 3 tonnes.

All the oils from the press were sent to a settling tank where the water was separated off and the oil from the tank sent to the liquid phase distillation plants. The solid residue had 2% oil in it.

They had tried to extract the residues from bituminous coal fugal residue and had achieved some success but the details are only available in the documents.

LABOUR STRENGTH.

From plant records it was noted that the plant had employed 670 staff, which must have included the drawing office staff for the central BRAPAG engineering office, and 2330 workers when the feed to the plant had been 266,000 Tnes/yr and the make of finished products 228,000 Tnes/yr.

They estimated that under peace time conditions with the D.H.D. and A.T. installations in commission they would require 3,000 men.

They had been employing 4,500 men recently because of bomb damage and because of the construction programme. The men had been switched from one class of work to the other according to the need.

PUBLIC COSTS.

The following costs for raw materials were given:-

| | |
|---|-----------------|
| Steam at 1.5 Ats. pressure from A.S.M. Böhlen | 1.20 R/Tne. |
| Grude | 8.20 R/Tne. |
| Water at 4.0 Ats. pressure from A.S.M. Böhlen | 0.05 R/Tne. |
| Electricity from A.S.M. Böhlen | 0.015 R/1000. |
| Tar from A.S.M. Böhlen | 62.50 R/Tne. |
| Tar from other sources | 78 to 95 R/Tne. |

The plant was said to require about 2,000 Tnes/hr of fresh water but not all this was for cooling purposes. They did not want to go in for cooling towers and this quantity of water was the maximum they could draw from the local river without prejudicing the cold water supplies to other plants in the area.

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The make of catalyst was quoted as follows when making Petro
& Diesel Oil.
10-12% " " when making Motor
petrol.
15-18% " " when making
Aviation Petrol.
When making Motor Petrol the make was 50% Propane 50% Butane.
" " Aviation " " " " 42% " 58% "

In 1941 the Liquid Phase Stills took 45% of the fuel gas
consumed, the Liquid Phase Stills 25%, the Vapour Phase Stills 20%
the remainder going to the Claus Sulphur Plant.

TREATMENT OF H₂S.

The H₂S recovered from the plant gases and not recycled to
the stalls is sent to a Claus plant where it is converted into
Sulphur. Some of the Sulphur is converted into Sulphuric Acid
but the process used was not ascertained.

TREATMENT OF WATER.

Certain of the waters draining from the plants, such as
the liquid phase distillation units and the Winkler generators are
washed with Tri-cresyl-phosphate to remove the Phenols. These
Phenols are sold to the I.G. at Leuna.

ISO-BUTANE PLANT.

On the West site there is a small plant for the production
of Iso-Butane for export to Leuna. The plant consists of two
towers, the one separating Iso-Butane from the Normal-Butane and
the other separating propane from the Iso-Butane. The butane
separation tower is filled with Raschig rings and is short in
length. The final product had a purity of 96% Iso-Butane and
they did not consider the unit a very good one for the production
of Iso-Butane.

AT-244 INSTALLATION.

This installation was not complete and appeared to be about
70% finished. The installation comprised a Normal-Butane Dehydro-
genation plant, a Butane Isomerisation Plant, an Alkylation Plant
and the necessary distillation units for these plants. Output
figures for the plants were not obtained for the sections by inter-
rogation as it was felt that sufficient information would be in the
documents. The documents are not at present available.

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The Isomerisation unit was a unit with three reactors and
the usual HCl stripping tower. The catalyst to be used was
Aluminium Chloride in the form of chips.

The Butane dehydrogenation plant was made up of four
dehydrogenation furnaces with two regeneration furnaces. The lower
sleeves at the base of the furnace tubes were not slitted as at
Leuna but had long teeth at the lower end. This arrangement of
slitted or serrated tubes is necessary for the separation of the
catalyst and the reactants.

The Alkylation plant was made up of two six unit
batteries the design being similar to that at Leuna except that
the fresh acid feed eggs were independent for each reactor instead
of a common one for the whole battery. The compressors were
single stage electric driven reciprocating machines.

The gases from the Isomerisation unit were to be distilled
in a 70 tray tower, the Iso-Butane being fed to the Iso-butane
stream going to the Alkylation plant after depropanising. There
were also to be two similar 70 tray towers working on the product
from the Alkylation plant. The Normal Butane from these three
towers was split up, one portion going to the dehydrogenation plant
from whence the products went to the Alkylation plant, and the
remaining Normal-Butane fed to the Isomerisation unit.

The crude Alkylate from the first distillation plant was
to be sent to another unit where the remaining Normal-Butane was
to be stripped out and the resulting product distilled to give the
desired Alkylate for blending into Aviation Petrol.

D.H.D. PLANT.

This installation comprises two stalls with the attendant
control room, compressors and distillation units.

One stall was almost ready for operation and the stills
appeared to be in the same state of readiness. It was the hope
that they would be given the opportunity to run the one unit, but
they were of the opinion that the process would be uneconomic under
peace time conditions.

Each stall consisted of six converters having a total
volume of 36 M³ of catalyst. The arrangement of the converters was
markedly different from the arrangement seen at Leuna in that there
were three converters each with a reaction volume of 5 M³ followed
by two of 7 M³ each and the sixth converter, placed between the two
interchangers, had a reaction volume of 7 M³. The stall had the

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There were also other pumps in the building for the injection of water, wash oil, and for other services.

LIQUID PHASE DISTILLATION PLANT (A, C & UHDE Distillation).

The crude oil from the liquid phase stalls is collected in tanks and mixed with the cleaned tar and the Schwelbenzin. This mixture is then distilled at atmospheric pressure to give an overhead boiling below 310°C and the remainder being taken off at the bottom of the tower to be fed to the liquid phase stalls. The stills, of which there are three, two being on the old site and one on the new West site, are normal fractionating units with gas-fired heaters.

The old stills are fitted with bubble cap trays but the new still is fitted with Raschig rings. They had made the alteration to the packing of the towers because towers packed with earthenware Raschig rings coked up less under operating conditions and can be overloaded more without losing general cutting efficiency, despite the fact that at specified loads the bubble tray towers will cut better.

The new distillation unit has a capacity of 100 Tnes/hr, the old ones having capacities of 75 M²/hr each.

The furnaces of the stills are fitted in the main with Sicromal steel tubing in order to reduce the corrosion of the metal by the tar acids in the feed oil. Some of these tubes have lasted almost ten years.

The old units are fitted with a side stream take off so that the oil overhead is lighter than it would be if all the A middle oil were taken overhead thereby achieving an easier separation of the water from the overhead oil. The new still is not fitted with a side stream take-off but they do not have any trouble from bad water separation, which is not surprising with an oil specific gravity of 0.860.

VAPOUR PHASE DISTILLATION.

The vapour phase distillation units are known as the B and D units, and the various towers are numbered according to their duty.

B.1 is a tower running at 2 Ats pressure with a steam heated reboiler to stabilise the vapour phase crude oil before it is distilled in the B.2 tower.

B.2 is an atmospheric pressure tower cutting the stabilised oil from B.1 into Petrol and Middle Oil. No gas is evolved from the top of this tower.

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B.4 is a tower operating at 8 Ats pressure to remove the propane and butane from Petrol which is being worked up as Aviation Petrol and is only used when the plant is making Aviation Petrol.

D.1 is a tower operating at atmospheric pressure working on wild crude from the vapour phase stalls cutting the oil to wild petrol and middle oil.

D.2 is a tower operating at 12 Ats pressure stabilising the petrol and giving butane overhead.

The B.2 and D.1 towers are heated by means of gas-fired furnaces which are very big for the amount of heat put into them.

GAS DISTRIBUTION AND RECOVERY SYSTEM.

In all hydrogenation plants there is a make of gas in the process and this make is split up into two kinds of gas, the one relatively poor in propane and butane and the other relatively rich in these gases.

At Böhlen certain of the gases are recycled to the stalls as mentioned in the section on Gas Circulation. The lean gases and the rich gases, as they are known, are passed separately through an Alkacid plant. In this plant they are scrubbed with the I.C. DIK liquor to remove H₂S. The regenerator of the plant is built of Aluminium and they have little corrosion on this regenerator. The two liquors from the two absorbers are mixed and are regenerated in a common regenerator.

The lean gas after removal of H₂S is then sent to the factory fuel-gas system.

The rich gas is sent to either a tower known as the B.3 tower working at 20 Ats. pressure to have the pentane and heavier hydrocarbons removed, or another similar tower on the West site for which no number was obtained.

After the removal of the heavy ends from the gas it is sent to the Linde fractionation plant where the Propane and Butane are separated out. There is also a similar plant under construction, about 80% complete, on the West site. The residual gas from the Linde plant is then sent to the fuel gas system.

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stalls but this is normally kept closed. There is a similar normally closed connection on the exit headers of the stall systems. The gas washing plant is connected into the liquid phase system only and is placed on the exit line from the stall so that the gas is washed to remove the nitrogen and hydrocarbons before the make-up gas is added to the system.

The make-up gas to the vapour phase system is controlled so that the density of the inlet gas to the stall is held at 0.280 Kg/M³ which will be approximately 82% hydrogen. To this system is also added the gas from the "Rückgas" compressors. Both these gases are added to the suction of the circulators.

There is also a connection from the inlet of the vapour phase machines, before the point of addition of the fresh gas, to the exit of the gas washing plant and the valve on this was permanently open. Because the gas to the vapour phase system was more than the operation of the stall required on the basis of the absorption of hydrogen by the oil, there was a purge of gas from the vapour phase system to the liquid phase system via this latter line.

The wash oil rate to the gas washing plant was normally run at 40 M³/hr. to maintain the purity of the inlet gas to the stalls at a density of 0.28 to 0.34 Kg/M³ i.e. 82 to 76% hydrogen. If the stall rate was low the wash oil rate was halved. The make-up gas to the liquid phase system was then added to keep the system pressure constant at 300 Ats at the inlet side.

The circulators were run to maintain a boost of 30 Ats. under normal operation but they had been run at 25 Ats on a number of occasions.

In the main compression plant there are also four "Rückgas" compressors which actually work in connection with the hydrogenation stalls circulating gas system.

The Rückgas was made up from the 18 At let-down gas from the vapour phase stall product, some H₂S from the Alkacid plant, the H.O.L.D. let-down gas, the 1 At. gas from the Gas-Washing Plant, the gas from the Liquid Phase Distillation units, and the water-washed gas from the 1 At. let-down gas from the liquid phase stall product. The mixture had a hydrogen content of 30%. The H₂S was regulated to give 0.2% H₂S by volume in the vapour phase stall exit gas, which corresponds to 0.4% at the inlet. The recycle was said to be carried out to save hydrogen, which is doubtful from the hydrogen content of the gas, to get the H₂S requirement for the stalls and to break down the organic sulphur content of the propane and butane obtained from the Liquid Phase Stalls.

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The ammonia in the system was kept down by the injection of water to all the stall coolers at a total rate of 6 M³/hr.

The ammonia contents of the circulating gases were quoted as

| | |
|------------------------------------|-----------------------|
| Inlet liquid phase stalls | 5.5 mg/M ³ |
| Exit | 10 |
| Inlet A system vapour phase stalls | 7.7 |
| Exit | 8.0 |
| Inlet B | 3.0 |
| Exit | 1.1 |

The ammonia figures for the vapour phase stalls are very high but without temperatures of the catalyst it is difficult to comment as to their probability.

They were hoping to wash the whole of the Rückgas with water in order to reduce the amount of ammonia being recycled to the vapour phase stalls but they had not got any design work in hand. They were however convinced that it would be a good thing, because it would reduce the ammonia content of the vapour phase stall circulating gas still further.

INJECTION PLANT.

For the injection of the feed to the liquid and vapour phase stalls they used injectors of the same design, each being electrically driven by variable speed motors and having a maximum capacity of 25 M³/hr. There were four machines available for the liquid phase system and six for the vapour phase system. One of these latter machines was also available as a spare for the gas washing injectors. All these machines were fitted with gland packing of the type used by the I.G. for their paste injectors, i.e. a U shaped metal ring with a conical spreader; a number of these sets of rings being used to make up one stuffing box.

In addition to these pumps there were three three-throw pumps and two oil-driven I.G. paste pumps, of the direct acting type, all of which had a capacity of 5 M³/hr. each. The three-throw pumps were the original pumps and had given considerable trouble owing to the high speed and the cramped nature of the construction and they had in general been superseded by the I.G. paste pumps which run very much more slowly. These pumps were used for the injection of the catalyst paste.

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Table II (cont'd)

| | Type of Production | | |
|-----------------------------------|---------------------|--------------|-----------------|
| | Petrol & Diesel Oil | Motor Petrol | Aviation Petrol |
| Gas made on A middle oil. | | | |
| Stall temperature A system in | 370°C. | | |
| out | 410°C. | | |
| Stall temperature B system in | - | | |
| out | - | | |
| Ratio of feeds A and B | - | 2:1 | 1:1 |
| Label | - | 0.25 | 0.10 |
| <u>Petrol Quality.</u> | | | |
| Specific Gravity | 0.751 | 0.740 | 0.718 |
| I.B.P. | 51 | 38.5 | 48 |
| F.B.P. | 185 | 195 | 145 |
| Aniline Point | 39 | 43 | 46.6 |
| Octane Number-Motor Method | 63 | 65 | 68 |
| Reid Vapour pressure at 40°C. Ats | 0.6 | 0.7 | 0.5 |
| % distilling up to 100°C. | 32 | 41 | 59 |
| <u>Diesel Oil Quality</u> | | | |
| Specific Gravity | 0.856 | | |
| Aniline Point | 42 | | |
| Cetane Number | 40 | | |
| Setting point | -15°C | | |
| Sulphur % Wt. | 0.21 | | |

(Flowsheets showing the three types of operation are shown on Fig IV)

The output of the plant as a whole was limited by the sections as follows:-

When making Petrol and Diesel Oil the liquid phase stalls and the A system vapour phase stalls limited the plant about equally.

When making Motor Petrol only the limitation was the A system vapour phase stalls.

When making Aviation Petrol the limitation was the B system vapour phase stalls.

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The catalyst in the A system stalls lasted about $\frac{3}{4}$ to 1 year on the average.

The life of the catalyst in the B system stalls depended on the type of petrol being made. When making Motor petrol the life was $\frac{3}{4}$ to 1 year but when making Aviation petrol the life was $\frac{1}{2}$ to $\frac{3}{4}$ year. The difference in life of the catalyst in the B stalls was explained by the fact that the oil fed to the B stalls when making Aviation Petrol had a lower I.B.P. than when making Motor Petrol and therefore a higher phenol content and this phenol content was blamed for the lower life.

The B stalls always had the newer catalyst when the two systems were in operation.

GAS CIRCULATION PLANT AND GAS WASHING PLANT.

The operation of these two plants is so interconnected that they will be treated from the operation point of view as one unit.

In the circulation plant there are five circulators with a capacity of 80,000 M³/hr. each and two with capacity of 50,000 M³/hr. each. The machines are constant speed electrically driven. Three machines were on liquid phase, and four on vapour phase operation.

The gas washing section of the plant consists of two 20 m³/hr three-throw injectors delivering against 300 Ats. The towers themselves are 800 mm. bore and 12 M long and there are two of them operating in parallel. The towers are packed with Raschig rings and the gas and oil flow in countercurrent directions. The oil from the towers is let-down to atmospheric pressure in two stages with the provision that the liquid can be further de-gassed by passing it through a third let-down stage under vacuum. The gas from the first stage operating at 20 Ats goes to the light-ends recovery unit. The gas from the second stage at atmospheric pressure is fed back to the stalls via the "Rückgas" compressors.

The de-gassed oil is then pumped back to the wash towers by the injectors.

The operation of the gas system is as follows:-

The two types of stall are operated with separate gas circulating systems as it is essential that the vapour phase catalyst does not get into contact with too much ammonia. Each system is run independent of the other apart from the cross connections. There is a connection between the inlet headers to the

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valve on the top of the cold catchpot to reduce the pressure from 300 to 100 Ats in 2 1/2 hrs. This had been done 40-50 times and they had been able to heat up straight away after the raid. This was attributed to the excessive quantities of catalyst used and the low asphalt in the system.

DESCRIPTION AND OPERATION OF THE VAPOUR PHASE STALLS.

There are four vapour phase stalls each comprising four converters, two interchangers, an electric preheater, a cooler and a cold catchpot. The catalyst volume of each converter is 8 M³.

Each converter is 1000 mm. bore and 18 M long with an insulation of 60 mm of the same material as the liquid phase stall converters. In the first converter there are seven beds of catalyst, in the second, third, and fourth five beds of catalyst. In all the converters there is provision for the injection of cold circulating gas to the reactants between the beds of catalyst, with the normal Leuna design of mixing plates. The direction of flow is either up or down except for the last converter, where they arrange that it shall always be downwards. They said that providing that the total gas flow did not exceed 40,000 M³/hr. they had no trouble on upward flow converters with catalyst carry-over.

The interchangers are the same as on the liquid phase stalls.

The preheaters are the I.G. electric type and are fitted in the stall chamber itself. They are run with a voltage of about 40 to 60 volts so that the workmen are not subject to electric shock from the plant. This precaution is necessary because it is not possible to insulate the preheater from the stall electrically. The preheater consists of a number of steel tubes arranged in the form of hairpins connected in series and packed into a sheet steel cylinder and insulated against heat losses. The electric heating current is passed into the steel tubing in four places and taken out in four places, i.e. there are four parallel paths. It is so arranged that the current does not have to pass through the return bends at the end of each length of tubing but is led from one tube to another by leads which by-pass the return bends. They had trouble at the start up of the plant owing to coking up of the preheater tubing giving rise to burst tubing. They had overcome the difficulty by fitting temperature points to all the lengths of tubing and regulated the current to each section according to the load and the temperature of the local elements, and since adopting this procedure they had not had very much trouble with the electric preheaters. The cold product was let down in two stages. 20 Ats & 1 At. to give some fractionation of the dissolved gases.

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Each stall was run with a reaction gas rate of 30,000 M³/hr under all running conditions. The feed rate varied, with a normal feed rate of 16 M³/hr. and maximum rates of 25 M³/hr. They had been up to 30 M³/hr. but had found that they were getting very unsteady temperatures and that they could not run the stall under such conditions.

The stalls were run under three different sets of conditions according to the products being made.

When making Petrol and Diesel Oil they ran the stalls on straight through operation, feeding A middle oil from the liquid phase still and distilling the stall cold catchpot product to give Petrol and Diesel Oil. There was no recycle of the middle oil from the vapour phase still to the stall.

When making either Motor Petrol alone or Aviation Petrol alone (they were never made in conjunction) the vapour phase stalls were split into two sections, one section operating on A middle oil under straight through conditions and the other section operating on the middle oil from the vapour phase still. The products from both sections were distilled together so that the second stall system was really operating under recycle conditions with middle oil from the first stall system as make-up.

Running conditions are summarised in Table II.

Table II.

| | Type of Production | | |
|---|---------------------|---------------------|---------------------|
| | Petrol & Diesel Oil | Motor Petrol | Aviation Petrol |
| Feed to A system Spec. Grav. | 0.860 | 0.860 | 0.860 |
| Product A system Spec. Grav. | 0.795 | 0.795 | 0.795 |
| Feed to B system Spec. Grav. | - | 0.848 | 0.805 |
| Product B system Spec. Grav. | - | 0.735 | 0.735 |
| B middle oil. Spec. Grav. | - | 0.848 | 0.805 |
| I.B.P. °C. | - | 198 | 125 |
| F.B.P. °C. | - | 310 | 300 |
| Sulphur % Wt. | - | 0.55 | 0.28 |
| % Petrol in Still Feed. | 58 | 60 | 45 |
| Circulating Gas per tonne feed to all stalls. | 2150 M ³ | 2150 M ³ | 2150 M ³ |
| Hydrogen consumption per tonne A Mid. Oil. | 270 M ³ | 525 M ³ | 660 M ³ |

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The converters were 1000 mm bore and 18 M long with an insulation of either stamped asbestos cement or diatomite bricks the lining being 60 mm thick. They said that they had to be very careful to see that the steel lining, which protected the insulation, was impervious or else they had trouble with the insulation when using the diatomite brick. They had seven temperature points in each converter.

The interchangers were 18 M long and had a bore of 600 mm. There were 199 tubes in each tube bundle.

The preheater tubes were made from N.8 steel and were 120 mm bore 171 mm outside diameter and the elements were about 15 M long. The hairpin elements were centred at 500 mm distance and the fins were 4 mm thick and 10 mm apart and 350 to 400 mm square. There are 15 hairpins all told in the preheater with 5 in each pass.

The operation of a stall is carried out as follows:-
15 to 20 M³/hr. of heavy oil, sp. gr. 0.907 from the base of the liquid phase still is fed to the stall with 5 M³/hr. of catalyst paste together with reaction gas. These are fed through the interchangers and then into the preheater having been mixed at the inlet to the preheater with 5 to 10 M³/hr. of hot recycle from the base of the hot catchpot. They are heated up to 460°C at the inlet to the first converter in the preheater and allowed to heat up to 475°C in the converters. The catalyst used is the I.G. 10927 Iron Catalyst supplied from Leuna. The make up of this is about 0.5 to 1.5% on the feed according to the asphalt in the H.O.L.D. and is varied to hold the asphalt at 0.3 to 0.5% in the H.O.L.D.

The H.O.L.D. has 25% of solids (ash) in it and 4 to 5 M³/hr are let down each hour. A portion of this is fuggled to remove solids and the clean oil used to make fresh catalyst paste. The residue from the fuggals contains about 50% oil and is dumped. At one time there was a plant for the extraction of the oil from this residue but it was destroyed in an air-raid.

The catalyst paste is mixed in 3 ball mills run in series to give a paste with 40% solids in it and mixing 8 Tnes of catalyst in 30 Hours. This paste is mixed with the remainder of the H.O.L.D. not treated in the fuggals and injected to the stall as the catalyst paste, containing on the average about 25% solids.

The cold catchpot product from the stall is mixed with the cleaned tar and the Schwelbenzin and distilled in the liquid phase still to give an end point of the A middle oil of 310°C.

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About 45% of the cold catchpot product boils below 310°C. and has a sulphur content of about 1.3% and a specific gravity of 0.922.

The hydrogen consumption was 230 M³/Tne of Liquid Phase Still Heavy Oil fed to the Stalls, the circulating gas being 1540 M³/Tne of Oil. This liquid phase heavy oil was made up of 49% make-up and 51% recycle from the Stall Cold Catchpot product.

The feed leistung was 0.65 Tne/M³/hr and the "A" middle oil leistung 0.254 Tne/M³/hr, which means that the L.O./H.O. efficiency was 80%. A typical flowsheet is given in Fig. III.

The cold product was let down in two stages i.e. 20 Ats and 1 At. to give some fractionation of the dissolved gases.

They had noticed very little corrosion in the preheaters and estimated that the various elements had a life of 5 years in the hot pass and about 10 years in the other passes. They held the maximum gas temperature at not greater than 560°C in order to avoid over-heating the metal of the tubing. They had average values for the heat transfer across the elements of 10 KgCals/M²/hr/°C based on the outside surface of the element. They had made trials with five elements to improve the heat transfer and distribution by fitting along the whole length of the elements between the fins of the two legs a steel spacing piece which filled the whole of the space between the fins. The trials with this modification had shown no marked difference from the normal type of element.

The interchangers on the liquid phase stalls are changed about every six months to be rebuilt because the K value for the tube bundle has fallen off from 300 to 100 Kg Cals/M²/hr/°C.

They had had some trouble with the hot catchpots resulting from the coking up under operating conditions. They were cleaned out on the average once every six months. They had made alterations to the pots so that there was a continuous injection of cold circulating gas to the base of the pot via a gas ring in the bottom of the cone and had also arranged to add cold gas to the reactants, between the last converter and the hot catchpot in order to cool them. This resulted in a lower temperature in the hot catchpot and therefore less coking.

An interesting feature of the operation of the liquid phase stalls was the fact that they were not blown down during air-raids and yet they had no coking troubles. On receipt of a warning they shut off oil and then shut off the gas after 5 minutes circulation. A small blow-down was then commenced by opening a

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Each tower has a normal capacity of 8,000 M³/hour inlet gas, taking 30 M³ water/1,000 M³ in Winter (10°C) and 60 M³/1,000 M³ in Summer (20° to 27°C). The CO₂ is reduced from 40% to 1.6%.

Centrifugal pumps are used, with pump, motor and Pelton, letting down the water, on the same shaft. The water is recirculated after regeneration in two wooden towers.

(g) CO Removal. There are 3 towers operating at 300 ats. The copper liquor used contains no formic acid and has the composition:

| | |
|---------------------------------|---------------------|
| 1.2 g. atoms Cu ⁺ /1 | } 9% Cu in solution |
| 0.2 g. atoms Cu ⁺ /1 | |
| 5 g. mols NH ₃ /1 | |
| 2.5 mols CO ₂ /1 | |

There is no trouble in controlling cupric content and there is no trouble from oil or sludge.

Three let-down engines are used for injecting 90% of the copper liquor: each engine has a capacity of 72 - 80 M³/hour, with a bore of 0.5 m. and a stroke of 1 m: they are supplemented by three 3-throw injectors, each with a capacity of 15 M³/hour against 325 ats. and three centrifugal pumps, each having a capacity of 80 M³/hour against 12 to 15 ats. These let-down engines gave little trouble. The original valves were of the normal mushroom type which had not been entirely satisfactory and they had changed them over to the Piston valve type which was becoming standard in Germany. After two years' experience they had come to the conclusion that the new type of valve would last about a year and would then have to be changed because of heavy wear. They said that this wear was not in the form of erosion. For the packing of the glands of these machines pumping copper liquor they use waxed chrome leather. This gives them a life of about 2,000 hours on the let-down engines and 3,000 hours on the three-throw pumps. The rods on the let-down engines are of carbon steel unhardened and ground, whilst the rams on the injectors are the same steel hardened and ground. In both cases they re-grind the rods every 18 months. The rams on the let-down engines are lubricated with an ammonia solution continuously and are swabbed once a shift with lubricating oil.

The engineer also said that it was not safe to weld branches into lines which had already been used for copper liquor and use them again for the same job. It was not possible to remove all traces of the copper which had been deposited on the steel and after welding there was a crack with copper in it and in service this crack grew in size and caused a burst.

Power consumption on both CO₂ and CO removal is 232 KWH/1,000M³.

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PREPARATION OF THE TAR FOR HYDROGENATION.

The raw tar imported to the plant is first fuggled to remove the solids and water. The average tar, which composed about 74% of the oil fed to the works, had about 25% distilling off below 310°C, contained about 5% phenols, 5% asphalts, and had a gravity of 0.966, these being average figures. The solids content averaged about 1.0%.

The raw tar was heated up to a temperature which would make settling easy and passed in charges of 500 to 600 litres to the fuggals. There are 11 solid bowl fuggals made by Haubolt of Chemnitz having a diameter of 2000 mm and a speed of 600 RPM. Of the 11, 8 to 9 were required to handle the feed to the tar stall and the remainder to handle the residues, sludge and dirty oil which were accumulated in the other sections of the works.

The charging of the fuggals required about 1½ to 2 minutes, the fuggalling time 3 minutes, the time to remove the clean oil 2 minutes and the time to remove the emulsion about 3 to 4 minutes. The oily solid sludge at the rim of the bowl was only removed once or twice a shift. The fuggals are kept under an atmosphere of CO₂ as a fire precaution. They had had some trouble with air entrainment in the oil leaving the fugal when it was being discharged and had finally fixed up a hood which totally enclosed the exit from the fugal and arranged for a small quantity to be by-passed through a sight glass so that they could see whether they were getting oil, water, emulsion or solids.

The clean oil from the fuggals (amounting to 98.7% of the raw tar fed) had a solid content of about 0.3%. This oil was then sent with the Schwelbenzin, amounting to 26% of the total oil fed to the works, and with the crude oil obtained from the liquid phase stalls to the liquid phase stills.

The Liquid Phase Stalls were operated to cut the oil into two fractions, one boiling below 310°C and the other above 310°C.

DESCRIPTION AND OPERATION OF THE LIQUID PHASE HYDROGENATION SECTION.

There are three liquid phase stalls each comprising 3 converters of reaction volume 8.8 M³ each, 2 interchangers of 200 M² area (approx), a gas-fired preheater, a cooler, a cold catchpot, and a hot catchpot. There were also 5 hot recycle pumps on the three stalls for the recycle of hot oil from the hot catchpot to the inlet of the preheater.

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L.P. steam usage is about 21 kg/kg. S, about one-third being direct and two-thirds indirect.

(iv) Oxide Boxes. The plant consists of the standard BAMAG design of tower boxes, used chiefly to save ground space. There are two sets in parallel, each set consisting of four boxes, each box having an I.D. of 6.8 m. and containing 240 T (320 M³) of Lantamasse in 12 trays. The maximum space velocity is 15 hrs⁻¹. All 12 trays in a box run in parallel, not in series. Sequence and flow can be changed as required; oxygen is added to the inlet gas for revivification but not NH₃. The mass is charged with 50% H₂O but comes out with 5% and containing 40 to 45% sulphur, when it is then sold. The inlet H₂S is 5 to 12 g/M³ and the exit is about 20 mg/M³.

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(d) CO Conversion. The plant consists of 8 sets working at ordinary pressure, together with three saturation towers and three combined water heating and gas cooling towers.

Each set has a normal throughput of 6,000 M³/hour water gas, with a maximum of 8,000 M³/hour (or 7,500 and 10,000 M³/hour catalysed gas respectively). Each set consists of a single tower, with No.1 converter at the top, then water injection with a tray of rings, then No.2 converter and finally the single interchanger at the bottom. Saturated gas, injected by steam, passes through the interchanger and enters No.1 converter at about 350°C. Gas leaves No.1 converter at 460 to 500°C and is then cooled to about 420°C by injection of condensate through a single spray playing on to 1 m. of steel rings. At the bottom of the tray of rings the whole gas is brought through a restriction to ensure good mixing. The gas leaves the No.2 converter at about 430 to 450°C, passing out through the interchanger.

The catalyst used is I.C. brown oxide, costing 1.65 to 2 RM/kg. Catalyst life averages 2 years, but is sometimes 4 years; the spent catalyst is thrown away. Each converter contains 12 T of catalyst and (assuming a density of 1.2 T/M³) this corresponds with a space velocity of 400 hrs⁻¹ on inlet gas at maximum rate of 300hrs⁻¹ on H₂ + CO₂. The CO content of inlet gas (a mixture of purified water gas and regenerated gas ex CO removal) is about 32%, which is reduced to about 3% in catalysed gas, which is close to equilibrium. The pressure drop across each converter is about 50 to 70 mms Hg. No troubles result from the use of condensate, provided it is kept free from oil, etc.

Water is circulated round the water heating and saturation towers, giving a saturation temperature of 65° to 70°C; both are wood-packed towers. The gas cooling tower, which forms the upper half of the water heating tower, is wood-packed and fed with two streams of circulating water; water at 10 to 20°C is fed into the top and water at 20 to 30°C, after use elsewhere, is fed half-way down.

| | |
|-------------------------------|--|
| Steam used (1.9 ats.g, 154°C) | 0.32 to 0.40 kg/M ³ catalysed gas |
| | 0.55 to 0.72 kg/M ³ H ₂ + CO |
| Condensate used | 0.057 kg/M ³ catalysed gas. |

(e) Compression. Seven six stage compressors are installed, with CO₂ removed after the third stage at 28 ats.

(f) CO₂ Removal. There are 7 washers, having an I.D. of 1.75 m. at the bottom and an I.D. of 1.44 m. at the top, with a total height of 18 m. They are filled with 50 x 50 mm. stacked rings, packed to a total height of 12 to 14 m. in two halves.

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is about 50 mms water. The fuel bed is maintained at a temperature of about 900°C, at which temperature gas also leaves the generator.

The dust-laden gas passes through a 1.4 m. I.D. pipe to a waste heat boiler, raising steam at 18 ats. The water-tube boiler has two drums, the lower of which is insulated and hangs in the gas space. A baffle wall from drum to drum protects the cold down flow tubes and also forces the gases to take a U-shaped path. The gases then pass through an economiser and, now cooled to 200° to 300°C, then pass through a set of multicyclones. After passing through a water seal, final dedusting is carried out in a water wash tower and a Theisen disintegrator. The dry dust collected (C.V., 4000 T.cals/T, 40% carbon) is used as boiler fuel, either at the ASW works or at Zeitz. The wet slurry is sometimes dried and used as boiler fuel, but is often dumped. The dust content of gas leaving the generator is about 200 to 250 g/M³ and about 25% of the calorific value of the fuel is lost in this way. 90% of the ash is blown overhead and only 10% is abstracted through the grate. The gas after the multicyclones has an ash content of 25 gms/M³ of gas.

Typical operating data are as follows:

Gas Analysis.

| | | |
|------------------|--------------|-----------------------------|
| CO ₂ | 24.7 | } H ₂ + CO 72.5% |
| CO | 27.1 | |
| H ₂ | 45.4 | |
| N ₂ | 0.8 | |
| CH ₄ | 1.5 | |
| H ₂ S | 0.45 to 0.85 | |

CONSUMPTIONS/10³ WATER GAS

| | | |
|--------------------------------|--------------|----------------|
| Grude | 0.32 to 0.56 | Kg |
| 98% Oxygen | 0.22 to 0.26 | M ³ |
| L.P. Steam (1.9 Ats. G. 162°C) | 0.61 to 0.69 | Kg |
| Cooling Water | 0.016 | M ³ |
| H.P. Steam Produced | 0.55 - 0.68 | Kg |
| Ashes (4.0% carbon) | 27 to 32% | of grude. |

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(o) H₂S Removal

- (i) SO₂ Removal This is removed by water in a 3 m. I.D. tower; the tower also cools the gas.
- (ii) HCN Removal It is necessary to remove HCN before using alkacid, since otherwise KCN would be formed in the alkacid liquor, thus removing potassium and destroying the liquor. Dr. Hausmann said the plant was poorly designed and inadequate. A single tower of 3 m. I.D. is used to purify 40,000 M³/hour gas; it has two depths, each 4 to 5 m, of grids, but rings would be better. A make-up of 10 to 15% K₂CO₃ solution to the circulating system is used.

Typical performance figures are:

| | |
|--------------|---|
| HCN in | 10 to 20 mg/M ³ |
| out | 5 to 10 mg/M ³ |
| Recirculated | 10 to 15% K ₂ CO ₃ solution |
| | 3 M ³ /1,000 M ³ gas. |

- (iii) Alkacid. Dr. Hausmann said this was also poorly designed, and especially inadequate in Summer. A single 3 m. I.D. tower is used for 40,000 M³/hour gas, but in effect the tower is run as three superimposed towers in parallel, for it contains three lots of ring packing, each 1 m. deep, each taking one-third of the gas. With an inlet H₂S of 12 gms/M³ the heat unit H₂S obtained is 5 gms/M³ and the worst 12 g/M³. In fact the plant is often by-passed. The circulating liquor rate is limited by the reboiler to 30 M³/hr and this is used for gas rates from 25,000 to 40,000 M³/hour.

DIK liquor is used, of specific gravity 1.165. The scrubber is made of iron, the rings of porcelain, and the boiler, pumps and distributor of aluminium. Corrosion is small but they were going to replace the aluminium reboiler by one in V2A. A recent D.I.N. sheet for Alkacid plants showed that the considered opinion in Germany was that ordinary steel or iron was satisfactory up to 50°C, 17% Cr. steel up to about 95°C, whilst for boiling solutions they specified a ferrous alloy containing 18% Cr, 9% Ni, with a small amount of carbon.

The poor performance is tolerated only because the plant is followed by oxide boxes. The usage of DIK liquor depends on the performance of the oxide boxes; in some years they purchased 10 T/year.

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vapour phase stalls, and no B vapour phase stalls are run.

When running to make either motor or aviation petrol the product from the A vapour phase stalls is distilled with the product from the B vapour phase stalls. The B vapour phase stalls are run on the middle oil obtained from the bottom of the distillation unit and no oil from the liquid phase still is fed to the B stalls.

The richer gases from the let-down of the hydrogenation crude products and the distillation of the products are collected and the H₂S scrubbed out in an Alkacid plant. They then pass to the light-ends recovery unit where the Pentane and heavier hydrocarbons are scrubbed out and then on to a Linde plant for the separation of the propane and butane. The residual gas then goes to the fuel gas system.

Some of the gases containing higher proportions of hydrogen and H₂S are recycled via the recycle compressors to the vapour phase circulating system to save hydrogen, put H₂S into the gas, and to achieve some removal of organic sulphur from the propane and butane. One or two of the gases recycled this way are water washed to remove the ammonia so that the vapour phase catalyst is not poisoned.

The other lean gases are collected and scrubbed in another Alkacid plant to remove the H₂S and then sent to the Fuel gas system.

The drain waters from the various plants are collected and treated to remove the phenols. The liquid phase distillation water drains are treated with tri-cresyl-phosphate and the resulting phenols are sent to Leuna.

The H₂S is treated in a Claus plant to get sulphur.

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WATER GAS PRODUCTION.

(a) Oxygen. There are five Line-Frankl units, each having a capacity of 2,300—3,000 M³/hr. of 98% O₂. Typical performance figures for one unit are as follows:-

| | |
|-----------------------------|--|
| Low pressure air | 13,150 M ³ /hr. (of which 350 M ³ /hour sent as such to factory) |
| High pressure air | 671 M ³ /hr. |
| 98% oxygen made | 2,275 M ³ /hr. |
| Pure nitrogen made | 196 M ³ /hr. |
| % oxygen recovery | 70 to 83.6% |
| Cooling water | 470 |
| Power for turbo-compressors | 0.60 to 0.64 KVH/M ³ O ₂ . |

(b) Winkler Generators. There are three Winkler generators, including one spare. The output of each can be varied from 12,000 to 25,000 M³/hour, but the normal output is about 20,000 M³/hour.

There are no outstanding design features; they were designed by Leuna and are very similar to the type at Zeitz and Magdeburg. The generator itself is a bricklined vessel, with an I.D. of 4.5 m. and a height of 20 m, with the boiling bed maintained at a depth of about 1.5 m.

Grude coke (C.V. 5,200 to 5,400 T.cals/T) is obtained from the adjoining ASW works and has a grading of 60μ to 5 mms. All bunkers are kept under CO₂ pressure; the generator bunker, holding approximately 125 M³, is fed through a star-feeder and the level can be determined by dip-stick. The grude is fed into the generator, at a point about half way up the bottom cone piece, by screw-conveyor, operating at 2 to 5 r.p.m. The feed tube actually entering the generator is made of FF.128 steel. The stationary grate is made of cast iron bricks, about 12" long, 2" wide at the top and 1" at the bottom; they are packed in threes, with very fine openings (1/16" or less) at the top, with a spacer between every three. A water-cooled scraper arm revolves over the grate, sweeping ashes into a hole in the grate, from which they are removed by screw-conveyor, dumped into a water channel and sluiced away to a settling pit. The whole grate can be lowered and wheeled away for maintenance, whilst being replaced by a spare grate, without cooling off the whole generator.

The oxygen-steam blast enters the grate through a wind-box, fitted with explosion plates, and passes through the grate; at least 80% of the oxygen is added in this way, the remaining 20% being added above the fuel bed. Pressure drop through the fuel-bed

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When the plant was running to make Aviation petrol only, the petrol had:-

An IEP of 48°C
 An FEF of 145°C
 A distillation giving 59% at 100°C
 An Aniline point of 46.6
 A Reid Vapour pressure of 0.5 Atm
 An Octane Number of 68
 A Specific Gravity of 0.710

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DESCRIPTION OF PLANT.

The Böhlen plant of ERABAG consists of two sections, the old part containing the original plant and its extensions and the new war-time site known as the West Site containing the new plant which do not have a 300 Ats hydrogenation section.

The old section contains a Winkler water-gas generating plant, using Grude Coke and Oxygen, a gas purification section, a tar fugging plant, a 300 Ats liquid phase hydrogenation plant containing a gas-washing plant and dope preparation plant, a vapour phase hydrogenation plant, the distillation plants connected with these two hydrogenation plants, an alkacid plant for the hydrogenation gases, a Linde plant for the separation of butane and propane and a Claus plant for the recovery of Sulphur. See Fig. I for rough plan.

In the new section in various stages of completion there are a boiler plant, a light ends recovery unit, an iso-butane unit, a second Linde propane and butane separation plant, a two stall D.H.D. plant with distillation units, a complete butane dehydrogenation unit, an alkylation unit, an isomerisation unit, and the accompanying distillation units, and a third liquid phase distillation unit. The necessary tanks for the operation of the units are also in the area. See Fig. II for rough plan.

DESCRIPTION OF OPERATION.

Water gas is made in three Winkler generators, using grude coke and oxygen made in Linde-Frankl units. After dedusting H_2S is removed in an Alkacid plant, followed by dry oxide boxes, using Lautamasse. After conversion of CO with steam the catalysed gas is compressed to 300 ats, CO_2 being removed by a water wash at 28 ats and CO being removed by copper liquor at 300 ats. The make-up hydrogen passes to the suction of the hydrogenation plant circulators.

The raw tar delivered to the plant is fuggled to remove water and dust and then passed to the feed tanks of the liquid phase stills where it is mixed with the crude product from the liquid phase stills, and the Leichtöl or Schwelbenzin. The mixture is out in the still so that the portion boiling heavier than 340°C is fed to the liquid phase stills and the portion boiling below 340°C is sent on to the A section of the vapour phase stills.

When running to make petrol and diesel oil the product from the A vapour phase stills is distilled to give the desired end point of the petrol and the bottoms from the still are exported as diesel oil. There is no recycle of oil from the still to the

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| YEAR | MONTH | MOTOR PETROL | AVIATION PETROL | DIESEL OIL | TREIBGAS | TOTAL |
|------|-----------|-----------------|--------------------|---------------|---------------|----------------|
| 1942 | January | 8,500 | T-Fuel | 10,000 | 400 | 19,900 |
| | February | 9,600 | 5,000 | 3,200 | 400 | 18,200 |
| | March | 12,700 | 4,700 | 7,800 | 1,150 | 26,350 |
| | April | 10,500 | 4,600 | 5,600 | 1,000 | 21,700 |
| | May | 11,700 | 5,200 | 4,800 | 1,000 | 21,700 |
| | June | 12,700 | 8,400 | 3,600 | 1,100 | 25,800 |
| | July | 9,800 | Aviation | 9,200 | 850 | 19,850 |
| | August | | 14,800 | | 2,100 | 16,900 |
| | September | | 15,600 | | 2,400 | 18,000 |
| | October | | 16,400 | | 3,000 | 19,400 |
| | November | | 16,000 | | 2,600 | 18,600 |
| | December | | 3,000 | 13,100 | 2,400 | 18,500 |
| | | <u>78,500</u> | <u>103,800</u> | <u>44,200</u> | <u>18,400</u> | <u>244,900</u> |
| 1943 | January | 19,800 | | | 1,600 | 21,400 |
| | February | 19,400 | | | 1,800 | 21,200 |
| | March | 4,200 | 12,800 | | 3,850 | 20,850 |
| | April | | 16,400 | | 3,950 | 20,350 |
| | May | | 16,400 | | 3,850 | 20,250 |
| | June | 6,800 | 8,200 | 5,000 | 2,200 | 22,200 |
| | July | 15,000 | | 11,000 | 900 | 26,900 |
| | August | 12,400 | | 9,000 | 750 | 22,150 |
| | September | 15,000 | | 7,000 | 1,100 | 23,100 |
| | October | 17,100 | | | 1,100 | 18,200 |
| | November | 4,300 | 13,500 | | 2,750 | 19,550 |
| | December | 10,000 | 9,000 | | 1,700 | 20,700 |
| | | <u>125,000</u> | <u>76,300</u> | <u>32,000</u> | <u>25,550</u> | <u>258,850</u> |
| 1944 | January | 12,000 | 6,400 | | 2,300 | 20,700 |
| | February | | 11,200 | | 1,800 | 13,000 |
| | March | 10,600 | 11,000 | | 2,800 | 24,400 |
| | April | 26,200 | | | 1,800 | 28,000 |
| | May | 8,700 | | | 650 | 9,350 |
| | June | 11,000 | | 6,000 | 850 | 17,850 |
| | July | | | | | NIL |
| | August | 5,200 | | | 150 | 5,350 |
| | September | 10,600 | | | 850 | 11,450 |
| | October | 3,600 | | | 400 | 4,000 |
| | November | 4,200 | | 4,000 | 100 | 8,300 |
| | December | 5,400 | | 11,000 | 300 | 16,700 |
| | | <u>97,500</u> | <u>28,600</u> | <u>21,000</u> | <u>11,900</u> | <u>159,000</u> |
| 1945 | January | 4,400 | | 7,600 | ? | 12,000 |

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EXPORTS

The petrol and diesel oil were sent to WIFO depots according to instructions. The Treibgas exports were split up so that the pure Propane was sent to ZETZ for use on the Propane Dewaxing plant there and for use as fuel for house heating, the Iso-Butane to Leuna until May 1944, and the remainder was sent away as liquified gas for motor fuel. The composition of the latter varied considerably but the only limit set was the maximum vapour pressure - 25 Atmospheres.

The T-Fuel was a special fuel made for the Campaigns in North Africa. The Diesel Oil made in Nov. and Dec 1940 and Jan and Feb. 1941 was a special fuel made with a low flash point at the expense of the heavy ends of the petrol, with an I.P.P. of 110°C., a specific gravity of 0.8, a flash point of 27°C and a Cetane No. of 42.

QUALITY OF FUELS - 1943.

When running to make petrol and diesel oil the petrol had:-

An I.P.P. of 51°C
 An F.P.P. of 185°C
 A distillation giving 32% at 100°C.
 An aniline Point of 39.5
 A Reid Vapour Pressure of 0.5-0.7 Ats at 40°C.
 An Octane Number of 63 (Motor Method)
 A Specific Gravity of 0.75

The Diesel Oil had:-

A setting point of -15 to -20°C.
 An Aniline point of 42.2
 A Cetane Number of 40
 A Specific Gravity of 0.856 at 20°C.
 A Flash Point of 45°C.

When the plant was running to produce Motor Petrol only, then the petrol had:-

An I.P.P. of 38.5°C
 An F.P.P. of 195°C.
 A Reid Vapour Pressure of 0.6-0.8 Ats.
 An Octane number of 65
 A Specific Gravity of 0.74
 A distillation giving 41% at 100°C.
 An Aniline point of 43

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TABLE I

PRODUCTION FIGURES

| YEAR | MONTH | MOTOR PETROL | AVIATION PETROL | DIESEL OIL | TREIBGAS | TOTAL |
|------|-----------|-----------------|--------------------|------------|--------------|----------------|
| 1936 | August | 12,000 | | | 100 | 12,100 |
| | September | 11,800 | | | 150 | 11,950 |
| | October | 13,900 | | | 160 | 14,060 |
| | November | 12,300 | | | 200 | 12,500 |
| | December | 13,500 | | | 350 | 13,850 |
| | | | <u>63,500</u> | | | <u>960</u> |
| 1937 | January | 13,500 | | | 150 | 13,650 |
| | February | 12,300 | | | 400 | 12,700 |
| | March | 11,100 | | | 400 | 11,500 |
| | April | 16,900 | | | 600 | 17,500 |
| | May | 9,800 | | | 650 | 10,450 |
| | June | 8,100 | | | 400 | 8,500 |
| | July | 12,200 | | | 600 | 12,800 |
| | August | 12,450 | | | 680 | 13,130 |
| | September | 15,600 | | | 400 | 16,000 |
| | October | 16,000 | | | 400 | 16,400 |
| | November | 9,700 | | | 300 | 10,000 |
| | December | | 9,100 | | 300 | 9,400 |
| | | <u>137,550</u> | <u>2,100</u> | | <u>5,280</u> | <u>151,930</u> |
| 1938 | January | 17,200 | | | 500 | 17,700 |
| | February | 12,600 | | | 500 | 13,100 |
| | March | 13,200 | | | 600 | 13,800 |
| | April | 11,800 | | | 600 | 12,400 |
| | May | 14,100 | | | 800 | 14,900 |
| | June | 13,000 | | | 800 | 13,800 |
| | July | 16,100 | | | 1,280 | 17,380 |
| | August | 15,300 | | | 1,100 | 16,400 |
| | September | 13,500 | | | 1,050 | 14,550 |
| | October | 14,000 | | | 1,000 | 15,000 |
| | November | 8,300 | | | 600 | 8,900 |
| | December | 15,000 | | | 950 | 15,950 |
| | | <u>164,100</u> | | | <u>9,780</u> | <u>173,880</u> |

RESTRICTED

| YEAR | MONTH | MOTOR PETROL | AVIATION PETROL | DIESEL OIL | TREIBGAS | TOTAL |
|------|-----------|-----------------|--------------------|----------------|---------------|----------------|
| 1939 | January | 15,300 | | | 1,100 | 16,400 |
| | February | 14,800 | | | 1,000 | 15,800 |
| | March | 15,800 | | | 1,200 | 17,000 |
| | April | 12,700 | | | 1,100 | 13,800 |
| | May | 14,400 | | | 1,350 | 15,750 |
| | June | 14,400 | | | 1,600 | 16,000 |
| | July | 13,400 | | | 1,350 | 14,750 |
| | August | 15,300 | | | 1,600 | 16,900 |
| | September | 7,800 | | | 500 | 8,300 |
| | October | | 12,600 | | 1,000 | 13,600 |
| | November | | 10,800 | | 1,500 | 12,300 |
| | December | | 11,400 | | 1,600 | 13,000 |
| | | <u>123,900</u> | <u>34,800</u> | | <u>14,900</u> | <u>173,600</u> |
| 1940 | January | 7,600 | 3,800 | | 950 | 12,350 |
| | February | 10,200 | | | 600 | 10,800 |
| | March | 13,200 | | | 1,100 | 14,300 |
| | April | 15,900 | | | 1,600 | 17,500 |
| | May | 16,300 | | | 1,800 | 18,100 |
| | June | | 11,500 | | 2,600 | 14,100 |
| | July | | 13,200 | | 3,100 | 16,300 |
| | August | | 14,900 | | 3,350 | 18,250 |
| | September | 7,000 | | 12,000 | 650 | 19,650 |
| | October | 8,600 | | 13,000 | 850 | 22,450 |
| | November | 3,600 | | 12,000 | 600 | 16,200 |
| | December | 4,400 | | 13,000 | 600 | 18,000 |
| | | <u>86,800</u> | <u>39,600</u> | <u>50,000</u> | <u>17,700</u> | <u>194,100</u> |
| 1941 | January | 4,000 | | 12,600 | 650 | 17,250 |
| | February | 4,600 | | 14,000 | 850 | 19,450 |
| | March | 8,100 | T-Fuel | 6,300 | 600 | 15,000 |
| | April | 9,700 | | 3,500 | 400 | 16,400 |
| | May | 10,200 | 4,600 | 4,700 | 400 | 19,900 |
| | June | 9,900 | 4,600 | 3,600 | 550 | 18,650 |
| | July | 10,500 | 4,900 | 3,300 | 500 | 19,300 |
| | August | 9,300 | | 7,700 | 300 | 17,300 |
| | September | 6,000 | | 7,300 | 600 | 13,900 |
| | October | 13,800 | | 14,200 | 850 | 28,850 |
| | November | 8,600 | | 10,800 | 400 | 19,800 |
| | December | 11,200 | | 12,600 | 450 | 24,250 |
| | | <u>105,900</u> | <u>16,900</u> | <u>100,600</u> | <u>6,550</u> | <u>229,950</u> |

RESUMÉ

HISTORY OF PLANT.

The BRABAG concern was founded in 1935 to make liquid fuels from brown coal by the Hydrogenation and Fischer-Tropsch processes or by any other processes which might seem to be economic under the prevailing conditions in Germany. The first plant built by the company was at Böhlen and was begun in 1935. The first petrol stall started up in the early part of 1936. nine months after the start of erection. Full output was reached in August 1936.

The original plant had four stalls, two liquid phase and two vapour phase, and the minimum equipment for the production of Hydrogen and other services, in order to keep the capital cost of the plant down to a minimum. All the stalls were to operate at 300 Atmospheres and 5058 catalyst was used in the vapour phase.

In July 1937 the vapour phase catalyst was changed to 6434 in order to improve the quality of the petrol. Until the extension of the plant in 1939 the plant ran to make Motor Petrol only, with one occasion when they made a trial run on the production of Aviation Petrol. The average output when on 5058 catalyst was 150,000 Tonnes/year of Motor Petrol and 6,000 Tonnes/year of Treibgas. When they changed to 6434 catalyst they made on the average 165,000 Tonnes/year of Petrol and 12,000 Tonnes/year of Treibgas. The quality of the petrol with 5058 catalyst was 59 Octane Number with an FBP of 180°C, and with 6434 catalyst 66 Octane Number with an FBP of 130°C.

The plant was extended in 1939 by installing some spare equipment and then extending the stalls to make use of the greater average gas production available and also to ensure steadiness of production. The four stalls were increased to seven, three liquid phase and four vapour phase, the two Winkler generators to three and various other small additions made. After these extensions the plant did not run to produce any single fuel as had been the case but ran to produce Motor Petrol, Aviation Petrol, Diesel Oil, according to the requirements of the Reich.

Since 1943 a new site to the west of the Main Site has been developed to enable the works to produce higher grade Aviation Spirit but this development has not been in production.

RESUMÉ

SOURCES OF RAW MATERIALS.

The plant imports the following raw materials:

| | |
|-------------------------------|---|
| Brown Coal Tar | A.S.W. Böhlen A.S.W. Hirschfeld A.S.W. Espenhain Kulkwitz |
| Lurgi Gas Tar Schwalbstein | A.S.W. Böhlen A.S.W. Böhlen A.S.W. Hirschfeld A.S.W. Espenhain Kulkwitz |
| Steam (Low pressure) | A.S.W. Böhlen |
| Electricity | A.S.W. Böhlen |
| Water | A.S.W. Böhlen |
| Crude | A.S.W. Böhlen |

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OBJECT OF VISIT

The plant was visited between May 4th and 13th 1945 for the purpose of obtaining information on the operation of the plant.

The investigators visited the plant during the following period:

| <u>British</u> | <u>American</u> |
|---------------------|--------------------|
| Lt. Col. H. Hollins | Dr. H.H. Weir |
| " J.G. Kin | Dr. L.L. Hirst |
| Major R.J. Morley | Dr. L.L. Newman |
| " B. Spivey | Dr. W.A. Horne |
| " H. Berdgett | Mr. C.F. Bays |
| " T.W. Harler | Mr. B.L. Mackenzie |
| " J.P. Ellis | Mr. P.K. Kuhne |
| | Mr. F.H. Peck |

The report has been written from notes given by Major Berdgett, Major Morley, Major Simpson (C.A.S.) and Major J.P. Ellis.

SUMMARY

The Böhlen plant of the Braunkohle Benzin A.G. (BRABAG) is situated near the village of ROTHA south of Leipzig. It makes from Brown Coal Tar by the Hydrogenation Process, in two stages at 300 Atmospheres, Petrol and Diesel oil. The plant was capable of producing either 300,000 Tonnes/year Motor Petrol and Diesel Oil, 250,000 Tonnes/year Motor Petrol, or 200,000 Tonnes/year Aviation Petrol.

PERSONNEL INTERVIEWED

| | |
|------------------------------------|---------------------------|
| Managing Director | Dr. von Felbert |
| Works Manager | Dr. Friedrich Lott. |
| Deputy Works Manager | Dr. Schneider. |
| Brabag Chief Design Engineer | Herr Richard Lackner. |
| Works Engineer | Dr. Wolf |
| Deputy Works Engineer | Herr R. Hertmann |
| Gas Section Manager | Dr. Hausmann |
| Gas Section Asst. Engineer | Dr. Müller |
| Low Pressure Section Manager | Dr. Wilhelm Saruck |
| Low Pressure Section Asst. Manager | Dr. Heinz Fleissig. |
| High Pressure Section Manager | Dr. Carl Schwitzer. |
| Compression Plant Engineer | Dipl.Ing. Rudolf Vollmer. |

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REPORT ON VISIT TO BRABAG I PLANT
AT BOHLEN

Reported by:

MAJOR J. F. ELLIS, MINISTRY OF FUEL AND POWER

On behalf of:

BRITISH MINISTRY OF FUEL AND POWER AND
TECHNICAL INDUSTRIAL INTELLIGENCE COMMITTEE

CIOS TARGET NO. 30/4.05
FUELS AND LUBRICANTS

1945
COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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BRABAG I PLANT
BOHLEN

Ellis, J. F.

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

2. CONCLUSIONS AND RECOMMENDATIONS

The development of the Fischer-Tropsch process as a producer of iso-paraffins, and thus a source of high quality aviation and automotive fuels, opens the way for it to become the most versatile of all synthetic fuel processes. It would then be capable of producing products which range all the way from high quality diesel fuels and lubricants to excellent aviation gasoline. No other process could make such a claim. It is therefore recommended that this information be made available to all interested agencies, and that the work of Dr. Pichler and Dr. Koch of KWI, which is still continuing at the Institut be closely followed in order to keep American technology as well informed as possible.

3. DETAILS OF SHIPMENTS

Small samples of the thoria-alumina and the ZnO-Al₂O₃ catalysts used in iso-paraffin synthesis are being forwarded to Bureau of Ships (Code 341) on Consignment Tag No. 3655. Under the same consignment is also being shipped small samples of n-octane, n-nonane, and n-decane for use in the standardization of mass spectrographic analysis procedure.

Prepared by:

D. R. DEWEY,
Lieut. (jg), USNR

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1. DETAILS OF RESEARCH (A) (cont'd).

(8) On the other hand the $\text{AnO-Al}_2\text{O}_3$ catalyst (1:1 by weight) is believed to carry more promise as a commercial catalyst because of its cheapness. It does give slightly less yield than the thoria type, and produces about ten (10) percent alcohols. There is less tendency to form carbon deposits on the catalyst. The same tendency holds true as with thoria catalysts, i.e., higher alumina content promotes methane formation. However, experiments have been made over a range of Al_2O_3 content from 2:1 to 1:2 without much change in results. Additions of K_2CO_3 do not appear to give any advantage.

(9) The thoria type catalyst was prepared by using two (2) liters of thorium and aluminum nitrates at their boiling point and in the right proportion to give a 1:4 weight ratio of thoria to alumina. This was added one (1) liter of a boiling Na_2CO_3 solution of proper strength to give one hundred (100) grams of dry co-ppt., in stoichiometric amounts. More dilute solutions gave greater density catalysts. The resultant precipitate was washed, dried, and sized to two (2) to four (4) millimeters.

(10) The same method was used for preparing the ZnO type catalysts, except it was found that the addition of the nitrate solution to the soda solution instead of the normal method gave increased liquid hydrocarbon yields.

(11) An alternate method for preparing thoria catalysts, and the one which gave the best yields, was to make sodium aluminate and then precipitate with sulphuric acid. Thorium hydroxide precipitate was also made, and these two ppts., were washed separately and mixed while wet. They were then air dried at one hundred (100) degree centigrade and ground to normal size.

(B) Aromatic Synthesis

(1) Aromatics were successfully synthesized by the use of Cr, Mo, Th oxides as catalysts. Five (5) to ten (10) percent K_2CO_3 was added to reduce carbon formation. It also reduced activity. A CO/H_2 ratio of 1:1 was used, at thirty (30) atmospheres and five hundred (500) degrees centigrade. Any higher temperature or pressure gave excessive carbon formation on the catalyst, while

1. DETAILS OF RESEARCH (B) (cont'd).

lower temperatures gave no aromatics, and reduced the reaction rate. Lower pressure similarly reduced the reaction rate.

(2) The yields were very poor, being only about eight (8) to ten (10) gm./m³ of liquids which were about fifty (50) percent aromatics and fifty (50) percent naphthenes. There was lots of methane formed, and much unconverted gas. The aromatics were principally toluene, xylene, and alkyl benzenes. The naphthenes were a grand mixture not yet identified.

(3) Because of the poor yields and the excessive carbon formation when working at such high temperature, the KWI staff had temporarily abandoned this line of research to concentrate on more productive wartime work.

(c) Iron Catalyst Synthesis

(1) The entire staff of the KWI was quite emphatic in stating that the use of iron catalysts in Fischer-Tropsch synthesis was considered only as a wartime necessity because of the shortage of chromium. Cobalt catalysts were stated to be definitely superior, cleaner to use, and capable of operating at lower temperatures. They did not know of a single instance where iron catalysts were actually used in commercial installations, although it had been planned in the event that chromium became unobtainable.

(2) Iron catalysts are prepared by precipitation from its nitrate with soda solution, drying at one hundred ten (110) degrees centigrade, grinding and sizing, and then treatment with 1:1 CO/H_2 gas at reduced pressure (from 0.1 to 1 atmosphere) and two hundred forty (240) to three hundred twenty (320) degrees centigrade. This treatment forms an iron carbide on the catalysts surface, and is continued until a sharp drop in CO_2 content of the exhaust gas indicates cessation of carbide formation. It is generally used in sizes ranging two (2) to five (5) millimeters.

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WARTIME RESEARCH ON SYNTHETIC FUELS BY THE
KAISER WILHELM INSTITUT FÜR KOHLENFORSCHUNG

1. DETAILS OF RESEARCH

(a) Iso-Paraffin Synthesis

(1) Operating conditions for the synthesis of iso-paraffin have been found to lie in the range of three hundred (300) atmospheres pressure and four hundred twenty (420) to four hundred fifty (450) degrees centigrade. For pressure under three hundred (300) atmospheres the yield falls off rather sharply, although a very slow reaction will take place at thirty (30) atmospheres or over. Higher pressures than three hundred (300) atmospheres give increasingly greater percentages of oxygenated products, until at one thousand (1000) atmospheres the principal product is dimethyl ether. Similarly, lower operating temperatures give slower reaction rates, more unsaturated compounds, a very high percentage of alcohols, and less carbon formation on the catalyst. For temperatures slightly greater than four hundred fifty (450) degrees centigrade, the products become principally naphthenic, and carbon deposition becomes excessive. Instantaneous reaction rates, however, are increased.

(2) It has been found that the best synthesis gas has a CO/H₂ ration of 1.2 volumes of CO to one (1) of H₂. Increasing the hydrogen increases methane formation; decreasing the hydrogen lowers the overall yield.

(3) The best catalyst found for this synthesis has been an aluminathoria co-precipitated one, although ZnO-Al₂O₃ appears to be nearly as good and much cheaper. Using either of these materials it was found necessary to burn off the carbon deposits about every two weeks of steady operation. This was accomplished with air at the temperature of the synthesis (450) degrees centigrade. Catalysts so treated have been used continuously for over six (6) months without appreciable decline in activity. Their heat sensitivity is also quite good, as they have been held for prolonged periods at eight hundred (800) degrees centigrade without damage.

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1. DETAILS OF RESEARCH (A) (cont'd).

(4) Heat evolution during synthesis is approximately the same as with the normal synthesis, i.e. one-fifth (1/5) of the heat of combustion of the products. Since it is possible to work in a twenty (20) to thirty (30) degree centigrade temperature range, this lessens the problem of very close temperature control normally encountered in this process. Another advantage is found in the fact that sulphur does not seem to be nearly as deleterious as in former syntheses, although the upper allowable limit has not yet been determined.

(5) Gas velocities have been fairly accurately studied, and it was determined that twenty (20) cubic centimeters of a two (2) to four (4) millimeter catalyst were necessary for each ten (10) liters of synthesis gas per hour. Any increase above this velocity gave sharp decreases in yield and also tended to form increasing amounts of alcohols.

(6) Product yields are as follows from a ZnO-Al₂O₃ catalyst using a CO/H₂ ratio of 1, at three hundred (300) atmospheres and four hundred fifty (450) degrees centigrade:

| | |
|--|------------------------------|
| Total yield of C ₃ and higher | 120 - 130 gm./m ³ |
| C ₂ | 20 gm./m ³ |
| C ₄ (90% isobutane) | 50 - 80 gm./m ³ |
| C ₅ and higher (over 97% iso) | 30 - 60 gm./m ³ |

(7) A comparison of the two (2) catalysts shows that for a thoria-alumina catalyst (1:4 by weight) the best yields of all are obtained. Higher gas velocities can be used, and there is little tendency to form alcohols. However, there is a greater tendency to form carbon on the catalyst, meaning shorter burn-off times. Experiments have varied the ratio of the two materials in the catalyst from twenty (20) to forty (40) percent Al₂O₃, with very little change in overall yields. However, increased alumina does increase methane formation somewhat. The addition of one-half (1/2) to one (1) percent K₂CO₃ to the catalyst will give a slight increase in yield.

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WARTIME RESEARCH ON SYNTHETIC FUELS BY THE
KAISER WILHELM INSTITUT FUR KOHLENFORSCHUNG

SUMMARY

Wartime research by the Kaiser Wilhelm Institut fur Kohlenforschung on the Fischer-Tropsch synthesis has been primarily directed along the line of iso-paraffin synthesis. Good yields of iso-paraffins have been obtained using the normal synthesis gas at three hundred (300) atmosphere pressure and four hundred twenty (420) to four hundred fifty (450) degrees centigrade over an alumina-thoria catalyst. Zinc oxide-alumina has also been used successfully. About one hundred twenty (120) to one hundred thirty (130) grams per cubic meter of synthesis gas have been obtained as liquid products of over ninety (90) percent iso-paraffins.

Synthesis of aromatics has been studied, using thirty (30) atmosphere pressure and five hundred (500) degrees centigrade. However, very poor yields and naphthene side reactions do not make this process look promising. Further work was abandoned by KWI for the duration of the war.

Fischer-Tropsch synthesis using iron catalysts was studied, but inferior operations resulting from this catalyst made it only desirable as a wartime substitute for scarce chromium.

June 1945

No. 289

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WARTIME RESEARCH ON SYNTHETIC FUELS BY THE
KAISER WILHELM INSTITUT FUR KOHLENFORSCHUNG

June 1945

Reported By

Lieut. (jg) D. R. DEWEY, USNR
NavTechMisEu

CIOS Target Number 30/6.01
Fuels & Lubricants

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

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WARTIME RESEARCH ON SYNTHETIC FUELS

KAISER WILHELM INSTITUT FÜR

KOHLNFORSCHUNG

Lieut. (jg) D. R. Dewey, USNP
NavTechMisc

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

NOTE: The Publication Board, in approving and disseminating this report, hopes that it will be of direct benefit to U. S. science and industry. Interested parties should realize that some products and processes described may also be the subject of U. S. patents. Accordingly, it is recommended that the usual patent study be made before pursuing practical applications.

Price \$.10

Carbide Production Capacity of Germany
(metric ton per month)

| <u>Group</u> | <u>Plant</u> | <u>In 1936</u> <u>tons/yr.</u> | <u>In May</u> <u>1942</u> <u>tons/mo.</u> | <u>AWA</u> <u>Planned</u> <u>in 1942</u> <u>tons/mo.</u> | <u>Reported</u> <u>in 1945</u> <u>Reference to</u> <u>1945 Report</u> |
|---|-----------------------------------|-----------------------------------|---|---|--|
| I. G. | Knapsack | 250,000 | 25,000 | 35,000 | 1,000 tons/da. I. I. O. S. |
| | Schkopau | 120,000 | 21,000 | 34,000 | 30,000/mo. Rasche (TIIC) |
| | Ludwigshafen | 4,000 | - | 3,500 | 3,000/mo. I. G. record 1944 |
| | Ausschwitz | - | - | 3,000 | |
| Bayerische-Stickstoffwerke | Piesteritz Deggendorf | 350,000 | 27,000 | 27,000 | |
| Wacker | Burghausen } Münchenberg } | 70,000 | 5,000 3,500 | 5,000 3,500 | 50,000/yr. Hunter (TIIC) |
| Ch. Werke Aussig | Falkenau | | 3,500 | 3,000 | |
| Total of I. G. and affiliated groups | | | 79,000 | 124,000 | |
| Viel | SKW-Hart Königshutte | | 15,000 13,500 | 14,000 16,000 | 340,000/yr. Bidlack (TIIC) |
| Lonza | Waldslurt } Spremberg } | 100,000 | 3,000 2,000 | 21,000 3,000 | 50/da. C. A. F. E. 21 |
| Degussa | Wyhlen Fürstenberg | 10,000 | 1,000 | 1,000 | |
| Scharfgothsche Heyden | Bobrek Hirchfelde | 30,000 10,000 | 3,000 1,000 | 3,000 1,000 | |
| Elektrowerke Cont. Ges. El. Wiede | Oberlaziisk Laudeck Freyung | 9,000 10,000 | 500 500 | 500 500 | |
| Totals | | | 122,550 | 172,150 | |

-40 - Ash & Cross Ltd., London.

silica and other impurities. The longest hold-up in production with the No.1 furnace came about soon after initial operation, and was caused by the explosion of a load switch on a transformer; and, for the same length of time, namely about 8 weeks, with furnace No.2, due a general overhauling, during which time the substitute insulation was replaced and a water-cooled support, similar to that used at Sohkopau, constructed for the suspended poker. Meanwhile the furnace No.1 has improved in operation, and in the last three months has reached an output of more than 4,000 tons of carbide, with a power consumption as low as 3,200 kw per ton of normal carbide. (The author obviously means kw.hr. T.T.)

The British report on carbide production gave Ludwigshafen a production of 4,000 tons per year in 1936. Presumably this was from a very small furnace if there was in fact any production at Ludwigshafen. The I.G. record of 1942 shows no carbide capacity at Ludwigshafen. The carbide capacity planned for Ludwigshafen by the R.W.A. was 13,500 tons per month. When the difficulties mentioned by Niemann had been cleared up, the two Ludwigshafen furnaces would presumably produce at least 8,000 tons per month if both were operated continuously.

During the past few months a number of the German carbide plants were visited by C.I.O.S. teams, and the production capacity of the furnaces noted. For example, in C.I.O.S. Report File XIX-I-Item 22, it is stated that the carbide plant near Ludwigshafen is equipped with two 20,000 kw furnaces, with an annual production capacity of 45,000 tons of carbide per year, but one of the furnaces is held in reserve. This statement is not quite correct, for a single 20,000 kw furnace will produce more than 45,000 tons of carbide per year, and the installed capacity at Ludwigshafen must now be at least 90,000 tons per year.

In the following table, the British estimate of carbide capacity of Germany in 1936, the I.G. statement as to capacity in May, 1942, the I.G. statement as to the ultimate capacity planned by

R.W.A. and the capacity reported by the recent investigations (so far as noted) are shown in parallel columns. The information on current production capacity (or perhaps it should be said the capacity reached before the bombing of the German plants) is not complete. It is evident, however, that the capacity planned by the R.W.A. in 1942 was actually reached in many of the plants by the middle of 1945.

III. Developments Since May, 1942

Evidently the goal of 210,000 tons of carbide per month, projected in the foregoing I.G. report, had not been fully attained as late as the end of July, 1944. On July 29, 1944, address at a "Bunatagung" (auf dem Kohlhof bei Heidelberg) Dr. Ambros of the I.G. said: "German economy depends on a capacity of about 210,000 tons of carbide per month when all the expansion is completed. The allocation of this will

55,000 tons per month for cyanamid
35,000 tons per month for granular
carbide and carbon black.
20,000 tons per month for acetylene
chemistry

These 120,000 tons per month of carbide correspond to 35,000 tons per month of acetylene, to which is to be added some 5,000 tons per month of acetylene from the H&A arc-light process. Of the total of 40,000 tons of acetylene per month, 60 per cent, that is about 25,000 tons per month goes into buna plants. The remaining 15,000 tons of acetylene per month go to other plants for acetylene chemistry, such as those of Knapsack, Wacker, etc. Of the 25,000 tons per month of acetylene, 17,000 tons go for buna synthesis alone and 8,000 tons per month for other acetylene derivatives, such as ethylene, synthetics, etc."

At the same "Bunatagung", in late July, 1944, there was a discussion as to the selection of processes used at Auschwitz, including the kind of carbide furnaces set up. The following paragraph discusses the furnaces:

"The 25,000 kw furnaces of the Bayerische Stickstoffwerke, with Söderberg electrodes were selected for the carbide plant. The triangular furnace at Knapsack has up to date encountered the drawback that a carefully sized raw material is required, and a fault-free gas offtake has not as yet been fully developed. The merit of the Piesteritz construction is, without question, the

central tapping. In spite of the frequent difficulty which Schkepau encountered in the beginning, we have concluded that Schkepau has accomplished a very satisfactory development of this furnace.

Regarding further details of the carbide plant I may not speak at this time, since the complete discussion of carbide production is not included in the day's agenda."

A little further information on carbide furnaces is to be found in a report presented by Dr. Niemann of the Ludwigshafen plant at the I.G. "Bunatagung" held near Heidelberg, July 29-31, 1944. He was presumably discussing the carbide furnaces at Ludwigshafen. The following is a translation of the pertinent paragraphs of this paper:

"I might now go through the industrial development of the several manufacturing steps in "diol" production, beginning with carbide production from one furnace in March, 1943. The second furnace went into operation in October. The first furnace was modeled on the Piesteritz construction and was accepted after a short test under our industrial guidance. During various tests, particularly of the coke and lime in the furnace charge, a series of difficulties were encountered, which often caused shut-downs. Overloading of the heavy-current circuits was at first frequent. Insulation defects had meanwhile been found on furnace No.2 to be due to the use of an unsuitable insulation substitute. We have had a succession of electrode breakages and have had to endure all the destruction and danger which result from terrible gas outbursts. In the end it was evident what the essential of a well-equipped and serviceable furnace is, and its dependence on using a charge of dry coke and a high quality lime. In order to attain good operation it was necessary to carry through various improvements of the furnace; for example, the current fluctuations had to be ironed out, the working crews trained, the lump coke secured despite the demand and growing shortage, and finally, the lime producers at Steeden and Stromberg had to be persuaded to furnish good lime, free from

Existing Position and Prospective Development of
German Carbide Capacity

(Position as of May 1942 in tons of carbide per month)

| Group | Plant | Present available capacity | In % | Capacity after completing current projects | Capacity after completion of R.W.A. additional plans |
|-----------------------------|--------------|----------------------------|------|--|--|
| I G | Knapsack | 25,000 | | 3,000 | 3,000 |
| | Schkopen | 21,000 | | 3,000 | 3,000 |
| | Ludwigshafen | -- | | 2,500 | 300 |
| | Auschwitz | -- | | 2,500 | 300 |
| | | 46,000 | 38 | 6,000 | 30,500 |
| Bayerische-Stickstoff-Werke | Plesteritz | 17,000 | | 3,000 | 3,000 |
| | Deggendorf | -- | | -- | 3,000 |
| Wacker | Burghausen | 17,000 | 4 | 3,000 | 3,000 |
| | Muckenberg | 5,000 | | 3,000 | 300 |
| | Muckenberg | 8,500 | | 3,000 | 300 |
| C.A. Werke Aussig | Falkenau | 13,500 | 11 | 3,500 | 3,500 |
| | Falkenau | 2,500 | | 3,500 | 3,500 |
| I G and affiliated groups | | 79,000 | 64 | 19,000 | 24,200 |
| V ag | SKW-Hart | 15,000 | | 3,000 | 3,000 |
| | Königsbütte | 13,500 | | 2,000 | 3,000 |
| | | 28,500 | 23 | 4,000 | 50,000 |

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| Group | Plant | Present available capacity | In % | Capacity after completing current projects | Capacity after completion of R.W.A. additional plans |
|---|-------------|----------------------------|------|--|--|
| Lanza | Waldsart | 6,000 | | 1,000 | 1,000 |
| | Sprenberg | 2,000 | | 2,000 | 3,000 |
| Degussa | | 8,000 | 7 | 23,000 | 23,000 |
| | Wöhlen | 1,000 | | 3,000 | 3,000 |
| | Fürstenberg | -- | | 4,000 | 3,000 |
| | | 1,000 | 1 | 3,000 | 3,000 |
| Schaffgotsche Heyden Elektrowerke C.A. Ges. El. Wiede | Bobrek | 3,000 | | 3,000 | 3,000 |
| | Hirschfelde | 1,000 | | 3,000 | 3,000 |
| | Oberlaziak | 1,950 | | 300 | 300 |
| | Laudeck | 600 | | 300 | 300 |
| | Freyung | 600 | | 300 | 300 |
| Total of misc. plants | | 6,150 | 5 | 3,150 | 3,150 |
| Total | | 122,650 | 100 | 135,150 | 112,150 |
| Total per Year | | 1,471,800 | | 2,221,180 | 3,545,800 |

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GERMAN CARBIDE CAPACITY
for 1942

| Plant | Company | Capacity 1000 tons per year. |
|--------------------------------|---|------------------------------------|
| <u>West Germany</u> | | |
| Knappe | A.G.für Stickstoffdünger | 10 |
| Steele (shut down) | Carbidwerk Wilm. Vogel | 10 |
| Wils (from 1942) | Chemische Werke Huls, G.m.b.H. (under construction) acrylonitrile | - |
| | | <u>220</u> |
| <u>Central Germany</u> | | |
| Pfeisteritz | Bayerische Stickstoffwerke | 100 |
| Mückenberg | Dr. Alexander Wacker | 21 |
| Spremberg | Lonza G.m.b.H. | 16 |
| Hirschfeld | Elektrochemische Gesellschaft | 10 |
| Falkenau | Anssiger Verein | 30 |
| Schkopau | I.G. (Expansion to 330 planned) | <u>220</u> |
| | | <u>496</u> |
| <u>East Germany</u> | | |
| Bobrek | Schaffgotsch'sche Werke | 26 |
| Chorzow | Ver. Stickstoffwerke Moscice - Chorzow | 150 |
| Lasisk | Electrowerke, G.m.b.H. | <u>36</u> |
| | | <u>212</u> |
| <u>South Germany</u> | | |
| Hart | Bayerische Stickstoff-Werke | 220 |
| Schalchen | Bayerische Stickstoff-Werke | (20) |
| Burghausen | Dr. Alexander Wacker, G.m.b.H. | 58 |
| Walzlurt | Lonza G.m.b.H. | 85 |
| Wyhlen | Deutsche Gold und Silberscheideanstalt | 10 |
| Freyung | Wiede's Carbidwerke, G.m.b.H. | 10 |
| Deutsch- Matrei (shut down) | Carbidwerk Deutsch-Matrei, A.G. | (10) |
| Landeck | Const. Geo.f. angew. Elektrizität | <u>11</u> |
| | | <u>394</u> |
| Total Germany capacity.... | | <u>1322</u> |

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Present Situation and Prospective Further
Development of German Carbide Allocation.

(Situation as of May, 1942)

| Product Use | Situation as of Mid. 1942 (tons per month) | In % | Situation after completing the settled plan (tons per month) | In % |
|---|---|------------|---|------------|
| Cyanamide | 26,000 | 24 | 55,000 | 24 |
| Buna | 15,000 | 1 | 18,000 | 1 |
| Vinyl Products & chlorinated hydrocarbons | 6,000 | 5 | 10,000 | 5 |
| Hydrogenating ethylene not for buna or lubricants | 5,000 | 4.5 | 10,000 | 4.5 |
| Acetaldehyde not for buna | 23,000 | 21 | 28,000 | 13 |
| Miscellaneous chemicals (acrylonitril etc.) | - | - | 4,500 | 2 |
| Lubricants | - | - | 4,500 | 2 |
| Carbon black | 2,000 | 2 | 3,500 | 2 |
| Domestic use | 20,000 | 18 | 27,000 | 13 |
| Miscellaneous uses | 5,000 | 4.5 | 5,000 | 2 |
| Totals | 110,000 | 100 | 210,000 | 100 |

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The regional distribution of individual carbide-producing plants (which Chart II further sets forth) indicates that the I.G. Knapsack, Schkopau, and Ausschwitz plants, in their geographic placing and also with regard to raw materials, are favorably located in the neighborhood of large brown coal and bituminous coal production. Carbide plants must be located on the banks of cheap power and therefore near the coal mines.

For the selection of Ludwigshafen as a location for a carbide plant, the arguments were particularly sound.

So far as a further expansion of carbide production by the I.G. is concerned, the water power of Deggendorf ("Stw" project) and Oberaltaich (Austria) must be kept in view.

On the other hand, the selection of Fürstenberg for the new plant of Degussa appears to be very questionable. The interest of Degussa in a carbide installation, with attached acetylene chemical units, is obvious; the company will provide a new basis for their solvent chemistry entirely through synthesis, since the former basis of wood carbonization by Hiag, with increasing scarcity of raw materials, grows ever more limited.

In summary, it is well established that German carbide capacity, through the present and planned expansion, is likely in the future to bring about a scant covering of the needs in German markets. By its irregular behavior with respect to needs, the cyanamide demand will serve as an important compensating factor. From it also, carbide may be withdrawn for the developing needs for other processes in the future, without at once bringing about unfavorable situations. On the other hand, the cyanamide industry could also, through freed quantities of carbide, be expanded to a considerable degree, in case there should

suddenly develop out-backs in other large consuming processes.

(Note: The above is a free translation and may contain in error in minor details the tables which immediately follow.)

Indwighshafen will install three 25,000 Kw furnaces, which, with an average throughput of some 4,500 tons per month will furnish 13,500 tons of carbide per month.

A like capacity will be provided at Auschwitz.

BStW, Wacker and Falkenau will remain unchanged.

The Viag (Vereinigte Industrie-Unternehmen A.G.) will increase its capacity to 20,000 tons per month in Hart and 20,000 tons per month in Königshütte.

Ionza will increase capacity at Waldenut to 11,000 tons per month and attain thereby a total output of 13,000 tons per month. (Note: this evidently includes the lower Spremberg output. Tr.)

Degussa is to build a large furnace in Fürstenberg and thereby increase its capacity by 4,000 tons, i.e. to 5,000 tons per month. (In the two plants. Tr.)

The capacity of the small plants will remain unchanged.

These current expansions bring the German carbide capacity to 185,000 tons per month. That this will not meet the final demand has been taken into account in the planning of the R.W.A., which contemplates but does not yet provide definitely for the following:

Ausschwitz is to undertake the installation in its Montau works, of a further furnace to turn out 4,500 tons per month.

The BStW will add two furnaces of this same size to its Degendorf project.

Falkenau will erect a large furnace with a 4,000 tons per month throughput and shut down the small obsolete units.

Viag will erect two new furnaces of 4,000 tons per month, one at Hart and one at Königshütte.

And, finally, Degussa is to provide a large furnace at Fürstenberg.

After the accomplishment of all these plans the German carbide needs will be covered within limits of error.

Regarding the probable time for the establishment of these final facilities, so far in intermediate stages, no definite prediction can be made.

An inspection of the individual plant capacities shows that the I.G. part at present is 35 per cent of the combined German capacity, and with the accomplishment of the fully determined and the additionally planned construction, this will become 64 per cent of the total. It appears that this will not appreciably change in the future. Likewise Viag will maintain its part amounting to 23 per cent.

Before the war, German carbide production went one third each to cyanamide, welding, and acetylation. Today, the allocation is 54 per cent to acetylene chemistry and this may finally be increased to 57 per cent.

Twenty-five per cent of today's carbide production is from water power and this fraction will not be appreciably changed in the future.

Most of the large producers of carbide are now concerned with acetylene chemistry, and they continue to seek agreement (Degussa; Viag).

silicide, for export and for use in shops within the producing plants.

For the most part the urgent demands arise from the carrying out of the current increasingly important enterprises.

According to the planning of the R.W.A. (this is presumably the Reichamt für Wehrschaff Ausbau - Tr.) there will be an increased allotment of 59,000 tons per month of carbide for the production of cyanamide.

The buna demand of Schkopau, Ludwigshafen and Auschwitz will amount to 42,000 tons per month.

The quote for vinyl products and chlorinated hydrocarbons will be doubled in the drive for increasing vinyl polymer output.

The Wacker establishments will make 40,000 tons per month of aldehyde spirits, corresponding to a carbide requirement of nearly 7,000 tons per month.

There will be a tripling of the allotment for the hydrogenation of ethylenes. Ludwigshafen, Schkopau, Gendorf and Auschwitz will produce ethylene oxide from hydrogenated ethylenes; Ludwigshafen will also prepare polystyrol from ethylbenzol.

The increase of carbide allocation for acetaldehyde, in addition that for the buna units, seems to be remarkably small, i.e. from 23,000 only to 28,000 tons per month of carbide, in spite of the prevailing great scarcity of aldehydes. But it must be borne in mind that:

1. A supplemental supply of aldehyde will come from acetylene made by the Hills arc light process.
2. The planning for Buna may presently prove to have been too broad.

3. Furthermore, it must be kept in view that a shift of the aldehyde balance may come about through the substitution of acetic acid made from carbon monoxide and methane.

That in the future the possibility exists that there may be a readjustment of the current liberal allocation to the export units.

The concluding items may be mentioned only.

For "excess chemistry" there appears to be 4,500 tons of carbide per month. This includes the requirement for the acrylonitril plants at Ludwigshafen and Leverkusen using the new process of recovery from acetaldehyde and hydrocyanic acid.

A further 4,500 tons per month will be allocated at Schkopau for hydrogenation of ethylene to produce lubricating oil 55900. 3,500 tons per month for carbon black at the BStW (Bayerische Stickstoffwerke), 2,700 tons per month for welding and cutting, leaving a balance of 5,000 tons per month for calcium silicide, export, and "within-plant" use.

In all, there appears to be a future allocation, and therewith a demand for carbide, amounting to 210,000 tons per month. The meeting of this requirement as to capacity is provided for in the current expansion and the additional planning of the R.W.A. (Reichamt für Wehrschaff Ausbau).

The current expansion, which is either now in progress or at least firmly provided for, contemplates the following:

Schkopau will increase its capacity to 8 furnaces, producing at least 34,000 tons of carbide per month.

The installed capacity which depends on water power at Burghausen is considerably greater, but the effect of a dry winter was to reduce the throughput to 5,000 tons per month.

The I.G. affiliated Chemische Werke Auesig can produce 2,500 tons of carbide per month at Falkenau in an obsolete installation made up of several small furnaces.

Amongst the outside group of concerns, Viag (I think this means the Reich controlled "Vereinigte Industrie-Unternehmungen A.G." - Tr.) stands first with its plants at Hart (15,000 tons per month) and Königshütte (13,500 tons per month). The situation with respect to the Hart plant, which depends on water power, is the same as that at Burghausen, the relatively dry winter lowered the production to

Next follows Lönze with the plants:

Waldshut (water power).....6,000 tons/mo.
Spremberg (coal power).....2,000 tons/mo.

Degussa (Deutsche Gold- und Silberscheideanstalt - Tr.), with its plant at Wyhlen (water power, 1,000 tons of carbide per month), and a group of small plants with a combined carbide capacity of 6,150 tons per month.

Greater Germany has at present a total capacity of about 120,000 tons per month, which will turn out 110,000 tons of carbide at something over 90 per cent full production.

(A paragraph of the document is omitted, here, inasmuch as it refers to the charts which are not here included. Tr.)

Cyanamide production in the last year went down to about 26,000 tons per month of carbide equivalent, and remains at present at this level.

In second largest user is Buna. Schkopau produced 5,000 tons per month of Buna up to May, 1942, and thereby utilized its full output. Five thousand tons of Buna per month correspond to 10,000 tons of acetaldehyde per month, which, in turn, correspond to 20,000 tons of carbide per month. The remaining 3,000 tons per month went into styrol components. At Schkopau, ethylene or ethylbenzol is prepared by the hydrogenation of acetylene which is generated from carbide.

Next follows an allotment of 6,000 tons of carbide per month for vinyl products and chlorinated hydrocarbons. This includes the present carbide requirements for vinyl chloride, vinyl acetate, vinyl ether, oppanol C and Korezin and also for chlorinated hydrocarbons produced by the I.G. and outside firms.

Five thousand tons per month of carbide will shortly be required at Gendorf for the manufacture of hydrogenated ethylene in the production of 1,500 tons per month of ethylene oxide.

The "in-plant" use of the Gendorf aldehyde plant will be about 2,300 tons of carbide per month for the manufacture of acetaldehyde, in addition to that used in the buna units. This corresponds to 11,500 tons per month of aldehyde which, after deducting the allotment for buna, leaves, at most, 2,000 tons per month from the combined production of the Knapsack, Mfkenberg, Burghausen, Waldslurt and Gendorf plants.

For acetylene black the BStW (Bayerische Stickstoffwerke) allots 2,000 tons of carbide per month.

For welding and cutting purposes, 20,000 tons of carbide per month will be required.

A remaining quantity of 5,000 tons per month serves for the preparation of calcium

| Name of Manufacturer | Location of Works, etc | Carbide Capacity 1937 in |
|--|------------------------|--------------------------|
| 7. Wiede's Carbide- werk Freyung m.b.H. | Freyung W-13 | 10,000 |
| 8. Carbidewerke Wilhelm Vogelsang | | |
| 9. Gräflich Schaff- gotts'sche Werke | Bobreck Gleiwitz | 10,000 |
| 10. Elektrochem- ische Werke | e, Waldschaffau | 10,000 |
| 11. J.G. Farber Industrie | Ludwigshaf | 4,000 |
| | | 120,000 |
| Total: | | 1,015,000 |

In the above-listed plants there are some which do not appear in a list prepared by the I.G. in 1942. These are: The Rheinische Westfälisches Elektricitätswerk plant supposed to be at Knapsack; the Wacher plants supposed to be at Lechbruck and Techschmitz; the Carbidewerke Wilhelm Vogelsang plant supposed to be at Hurst a.d. Ruhr; and the Gräflich Schaffgotts'sche Werke, G.m.b.H., plant supposed to be at Gleiwitz.

In the text of "The Chemical Industry" cited above, the statement is made that the output of carbide in 1937 was estimated to have been about 900,000 tons, i.e. somewhat less than the capacity estimate shown in the table. The report states that the 1943 production is thought to have exceeded 2,750,000 tons. The evacuated document, to which reference will be made below, gives a better picture of the carbide situation in Germany during the war.

II. The Situation as of May, 1942 and Plans for Expansion.

A very complete review of the situation as of May, 1942, the expansion then definitely scheduled, and the final production goal set by the R.W.A. are revealed in an evacuated document bearing the title "Carbid-1942-1945".

There follows a free translation of most of the text of this document and copies of the tables. The charts have not been reproduced inasmuch as they may be reproduced readily from the tables.

I.G. Farbenindustrie Aktiengesellschaft Ludwigshafen

Internationale Products Group

Ludwigshafen a/R

May 10, 1942

Dr. Atw

Review of the Carbide Situation

(The first two paragraphs of the document refer to the charts and are omitted here inasmuch as the charts have not been included. The charts may be reproduced from the tables included herewith. Tr.)

*Within the strictly I.G. part (of the total) are the Knapsack and Schkopau plants, Knapsack has a capacity of 25,000 tons per month. Schkopau has six large furnaces, which should have given it the same capacity (as Knapsack). But during the month of 1942 one furnace was out of production undergoing repairs, lowering the Schkopau capacity to about 21,000 tons per month.

The BStW (Bayerische Stickstoffwerke) has a carbide capacity of 17,000 tons per month in the Piesteritz plant.

The portion of the Wacherchemie is 13,500 tons per month from two plants, namely:

Burghansen.....5,000 tons/mo.
Mückenburg.....8,500 " "

CONFIDENTIAL

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I. Situation in 1936-38

The printed report of the "Economic Advisory Branch of the Foreign Office and Ministry of Economic Warfare" (British) entitled "Economic Survey of Germany. Section J. The Chemical Industry", includes a brief part regarding calcium carbide production and uses in Germany and Austria.

The following table is made up from the above-named report, so indicating the situation in 1936.

Carbide Plants in Germany Proper
(About 1936)

| Name of Manufacturer | Location of Works, etc. | Carbide Capacity 1936 in tons |
|---|--|-------------------------------------|
| 1. A.G.für Stickstoffdünger. | Knapsack, near Cologne. | 250,000 |
| 2. Bayerische Stickstoffwerke, A.G. | Piesteritz, near Wittenberg. | 350,000 |
| 3. Carbidfabrik Wylen, G.m.b.H. | Wylen-Baden | 10,000 |
| 4. Ionzawerke Electrochemische Fabrikan, G.m.b.H. | Waldslurt- Spornberg | 100,000 |
| 5. Rheinische Westfälisches Elektrizitätswerk. | Knapsack, near Cologne | 50,000 |
| 6. Dr. Alexander Wacker, Gesellschaft für Elektrochemische Industrie | Lochbruck Burghansen Tachschnitz Mückenberg | 70,000 |

File XV-52

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PRODUCTION AND USES OF CERAMIC

INDUSTRIAL MATERIALS

HARRY A. CURTIS, U.S.

on behalf of the

U.S. Technical Industrial Intelligence Committee

[1945]

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

NOTE: The Publication Board, in approving and disseminating this report, hopes that it will be of direct benefit to U. S. science and industry. Interested parties should realize that some products and processes described may also be the subject of U. S. patents. Accordingly, it is recommended that the usual patent study be made before pursuing practical applications.

22 p.

TANNING CHEMICALS Gerbstoff G, Tanigan U N

TANIGAN EXTRA A tanning material produced by condensing di-oxydiphenylsulfone with ligninsulpho acid and formaldehyde.

TANIGAN (SK SK7 HN HZ G GB etc)(Ref CWS 4004) Tanning agents.

TARBUN (Trilon or T-83) Poison gas.

TEDESCAN A lac made from abietic acid B powder, maleic acid hydrate and pentaerythrite.

| | | |
|--------------------------|------------------|---------------------------|
| <u>TEXTILE CHEMICALS</u> | Emulphor MW | Ramasit BK |
| | Alipon B | Phenylmepasin |
| | Chrombeize MG | Scromin FL, FL, FEB, etc. |
| | Igepol M,B,C,W,F | Persistol N 940 |
| | Chlormepasin | Dewanil Zn 1674 |

TEXTILVEREDLUNGSMITTEL MA 424 46% of a condensation product of 1 mol of melamine with 4 mols of formaldehyde and 1/3 mol of triethanolamine. 54% water.

THIODIPHERYLAMIN S Crude phenothiazin.

TONALON

TORALIT SM 18 Styrene, methyl, acrylate and acrylic acid polymer.

TRIPEN (Ref CWS 3994) Hexachlorbutene.

TROLIT (Ref CWS 3937) A molding powder made from cellulose acetate and a plasticizer such as dimethyl phthalate.

TYLOSE (Ref CWS 3996) Cellulose-O-CH₂COONa. Same as Coloresin.

ULSTRASAN

URESIN B Plasticizer for nitrocellulose, made from urethane and formaldehyde.

VB-EMULSION 1/1 Made from a 1 to 1 mixture of vinyl acetate and acrylic acid butyl ester.

VESDICKUNGSMITTEL T 724 Made from vinyl methyl ether plus vinyl octodecyl ether.

VINIDUR Polyvinyl chloride without plasticizers.

VINOFLEX Same as Igelit PC, i.e., highly chlorinated (65%) polyvinyl chloride. Used in making non-flammable lacquers.

VINOFLEX S A relatively low molecular weight not rechlorinated polyvinyl chloride. Same as Igelit PCU except less polymerized.

VISCOL S Na Sodium salt of Viscol S.

VISCOL S NH₄ The ammonium salt of Viscol S.

WACKER 83 Insecticide containing copper and fine sulphur.

WASHING CHEMICALS Kalunit A,B, etc. Lu 3/115A, Igepal Na, etc.

WEICHLARZ XF A condensation product of xylol and formaldehyde.

SOFTENING AGENTS FOR SYNTHETICS

Weichmachungsmittels (with various designations)
Teolan P
Formilid
Diacetine H
Triactin H
Weichmacher (with various designations)
Dellatol

WEICHMACHER 77 Thiodiglycolic acid dibutyl ester.

WEICHMACHUNGSMITTEL 102 and 102a From glucose and butylene glycol.

WETTING AGENT 2406 (Ref CWS 3996) Wetting agent used with insecticides. Tri-isobutyl-beta naththyl polymerized with 10 molecules of glycol ether.

WOFATT KN A water softener made from "aldehyd disäure".

X-STOFF (Ref CWS 3997) Tetranitro methane

X F S HARZ Condensation product of xylene benzosulphamide and formaldehyde.

Z-STOFF (Ref CWS 3922) Sodium or calcium permanganate.

POROFOR 505A Azo dicarbonamide

POROFOR 476 Azo dicarbonic acid di-ethyl ester. Used in buna.

POVIMAL (with various designations) Polymers used as additions to acetate silk and other synthetics.

PROVIMAL A E, MA, AA All made from maleic acid anhydride with AE for vinyl formate, MA for vinyl methyl ether. AA for vinyl ethyl ether.

PROVIMAL C Mixed polymer of maleic anhydride and vinyl chloride.

PROVIMAL St Same as Povimal C except that styrene is used instead of vinyl chloride.

PROVIMAL EE Made from vinyl acetate and maleic acid anhydride. Used in making Provimal silk.

PVA-OEL (K-Wert 21-14) Made from vinyl ethyl ether.

PVJ OEL Made from vinyl isobutyl ether.

PUM OEL Made from vinyl methyl ether.

REGENERAT II

REFELIT A tar acid resin made by Siemens-Schuckert

RESIN A C (22-22-1) Made at Huls from acetophenone, formaldehyde and methanol.

RESIN S A X (Curtis and Folger 22-22-1) Made from styrene, acetophenone, xylene and hydrofluoroboric acid.

RESINIT A tar acid resin made at Raschig plant.

VIGOPAS Another resin from same source.

VIGORIT Ditto

RESOLE

RESOPAL A tar acid resin made by Siemens-Schuckert.

RIZIMAL Rizinusöl plus maleic acid anhydride. Used as a bonding material for sandpaper.

RONILLA Polymerized styrene.

SARIN Poison gas.

SB-SALZ O-sulpho benzoic acid

SCHAUMPULVER I.B. (Ref 22-23-9)

24.4% Igepon AP extra conc
30.9% Sodium bicarbonate
44.7% Benzoic acid

SELINON (Ref CWS 4001) Insecticide.

SELLOXINE (1,13,52 etc) (Ref CWS 3992) Material for making cloth protective against mustard gas.

SERVITAL AC CONC (Ref CWS 3996) Lubricant for acetate silk yarn.

SERVITAL OL CONC (Ref CWS 3996) Textile lubricating oil.

SILIRON

SOLAGEN A dryer for oils. The manganese or cobalt salts of higher fatty acids from the Fischer-Tropsch "Kogasin".

SCHWEFELÖL Dimethyl disulphide.

SOFTENERS-PLASTICIZERS

Teolan P Triacetin H and
Formanilid many number
Diacetin H "Weichmacher"

SOLVENT A (Made at Huls)

SOLVENT AH (Made at Huls)

SCROMIN FFB Elyester of adipinic acid

SCROMIN SG (Ref CWS 3996) Stearyl poly (6 molecules) glycolether.

STABILIZATOR D B G Polymer of di isobutyl phenol and epichlorhydrine.

STABILISATOR 5 2 phenylindol

STYRONIL (10,20, etc) Made from polystyrene with the indicated proportion of acryl nitrite.

STYRONAL B (10,20, etc) Mixed polymer from styrene and indicated proportion of maleic acid butyl ester.

SULFASTHAN The methyl oxyethyl amide of methane sulphonic acid.

T-STOFF German name for concentrated H₂O₂ (82%)
Neutralin } Other code
Subsidol } name for 80-86%
Aurocl } H₂O₂

NITRILE V6 An oil resisting polymer made from equal parts of acrylic acid nitrile and isohexylic acid vinyl ester.

NITROSO T32 4-Nitroso-mo-oxothyl ethyl aniline chlorohydrate 194.

NITRYLURE For dinitrodiphenyl carbonic acid.

NIVOSOL

NOVOACK

OPM-HARZ Condensation product of dipentene with phenol.

OPPANOL B Polyisobutylene.

OPPANOL C Polyvinyl isobutyl ether.

OPPANOL U Isobutylene copolymerized with isoprene of butadiene.

ORCINOL A small flying agent from a mixture of crude alcohols, C₂H₅OC₂H₅ 16.

P B N (Ref 22-22-7) Phenyl beta-naphthylamine. An anti-oxidant.

PAINTS AND OTHER PROTECTIVE COATINGS Luvinit.

PALAMOLL Polymer of fumaric acid diethyl ester plus butadiene.

PAPER CHEMICALS Bapierleim A, B, etc. Harz 3925.

PARALEUKANILIN 4-4'-4" - triamido triphenyl methane.

P B N Phenyl-beta-naphthylamine.

PERREN An isomerized natural rubber.

PERBUNAN Mixed polymer of 75 parts butadiene and 25 parts acrylic acid nitrile.

PERCACHINON (Ref CWS 3997)

FLUORILES

LUMOGENES Chemical used to detect mustard gas.

PERDURAN G BB. Diphenyl ether condensed with Naps.

PERDURAN H Dichloroethyl formaldehyde hydracetal plus Naps.

PERFUROL Made from methylene chloride.

PERLON Perlon B is probably same as Igamid B, i.e., a variety of nylon.

PERVINAN Polymer of vinyl chloride with vinyl acetate or maleic ester.

PERISTON A synthetic material used as blood plasma.

PHONOLITH

PHOSPHOSTRITAN Tri phenyl triphenyl methyl phosphonium chloride.

PLANT-PROTECTING CHEMICALS AND INSECTICIDES Netzmitt 2377 N

PLASTICIZER III Prepared from butyl crotonate, hydrogen sulphide.

PLASTIKATOR 32 A sodium polymerized butadiene as is also Buna 85.

PLASTIKATOR 88 The butyl ester of methylene dithioglycolic acid.

PLASTOMOLL SW Vinyl methyl ether.

PLASTOROBB UKK A softener made from coal tar.

POLEFTINAL

Lucrylan (200, 300 etc) } These are all emulsions of vinyl isobutyl ether, methyl acrylate with acrylnitrite.
Collacrel
Todescan

POLMIN Aqueous solution of polyethylene amine.

POLVAL (50 und 90) Polyvinyl alcohols
VINAROL

POLYSOLVAN (with various designations) All are solvents as the name suggests.

POLYSOLVAN AO A lac solvent made from chloroacetic acid and Polysolvan A.

POLYSOLVAN HS Made from isobutyl oil (Leuna oil).

POLYSOLVAN A Made from isoamyl alcohol.

POLYSOLVAN J Made from crude isobutyl alcohol.

POLYSTAL (Ref 22-22-16) Adhesives made from Desmodur T and Desmophen 1200 used in making plywood.

POLYSTAL H 1 Solution of Desmophen 900 in acetic ester.

POLYSTYROL E H Made from 50 parts styrol, 25 parts acryl nitrite and 25 parts of vinyl carbazole.

POLYSTYROL EN (70-30) Made from 70 parts styrol and 30 parts acryl nitrite.

POLYSTYROL 30B Made from styrene monomer with acrylic acid butyl ester.

POLYURETHAN 4153 B (Igamid U) Condensation product from hexandi isocyanate and 1,4 butylene glycol.

MAPRENAL MJB (Ref CWS 3957) Condensate of melamine and formaldehyde in butanol.

MAPRENAL BG (Ref CWS 3957) Condensate of benzoguanamine with formaldehyde.

MARFANIL (Ref CWS 3756) $\text{NH}_2\text{SO}_2\text{C}_6\text{H}_4\text{CH}_2\text{NH}_2\text{HCl}$

MAPASIN Resin of the phenol - formaldehyde type

ME 1700 (Ref CWS 3756)(3892)(Cl-C₆H₄)₂ CH CH Cl₂

MEPASIN (Ref CWS 3996) Intermediate made from Kogasin II and subsequently converted to Mersol.

MEPRANOL Melamine-formaldehyde condensation product. I.G.Barb.

MARKAPTO E Mercaptobenzene thiazol for "Englebert" shaped with Bulkazit DM

MERSOL (Ref CWS 3996) Raw material for making mersolates, which is a soap substitute. Mersol is made from Kogasin II, which is one of the higher boiling fractions of the Fischer-Tropsch oil.

METHYL STOFF (Ref CWS 3997) Aluminum chloromethyl.

MIPOLAM (Ref 22-21-3) Several types:

- I. Plasticized polyvinyl chloride
- II. Copolymers of polyvinyl chloride and acrylic esters
- III. Polyvinyl chloride and maleic esters.

MITTEL MA 20% Made from acetocyanhydride.

MITTEL S NA 100% A polymer made by the sodium polymerization of acrylic acid.

MORPHOLON 3,4,3', 4', dimorpholin diphenyl.

MOSQUITO REPELLENT 50/181 (Ref CWS 3892) Trichloroacetyl, chloroethylamines, $\text{C Cl}_2\text{CONHC}_2\text{H}_4\text{Cl}$

MOWILITH 20,30,50,70,90 (Ref CWS 4004)

MOWILITH 20,30,50,70,90 etc. (Hasche) Polymers of vinyl acetate with various proportions of propion-aldehyde. The 20 and 30 varieties are mass polymers, the 50,70,90 are emulsions polymers, the emulsifying agent being a low polymer sodium polyacrylate or polystyrene.

MOWILITH A B (Hasche) Emulsion polymer of 85% polyvinyl acetate and 15% polyvinyl benzoate. Use as a water repellent in treating paper.

MOWILITH ABC (Hasche) Polyvinyl acetate 81%, polyvinyl benzoate 15%, crotonic acid 4%. Mixed with phenol and used in coating tin cans.

MOWILITH D 300 (Hasche) Copolymer of 70% vinyl acetate and 30% vinyl chloride. Emulsion in water, compounded with pigments and plasticizer, used as washable paint.

MOWITAL (Ref CWS 4004)

| | | |
|---------|---------|--|
| MOWITAL | NNF | } These are all made from Mowiliths, the first letter indicating the Mowilith used. The second letter refers to aldehyde used, i.e., F is formaldehyde; A is acetaldehyde; B is butyraldehyde; E is a Mowilith emulsion. |
| | NF | |
| | NB NNF | |
| | NNB HF | |
| | HB EF | |
| | MxB ExF | |
| | HxF | |
| | MNA | |
| | HA | |
| | ED | |

NEKAL Sodium dibutyl naphthalene sulphonate, emulsifying agent used in compounding rubber.

NEKAL BX (Ref CWS 3829)

NETZMITTEL A (Intravan A) Sodium isohexylate, a fire protection material.

NEUTRIGAN Used in tanning. Tamol K, used in tanning.

NIBRENWACHS D 130 L II Hexachlor naphthalene mixed with Luvican - molding material.

NIBRENWACHS R N 88 Crude tetrachloronaphthalene.

NITRACYL 4 nitro, p-aceto, diphenyl.

NITRIL A H Z M-amido cinnamic acid nitrile.

NITRIL M G U An oil resisting material made by polymerizing equal parts of acrylic acid nitrile and methyl glycol vinyl ether.

NITRIL N H Z m-nitro-a-chlorhydrocinnamic acid nitrile.

LUCEX (Ref CWS 3756, 3892) Insecticide produced by ethylating chlorobenzene and chlorinating the side chain in light.

LUCYONAL (With various designations) Mixed polymers used in making lacquers and other synthetics.

LUCRONAL JFM (50-30-20 etc) Polymer from isobutylene or vinyl chloride with the indicated proportion of fumaric acid dimethyl ester.

LUCRYLAN I

ACRONAL)
COSIALGRUND D) All these are apparently polyacrylic
COSIALGRUND D) acid methyl ester. The I refers to
APPRETAN A) carbon atoms.

LUCRYLAN IV

ACRONAL IV) Polyacrylic acid butyl esters.

LUPOLEN N (I.G. WACHS A) A relatively low molecular weight polyethylene.

LUPRENAL Mixed condensation product of adipinic acid,
PASTOPAL trimethylol propane and urea, formaldehyde resin.

LUPRETIN

LURAN

LUTONAL 13 and 5 Polyvinyl isobutyl ether. If a number is given in the name, this is the K value.

LUTONAL M

LUTONAL A

LUTONAL MV Mixed polymer from vinyl methyl ether plus "wax alcohol" vinyl ether.

LUPAMID A (Igamid A) Condensation product of adipinic acid and hexamethylene diamine.

LUPAMID 13 (Perluran, Igamid 13) Condensation product of e-aminocaproic acid lactam.

LUAPMID 6A (Igamid 6A) Mixed condensation product from hexamethylene, adipinic acid plus e-aminocaproic acid lactam (LuranN)

LUPAMID 5A (Igamid 5A) Mixed condensation product from hexamethylene, adipinate and caprol lactam.

LUPAMID 85B, 70B, (Igamid 85B, 70B) Mixed polymer from e-aminolactam plus ketopimelinic acid hexamethyl diamine.

LURAN-N E-aminocaproic acid lactam

LURISIN F PT Condensate of ammonium sulphate, formaldehyde and acetaldehyde.

LUVICAN Name used either for polyvinyl carbazole or a mixed polymer with styrene.

LUVIMAL (With various designations) All are polymers of vinyl chloride with various other materials. The number in the name indicates the percentage of the second component. LL 10-20-30 etc. is the mixed polymer of vinyl chloride with maleic and "Lenua" ester. AG (10,15 etc) is with maleic ethyl glycol ester. BG (10,15 etc) is with maleic butyl glycol ester. MM (10,15, etc) is with maleic methyl ester.

LUVIMAL (with various designations) Mixed polymers in which vinyl chloride used as one of raw materials. Used in making lacquer and other synthetics.

LUVIMAL FM 20, FJB 20. ^{FMG 20} Made from 80 parts vinyl chloride polymerized with 20 parts of FM, ie, fumaric acid methyl ester; of FMG, ie, fumaric acid methyl glycol ester; of FB, ie, fumaric acid butyl ester; or of FJB, ie, fumaric acid isobutyl ester.

LUVIMAL MG Mixed polymer from 80 parts vinyl chloride and 20 parts maleic acid methyl glycol ester.

LUVIMAL BI (10,15 etc) Mixed polymer from vinyl chloride with indicated parts of maleic acid isobutyl ester. Luvimal B is from butyl instead of isobutyl ester.

LUVIMAL JP 18 Made from 82 parts of vinyl chloride and 18 parts of maleic acid di-isopropyl ester.

LUVIGAN Polymerized vinyl carbazole m.p. plus 200°C, M150 is pure polyvinyl carbazole. M125 is vinyl carbazole polymerized with small proportion of styrene. M100 is vinyl carbazole polymerized with 20% styrene.

LUVINIT A paint material made from 1 to 1 mixture of acrylic acid methyl ester and isohexylic acid vinyl ester.

LUZIMAL B2 (10,20 etc) Mixed polymer from vinyl chloride and indicated proportion of maleic acid benzyl ester.

MEDIALAN A (Ref CWS 3996) Essentially the sodium salt of the condensation product of oleyl chloride and sarcosine.

MAPRENAL A.G. A 66% solution of isobutylene of a condensation product of one benzoguaname with four molecules of formaldehyde.

IGEVIN D J Mixed polymer from vinyl isobutyl and dekolo1 vinyl ether.

IGEVIN J Polyvinyl isobutyl ether

IGEVIN M Polyvinyl methyl ether

INSECTICIDES (Ref CWS 4004)

| | |
|-----------|------------|
| Gralit | Brassian |
| Nosprasit | Bulbosan |
| Vitigran | Tristisan |
| Nitrosan | Dizan |
| Nikopren | Gwodyl |
| Venetan | Raupenleim |
| Brassikol | Gix |

INTRAVAN BK Fireproofing material made from ammonium sulphate.

INTRAVAN NK A fireproofing chemical made from diammonium phosphate.

INTRAVAN U. A fireproofing material made from zinc hydroxide slurry.

IPORNA Urea formaldehyde foam used as insulation - D15 Kg/cu.m. $K=0.22$ (U.S. units) Destroyed by heat but will not support combustion.

KARBIDSPRIT Acetaldehyde.

KAURIT MKF (Ref CWS 3957) Essentially tetramethylol melamine.

KLEBELÖSUNG V 80E Solution of "Desmophen 800 HH in ethyl acetate.

KLEBMITTEL TN A collar stiffener (starch) made from vinyl acetate and vinyl chloride in proportion of 70-30.

KOGASIN (Ref CWS 3828) Fischer-Tropsch fraction boiling in range 230-330°C. Straight chain hydrocarbon mixture with average of 15 carbon atoms per molecule.

KORESIN (Ref 22-22-7) Polymer made from acetylene and p-Isobutyl phenol.

KONDENSITE A tar acid resin. See Bakelit.

KUNSTHARZ G143 Made from phthalic acid, glycerine and adipinic acid.

KUNSTHARZ UX A shellac substitute made from craton aldehyde and acetic aldehyde.

KYBOL

LACQUER RESINS AND LACQUER RAW MATERIALS

Membranit FB 137 (etc)
Alkydal STK, S45, S100 (etc)
Kunsthartz He44, 18 PH, KPM
Weichharz 166 S
Luphen B, AT 50% neu, AMH, BW (etc)
Chlorbuna
Sulphone red

LATEKOLL Ammonium salt of polyacrylic acid - A thickener for latex

LAUSETO-alt (Ref CWS 3756, 3792) An analog of DDT made with 1 mol of chlorbenzol and 1 mol of benzol combined with chloral in presence of sulphuric acid.

LAUSETO NEU (Ref CWS 3756, 3892) $Cl-C_6H_4-CH_2-Cl$

LEATHER CHEMICALS Eukanoltop matt NJ
Eukanoltop I Teig
Eukanolbinder A.J.
Dispergiermittel Z
Lederöl H8 190

LEDERÖL 265 Condensation product of di-ethylbenzene and formaldehyde.

LEDERÖL 266 Ethylated product from Leuna alcohol (160-180°C) with "Weichharz XF". The "Weichharz XF" is a condensation product of Xylol and formaldehyde.

LENOROL ROH Mixture of mono-acid dipropyl dichorbenzene.

Li 1c A filler coating made by polymerizing methyl ethyl vinyl ether and di-ethylene glycol di-vinyl ether with subsequent addition of a resin.

LINAX A tar acid resin made by Siemen-Schnckert.

GESAROL German name for DDT

GIX (Ref CWS 3892) Like DDT except made from flourbenzene instead of chlorbenzene.

GM-1 (Ref CWS 3997) Nitrous oxide

GP 4243 (Ref CWS 3892) Active principle is p-dimethylamino phenyl diazo sodium sulphate, $(\text{CH}_3)_2\text{N}-\text{N}=\text{N}-\text{SO}_3\text{N}$

HARNSTOFF BR 1 methoxy, 2 ethoxy, 5 phenyl, 1 thourea

HOSTAPHEN (Ref. WS 3996) Mepasin sulphonic acid

HOSTALON O B (MOWITAL) Polyvinyl alcohols with cyclohexanone and acetaldehyde or partly acetalized butyraldehyde.

HYDRO Hydrazo diaarmonic acid di-ethyl ester.

IDONIT A tar acid resin. See Bakelit.

IGAMID A (Ref 22-22-7) Equivalent of American Nylon.

IGAMID B (Ref 22-22-7) A nylon-like material made from amino caproic acid.

IGAMID BS (Ref 22-22-7) Of lower mol. wt. than Igamid B, developed for tire cord.

IGAMID CA (Ref 22-22-7) A mixture of Igamid A and B inter-polymerized.

IGAMID U (Ref 22-22-16) A polyurethane of 1,6 hexamethylene di-isocyanate (Desmodur H) 1,4 butenedbl in monochlorobenzene and o-dichlorobenzene.

IGANIL Aniline formaldehyde resin.

IGELIT MP Polymer of 80 parts vinyl chloride, 10 parts di-methyl maleic acid ester, 10 parts diethyl maleic acid ester. Other esters of maleic acid may be used.

IGELIT MP-A 80 parts vinyl chloride polymerized with 20 parts dimethyl maleic acid ester. Stabilized with sodium carbonate and pressed into sheets. Sold under trade name "Astralon".

IGELIT MP-D Same as MP-K except emulsifier removed. Used for dentures.

IGELIT MP-K Copolymer of 80 parts vinyl chloride and 20 parts methyl acrylate. Cable covering.

IGELIT MP-AK Used in making battery jars.

IGELIT MP-S 60 parts vinyl chloride polymerized with 40 parts vinyl acetate.

IGELIT MP-400 Copolymer of equal parts of vinyl chloride and vinyl isobutyl extra.

IGELIT PC (Same as vinoflex) Rechlorinated polyvinyl chloride (65% Cl)

IGELIT PCU (Fest) A highly polymerized but not rechlorinated polyvinyl chloride (55% Cl) Basis for preparing "Vinidur".

IGELIT UJB 16 A cable material (Astralon) made by polymerizing 90 parts of vinyl chloride with acrylic acid isobutyl ester.

IGELIT V2, V6, 10JB, MM, K, DC, Cl. These are all mixed vinyl polymers. V2 is vinyl acetate with vinyl chloride; V6 is vinyl chloride with isohexylic acid vinyl ester; 10 JB is 86 parts of vinyl chloride polymerized with 14 parts of Leuna isobutyl ester; MM is from vinyl chloride with MM ester (50:50); the K is from vinyl chloride with vinyl methyl (80-20); the DC is vinyl chloride with ethylene glycol dicrotonate (80-20); Cl is vinyl chloride with 0.5% of symmetrical dichorethylene.

IGELIT VB 10 A mixed polymer of 86 parts vinyl chloride and 14 parts acrylic acid butyl ester.

IGELIT (VB) MPTYP Polymer of vinyl chloride, acrylic ester, maleic ester, etc.

IGELIT VV A synthetic lac bonding material made from 1 part vinyl chloride and 1 part vinyl acetate.

IGEPAL B,C,F,M,W, Etc (Ref CWS 3996) Washing materials. Typical formula: $\text{CH}_3(\text{CH}_2)_{11}\text{C}_6\text{H}_4\text{OCH}_2\text{CH}_2\text{OCH}_2\text{OCH}_2\text{OCH}_2\text{OCH}_2\text{OCH}_2\text{SO}_2$

IGEPONS Water conditioning chemicals emulsifiers etc. made from sodium sulphates of aliphatic glycols.

IGEPON A

IGEPON T (Ref CWS 3996) Essentially the sodium salt of oleyl methyl taride.

IGEPON AP EXTRA CONC (Ref 22-23-9) Sodium salt of the oleylester of oxy-ethane sulphonic acid containing very little salt. $\text{C}_{17}\text{H}_{33}\text{COOCH}_2\text{CH}_2\text{SO}_3\text{Na}$

IGETEX Igetex S is a 30% dispersion of Buna S

IGEVIN A Polyvinyl ethyl ether.

IGEVIN D Poly-beta-dekalol vinyl ether. Apparently same as Densodrin NH and I.G. Wachs NH

DICHLORD NITROCARBAZOLE FEUCHT 1-8 dichlor 3-6 dinitrocarbazole. An insecticide.

DIKRESYLMETHAN-S A 50% solution in styrene of a resin from phenol oil and formaldehyde.

DIMULGEN A demulsifying agent.

DISMULGAN III (Ref CWS 3996) Emulsion breaking agent.

DISMULGAN IV The sulphuric acid ester of oleic acid and ethyl-anilide.

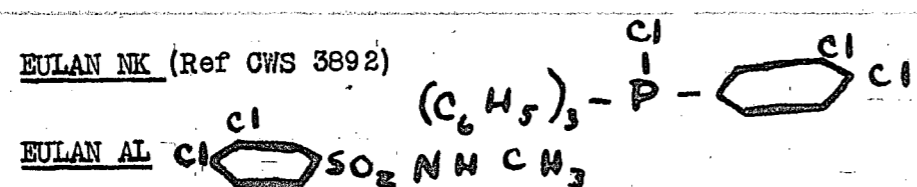
DITHIOSALZ Sodium dithiodiglycolate.

DOLANIN 1-methyl, 4 phenylpiperdin, 4 carbonic acid ethyl ester. An analgesic used in obstetrics.

EULAN (Ref CWS 3892) Moth repellants

EULAN NEU

EULAN NK (Ref CWS 3892)



EULAN CN A moth repellent. Sodium salt of 4,3', 5', 3'', 5'', pentachlor 6,6" dioxotriphenyl methane 2 sulphonic acid.

ELEUBRON SALZ 4 amino benzene sulphamino thiazol.

ELEUDRON (Ref CWS 3756)

EMULPHOR Emulsifying agent.

EMULPHOR A EXTRA (Ref CWS 3996)

EMULPHOR MW (Ref CWS 3996)

EMULPHOR ELW (Ref CWS 3996)

EMULSION MU Made from vinyl acetate and polyvinyl alcohol. 10% solution.

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EMULSION 614 Made from same as above.

EMULSION 683 Made from vinyl acetate and "amphoseife".

EMULSION L:355 Polymer from acrylic acid ethyl ester, vinyl isobutyric ether and styrene.

EMULSION MVIW Made from vinyl acetate, polyvinyl, alcohol and "Palatinol C".

EMP 58/6 Softening material made from polymerized acrylic acid ethyl ester and vinyl ethyl ether.

ESHALIT) Tar acid resins made by Siemens-Schuckert, Werke,
LINAX) A.G.
REPELIT)
RESOPAL)

ETROL (Ref CWS 3994) Ethyl acetate

EXPYROL Z A fireproofing agent made from ammonium sulphate

F-GELBKÖRPER Difurfurilidene acetone. A glycerine substitute.

FIREPROOFING CHEMICALS Expyrol z
Intravan BK, NK and several U's.
Aktivator

FLOTOL A Crude terpentine oil sulphate

FORMOSE A sugar-like condensation product from formaldehyde

FRIGEN Dichlorodifluoromethane I.G. Farbenindustrie
OR
FREON 12

GALLOPHENIN HB BASE 1 oxy-3 anilido-5 methyl-7 dieksylamido-2 phenoxazon-4 carbonamide

GELEVAN Chlorinated Mepasin®.

GERBSTOFF B'S A tanning material made by condensing a mixture of brenzcatechin and homobrenzcatechin with sodium bisulphite and formaldehyde.

GERBSTOFF BO Condensation product of benzene (40%) and oxybutane disulphonic acid with "formaldehyd"

GERBSTOFF KN Condensation product of sulphonated beta-naphthalene with cresol and formaldehyde.

GERBSTOFF NR Condensation product of a naphthalenic acid and benzene (40%) with formaldehyde.

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BUTYLWEICHMACHER N-butylamide of benzoisulphonic acid.

C-ACID Crotonic acid

C OR J₂R STOFF Hydrazine hydrate in methanol-water solution.

CARBOXYSAURE 1 carbethoxyamine, 7 naphthol, 3 sulphonic acid

CASITRIX (Ref CWS 3892) Rat poison.

CELLON (Ref CWS 3937) A cellulose acetate product used for gas masks.

CERESAN (Ref CWS 9001) Insect repellent, active principle is ethoxy ethyl mercuric phosphate.

CLOPHENHARZ ROH NEUTRAL Crude heptachlor terphenyl.

COHAERIT (Bindemittel LI 160) Condensation product of trimethylol propane, hexantriol and adipinic acid.

COLORESIN (Ref CWS 3996) Cellulose O-CH₂ COONa

CORIALOL K Solution of the dye in methyl alcohol.

COSAL W (400) Emulsion of the dye.

COSTALGRUND N Sodium salt of the dye.

CROVOLIT MB, AB, BB, MUC, AVC, BVC, MAM, AAM, BAM, MST, AST, BST.
All these are synthetic lac materials made from crotonic acid esters. The M indicates methyl, A the ethyl ester, B is butadiene B is butadiene and the butyl ester, UC is vinyl chloride, AM is acrylic acid, S ethyl ester, AAM is the acrylic acid ethyl ester, the St is styrene, as an example, Crovolit MST, is a mixed polymer of 25% Crotonic acid methyl ester with 75% of styrene.

CYOMYL 3,4-dicyandiphenyl.

CYSTOGAN (Ref CWS 4001) (CH₂)₂ NCSSNH₂ (CH₂)₂ Insecticide.

FORBIAT Insecticide with same active principle as Cystogan.

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DL210 (Ref CWS 3832) Mixture of 1,2-dichloromethyl phenyl sulphone and 3,4 dichlorobenzyl alcohol. "Beetle killer".

DELLATOL Mixture of 75% "Weichmacher, MMA" and 25% "Butyl-Weichmacher".

DESODRIN (Hasche and Boundy) A low mol. wt. polymer of vinyl isobutyl ether. Used in waterproofing.

DESODRIN NH) Poly-beta-decalol vinyl ether. I.G. WACHS NH

DESODRIN V) "Wax alcohol" vinyl ether. I.G. WACHS V

DESODRIN W Mixed polymer from vinyl octodecyl ether and vinyl coconut oil ether.

DESMODUR H (Ref. 22-22-16) Hexamethylene di-isocyanate.

DESMODUR R (Ref 22-22-16) Triphenylmethane-p-tri-isocyanate.

DESMODUR T (Ref 22-22-16) Toluene di-isocyanate.

DESMOPHEN O-0 Polyester of oxalic acid and "hexantriol".

DESMOPHEN 800S Polyester of trimethylol propane with adipinic and phthalic acids.

DESMOPHEN 100 A condensation product of 3 mols. of adipinic acid 2 mols of 1-4 butylene glycol with 2 mols of Hexantriol H or trimethylolpropane.

DESMOPHEN 300 HH Condensation product of "XF OIL" and "Desmophen 900 HH".

DESMOPHEN 900 (Bindemittel 28) (Ref 22-22-16) Ester of 3 mols of adipinic acid and 4.2 mols of trimethylol propane.

DESMOPHEN 1100 Polyester of 3 mol. adipic acid, 2 mols 1,4 butylene glycol and 2 mols of trimethylol propane.

DESMOPHEN 1200 (Ref 22-22-16) Polyester from 3 mols. adipic acid, 3 mols 1,4 butanediol and 1 mol trimethylol propane.

DESMODUR T Toluene di-isocyanate. (Ref CWS 3832)

DESMODUR H Hexamethylene di-isocyanate OCN-(CH₂)₆-NCO

DESMOSIT C (Ref 22-22-16) 1-chlorophenylene - 2,4 di-isocyanate.

DESMOSIT O Polynexamethylene di-isocyanate. A glue material.

DESMOSIT T 1-2-4 toluylene di-isocyanate.

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ALKYDAL Kn 67/XIII Made from paraffin oxydation fatty acids trimethylol propane, phthalic acid and adipic acid.

ALKYDON 50) Raw materials for lac synthetics made from
ALKYDON 65) "Sardineutrau".

AMIN T32 4 amino-n-oxethyl ethyl aniline 180 - a photographic developer.

AMIDONITROPAME Sodium salt of 1 amino, 4 nitroanthraquinone-2 sulphonic acid.

AMILIN 5 2 amido, 5 naphthol, 7 sulphonic acid.

APPRETAN H (Hasche) Polyvinyl acetate 95%, crotonaldehyde 5% in solution in alcohol, with ammonia added to for m emulsion. Used as lacquer and as stiffener for hats.

APPRETAN M A dye emulsion.

APPRETAN P Sodium salt of compound.

ARIGINAL (Ref CWS 4001) Methyl formate insecticide.

ARUBREN A fire extinguisher made from "Pergut", "Chlophen" and "Niprewachs".

ARUBREN (Nibrenswacher RN 88) Used in rubber industry.

ASPLIT (Ref CWS 4004)

| | |
|-----------------------------|-----|
| p-Toluene sulphone chloride | 10% |
| Silica | 20% |
| Quartz powder | 70% |
| Phenol-formaldehyde resin | 90% |
| Benzyl alcohol | 10% |

Cement for brick and tile

ASPLIT A (Ref CWS 4004)

| | |
|-----------------------------|-----|
| P-Toluene sulphone chloride | 10% |
| Barium sulphate | 70% |
| Quartz powder | 20% |

| | |
|-------------------------|-----|
| Phenol-formaldehydresin | 75% |
| Benzyl alcohol | 5% |
| Triethyl phosphate * | 20% |

* Or dichloropropanol or chlorobenzaldehyde

ASPLIT EI (Ref CWS 4004)

| | |
|--|------|
| P-Toluene sulphone chloride | 6.5% |
| Polymer of vinyl chloride and maleic anhydride | 1.5 |
| Kieselguler | 1.0 |
| Silica | 10.0 |
| Quartz sand | 41.0 |
| Quartz powder | 40.0 |

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ASPLIT EI, cont.

| | |
|-------------------------------|-----|
| Phenol-formaldehyde resin | 75% |
| Benzyl alcohol | 5 |
| Triethyl phosphate (or subst) | 20 |

ASTRALON See Igolit MP-A

AZAMIN 2,4,6, triamino 1,3,5 Eriazin (melamine)

B STOFF OR L.B. STOFF Hydrazine hydrate

BAKELIT

| | |
|------------|---|
| IDONIT |) Tar acid resins made by Bakelit, GmbH |
| KONDENSITE | |
| RESINIT | |

BARDIONAL (Ref CWS 3756) Sulfathiourea

BINDEMITTEL

| | |
|-----|--|
| AG3 | - Trimethylol acetoguanamine |
| AG4 | - Tetramethylol acetoguanamine |
| TL2 | - Condensation product of 2 amino, 4 methyl, thozol, formaldehyde and butanol. |

BINDEMITTEL 28 (Desmophen 900) (Ref 22-22-16) Polyester of 4.2 mols of trimethylol propane and 3 mols of adipic acid.

BINDEMITTEL 28H Corresponding ester.

BINDEMITTEL 38 Polyester of trimethylol propane, adipic acid anhydride.

BLADAN (Ref CWS 3756, 3892) Insecticide $(C_2H_5O)_2PO$

BRAUN-ANIL 4-5 benzo, 3 oxy-thionaphthene, 2 (p-dimethylonnido) aniline

BS-EMULSTON 1/1 A binder made from a 1 to 1 mixture of acrylic acid butyl ester and styrol.

BUNA CHEMICALS Alkydal 149/3, OKC-Harz, Trithinol roh flussig (1,2,4 trichlor, 6 phenol)

BUNA S-111 First quality bunas as made at Ludwigshafen

BUNA S-V Offgrade buna s

BUNA S-A Buna scrap

BUNALIT Chlorinated buna S

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the most part, the glossary on the following pages is a selection of the I. G. names for materials that are actually or potentially of industrial importance. Some of these names, such as nylon, vinillite, asplit, etc., are already familiar in America.

Information as to production of some of the synthetics mentioned in the glossary will be found in an evaluated I.G. document labelled "Kunststoffe und Lackharze" and in various C.I.O.S. reports which have been filed.

After preparing the glossary from the I.G. document "Neue Produkte", mentioned above, the reports of many C.I.O.S. investigators, were searched and the information contained therein was used to supplement the glossary. Where the products were mentioned in a C.I.O.S. final report, a reference has been included. Thus "Ref. 22-22-16" indicates C.I.O.S. Report Item No. 22, File XXII-16. Unfortunately, only a few of the hundreds or thousands of C.I.O.S. reports which will eventually be issued have as yet appeared, and therefore references to these reports are scanty. The London office of the Chemical Warfare Service, Intelligence Division, has to a considerable extent overcome the delay in issuing C.I.O.S. reports by getting out the reports in a ditto form. Where the C.I.O.S. report is not available at present, a reference has been given to the CWS ditto report, i.e., "Ref CWS 3996". Inasmuch as the CWS "quickie" will eventually appear as a formal C.I.O.S. report, it is planned later to issue a correlation of the references between the two series of reports. In a few instances, the information used had not appeared in either a C.I.O.S. report or in a CWS "quickie". In such cases the author of the report from which the information was taken is shown. Later on, all references may be given in terms of C.I.O.S. reports. Where no reference is given, it is to be understood that the information was taken from the German documents mentioned above.

GLOSSARY OF GERMAN NAMES FOR VARIOUS CHEMICAL PRODUCTS

ACRONAL 1,2,4 (Hasche and Boundy) Dispersion of mixed polymer of 1 part methyl acrylate, 2 parts ethyl acrylate and 4 parts of n-butyl acrylate.

ACRONAL B1 (10,20, etc) Mixed polymer from acryl nitrate and the indicated proportion of maleic acid isobutyl ester.

ACRONAL 500-D (Hasche and Boundy) Copolymer of 50% butyl acrylate, 50% vinyl acetate, with 2% free acrylic acid needed. Used for waterproofing textiles. Emulsified with ammonia and used as a paint.

ACRONAL V6 Mixed polymer from equal parts of methyl acrylate and isohexylic acid vinyl ester.

ACRONAL V26 Mixed Polymer from vinyl acetate and isohexylic acid vinyl ester (1-1) equal parts.

ADIPINSAURESESTER The methyl ester.

AGLUTOL 5 Softeners made from lump glucose (Brockenglukose)
AGLUTOL 6

AKARDIT II A stabilizer made from diphenylamine, phosgene, and methylamine.

ALAL (Ref CWS 3994) A liquid catalyst used in preparing ethyl acetaldehyde.

ALBERTOL (Ref CWS 3999) Phenolic resins made by the chemische Werke Albert

ALPHENATE
ALRESENE
ALNOVOL
PHANODURE
DUROPHENALRESAT
ALRESAT

ALIPAL (Ref CWS 3996) Soap substitute

ALIPAL CI 35% (Ref CWS 3996)

ALIPAL D 35% (Ref CWS 3996)

ALIPON OAN 70% sodium salts of ester from the chloride of oxidized fatty acids plus 2 to 4% of soap plus salt.

ALKYDAL K1397, K1388, 1-4, K1365, Lk339/4, K1339/5, K1328/1, K1339/2. All made from phthalic acid, and glycerine, with various oils such as "Tallol", "Läinol" "Holzol" etc. They are the raw materials for various resins.

RESTRICTED

Subject

Page

Product

RESTRICTED

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INTRODUCTION

Amongst the I.G. Farbenindustrie documents taken to London by one of the investigating teams is a quarterly record from all the I.G. laboratories listing all new chemical compounds or new chemical products made. The file containing the record is labelled "Neue Produkte - Meldungen 1935 - ". The period covered is from 1935 to 1944 inclusive. This document has been used as one source of information in the preparing of a glossary of some of the I.G. names for chemical products. No attempt has been made to include all the I.G. chemical names recorded in the document mentioned, but a selection has been made of some which may be found useful to readers of chemical records from I.G. and other German sources.

The naming of complex organic compounds has always been a troublesome matter. National and international agreements have been made with respect to systems of nomenclature, the object of which was so to name complex organic compounds that any organic chemist, on seeing the names, could at once write the structural formulas. The most commonly used system which has resulted from these efforts is a highly informative one but is very cumbersome, so cumbersome, in fact, that thousands of simpler names have been accepted in lieu of the more informative, strictly scientific, names. The simplified names so frequently used by organic chemists usually reveal something of the structures of the chemicals but often leave uncertainties, particularly in the case of isomers.

The I.G. record mentioned above gives the full name of the organic compounds of definite structure, and then gives the simplified name as an "I.G. Name". The I. G. chemists, however, went far beyond the common practice of organic chemists in adopting simplified names, as the following examples will illustrate:

HYDROSAURE is dimethyl tetrahydro diphenyl carbonic acid
ORKRONITRIL is 1 methyl, 2 cyan, 4 chlor, 5 nitrobenzene
NITROPYRAZOLMEKRYLSAURE is 2' methyl, 3' nitro, 5' sulpho
1 phenyl, 3 methyl, 5 pyrazolone
BENZA-GUANAMIN is 4,6 diamino, 2 phenyl, 1,3,5 triazine

ETC.

In the present glossary, very few of the simplified or "I.G. Names" for organic compounds have been included, primarily for the reason that there are literally thousands of such names in the I.G. documents, and at present the writer does not have time to unravel the extremely tangled records. For

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RESTRICTED

GLOSSARY OF SOME GERMAN NAMES
FOR CHEMICAL PRODUCTS

Reported by

HARRY A. CURTIS, U.S.

on behalf of the

U.S. Technical Industrial Intelligence Committee

CIOS Item No. 22
Miscellaneous Chemicals

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

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25 p.

Copy 1

REF ID: A66000

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by authority of
Joint Chiefs of Staff
Col. R. W. ...

REF ID: A66000

SAR: OF SOME GERMAN NEWS
... HISTORICAL ...

... ..

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

17

UNCLASSIFIED

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by authority of
Joint Chiefs of Staff
1 Oct 77

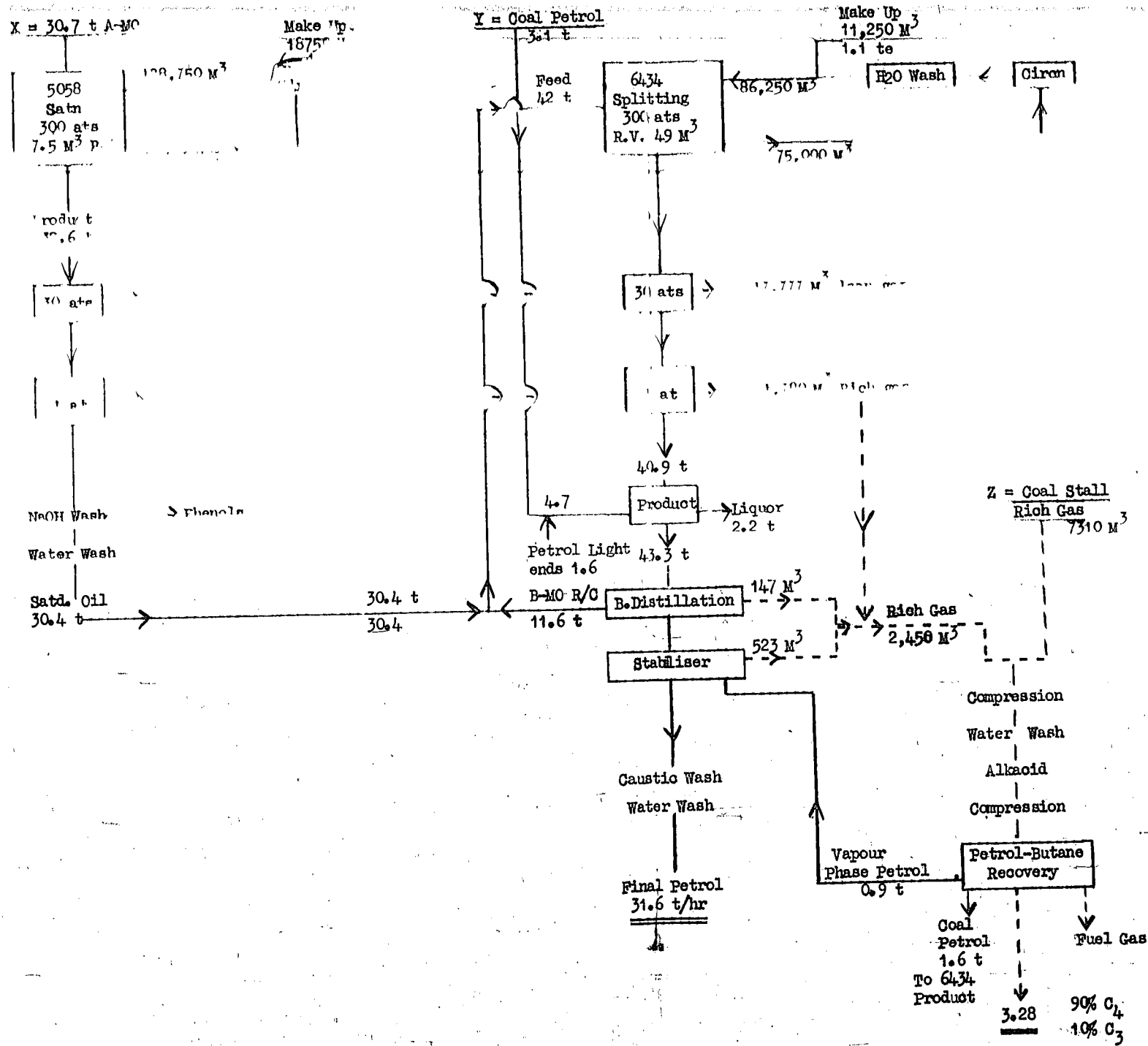
179

GLOSSARY OF SOME GERMAN NAMES
FOR CHEMICAL PRODUCTS

Curtis, Harry A.

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

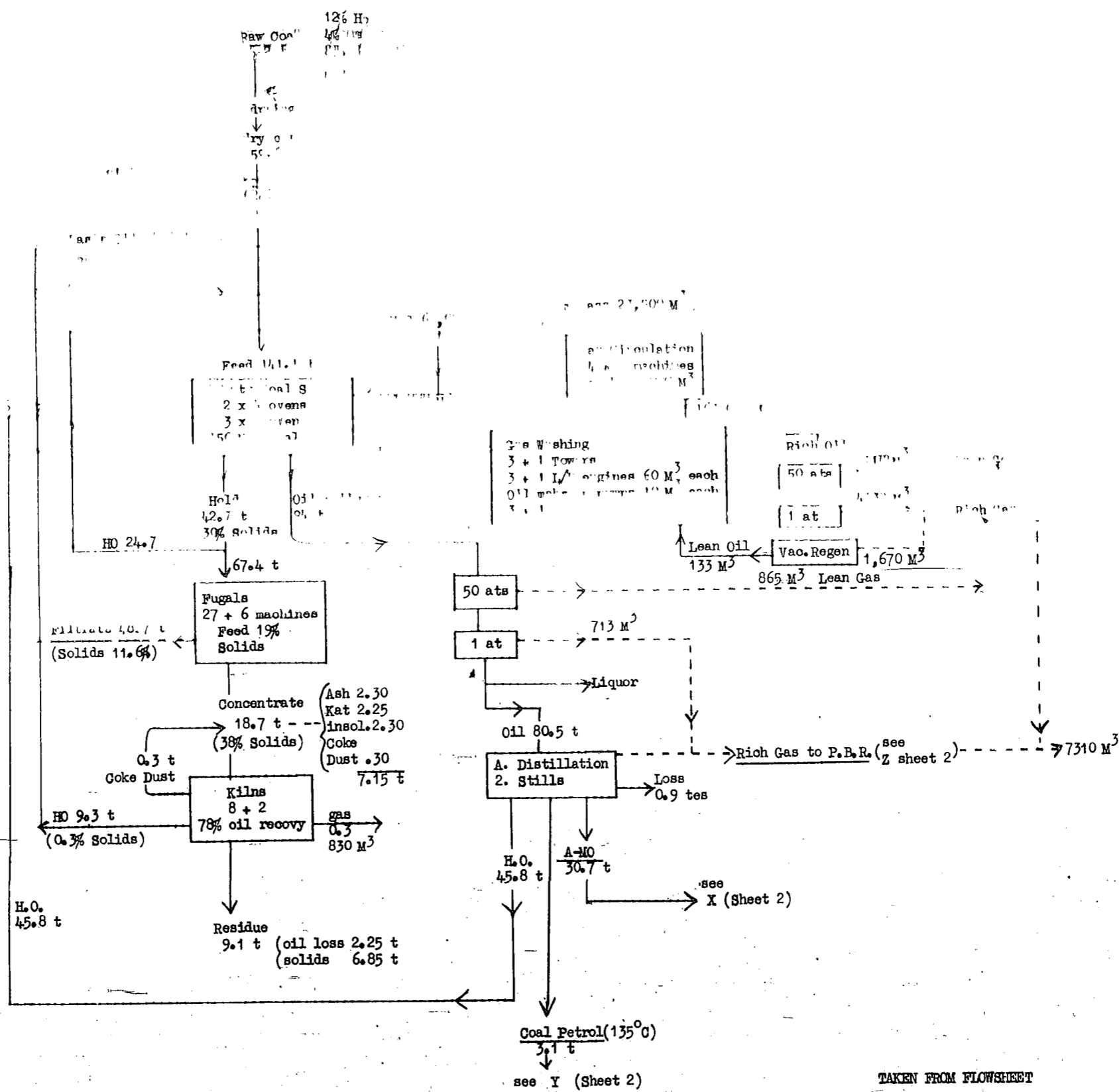
FLWSHEET FOR PRODUCTION OF 250,000 TES/YEAR MOTOR FUEL (330 days)



TAKEN FROM FLOWSHEET
 MA III 2001-2
 10-11-38

Flowsheet II

Flowsheet for Refraction



TAKEN FROM FLOWSHEET
 MA III 2001-2
 10-11-38

IX. SUMMARISED CAPITAL COSTS FOR SCHOLVEN

| | R.M. |
|---|-------------|
| <u>Hydrogen-Production</u> | 31,317,000 |
| <u>Conversion (shells, sludge plant etc.)</u> | 47,052,000 |
| <u>Refinery</u> | 16,678,000 |
| <u>By-products</u> (Phenols, hydrogenation and alcohol wash, Claus plant) | 1,723,000 |
| <u>Services</u> | 15,053,000 |
| <u>Power Plant</u> | 5,712,000 |
| <u>General</u> | 28,333,000 |
| | <hr/> |
| <u>TOTAL</u> | 145,868,000 |
| | <hr/> <hr/> |

FLWSHEET X

(Scholven & ...)

g Bu'ne
C₄ 1.32
C₄ .57
C₄ .16

g Bu'ne
C₄ 0.44
C₄ 7.66

Dehydrogenation

| Feed | Product |
|------------------------|------------------------|
| i C ₄ 0.97 | i C ₄ 0.73 |
| n C ₄ 24.64 | n C ₄ 18.47 |
| <u>0.14</u> | i C ₄ 0.20 |
| 25.75 | n C ₄ 5.23 |
| | C ₅ .14 |
| | <u>24.77</u> |

Conversion C₄ 25%

Iso Butane R/C
i C₄H₁₀ 30.83
n C₄H₁₀ 5.12
C₃H₈ 1.04
36.99

FLWSHEET FOR A.T. (ALKYLATE) PRODUCTION - From MA/SCHO 686-2 28/1/43 All quantities in Metric tons/hr.

35.5 i C₄
19.6 n C₄
.43 AT
4.47 C₃
60.00 tr/hr.

| Alkylation Unit | |
|------------------------|---------------|
| Feed | Product |
| i C ₄ 71.38 | 65.00 |
| n C ₄ 13.76 | |
| i C ₄ 0.20 | |
| n C ₄ 5.23 | |
| C ₃ 5.67 | 5.67 |
| AT .43 | 12.28 |
| C ₅ .14 | |
| <u>126.81</u> | <u>126.76</u> |

36.99 tes/hr.

R/C

66.76 tes
11.95 AT
29.50 i C₄
24.11 n C₄
1.20 C₃
66.76

i C₄ 0.53
n C₄ 16.98
17.51 tes/hr

Pre-Distillation
Butane Separation and Propane Separation

2.04 i C₄
3.87 n C₄
0.10 C₃
6.01 tes/hr.

AT 11.95 tes/hr
54.81 tes/hr
i C₄ 29.5
n C₄ 4.11
C₃H₈

0.18 i C₄
5.88 n C₄
6.06 tes/hr

Isomerisation
Loss 0.05 te/hr.

Residue 0.6 tes. (4800 tes/yr)
To Autobenzene

Redistillation

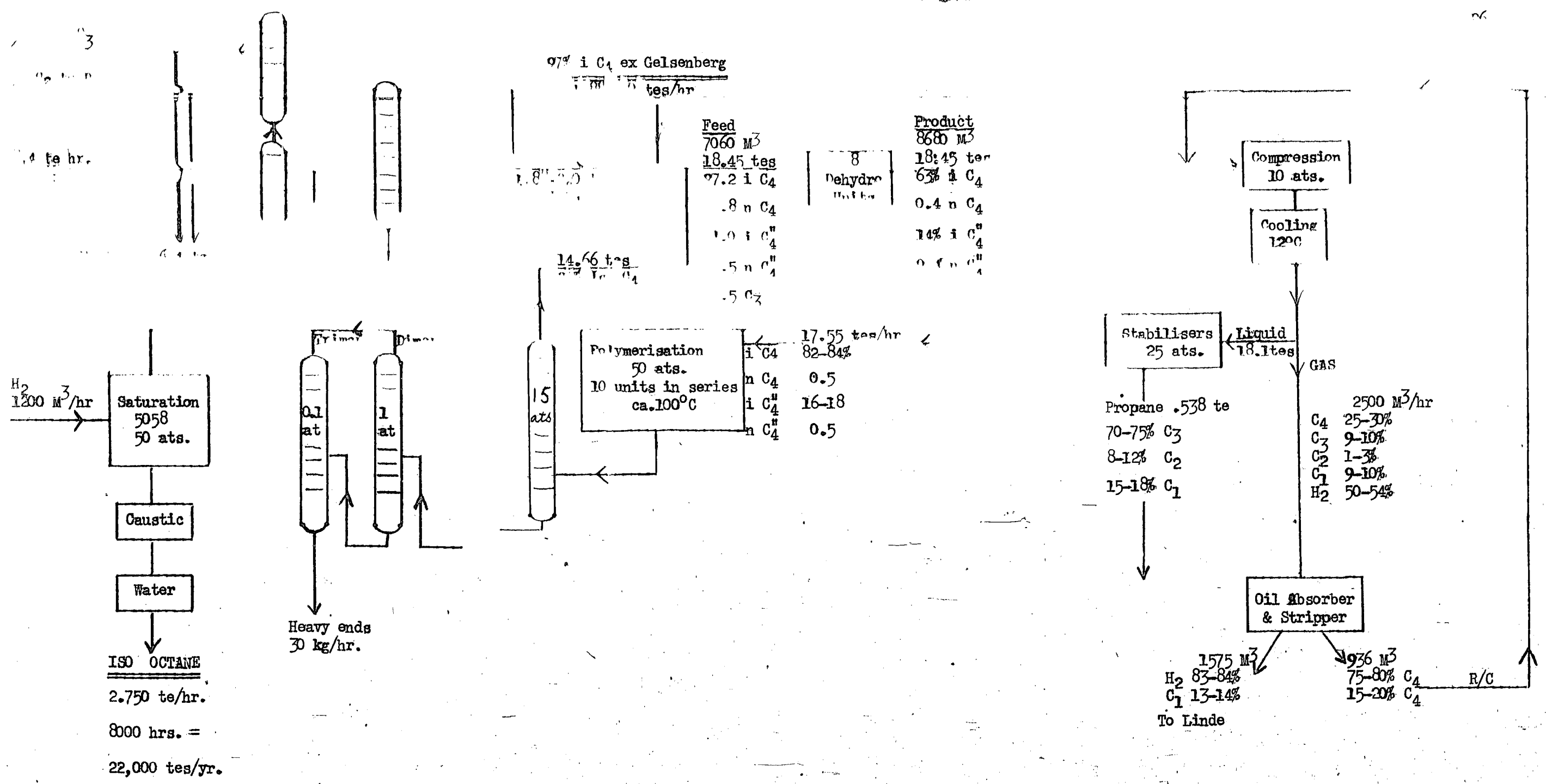
11.35 tes.

11.01 tes/hr
Alkylate
88,080 tes/yr

0.34 tes (2720 tes/yr)
to petrol recovery plant.

During the first six months of 1944, a cold acid alkylation unit was put into operation to replace the polymerisation unit. This was done to get over the limitation on iso-octane manufacture imposed by the dehydrogenation unit. The iso-butane fed to the dehydrogenation unit was replaced by n-butane. There was a shortage of iso-butane and plans had been made to build an isomerisation unit for the conversion of n-butane to iso-butane. Flowsheet X taken from drawing MA/Sche 686-2 dated 28.1.43 shows one scheme. In this case 6.4 tes/hr of n-butane was to be dehydrogenated at 25% conversion per pass and with a mole yield of butylene of 85%. Allowing for the higher conversion some extensions of the dehydrogenation units would have been required. 2 tes/hr of iso-butane was made by isomerising n-butane with a pass conversion of about 3%. In the alkylati

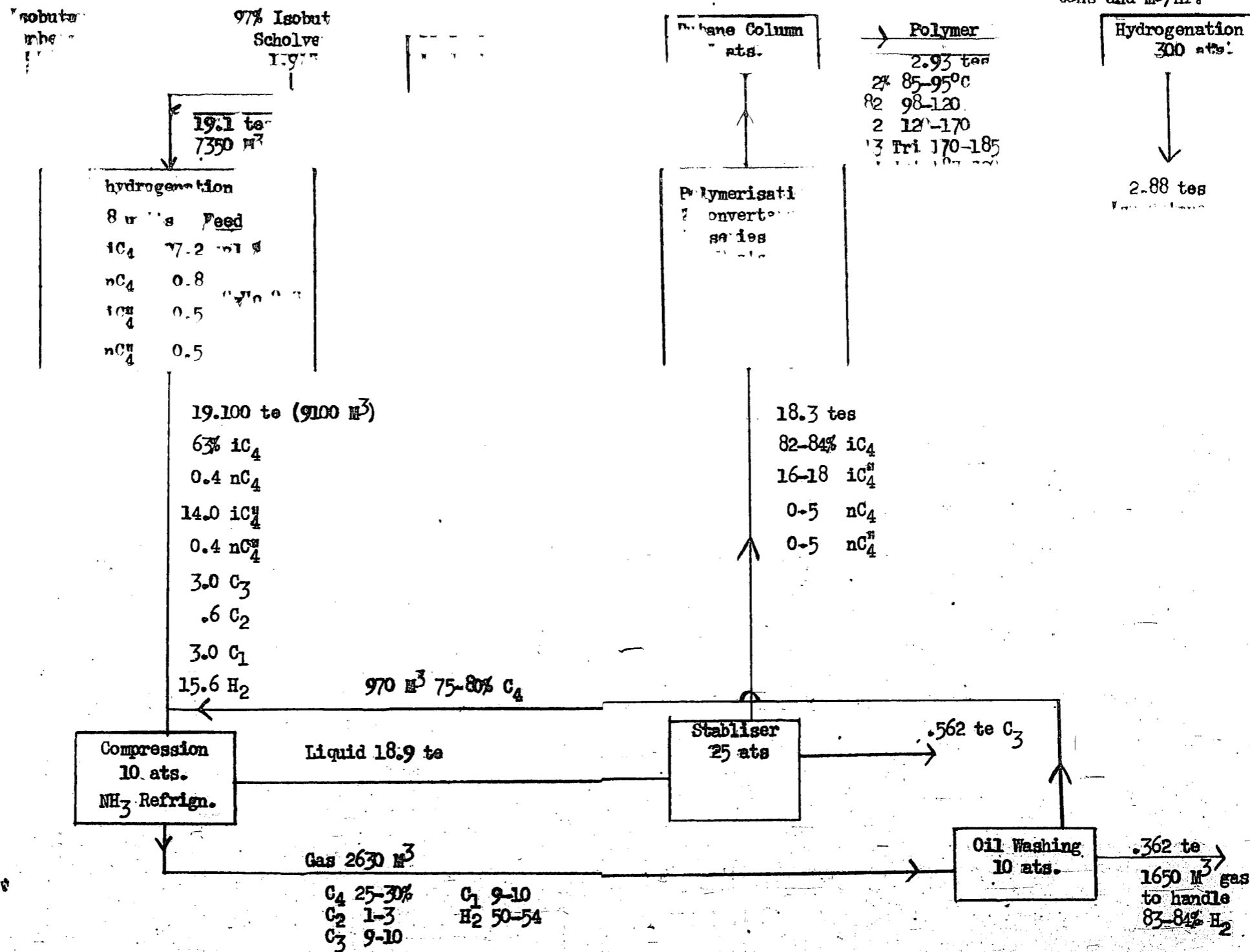
PROCESSES FOR THE PRODUCTION OF ISO OCTANE (continued) dated 16.1.42) All Quantities te/hr or M³/hr



FLOWSHEET VIII

T-52 UNIT FOR 22.0 ... OCTANE FROM MA-SCHO 161-2 1.12.39

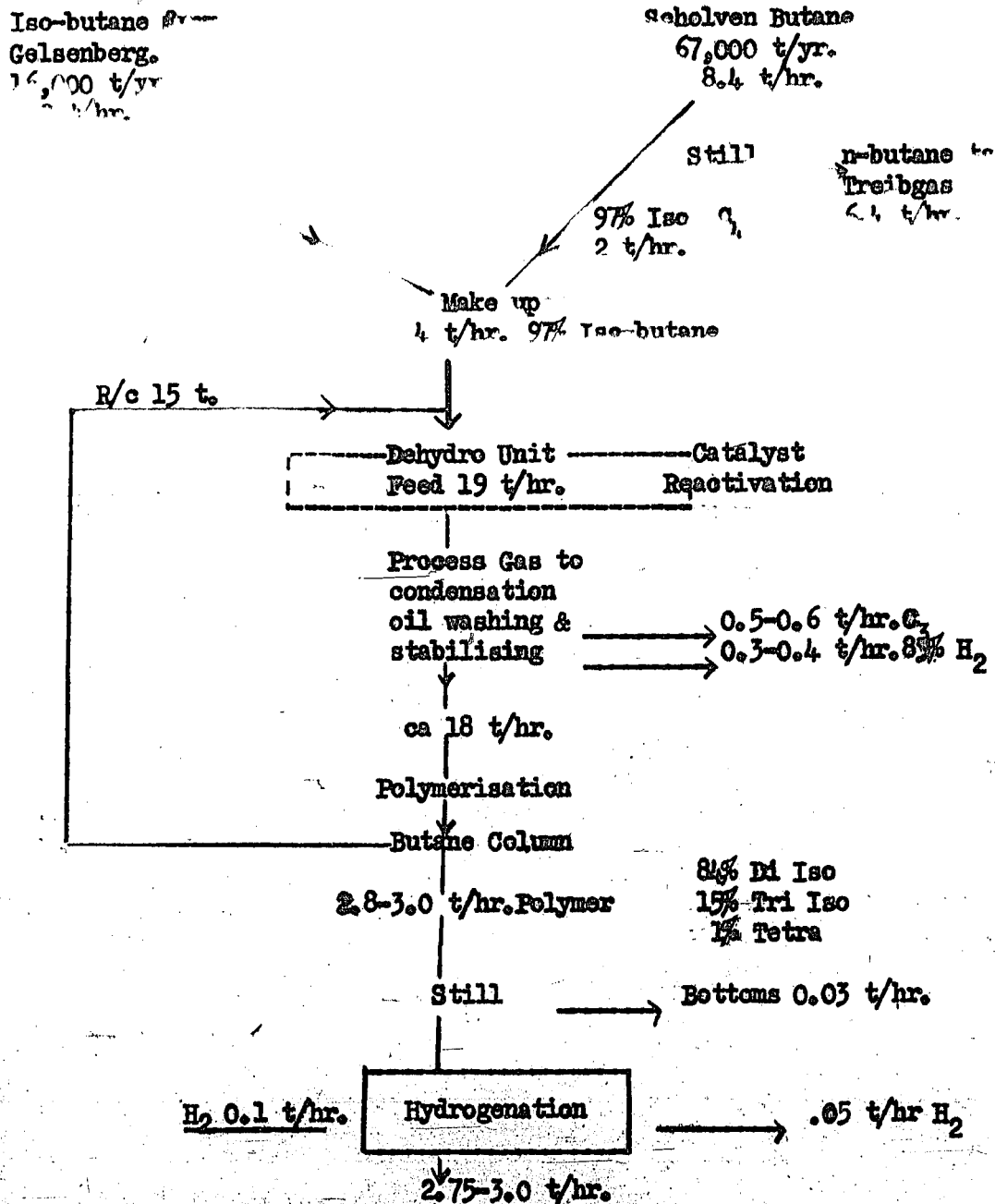
(All quantities in metric tons and M³/hr.)



FLOWSHEET VII

FOR SCHOLVEN BUTANE DEHYDROGENATION

UNIT T. 52.



Iso Octanes 22,000 - 24,000 t/year.
 Yield 69-75% on total butane feed
 Conversion - butane → butylene ca 21% per pass.

balance has been calculated as follows.

| Feed % Vol. | 7350 m^3/hr | Product % Vol. | 9100 m^3 | Make |
|-------------------|-----------------------------|-------------------|----------------------|--------|
| 7.2 $i\text{C}_4$ | 5143 | 63.0 | 5738 | = 1405 |
| 0.8 $n\text{C}_4$ | 59 | 0.4 | 36 | - 23 |
| 1.0 $i\text{C}_4$ | 74 | 14.0 | 1270 | - 1196 |
| .5 $n\text{C}_4$ | 37 | 0.4 | 36 | |
| | 37 | 3.0 | 273 | |
| | <u>7202</u> | C_2 0.6 | 54 | |
| | | C_1 3.0 | 273 | |
| | | H_2 15.6 | 1420 | |
| | | | <u>9,102</u> | |

$$\text{Conversion} = \frac{1428}{7202} = 19.8\%$$

$$\text{Mol yield of butylenes} = \frac{1196}{1428} = 84\%$$

$$\text{Iso-octane \% make-up butane} = \frac{2.88}{3.95} = 73\%$$

From Flowsheet IX taken from drawing MA Scho 538-2 dated 16.1.42, it can be calculated that the conversion per pass was 20.4%, the mole yield of butylenes 81%, and the yield of iso-octane 73.0% on the make-up butane.

FIGURE II
A DEHYDROGENATION REACTORS

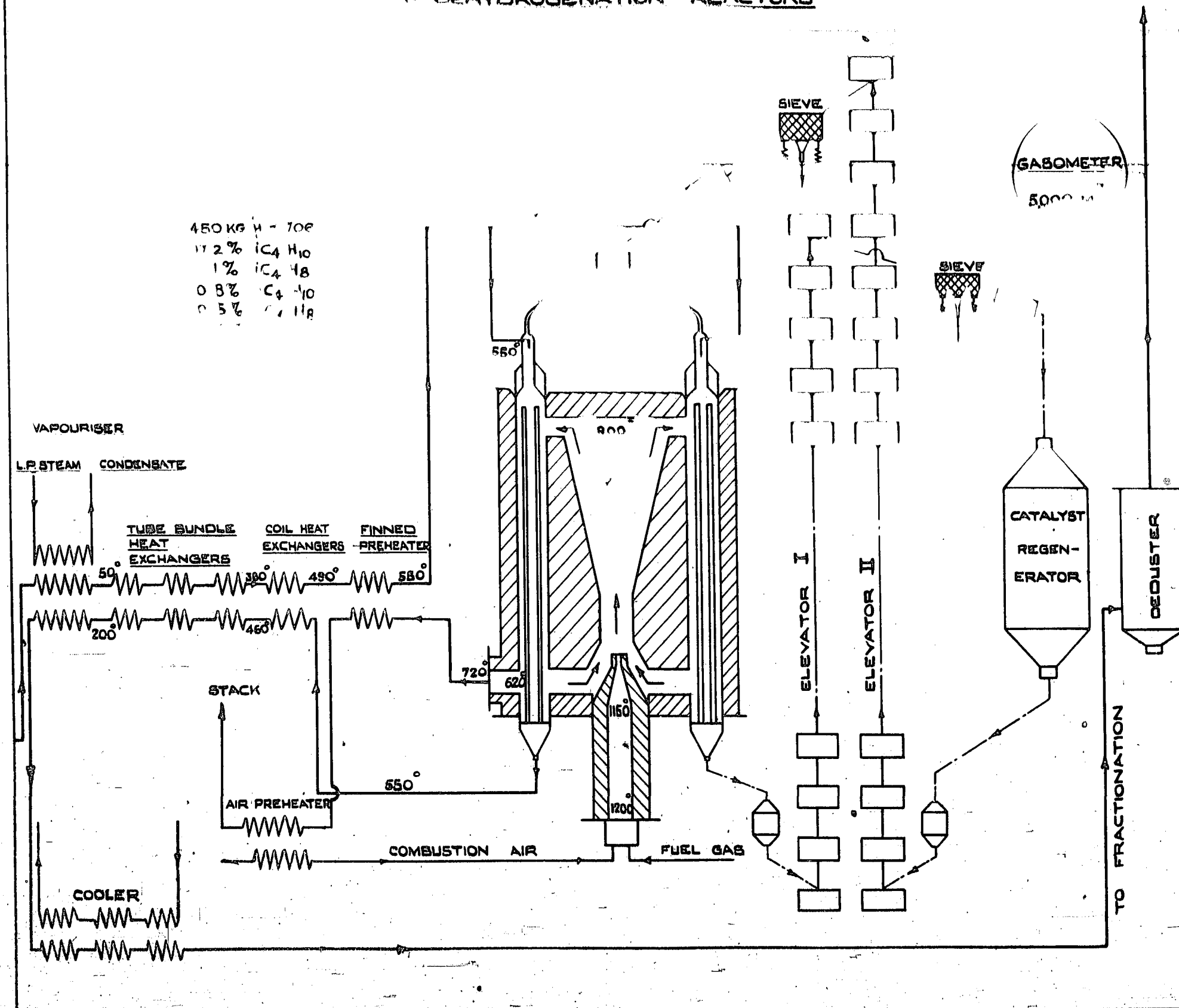
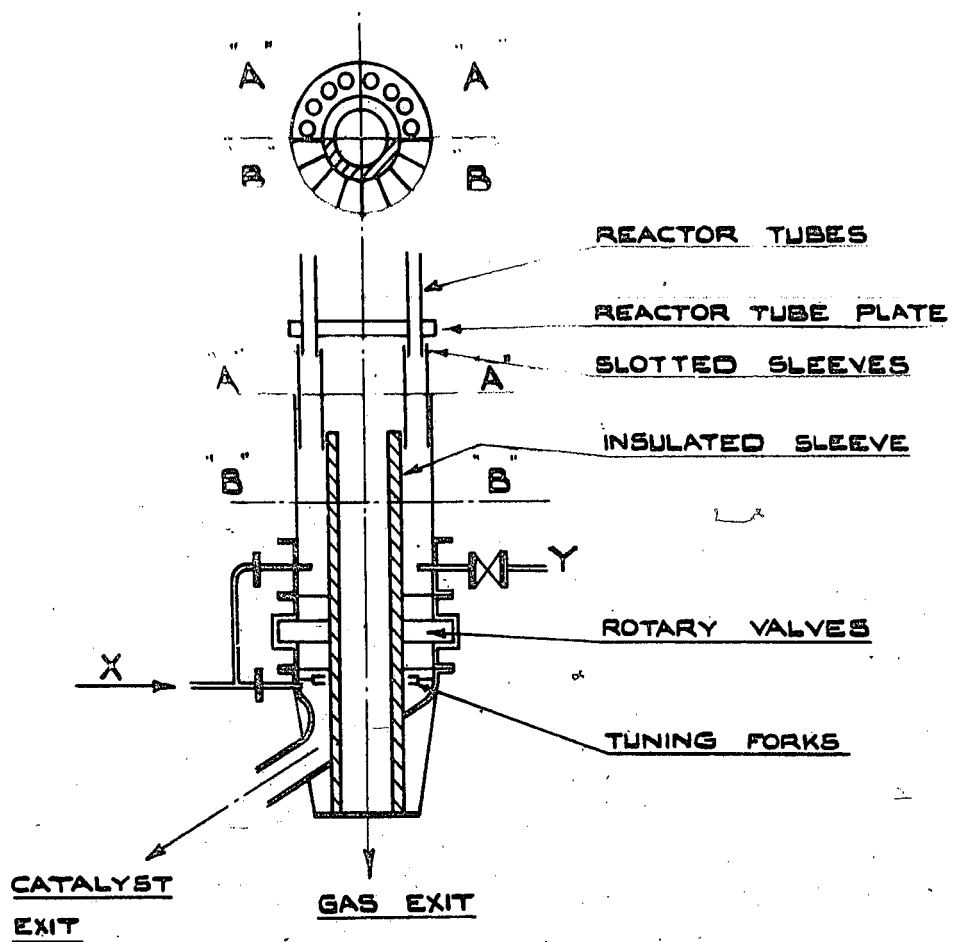
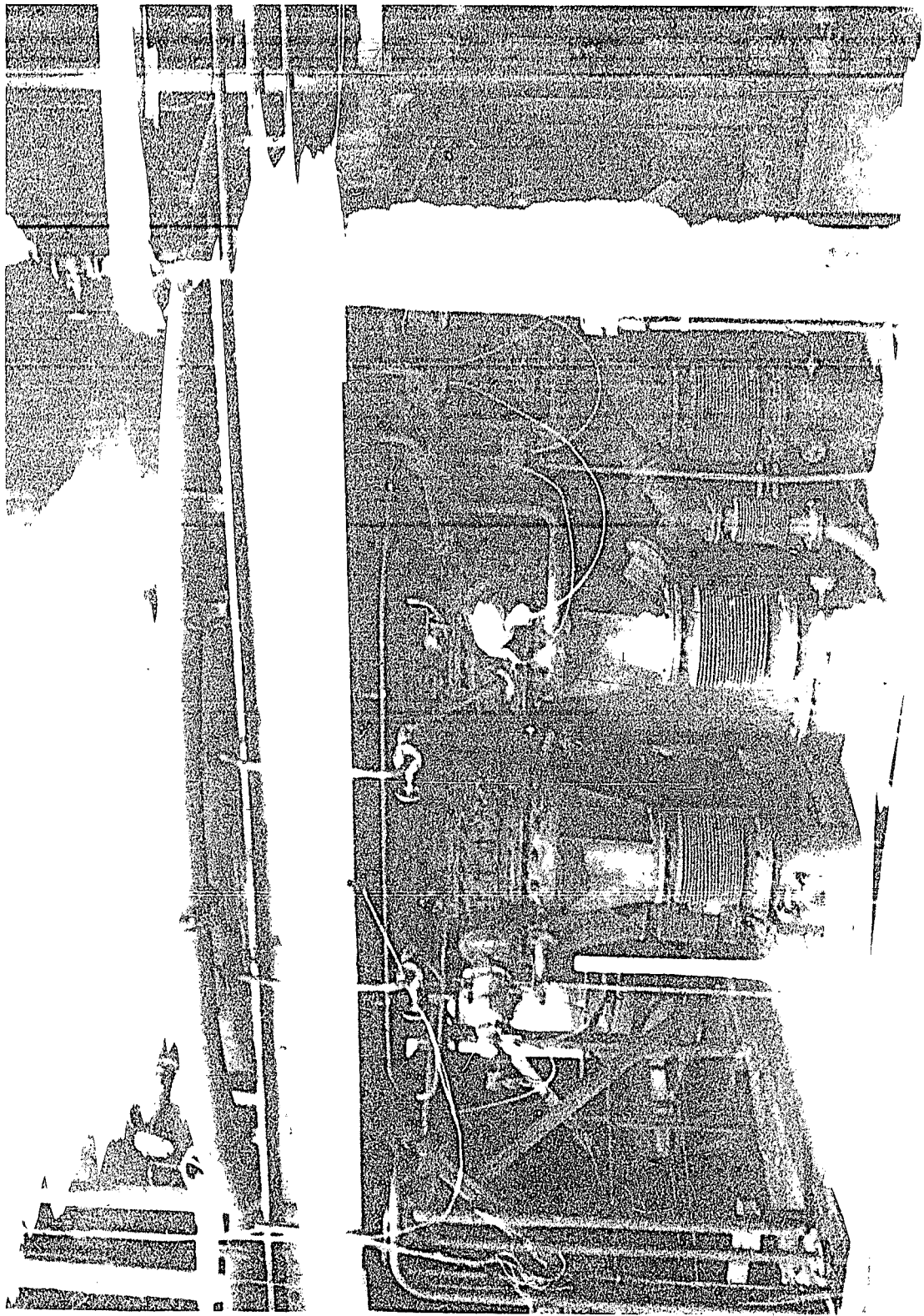


FIGURE I

**SECTION AT BASE OF BUTANE DEHYDROGENATION
REACTOR TUBE BUNDLE.**





catalyst and served to indicate when a valve became choked. Cold gas (x) was injected into the annular space around the insulated gas-offtake, this reduced the temperature of the catalyst before the valves. The connection (y) was probably used for sweeping out with inert gas. We did not find out how quickly the catalyst was moved through the tubes. The catalyst regenerators, of which there were four, consisted of a vertical cylinder into which the catalyst was fed. Hot flue gas containing a controlled amount of oxygen was circulated through the catalyst from a combustion chamber by a hot fan.

The catalyst was in the form of $3/16'' - 1/4''$ granules, and it is believed to be chromium on alumina. Samples of new and regenerated catalyst were obtained. It was noted that regeneration of the catalyst was not complete, the inside of the granules was quite black.

The feed to the dehydro units was vaporised with steam heating to 50 C, it was then heated to 490 C by interchange, and then heated to 550 C in a gas fired preheater. The temperature exit of the reactor was given as 620 C. The pressure was 1.5 ats inlet 1.3 ats exit.

The general arrangement of a unit is shown diagrammatically in Fig. II. From flowsheets it would appear that the conversion of butane was 20-25% per pass, and the mole yield of butylenes produced was 81-85% of the butane converted.

After separating butane plus butylene from the process gas, the butylenes were polymerised at 50 ats pressure over a phosphoric acid catalyst which was formed into rough rectangular blocks. A sample of this catalyst was obtained. A number of small reactors were used in series. The polymer was debutanised, and after removing about 1% of bottoms, dimers and trimers were hydrogenated together over 5058 catalyst at 50 ats pressure.

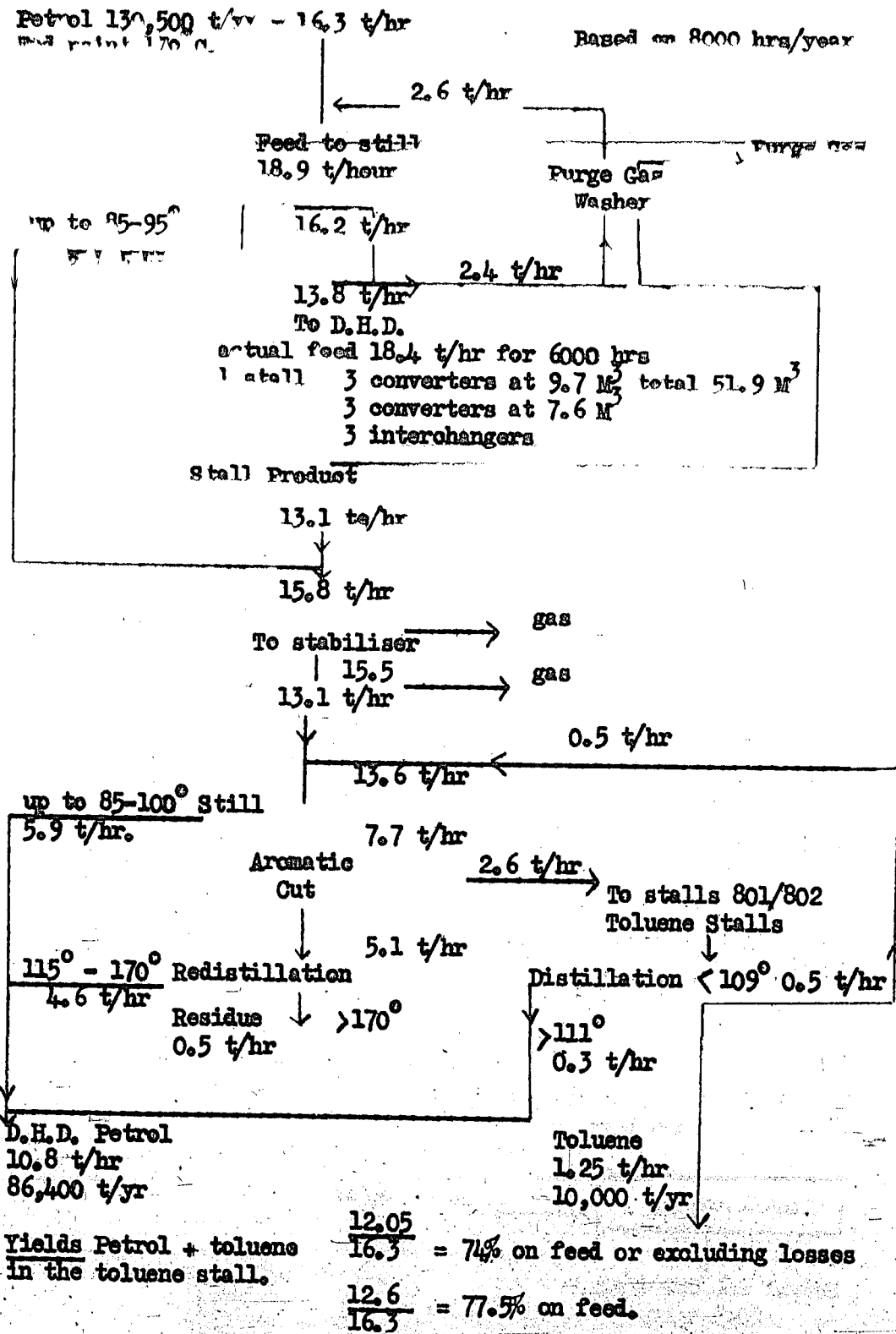
The following flowsheets were found on the plant.

Flowsheet VII shows a production of 22,000 - 24,000 tes/year (8000 hrs) of iso-octane from 4 tes/hr of iso-butane from Scholven and Gelsenberg. The conversion per pass is 21% and the yield of octane 69-75% on the make-up butane.

Flowsheet VIII taken from drawing MA-Scho 161-2 dated 1.12.39 shows the general plan of the operation.

D.H.D. FLOWSHEET VI.

For Petrol from Petroleum -
Ludwigshafen 19.2.43.



VIII. Butane Dehydrogenation and Octane Processes.

At Scholven there was a butane dehydrogenation plant which was originally used for treating iso-butane for the manufacture of iso-octane by polymerisation of iso-butylene. Later it was used for treating n-butane to make n-butylene for alkylation with iso-butane. The process had been developed by the I.G. The catalyst was moved by gravity through the reaction tubes and it was regenerated in separate units. It was therefore unnecessary to provide tube materials which were capable of withstanding the alternating oxidising and reducing conditions of the U.O.P. process. Sichelmal was used to stand up to the high temperatures employed and even this gave trouble with pinholing. Hot switch valves and time cycle control gear were eliminated at the expense of having to use a fairly complicated mechanical device for withdrawing the catalyst from the tubes. For its output of butylenes, equivalent to 22-24,000 tes/year of iso-octane, the plant was of considerable size compared with the equivalent U.O.P. unit. It was divided into 8 independent units each containing 128 reactor tubes.

Each of the 8 units consisted of a brick lined combustion chamber with the central burner in the bottom. The burner was supplied with preheated air. The flue gas was deflected by the roof down through an annulus containing eight bundles of tubes. Each tube bundle had 16 tubes 2 3/4" ID, approximately 15 ft long, pitched on a circle of radius 26". The inside of each tube bundle was occupied by a pipe closed at both ends, this acted as a support for the bundle, and it also increased the velocity of the flue gases. Each unit of 128 tubes contained about 2.2 M³ of catalyst or 17.6 M³ for the whole plant. Catalyst was lifted to the top of the reactor unit by a conveyor delivering into a closed hopper. This hopper was only filled at intervals and the unit was taken off stream while this was carried out, thus avoiding the necessity for providing gas seals. No mechanical device was used to distribute the catalyst to the tubes.

The method of extracting the catalyst from the bottom of the tubes is shown in Fig.I. Extraction gear was fitted to each tube bundle and two bundles fed into a common catalyst offtake pipe and gas sealing hopper. The bottom of each tube was covered by a slotted sleeve. The process gas passed through the slots into the insulated offtake pipe. The catalyst dropped into a chamber provided below with rotary valves. These valves formed a gas seal and consisted of a horizontal sleeve with a slot out in it. The valves were rotated by an electric motor through gearing. The tuning forks, one below each valve, were actuated by the falling

FLWSHEET IV (continued)

| | Purge M^3/hr | Gas B | Gas C | Total |
|-------------|----------------|-------|-------|-------|
| M^3/hr | 4160 | 1700 | 590 | |
| H_2 | 0.212 | .006 | - | .218 |
| CH_4 | .800 | .165 | - | .965 |
| C_2H_6 | .417 | .670 | - | 1.087 |
| C_3H_8 | .115 | .840 | 0.174 | 1.129 |
| C_4H_{10} | - | .170 | .193 | .363 |
| C_4H_{10} | - | .295 | .332 | .627 |
| C_5H_{12} | - | .575 | .835 | 1.410 |
| Total | 1.544 | 2.721 | 1.534 | 5.799 |

Equipment Installed

2 predistillation stills 3 ats. pressure, feed 18 t/hr max.
 2 injectors each $60 M^3/hr$.
 1 injector $35 M^3/hr$.
 2 stalls each with three converters $1400 mm \times 8 m. = 6 M^3$
 two converters $1400 mm \times 12 m. = 9 M^3$
 one converter for refining $1400 mm \times 12 m = 9 M^3$

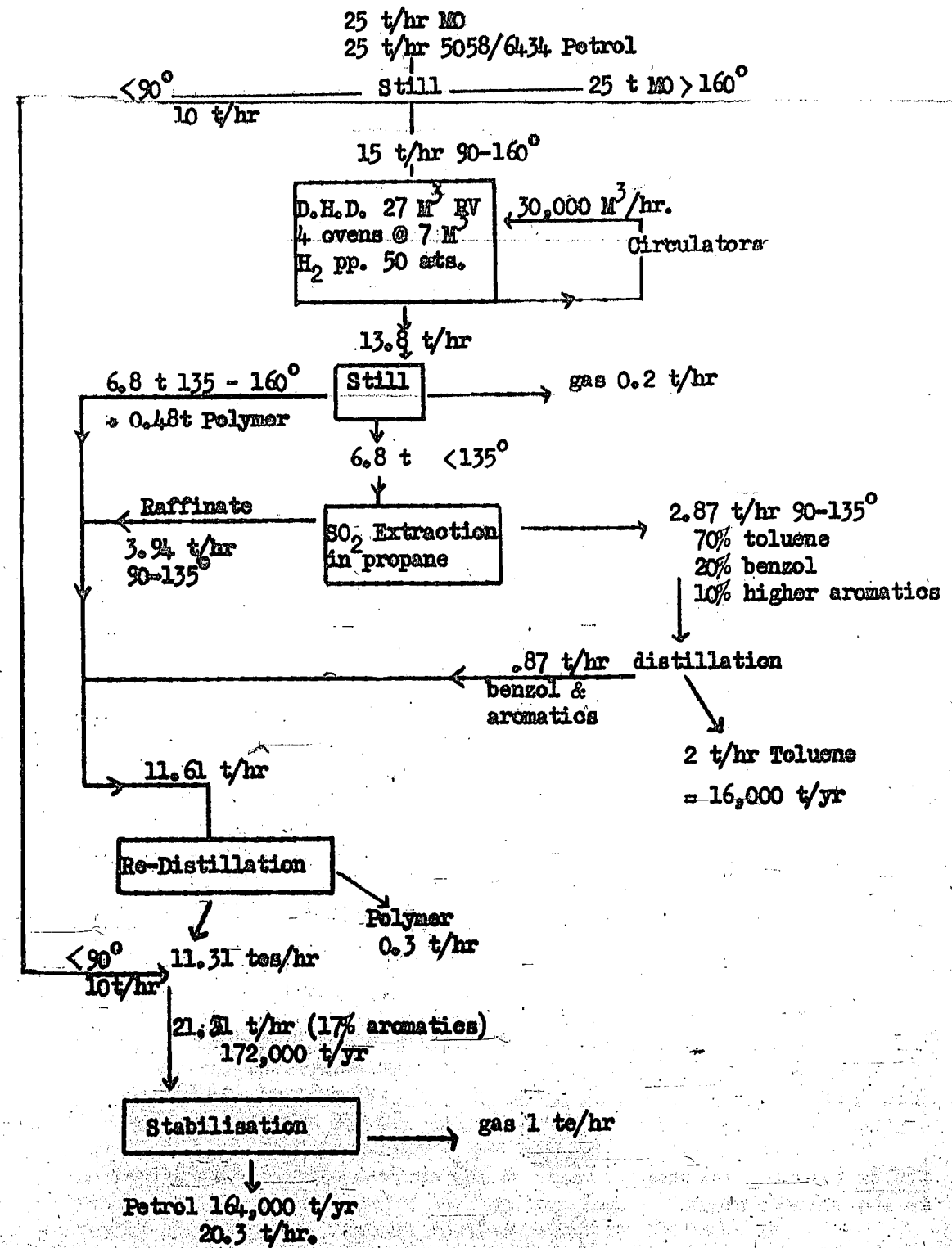
5 circulators $17,500 M^3/hr$ each boost 50 \rightarrow 66 ats.
 2 air compressors $1,700 M^3/hr$ each (70 ats)
 2 oil

The circulating gas rate was given as $26,000 M^3/hr$ per stall and the circulating regenerating gas $30,000 - 35,000 M^3/hr/stall$.

FLWSHEET V

FLWSHEET FOR SCHOLVEN D.H.D. UNIT

(from Ma Soho 148-2 dated 6.12.39.)



Product Side

Inlet 3rd inter or exit 5th converter 519°
 Exit 3rd inter 357° on processing
 Inlet 2nd inter or exit 6th converter 295°
 Exit 1st inter 134° C

Converters

| | | |
|------|-----------|------------------------|
| No.1 | inlet 520 | exit 491 |
| No.2 | inlet 523 | exit 496 on processing |
| No.3 | inlet 523 | exit 498 |
| No.4 | inlet 523 | exit 513 |
| No.5 | inlet 523 | exit 519 |
| No.6 | inlet 357 | exit 295 |

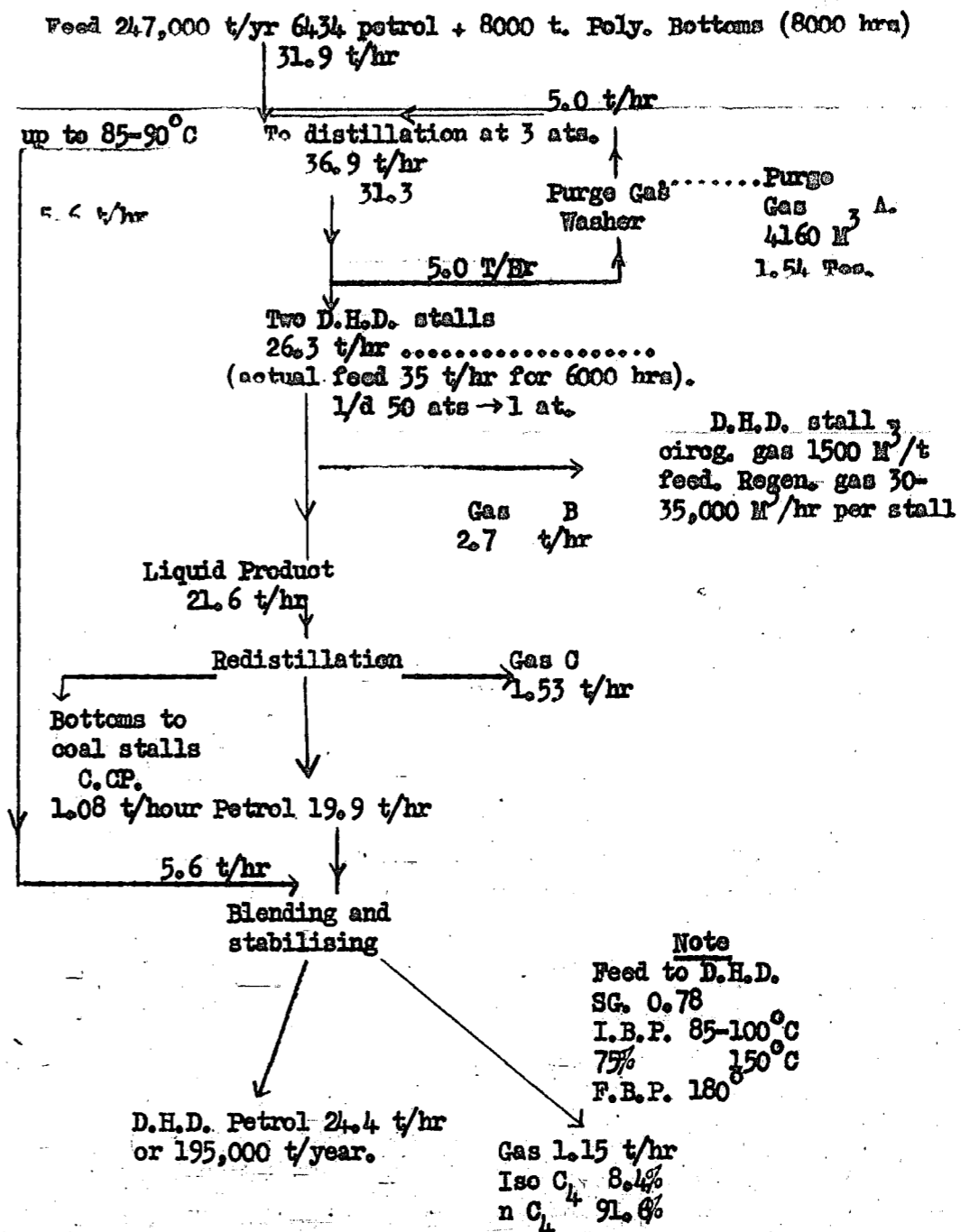
The interchangers were given as 12 m long by 600 mm, 313 tubes, area 158 M².

Flowsheets

Flowsheet IV dated 14.2.41 is a scheme for the production of aromatic petrol from the whole of the 6434 petrol being made at Scholven. At this time they were making auto benzol with an end point of 180° C. The yield of petrol on the fraction fed to the aromatising stall was estimated to be 71.5% and the overall yield of DHD petrol was 76.5%. 3.2% of bottoms from the poly units was included in the feed. It is curious that polymer bottoms were added to the feed as the paraffins contained therein would be cracked to butane. It is not what was the hydrogen absorption if any, but the flowsheet shows a gain without allowing for the carbon which was burnt off. Flowsheet V is a scheme for taking the 6434 stall cold catchpot product cutting it into three fractions (a) middle oil above 160° C (b) light petrol to 90° C and a fraction from 90-160° as feed to one small aromatising stall. Toluene was separated from the aromatised product by extraction with SO₂ in propane. The aromatising treatment was to be much less severe than that finally adopted at Scholven. Including the toluene, the aromatics in the product were only 25% by weight. The yield of product from the fraction aromatised was therefore high (91%). Flowsheet VI refers to a scheme prepared at Ludwigshafen dated 19.2.43. for aromatising hydro petrol from petroleum. This probably represents the operations which were to be carried out on the DHD plant under construction at Oppau.

FLWSHEET IV

ABSTRACT FROM SCHOLVEN D.H.D. FLOW SHEET DATED 14TH FEB. 1941.



Yield Final D.H.D. petrol 76.5% of total feed or 7% on petrol fed.
 Yield from D.H.D. stall feed alone 71.5%.

VII. D.H.D. Process for Aromatising Petrol.

At Scholven the whole of the hydrogenation-petrol was treated in two D.H.D. units. Dr. Schmidt said that he was not familiar with the details of operation, but he outlined the process. Further data was obtained from documents found on the plant.

Briefly the petrol was distilled to remove a fraction boiling up to $85-90^{\circ}\text{C}$, this amounted to about 30% of the petrol. The residue was treated at $500-520^{\circ}\text{C}$ over a molybdenum catalyst in hydrogen at 50 ats pressure. The operation was a straight-through process. The carbon formed during reaction was burnt off after 100-140 hrs processing. To do this the pressure was blown off the plant and nitrogen containing a controlled amount of oxygen was circulated.

By this process the aromatic content of the blend of light fraction and aromatised petrol was raised from 12% to 45-50%. The yield of final petrol was, according to Schmidt, about 80% on the hydro petrol.

The gas made in the aromatising process depends on the composition of the feed, naphthenes are the best feed stock, and paraffins are to be avoided. The extent to which aromatising is carried out also determines the gas make.

Dr. Schmidt said that the feed rate per stall was 14 tes/hr with 16,000 M^3/hr circulating hydrogen. The operating temperature was said to be $500-510^{\circ}\text{C}$ inlet converters falling to 470°C at the exit. The pressure was between 45 and 70 ats. After processing for 100-140 hrs the pressure was blown off the stall, and after sweeping out to get rid of hydrogen it was repressured with nitrogen. A controlled amount of air was admitted to burn off the carbon, if desired the converters could be reactivated in sequence. In order to maintain the activity of the catalyst it was necessary to limit the temperature during regeneration to 530°C max. From the flowsheets found on the plant it was deduced that the reactivating cycle occupied 20-30 hours.

Dr. Schmidt said that the catalyst was 7360, it is believed that it was molybdenum oxide on chemically precipitated alumina. Samples of catalyst were removed from a splinter hole in a converter for subsequent analysis. No catalyst was made at Scholven. The units had been in operation for 6-9 months before their destruction by bombing.

The Scholven units had converters designed for a working pressure of 70 ats. One stall had five converters and the other six converters. The preheaters were notable for their size. They were built in five vertical sections each with a burner at the base, and hot flue gas was circulated by fans.

The following information was obtained from a drawing found on the plant and dated 9.2.42.

The six converter stall had three converters 10-m-x-1,400 mm each 7.7 M^3 reaction volume, the last three converters were $12\text{ m x }1,400\text{ mm}$ each of 9.6 M^3 reaction volume giving a total of 50.9 M^3 catalyst space. There were three interchangers. The first five converters ran with a temperature gradient from about 520°C down to $470-500^{\circ}\text{C}$, the product from each converter was reheated in the preheater before passing to the next converter. The product from the fifth converter was cooled to about 360°C in the hot interchanger before passing to the last converter or refining stage. Cold circulating hydrogen was supplied as by-pass gas to the exit of each preheater pass, and to the exit of the hot interchanger to give control of the converter inlet temperatures. In the same way control on the reactivating cycle was obtained by using cold circulating regeneration gas as by-pass gas. Common circulators were used for hydrogen and regenerating gas. An oxygen controller on the exit of each converter controlled the addition of air to maintain the required concentration of oxygen at about 3-4%. Regenerating air was supplied to two units by two compressors, each $7,700 \text{ M}^3/\text{hr}$ at 70 ats. Six hydrogen or reactivating gas circulators were provided for the two stalls each capable of $16,000 \text{ M}^3/\text{hr}$ with a boost from 48 ats to 66 ats.

The following data was given on the flowsheet.

| | |
|-----------------------|--|
| Feed | 17.5 tes/hr |
| Product | 14.4 M^3 (not stabilised) |
| Total Circulating gas | 28,000 M^3/hr processing |
| | 17,000 M^3/hr regeneration |
| Cooling gas approx. | 2,000 M^3/hr processing |
| | 7,000 M^3/hr regeneration |

Interchangers

| | |
|-----------|------------------------------|
| Feed side | inlet 30° |
| | Exit NO.2 219° |
| | Exit NO.3 368° |
| | Exit preheater 520° |

The octane number of the 6434 petrol alone was 72 clear CFR. m.m, and the blend with liquid phase petrol 70-71%.

Like Gelsenberg shortage of tungsten had forced them to replace one converter of 5058 in both saturation stalls by 8376 - a catalyst containing only 2% W.S.₂. This change resulted in more nitrogen compounds passing on to the splitting stalls and deterioration of the 6434 catalyst. A plant to remove these small amounts of nitrogen, (a few parts per million), by washing the saturated oil with acid was under construction.

They had some trouble with ammonium chloride chokes in the saturation stalls which they said were caused by ammonium chloride in imported tar oils. Middle oil pump bodies had given the usual trouble with cracks developing. This had been overcome by designing the bodies so that the valve chest was independent of the pump cylinder. The main source of trouble on the saturation stalls had been leaking interchanger glands. They had tried sealing the gland by injecting steam cylinder oil once a day, but this was not entirely successful and they were changing to the injection of circulating gas continuously. They had also the design of a bellows type gland in hand but they thought that such a gland would not be satisfactory with frequent and rapid blowing down of the stalls in air raids. A rapid release of pressure may give rise to excessive pressure difference across the gland. The tightness of glands is of course more important on saturation stalls because a leakage of feed into the product has a bad effect on the splitting stalls because of the nitrogen compounds in the feed.

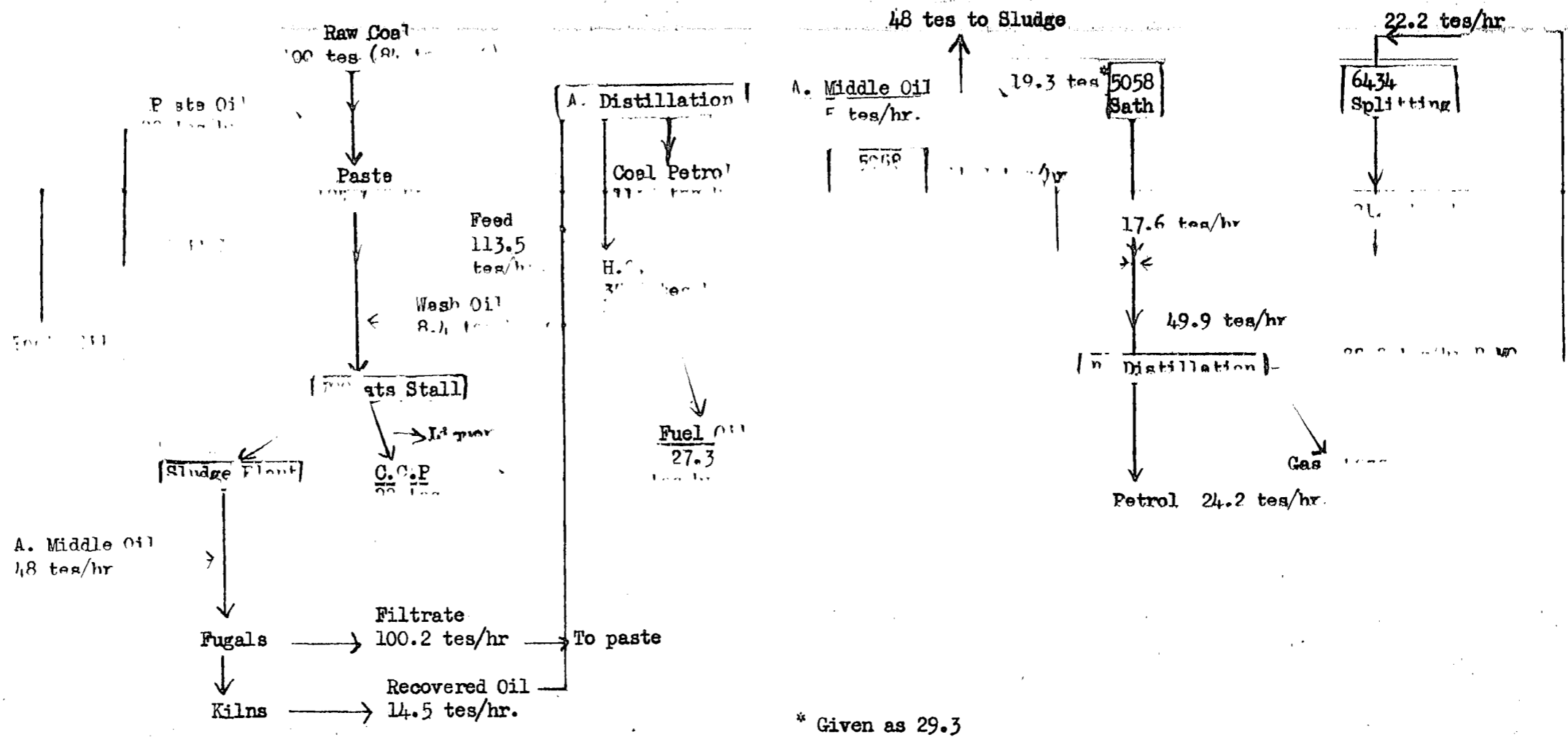
In connection with the general question of gland packings it may be noted here that they had substituted 80-20 lead-copper packing rings for the paste injector glands when tin became short. They admitted that they had no experience with glands packed entirely with the new packing, a mixture of the old and new type of rings had always been used. Their experience with gland packings for the hydrogen plant copper liquor injectors was very interesting. They used a Buna S.E.A. ring packing and lubricated the gland with water, which was fed onto the ram outside the gland. Only two rings were used to seal off 325 ats, and the gland lasted at least 6 months. If more rings were used the rams got hot. As an experiment they had tried running with only one ring but had decided that it was too dangerous to run in such a manner for a long time.

FLWSHEET III

MA/GL 9-2 23.8.41.

(All quantities metric tons/hr)

12A



* Given as 29.3

300 ats Vapour Phase Stalls

(a) Saturation Stalls (5058)
 Reaction Volume 57.5 M³
 Feed-A-MO t/hr 30.7
 Throughput t/M³/hr. .535
 Circulating gas M³/t feed. 4200
 app. H₂ used % feed 5.0

(b) Splitting Stalls (6434)
 Reaction volume 4.9 M³
 Feed sat. oil + B-MO t/hr. 4.2
 Make up in feed % 72.5
 Feed thro'put T/M³/hr. 0.86
 Petrol leistung T/M³/hr. 0.25 (overall 5058, 6434)
 Gas circulated M³/t feed 2050
 approx. H₂ used % make up 3.0

Overall H₂ used % A-MO 8.0
 Petrol made % A-MO 87.5

Total petrol t/hr 31.6
 amf coal t/hr. 55.5
 % petrol 56.9
 overall H₂ used
 % on amf coal 13.46

VI. Vapour Phase Stalls

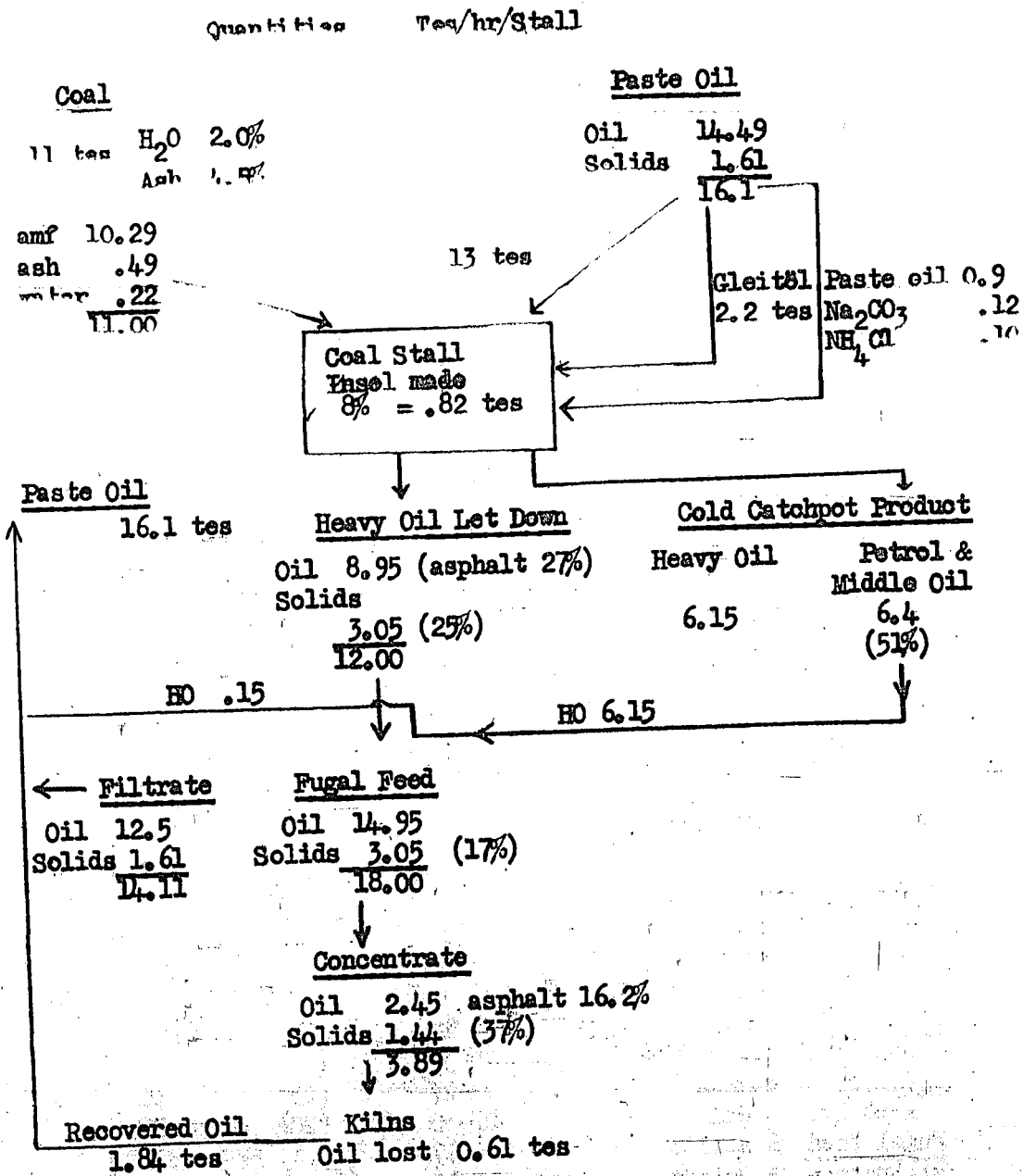
There were two 5058 saturation stalls and two 6434 splitting stalls, each with two or three converters in series. A converter contained 9.5 M³ catalyst. Each converter contained 7 beds of catalyst of uniform size with cooling gas points in between beds. Cooling gas was not fitted to all beds, in particular in the last converter of the splitting stalls the effective length of bed was doubled by leaving out alternate by-pass connections. Two interchangers, 18 m x 600 mm with 240-300 tubes 14 mm i.d. x 23 mm o.d. were fitted to each stall. The preheaters were all gas fired and were fitted with 8 hairpins 90 mm diam. 28 m long, with a heating surface of 1280 sq. metres.

The feed to the saturation stalls was 15-16 tes/hr for a three converter stall giving a leistung of 0.52-0.56 te/M³/hr. The splitting stalls worked to a conversion of 65% per throughput to petrol of 55% volatility. The products from the saturation stalls and from the splitting stalls were distilled together. In this way dissolved ammonia was removed from the saturated oil and the refined liquid phase petrol and the petrol made during saturation was not passed over the splitting stall catalyst.

| | Scholven | Gelsenberg |
|--------------------|------------|------------|
| Kiln feed tes/hr | 2.5-3.2 | 2.7-3.0 |
| Kilns Oil Recovery | 75-80 (80) | 78 |
| Cold catchpot % P | 8 | |
| % MO | 47 | |

FLWSHEET I

Scholven Coal Stalls (Data from Dr. Schmidt)



V. The Gladbeck Project

It was the intention to extend Scholven on an adjacent site. Later a new site was chosen at Gladbeck. The scheme was sanctioned in 1938 for the production of 250,000 tes/yr of petrol. Erection was started but was abandoned in April 1942 owing to the shortage of building construction labour. Many of the detailed drawings were found at Scholven. The main interest in this project is that it was intended to operate the coal stalls at 700 ats pressure. The use of the Welheim 700 ats vapour phase process was still under discussion when work was stopped. Flowsheet II at the end of this report, was taken from drawing MA.III 2001-2, dated 10.11.38. A summarised version of this is given in table II below.

A second flowsheet III taken from drawing MA/CL 9-2 23.8.41 indicates that they had in mind the possibility of working with a middle oil paste to produce excess heavy oil (32% on the coal) for use as fuel oil. The vapour phase side of this flowsheet seems to be in error as the yield of petrol appears to be less than 70% on the middle oil treated.

TABLE II

Summary from Flowsheet MA.III 2001-2 (10.11.38)

| 700 ats coal stalls | |
|--|-----------------------|
| Reaction volume | 150 M ³ |
| a.m.f. coal/hr | 55.5 |
| dry coal(2% H ₂ O)t/hr | 59.6 |
| Coal in paste | 45.5 |
| Total catalyst % on coal | 4.0 |
| Total paste throughput | 0.94 |
| | Te/M ³ /hr |
| amf. coal Te/M ³ /hr. | 0.37 |
| Circulating Gas/te coal | 3740 |
| approx. H ₂ used % a.m.f coal | 9.0 |
| | |
| <u>Yields % a.m.f. coal</u> | |
| Petrol | 8.5 |
| A-Middle oil | 55.3 |
| HO purge at sludge | 4.1 |
| Insols. | 4.0 |
| Gas | 24.0 |
| Liquor (est ^d) | 8.0 |
| | 103.9 |
| Petrol from A-MO | 87.5% |
| = 48.4% amf. coal | |
| Overall petrol | 57.2% |
| As motor fuel | |

The fugal concentrate was carbonised in 12 Kugel ovens, 10 of which were usually required to balance 5 coal stalls on line. The feed to an oven was 2.7-3.5 tes/hr, and 0.5 tes/hr of superheated steam was added. The amount of steam used at Gelsenberg was only half this quantity. The temperature inside the Kugel ovens was not measured, but the shell temperature was said to be 590-610°C. Each oven had to be shut down at intervals of 4 weeks to remove the coke accumulated on the shell. This compares unfavourably with Gelsenberg where the ovens ran for 6-12 weeks because the ratio of solids to asphalt was higher. For the same reason the efficiency of oil recovery was only about 75% on the oil plus asphalt fed compared with 78% at Gelsenberg. Experiments on grinding charges indicated that the most satisfactory charge consisted of ribbed balls.

The design and arrangement of the Kugel ovens was like that at Gelsenberg. A more detailed description is given in the report on Gelsenberg.

The coal stalls were operated under such conditions of temperature and paste throughput that the heavy oil in the new formed oil was just sufficient to balance the loss of oil at sludge. Also the end point of the middle oil was varied over the range 325-330°C in order to balance heavy oil stocks.

Dr. Schmidt gave us the following data for the operation of the coal stalls, see Table I. The figures given in brackets are those obtained in 1937. It is evident that there had been little change in the method of operation except for the increase in asphalts in H.O.L.D. which has already been discussed. For comparison some figures for the 700 ats coal stalls at Gelsenberg are included.

Flowsheet I has been calculated from Dr. Schmidt's figures. The overall yields are calculated to be :-

Yields % amf. Coal

| | | |
|-------------|--------------|----------------------|
| Petrol + MO | 62.2 | (10% petrol on coal) |
| HO lost | 6.0 | |
| Insols. | 8.0 | |
| Gas | 24.0 | |
| Liquor | 9.0 | |
| | <u>109.2</u> | |

H₂ chemically absorbed 9.2

By conversion of the middle oil to petrol in the vapour phase stalls, the overall yield of petrol of 55%

volatility at 100°C would be expected to be 5% on the amf coal. We know that the actual yield of petrol at Scholven was between 49 and 50% of the amf coal. This does not allow for additional losses incurred in the D.H.D. units.

The cold catchpot product after separation of liquor was distilled to take off middle oil and petrol together. After stabilising the distillate this was fed to the saturation stalls. Up to 120 t/d of tar oils and creosote-middle oil were imported, these were either added to the pasting oil or put to the liquid phase stills.

The liquor from the stalls was treated for the recovery of phenols by benzol extraction. Some 500 tes/yr of phenols were recovered for use in the manufacture of resins.

Summary of Operating Data - Coal Stalls.

| | Scholven | Gelsenberg |
|--|---------------|------------|
| | 1944 | (1937) |
| Stalls | 6 | (3) |
| Average on line | 5 | |
| Reaction Vol/stall M ³ | 39 | (36) |
| Dry Coal tes/hr | 11 | (9.5) |
| Carbon content % amf. | 82-83 | 83 |
| Coal paste tes/hr | 24 | (20) |
| Gleitöl tes/hr | 2.2 | (2) |
| Coal in paste % | 46 | (47) |
| Coal in paste + Gleitol | 42 | |
| Pressure inlet preheater ats. | 300 | 700 |
| Gas to preheater M ³ /hr. | 27,000-30,000 | |
| Cooling gas M ³ /hr. | 6,000-7,000 | |
| Average Temp. °C | 465-470 | 475-480 |
| H.O.L.D. te/hr. | 11-13 | 14-15 |
| Paste & Gleitöl te/M ³ /hr. | .67 | 1.1 |
| amf. coal/M ³ /hr. | .264 | .39 |
| Petrol + Middle oil te/M ³ /hr. | .157 | .253 |
| organic insols. % amf coal | 8-8.5 | 4.0 |
| Paste Oil % Solids | 10 | (10.6) |
| Ash % of solids | 25-30 | (30) |
| H.O.L.D. % solids | 22-27 | (25) |
| Asphalts in oil % | 27-30 | (20) |
| Fugal feed % solids | 17 | (19.1) |
| Filtrate % solids | 11-12 | (14.3) |
| Concentrate % solids | 37-39 | (33.9) |
| % asphalts in oil | 17-20 | (14) |
| ratio Solids asphalts | 3.6 | 4.5 |

was greater. The final preheating of paste and the remainder of the inlet circulating gas, which was already preheated in the interchanger, was done in 120 mm diam. tubes. There were usually 17 hairpins in a preheater.

About 2 tes/hr/stall of pasting oil was fed direct to the preheater, this was called Gleitöl. This is common I.G. practice and it is supposed that the oil acts as a lubricant for the paste.

Originally the catalysts, tin oxalate and ammonium chloride, were put into the coal paste. As might be expected this caused considerable trouble with corrosion in the preheater and the ammonium chloride was then injected as a 20% paste in pasting oil, direct to the inlet of the first converter. The top bends of the preheaters were enamelled and the bottom bends were forged pieces fitted with hard erosion resistant linings. Corrosion-erosion took place only in the temperature region of 250-350°C, and this difficulty was largely overcome by changing the point of injection of the ammonium chloride.

In order to improve the performance of the coal stall preheaters, one or two preheaters had been fitted with axial flow fans. These had given trouble with inadequate clearances due to expansion, and they were going to change back to the original type of centrifugal fan.

The converters were hot walled and were fitted with V2A liners, and the usual cement lagging internally.

In order to save tin which was in short supply, a lead catalyst had been tried but it was not satisfactory because the amount of unconverted coal (organic insols) was too high. From the stall record books it was noted that there had been changes in the concentration of tin oxalate and ammonium chloride. In 1937 they were using .07% tin oxalate and 1% ammonium chloride, expressed on the coal. A reduction in ammonium chloride to reduce corrosion troubles was probably the reason for the increase in asphalts in H.O.L.D. after that date; in 1937 asphalts in oil were 20% compared with 27-30% for more recent conditions.

Because they were using hydrochloric acid as a catalyst it was necessary to remove it before the reactants cooled below 350-400°C. At Scholven this was done by injecting 120% of the theoretical amount of sodium carbonate required for complete neutralisation, as a 20% suspension in pasting oil. This was added to a point about 10 ft from the end of the last converter. A baffle across the converter below the point of injection of the alkaline paste reduced the

possibility of alkali contaminating the whole converter.

The I.G. had considered the possibility of using a separate vessel after the converter but we do not think that this had been installed. They did not employ the method adopted at Billingham of scrubbing the vapours from the hot catchpot using a mixing vessel, a second hot catchpot and separate alkali let down. For this reason it was essential to centrifuge the whole of the H.O.L.D. in order to remove the excess of sodium carbonate. From the I.G.'s point of view this in itself was not a disadvantage because they always favoured centrifuging all the H.O.L.D. but the whole of the Kugel oven residues were contaminated with an alkaline ash. To burn this residue they had found it essential to install special boilers which discharged the ash in the form of a fluid slag.

In complete removal of sodium carbonate in the fugals could give an alkaline condition in the preheater. At 300 ats pressure this might lead to a higher make of insoluble residue. It is known that the pasting oil contained 0.2-0.3% free sodium carbonate, which was more than sufficient to neutralise the whole of the chlorine in the coal (0.1-0.12%).

The I.G. system for hydrochloric acid removal was cheaper in capital and maintenance costs, but it was not as efficient.

The hot catchpots were fitted with an arrangement to recycle hot H.O.L.D. in order to prevent solids settling and coking on the walls. They had tried gas cooling coils to provide reflux on the walls, but this was discontinued because of corrosion. The average temperature in the converters was usually 465-470°C, and in the vapours from the hot catchpot 450°C.

The H.O.L.D. was let down from the hot catchpot through a finned convection cooler. After separation of dissolved gases the H.O.L.D. was diluted with liquid phase heavy oil (i.e. heavy oil from the distillation of the vaporised oil collected in the cold catchpot) to reduce the solid content from about 27% to 17%. The mixture was centrifuged in Alfa Laval continuous centrifuges fitted with Widia nozzles for discharging the concentrate which contained 37-39% solids. The filtrate was used for making up pasting oil, this was done by diluting the filtrate further with recovered oil from the kilns and with the remaining liquid phase heavy oil. The feed rate to a fugal was 2-2.2 tes/hr.

CO₂ removal was done at 25 ats and CO removal at 300 ats. CO₂ in water gas hydrogen was stated to be 0.05-0.1%.

The Linde units each took 9,000 M³ of gas and produced 4,500-5,000 M³/hr of hydrogen. The total feed to the Linde units was about 55,000 M³/hr made up of 41,000 M³/hr purified coke oven gas, 5,000 lean gas and the remainder N-water gas from the hydrogen sets. CO₂ was removed by compression to 12 ats and water scrubbing. The residual CO₂ about 0.5% was removed with caustic soda. Of the 27,000 M³/hr of hydrogen made on the Linde Units, 16,000 was compressed to 300 ats in one set of machines. The remainder was compressed to 150 ats first and then recompressed to 300 ats in another set of machines. This was done to make use of old machines which were available when Scholven started construction.

About 1,000 M³/hr of ethylene was produced by the Linde Units for export to Hils.

The gas system at Scholven can be summarised as follows.

| | | |
|--------------------------------|---|----------------------|
| Coal Stall 40 ats let down gas |) | To fuel gas & Linde |
| Gas Washing 25 ats |) | " |
| 5058 25 ats gas |) | " |
| 6434 25 ats gas |) | " |
| Coal Stall 5 ats |) | To fuel gas |
| Gas Washing 5 ats |) | " |
| Lean Gas from DHD |) | " |
| and octane units |) | " |
| Coal stall 1 at |) | To petrol recovery |
| Gas washing 1 at |) | and propane & butane |
| 5058 1 at |) | separation. Residual |
| 6434 1 at |) | gases to fuel gas. |
| Liquid phase and vapour phase |) | |
| distillation gases. |) | |

On the 5058 and 6434 stalls there were let down stages operating at 100 ats pressure. The dissolved gases from these stages were recompressed to 300 ats to join the main stream of make-up gas.

The total fuel gas available was about 230,000 t cal/hr, of this 120,000 t cal/hr were used on the site and the rest was exported to Hibernia.

IV. Coal Stalls

There were 6 coal stalls, on the average 5 stalls were on-line together. A seventh stall was under construction. Each stall had a throughput of 11 tes/hr of dry coal.

The coal was obtained from Hibernia Colliery and it had the composition -

| | | |
|-----------|----------|----------------------------|
| C | 82-83% | ash moisture free (a.m.f.) |
| Volatiles | 37-38% | |
| Ash | 4.5-5.5% | (on dry coal) |
| Water | 8-12% | |

The coal was cleaned in a Baum washer before being received at the Hydrogenation plant. Schmidt gave the ash content as 4.5-5.5%, this is considerably higher than it was in 1937 when it was 4%.

The arrangement for coal grinding and pasting was similar to that seen at Gelsenberg. Grinding and drying to 2% water was done in 4 Knapp-Greson (Magdeburg) mills each of capacity 22 tes/hr coals. There were 4 Concentra rod mills for paste making. The pasting oil contained 8-12% solids and the paste 4% coal.

The general arrangement of the stalls, control rooms, injection and gas circulation buildings was similar to that at Gelsenberg and Welheim (see photograph in the report on Welheim.)

Each stall had two preheater fans, three converters each 13 M³ reaction volume, gas-products interchanger and hot catchpot. The usual size of converter was 18 metres long by 1200 m.m ID giving 13 to 13.5 M³ reaction volume. Some of the converters in use were 18 metres x 1000 m.m giving 9.5 M³ reaction volume. Four converters had been tried but the preheaters were not of sufficient capacity to allow the feed rate to be increased to make use of the extra reaction volume. The interchangers were tube stacks. Paste interchange had not been tried as at Gelsenberg where alkaline conditions were employed, and where there was not the risk of corrosion at low temperatures that there was at Scholven. The preheaters had 6 hairpins with 90 mm tubes in the paste section (the breiteil), 2000-3000 M³/hr of circulating gas, which had been preheated in a single hairpin, was added to the inlet of the breiteil. This was done to increase the rate of heating of the paste through the dangerous range below 250°C, in which range water was being evaporated and risk of corrosion

to maintain the output of petrol at about 220,000 tes/year.

A plant for the dehydrogenation of butanes had been in operation. This was the German Version of the U.O.P. catalytic process and it compared very unfavorably in size and output with U.O.P. plants. It employed a moving bed of catalyst with external regeneration of the catalyst. 22-24,000 tes/year of octanes were made by polymerisation of the butylenes from iso butane. The polymerisation plant was replaced by an alkylation unit and n-butane was dehydrogenated. In this way it was expected to increase the output of octanes to 45-50,000 tes/year. This alkylation unit ran during the first six months of 1944 and was not over the teething troubles when it was destroyed. The general impression was that the L.G. had used much more steel in the alkylation units than was really necessary because they did not realise the benefits to be derived from efficient mixing of the reactants and from having a very high ratio of iso butane to butylene. For octane manufacture 16,000 tes/yr. of iso butane¹⁹³⁸ separated at Scholven and another 16,000 tes/yr. were imported from Gelsenberg. When the alkylation unit was put into operation there was a shortage of iso butane and preparations were being made to isomerise n-butane to iso-butane.

Some propane and all the ethane produced were exported to the rubber plant at Hils. Spare n-butane and propane were bottled for use in cars. 500 tes/yr of phenols were extracted from the liquor produced by the hydrogenation units, this was used for manufacture of resins.

II. Production

(a) From the auditor's annual statements the following data was obtained. These statements were removed for further examination of the cost data contained in them.

| | 1938 | 1939 | 1940 |
|---------------------|-------------|-------------|-------------|
| Auto benzine | 127,080 tes | 106,377 tes | 103,670 tes |
| Aviation | - | 71,018 " | 74,060 " |
| Treib gas for cars. | - | - | 10,285 " |
| | 127,080 " | 177,395 " | 188,015 " |

After August 1940 the whole of the petrol was produced as aviation grade.

(b) Jost said that in 1943 the output was 200,000 tes, also that in 1944 when DHD was running and there were 6 coal stalls, the output was 185,000-200,000 tes/yr.

(c) Our estimate of production, assuming an average of 5 coal stalls running is :-

| | |
|-------------------------|---|
| Raw coal treated | 1,320 tes/d. |
| a.m.f. coal | 1,240 " |
| Hydro petrol from coal | 620-645 " |
| Tar treated | 120 " |
| Petrol from tar | 100 " |
| Total petrol before DHD | 720-745 tes/d = 262,000-272,000 tes/yr. |
| D.H.D. Aviation petrol | 210,000-218,000 tes/yr. |

These figures represent the maximum output possible. They do not include octane or alkylate made.

A heavy raid on March 15th, 1941 put the plant out of commission for 3-4 weeks. After a heavy raid in July 1944 it was partly restored, but it did not get onto production. Another raid in October 1944 made further attempts to repair the damage hopeless. Of the three hydrogenation plants in the Ruhr, Scholven, Welheim and Gelsenberg, Scholven appeared to have suffered the most severely. Although equipment which had not been damaged was urgently needed elsewhere it had been impossible to remove it because of lack of railway facilities. Light equipment such as instruments had been removed. The instruments from the stalls were found in the Hibernia Mine.

III. Hydrogen Production and Fuel Gas

The average amount of hydrogen required was about 6.5 tes/hr or 78,000 M³/hr of make-up gas. This was supplied as 50% from water gas from coke and the remainder from Linde units operating on coke oven gas, hydrogenation plant lean gases and N-water gas; also 7,500 M³/hr of hydrogen was imported from Hils. 5 Demag and 5 Pintsch generators produced about 58,000 M³/hr of blue water gas with 7 generators on line. The raw coke consumption was given as 0.55 tes/1,000 M³ B.W.G. 20% of this gas was N-water gas which was purified in oxide boxes, about 50% of the N-water gas went to the Linde units after conversion of the CO, and the rest was used as fuel gas. N-water gas after sulphur removal by oxide boxes and alkacid scrubbing was treated over Brown Oxide catalyst (iron plus chromium) for conversion of CO to hydrogen.

¹⁹³⁸ H water gas and N water gas are terms used to denote water gas containing a small quantity (usually 1-2%) of nitrogen and a large quantity (above 10%) of nitrogen respectively; the N water gas is produced during the purge periods at the beginning and end of the water gas cycle of a water gas generator.

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PERSONNEL OF TEAM

| | | |
|--------------------------|---|--------------------------------------|
| Mr. E. L. Baldeschweiler | - | U.S. Petroleum Administrator for War |
| Mr. L. L. Hirst |) | - U.S. Bureau of Mines |
| Mr. G. Von Elbe | | |
| Major C. Cockram |) | - Ministry of Fuel and Power |
| Major F. A. Williams | | |
| Capt. C. M. Cawley | | |

I. INTRODUCTION

The information given in this report was obtained by interrogating plant personnel. Some information was extracted on the site from documents for the purpose of getting a general picture of the operations carried out at Scholven. Some data has been incorporated from a study of drawings collected by Colonel L. L. Hirst. Notes taken by Major H. G. Simpson (C.A.F.T.) on operating details and notes on Engineering matters from Major J. F. Ellis, who visited the plant at a later date, have been used in preparing the report.

Dr. Jöst the managing director and Dr. Schmidt were interrogated. Dr. Jöst proved very difficult. Dr. Schmidt gave us most of the detailed information about the hydrogenation stalls. He claimed to have only general information about the DHD and octane units.

The plant was visited between April 6th-9th by a team comprising Messrs. E. L. Baldeschweiler, C. M. Cawley, C. Cockram, G. Von Elbe, L. L. Hirst and F. A. Williams.

The Scholven plant was started up in 1936 and extensions were commenced in 1937. The main purpose of the plant was to hydrogenate coal to produce aviation petrol. A small amount of tar oils (120 Tes/day) were also treated, these were imported from Rauchsels and Meiderich to help the coke oven people. The hydrogenation process for coal was the standard I.G. process operating at 300 ats. pressure with tin and hydrogen chloride catalyst. The conversion of the middle oils produced was done in the vapour phase at 300 ats. pressure with 50% catalyst for saturation and 60% catalyst for splitting. Apart from increasing the capacity of the hydrogenation plant by the addition of new stalls there had been only minor changes in the process since 1937; no changes had been made which had given an increased yield of petrol from coal.

Two new processes were in operation at Scholven. Two stalls for the D.H.D. process, a modification of the I.G. high pressure, high temperature aromatising process, started operations at the end of 1944. The fraction of the hydrogenation petrol boiling above 80-90°C was treated at 510°C and 50 ats. pressure to raise the aromatics in the total petrol from 12% to 45-50%. The overall yield was 77-79% on the hydrogenation petrol. The octane rating of the final petrol was raised from 72 clear CFR. M.M to 76-78. The loss of petrol resulting from the introduction of this process necessitated the installation of an additional coal stall (5 running instead of 4) in order

RESTRICTED

Visit to
CUMMER HYDROGENATION PLANT.

Reported by C. Cockram on behalf
of British Ministry of Fuel and
Power and U.S. Technical and
Industrial Intelligence Committee.

CIOS Target NO. 30/4.09

Dated August 8th. 1945.

Combined Intelligence Objectives Sub-Committee.

G-2 Division SHAEF (Rear) APO 413.

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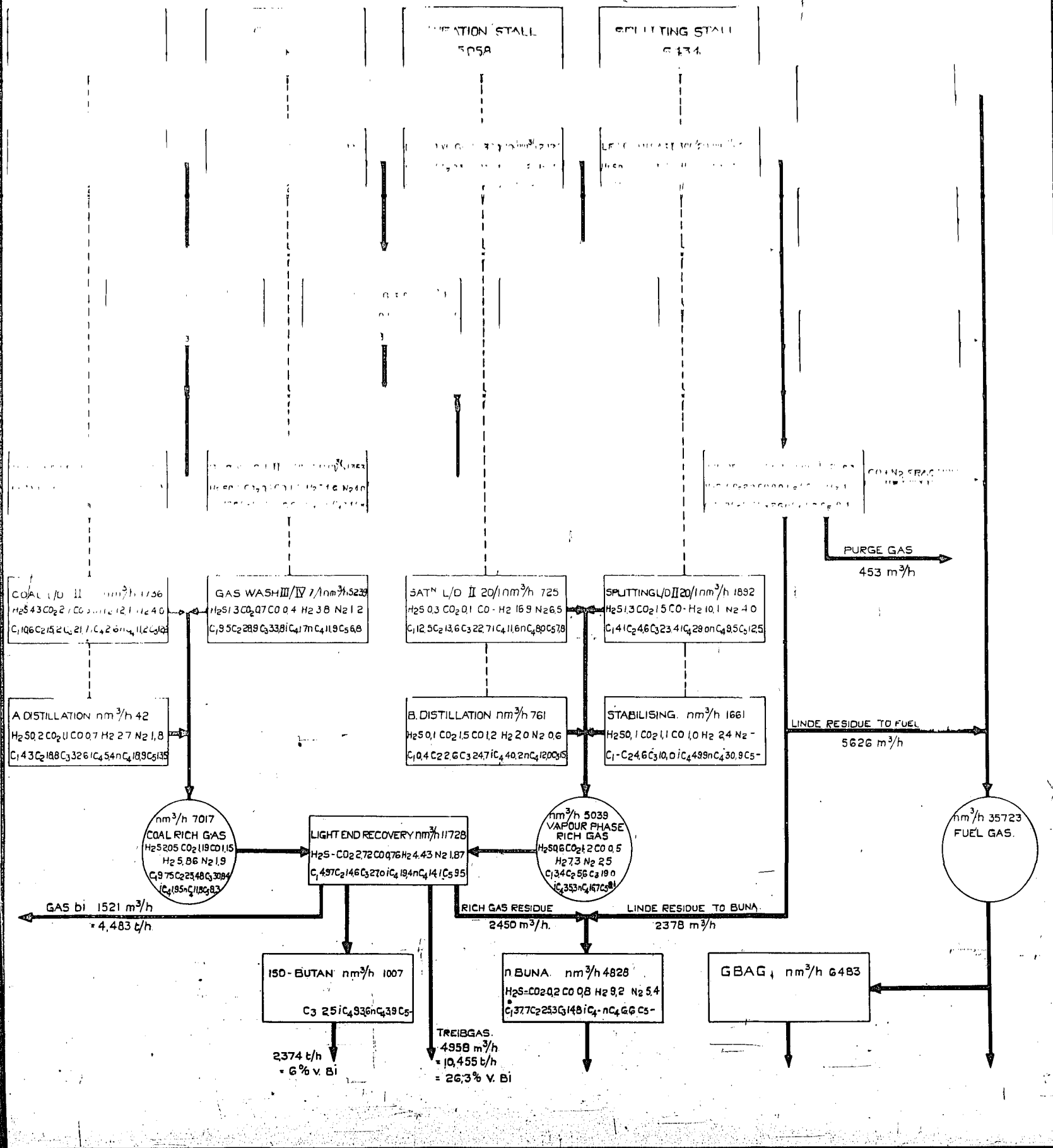
SCHOLVEN HYDROGENATION PLANT

Cochran C

~~RESTRICTED~~

COMBINED INTELLIGENCE OBJECTIVES
SUB - COMMITTEE

FUNCTION AND USAGE OF THE PRODUCTION OF 39.7 TE/HR. M. 1977



VII. SUMMARISED CAPITAL COSTS FOR GELSENBERG

1937 - 1939

Capital Costs at 30.6.1944.

| | R.M. |
|--------------------------------------|--------------------|
| Hydrogen Production | 41,887,000 |
| Linde Hydrogen Plant | 7,517,000 |
| Coal Pasting | 4,994,000 |
| Coal Stalls including distillation | 53,314,000 |
| Sludge & Fugals | 8,096,000 |
| Vapour Phase Stalls and distillation | 29,363,000 |
| Export Tanks | 3,225,000 |
| Treibgas | 9,557,000 |
| H ₂ S Removal | 2,512,000 |
| Sulphate Plant | 1,680,000 |
| Power Plant | 42,880,000 |
| Power Distribution | 21,315,000 |
| Workshops | 3,594,000 |
| Railways | 4,857,000 |
| Laboratories | 3,124,000 |
| General | 10,177,000 |
| Total | 248,092,000 |
| Starting up Charges | 7,409,000 |
| Construction | 37,612,000 |
| | <u>293,113,000</u> |

Stall 307 was arranged with three converters containing 7019 catalyst and one converter with 7360 catalyst. The middle oil was not saturated over 5058 first and the liquid phase petrol had been removed. The operating temperature was 500 - 510°C. The catalyst had a short life, 3-6 months. 7019 required lower temperatures than 3510 catalyst and the gas make was rather lower. It was decided to abandon the experiment because at that time the demand for aromatics had fallen and the output of petrol was seriously reduced by this aromatising treatment. Details of the conditions of running the stall are available from the documents.

Typical analyses of the petrol made were:-

| | 29th April 1941 | 8th July 1941 | 2nd Oct. 1941 | 24th Sept. 1941 |
|-----------------------|--------------------|-------------------------|--------------------|--------------------|
| | Pet. Bi. 7019 | Pet. CV ₂ BL | CV ₂ BL | CV ₂ BL |
| Density | .788 | .795 | .788 | .779 |
| % Aromatics by vol. | 45.5 | 50 | 45-46 | 41.5 |
| by wt. | 49.2 | 52 | | 46.5 |
| Naphthenes by vol. | 37.8 | 33.6 | | |
| " by wt. | 34.7 | 31.5 | 34 | 33.6 |
| Distillate 98% at | 166 | 165 | 96.5% 150° | 98% 150° |
| % to 100°C | 35 | 30 | 42 | 42.5 |
| O.N. CFR M.M. | 77-79 | 77-79 | 77.7 | 78 |
| do. + 0.12% vol. TEL. | | | 91.2 | 91.6 |

Note: Analyses found at Welheim -

for 7019 catalyst showed

(a) 7019 dated 7.3.40

Charcoal 81.8%
Ash 18.2%

Ash

Sulphur 0.8%
Cr₂O₃ 10.6% = 7.3% Cr
Al₂O₃ 6.8% = 3.5% Al

(b) 7019 dated 30.7.41.

Total ash 18-23%, total sulphur 2%
Active charcoal + 7% Cr, 1% Va, 1.75% Fe,
4.5% Al.

VAPOUR PHASE DISTILLATION ("B" Distillation)

5 units were available for the distillation of the combined saturation stall and splitting stall products. The capacities of these plants were:-

| | |
|---------------------------------|------------------|
| B ₁ + B ₂ | 18.4 te/hr. each |
| B ₃ B ₄ | 28 te/hr. each |
| B ₅ | 12.3 te/hr. |

Four units were said to be capable of taking the normal load with five coal stalls on line, but as the average feed to the vapour phase stills was 85-90 tes/hr. the distillation position must have been tight when one of the large stills was shut down. Rich gas from the stabilisers was combined with the rich gas from the liquid phase stills for separation of propane and butane. The butane was further fractionated into iso and butane, the iso butane being exported to Scholven.

Table VII (cont'd)

Petrol from splitting alone

| | |
|------------------|-------------|
| Volatility 100°C | 57 - 60.5 |
| O.N. clear | 73.5 - 73.7 |
| Vol. % aromatics | 3.3 - 3.5 |

At this time stalls 305 and 307 were doing -

| Stall | 305 | 307 |
|--|---------|---------|
| Age - days | 334-364 | 125-155 |
| Throughput | .36-.39 | 1.08 |
| Av. Temp. | 422 | 395 |
| Petrol Leistung te/m ³ /hr | .185 | .51 |
| Petrol to 155° in C.C.P. | 46-47 | 48-51 |

(B) Petrols Produced

- (i) During 1940 and early 1941 (information from shipping memos). At this time liquid phase petrol was

| | Petrol L-BC sq .735- .739 | refined separately |
|------------------|------------------------------|--------------------|
| Vol % at 100° | 54 | |
| 75° | 20 | |
| FBP 98% to 155°C | | |
| Aromatics | 13-15% | |
| Naphthenes | 48 | |
| O.N. CFR MM | | |
| Clear | 72-74 | |

- (ii) From Oct. 1942- Sept. 1943 (From annual report on distillation). At this time liquid and vapour phase petrol were being distilled off together.

| | |
|------------------------|------------------------|
| SG | .733 - .736 |
| Volatility at 100°C | 54-58 (average 55) |
| FBP | 154-160 (average 159) |
| Vapour Pressure (ats.) | 0.419-0.445 (av. 0.44) |
| Octanes number | |
| CFR MM clear | 71.9-72.4 |
| Vol % aromatics | 5.2-5.8 |
| Naphthenes | 54-59 |

(iii) Petrols cut from saturation stall product.

| ON | CFRMM | Clear | Leaded |
|--------|--------|-------|--------|
| End pt | 140 °C | 68.6 | 86.4 |
| | 145 | 68.2 | 85.8 |
| | 150 | 67.3 | 85.1 |
| | 155 | 66.2 | 84.1 |
| | 160 | 66.1 | 84.2 |

At this time Scholven VT 705 petrol was 68.8 clear and 89.7 leaded.

(iv) Petrols produced in 1944

| | |
|---------------------|---------------------------------|
| 21.1.44 to 18.2.44. | |
| Octane no CFRMM | 71.6-72 clear |
| | 89.5-90 leaded (1.15 ccs/litre) |

Note: Petrol L.Bi made in 1940 and early 1941, before the aromatising experiment started, is known to be a blend of all the petrol made including the refined liquid phase petrol. The fact that it had a higher aromatic content and higher octane number than the petrols made after Oct. 1942 requires further investigation.

(C) PRODUCTION OF AROMATIC PETROL

From April 1941 to January 1942 a part of the petrol was made by treating coal middle oil under aromatising conditions with 7019 and 7360 catalysts. Small scale experiments had been done at 320 ats. and 510°C with both 421 and 7019 catalysts. The analysis of 421 was given in documents as 4.4% MO, 1.2% Al, 0.5% Zn, 93.9% aluminium silicate. Jacob thought that 7019 was an active carbon plus chromium and vanadium. It was concluded that they gave similar results.

They had not given serious consideration to the use of the Welheim catalyst at 700 ats. because it would have required too much new plant.

TABLE VI

Typical Operating Conditions For Vapour Phase Stalls. From Monthly Reports.

| | | Saturation Stall 302 March 1944 | |
|--|--|------------------------------------|------|
| Catalyst Age days | | 367-397 | |
| Throughput tes/m ³ /hr | | 0.5-0.65 | |
| Average Temp. | | 422-434 | |
| H ₂ p.p. ats. | | 267-270 | |
| Total gas m ³ /te/feed | | 4080-3520 | |
| Petrol leistung tes/m ³ /hr | | 0.15-0.19 | |
| Petrol % product oil | | 39.8-39.0 | |
| Gas make | | 11.4 - | |
| H ₂ used/te MO | | 600-600 | |
| te petrol | | 1910-2030 | |
| <u>Feed</u> | | | |
| Specific gravity | | .943 | .943 |
| FBP | | 335 | 336 |
| % Sulphur | | 0.34 | 0.31 |
| Phenols | | 12.3 | ? |
| Petrol in feed - volatility at 100°C | | 42 | 38 |
| Octane No. | | 68.3 | 67 |
| <u>Product</u> | | | |
| Specific gravity | | .804 | .800 |
| Aniline pt °C | | 46.7 | 45.0 |
| % petrol to 155° | | 37.5 | 41.7 |
| S.G. of petrol | | .736 | .741 |
| Aniline pt °C petrol | | 45.4 | 44.4 |
| petrol aromatics vol% | | 4.3 | ? |
| B - MO S.G. | | .855 | .856 |
| FBP | | 300 | 298 |
| An. pt °C | | 50.3 | 48.2 |
| Phenols % | | .01 | .01 |

At this time the throughput on the other stalls was -

| | |
|-----------|------------|
| Stall 303 | 0.4 - 0.42 |
| 304 | 0.58-0.63 |
| 306 | 0.59-0.7 |

(c.f. figure above)

B - MO = feed oil to splitting

Table VI (Cont'd)

Note : Since the saturation stall product is distilled with the splitting stall product the petrol leistung given above must refer to an arbitrary cut taken in a laboratory column for the purpose of estimating the performance of the saturation stalls. The properties of the B-middle oil are determined in the same way. The petrol leistung appears to refer to the total petrol including the 15% liquid phase petrol in the feed. The figures quoted for petrol leistung, petrol in product and hydrogen consumption are not entirely consistent.

TABLE VII

Splitting - Stall 301

March 1944

| | | | |
|---------------------------------------|--|---------------|------|
| Catalyst Age - days | | 346 - 371 | |
| Throughput tes/m ³ /hr | | 0.76 - 0.85 | |
| Average temp. °C | | 406 - 420 | |
| H ₂ p.p. ats. | | 269 - 258 | |
| gas/te feed | | 1850 - 1580 | |
| Petrol leistung te/m ³ /hr | | 0.36 - 0.365 | |
| Petrol in CCP % wt. | | 50.5 - 48.2 | |
| Gas make % on MO | | 22.6 - 23.7 | |
| Gas make satn. + splitting | | 17.95 - 16.45 | |
| H ₂ used/te feed | | 300 - 300 | |
| /te petrol | | 637 - 703 | |
| <u>Feed</u> | | | |
| an pt °C | | 50.7 | 49.5 |
| IEP °C | | 160 | 158 |
| FBP °C | | 300 | 300 |
| % S | | .47 | .50 |
| <u>Product</u> | | | |
| SG | | .755 | |
| Petrol | | 50.5 | 48.2 |
| Residue % of feed | | 46 | 45.8 |
| <u>Petrol from stills</u> | | | |
| FBP | | 160 | 158 |
| Octane number clear | | 71.4 | 71.4 |
| leaded | | 89.8 | 89.9 |
| vol % aromatics | | 5.5 | ? |
| volatility at 100 °C | | 55 | |

The let down engines were of standard I.G. design. Each engine consists of two vertical cylinders, each with a piston and rod through the top cover to act as a guide and to provide a differential area top-bottom side of the piston. The 700 ats. rich oil from the gas washing towers enters the bottom of the cylinder and drives the fresh wash oil into the gas washing tower. The rich oil is discharged to the let down sets at 40 ats. pressure and the cylinder is supplied with fresh oil on the suction stroke at 65 ats. by a centrifugal pump. This pump and the rich wash oil supply the motive power. The two cylinders operate alternately to provide a constant supply of wash oil. The engines are only 80% efficient and a make up of wash oil is provided by three throw 700 ats. injectors. The inlet and exhaust valves on the let down engines are operated by oil relay from the piston rods.

VI. VAPOUR PHASE, MIDDLE OIL HYDROGENATION.

(a) The vapour phase stalls employed a two stage process, 5058 saturation followed by splitting over 6434 catalyst. Both operations were carried out at 300 ats. pressure. It was noted that the throughputs in relation to catalyst volumes in both stages were low as is usual in I.G. practice. The overall petrol leistung for the two stages was only 0.2-0.25 tes/m³/hr. There were four saturation stalls (Vorkammer) each with three converters with a total of 24 M³ catalyst space, and 3 splitting stalls (Benzinierungs Kammer) also with three converters and 24 M³ catalyst space. Figs. 2 & 3 show typical line diagrams for these stalls.

Six of the seven vapour phase stalls had gas fired preheaters. The seventh stall had been erected between two others and there was no room for a preheater outside the converter bay. An electric preheater had been installed in the bay. The I.G. once favoured the electric preheater but now gas fired heaters are preferred. The electric preheater is most suitable for stalls which are almost autothermic, with heavy preheater loadings great care is necessary if high metal temperatures are to be avoided.

The vapour phase converters were of the hot wall type with downward flow and crossed slit mixing chambers between the beds. The cooling gas connections were made through the top cover.

The maximum feed rate to a saturation stall was 0.7 tes/m³/hr of catalyst volume, the feed contained approximately 15% of coal stall petrol. The average feed rate was 0.5 - 0.6 tes/m³/hr, temperatures varied from 400°C to 442°C according to the age of the catalyst. The inlet pressure was 300 ats. with 250 ats. hydrogen partial pressure. The gas circulators were capable of 40 ats. boost. It can be calculated that with four saturation stalls for a production of 320,000 tes/yr of petrol, the feed to saturation would be 48 tes/hr or an average rate of 0.5 tes/m³/hr.

Until November 1942, 5058 catalyst was used in all converters for saturation. Because of the shortage of tungsten one converter in each stall was replaced by catalyst, 7846 (also known as 8376). This was 25% WS₂, 3% NiS on active alumina. The change over on all four stalls was complete by March 1943. This catalyst was better than 5058 for removing phenols but its activity for removing nitrogen was poor. Although the throughput on saturation stalls was reduced, a bigger load was put on the splitting stalls which had to be run with higher temperatures and the life of splitting stall catalyst was reduced. The proportion of saturation stall petrol in the final petrol made fell from 42% with 5058 catalyst alone to 36% when all four stalls had one converter of 7846. After June 1943 some change was made in the method of operating the saturation stalls which raised the percentage of saturation stall petrol to 40%. Examination of the stall record books is required to explain this. The overall gas make from the vapour phase stalls rose from 16% to 19% on the middle oil converted.

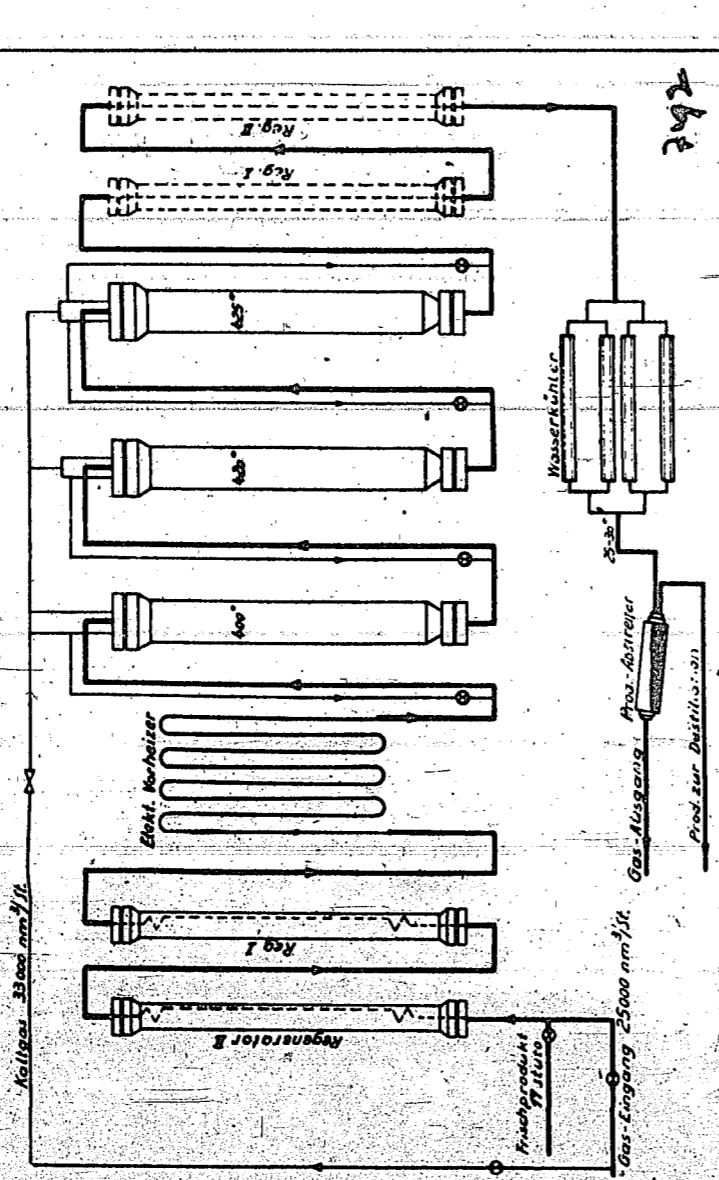
In splitting stalls the throughput was 1.1 tes/m³/hr decreasing to 0.8 as the catalyst aged. The petrol leistung was on the average 0.5 tes/m³/hr. The average catalyst temperatures were raised from 374°C to 434°C during the life of the catalyst.

The catalyst charges in use at the end of 1942 were given as:-

| | | |
|-----------|-------------------|--|
| Stall 301 | 16 M ³ | 2 x 5.8 tes 6434 |
| 302 | 24 " | 18.3-18.8 tes/converter 5058 |
| 304 | 24 " | 18.2-18.7 tes/converter 5058 |
| 305 | 24 " | 5.8 tes/converter 6434 |
| 306 | 24 " | 18 tes/converter 5058 |
| 307 | 32 " | 3 @ 5.5 tes 7019) see note on 1 @ 6.3 tes 7360) aromatising |

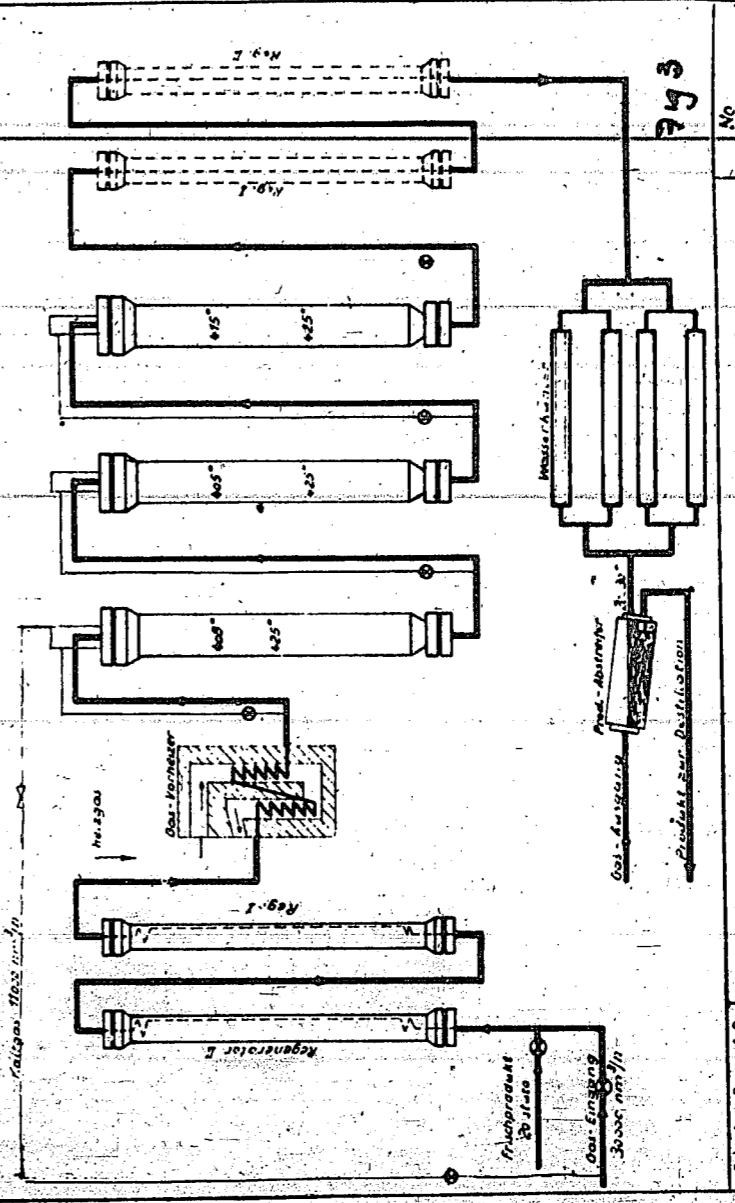
Typical operating data for saturation and splitting stalls are given in Tables VI and VII.

Vorkammer mit Elektro-Vorheizer u. 3 Öfen = 24 cbm Kontakt 3058



Gas-Eingang 25000 nm³/h
 Göttinger Werke A.G.
 Siedewerk
 No. 342

Benzinierungs-Kammer m. Gas-Vorheizer u. 3 Öfen = 24 cbm Kontakt 6434



Gas-Eingang 30000 nm³/h
 Göttinger Werke A.G.
 Siedewerk
 No. 343

about 60-70% hydrogen, a total of 5-6000 m³/hr was returned to the compression plant to be added to the make-up gas. The H.O.L.D. was measured by Eckardt displacement meters on its way to the main storage tanks. The whole of the H.O.L.D. was diluted to 16% solids by the addition of liquid phase heavy oil before feeding it to the fugals. The fugal feed was maintained at about 170°C. The filtrate containing 9-11% solids was used for making up pasting oil. The concentrate containing 35-36% solids was obtained at a temperature of 160 C. The fugals were fitted with Widia nozzles to stand up to the erosion by the concentrate discharged continuously. There were 12 kilns (Kugel ovens) installed, similar to those used at Scholven. They were 11.3 metres long, 2.2 metres diameter inclined to the horizontal 2° down to the exit end. Each kiln had two baffles for retaining the grinding charge, these heavily ribbed to reduce the hammering effect of the charge and the baffles were placed 2 metres and 5 metres respectively from the exit end. Experiments on various types of charge indicated that heavily ribbed diamond shaped iron bodies, 4-5 inches long and across, gave the best results. The kilns shells were arranged in a gas heated brick setting with the flined feed preheater and steam heater arranged longitudinally on the top. The feed was preheated to 350°C by the preheater, there was no interchange with the products. The kiln shell was run at 560-610°C. The oil vapours and steam from the kiln were first scrubbed with hot oil to remove coke dust carried forward. The scrubbing oil was sometimes returned to the kiln if the solid content was abnormal. The vapours were then condensed and after separation of water (introduced as steam) the oil was used as a constituent of pasting oil. The coke residue was discharged through a water seal onto a belt conveyor from which it was loaded into trucks. It was burnt on boilers as it had a low alkali content. At Scholven special boilers had been installed to stand up to the sodium salts in the ash.

8 - 9 kilns were required to deal with the concentrate from 5 - 6 stalls. The normal feed was 2.7 - 3.0 tes/hr with the addition of 150-250 kgs/hr of steam preheated to 450°C countercurrent to the feed.

Because of the low asphalt content of the feed, solids/asphalt 4.5/1, the kilns ran 6 - 12 weeks before decoking was necessary. Scholven running with a lower ratio of solids to asphalt, 3.5/1, could get only about a months run. The kilns at Gelsenberg had never limited the stalls. The main problem in carbonising the concentrate is bound up with the asphalt content since the asphalts determine the plastic range. No kiln shells had been changed but every year they were relined with "coronit", the weld metal being laid in the form of short runs along the axis of the kiln. The ribbed partitions were changed once a year.

(E) Liquid phase distillation ("A" Distillation)

There were four units for the distillation of coal stall cold catchpot product. Three units could deal with the full output of the stalls. The tar oils imported were added to the cold catchpot product before distillation, these oils usually contained 90-95% of middle oil boiling to 330°C.

Until August 1942, the liquid phase petrol which amounted to about 10% on the coal, was removed as a separate fraction and it was refined separately from the vapour phase petrol. After August 1942 petrol and middle oil to 335-343°C were taken off together and the liquid phase petrol was refined with the middle oil in the saturation stalls. The refined petrol was not passed through the 6434 splitting stalls. The rich gas from the coal stall let down sets and from the stills was combined with the vapour phase rich gases for petrol recovery and gas fractionation.

(F) Gas Washing

At Welheim where the vapour phase stalls were run at 700 ats. no separate washing of the circulating gas was necessary. At Gelsenberg it was usual to wash the circulating gas from the coal stalls with wash oil equivalent to 3.3 m³/oil/tonne coal. When there was a shortage of hydrogen the amount of wash oil was reduced to 3 m³/tonne of coal in order to reduce the hydrogen partial pressure and reduce the loss of hydrogen as dissolved gas. The wash oil with its dissolved gas was let down to 40 ats. through the let down engines which provided about 80% of the power needed for wash oil injection; three let down engines were provided. The wash oil at 40 ats. was then let down in two stages to 7 ats. and 1 at.

TABLE IV (cont'd)

B. a.m.f. coal tes/m³/hr

| | 1942 | 1943 | |
|---------|------|------|------|
| Jan. | .375 | .385 | .360 |
| Feb. | .380 | .366 | .400 |
| March | .400 | .336 | .340 |
| April | .420 | .392 | .340 |
| May | .430 | .355 | |
| June | .380 | .374 | |
| July | .370 | .365 | |
| Aug. | .360 | .395 | |
| Sept. | .362 | ? | |
| Oct. | .360 | .365 | |
| Nov. | .387 | .380 | |
| Dec. | .395 | .350 | |
| Average | .384 | .369 | .360 |

(C) Notes on Preheater Troubles

The preheater tubes had proved to be a major difficulty in the operation of coal stalls at 700 ats. pressure. Preheater bursts started as soon as the plant was put into operation. The troubles could be divided into two sections, (a) with the return pieces at the bottom of the preheater elements and (b) failures of the tubes themselves. Failures from the two causes were about equal. We saw a photograph of the damage to a stall resulting from a burst tube, in this case the converters and interchanger had fallen out of the structure.

When the plant was built they took into consideration the experience at Scholven with corrosion of the bends at the bottom of the preheater elements and they fitted the special return pieces which had been developed at that plant. They had considerable trouble with the failure of these return pieces arising out of cracks starting at change of sections, an apparent weakness to which N.10 is particularly susceptible. The return pieces are characterized by the presence of plugs screwed into the forging and then welded. At first they had a sealing weld only but gradually changed to a plug which was held in by the welding but they still had trouble. After some time they agreed that as they

were working under alkaline conditions there was no need for the elaborate precautions they were taking against corrosion and they made experiments with preheater hairpins with the normal return bend as fitted to the I.G. preheaters at Leuna. Examination of the trial bends after a years service showed that they were getting no corrosion or erosion and they were changing all their elements to the normal design.

As to the failures of the preheater tubing itself they were of the opinion that they were due to two causes (a) the presence of inclusions in the steel, and (b) the use of an incorrect heat treatment for the steel. They had had no failure of the tubing outside the preheater and had had only one or two failures of the forged pieces such as Y-blocks and T-pieces. At the first they were getting failures after a stall had been on line about 4-6 weeks but as a result of the changes that had been made they were able to run at least six months without any bursts.

Changes were made in the manufacture of the tubing at the steelworks and in the method of heat treatment carried out at Leuna. They also limited the tube metal temperature to 520°C. They were questioned about their ability to measure metal temperatures accurately and they were satisfied that they were measuring the metal temperature and not some temperature intermediate between the flue gas and the metal temperatures. With a clean stall, without paste interchange, they could achieve the desired paste throughput with metal temperatures of 480°C. As the preheater became dirty it was necessary to limit the throughput to keep the metal temperatures below 520°C. The use of paste interchange made it possible to maintain full output.

The composition of N10 steel was varied from time to time, the approximate analysis was C 0.23, Cr 3.0, Va 0.75 Mn 0.4 Si 0.4 Mo 0.4 WO.4.

(D) Sludge Plant

The H.O.L.D. was let down from the hot catchpot via a H.P. finned air cooler to the fugal house. The let down from 700 ats. was carried out in one stage by a valve fitted with a widia nose and seat and a pressure reducing nozzle of widia. This was changed every 800 hours. The H.O.L.D. was received in a let down vessel, one to each stall and three spares. The dissolved gases from these vessels contained

Table V shows the composition of Bayer Masse (Kontakte II).

TABLE V
Analyses of Bayer Masse (Kontakte II)

| | From Hilsch. | | |
|--------------------------------|--------------|-------|-------|
| | % | % | % |
| H ₂ O | 22 | ? | ? |
| Loss on Ignition | 38.05 | 6.21 | 15.34 |
| CO ₂ | - | 2.2 | 2.62 |
| SiO ₂ on residue | 6.42 | 11.62 | 12.50 |
| Fe ₂ O ₃ | 39.57 | 63.87 | 46.31 |
| Al ₂ O ₃ | 11.73 | 5.92 | 14.20 |
| TiO ₂ | 0.99 | 11.20 | 5.79 |
| Cr ₂ O ₃ | 0.99 | | |
| CaO | 0.92 | | |
| SO ₃ | 1.36 | 2.96 | trace |
| MnO ₂ | | 0.31 | nil |
| Na ₂ O | | 3.88 | 4.76 |

TABLE III

Data abstracted from monthly graphs

| | March 1944 | April 1944 |
|--|-------------|-------------|
| | 15th - 31st | 1st - 30th |
| Coal and Oil throughput tes/m ³ /hr | 1.0 - 1.1 | 1.05 |
| H ₂ partial pressure ats. | 545 - 560 | 550 |
| Average temperature °C | 476 | 478 |
| H ₂ used m ³ /te a.m.f. coal | 1200 - 1250 | 1200 - 1300 |
| Heavy oil let down % of coal | 110 - 130 | 120 |
| Solids in H.O.L.D. | 23 - 25 | 23 |
| Asphalt in oil | 17 - 20 | 20 - 23 |
| Cold catchpot product wt % to 330°C | 47 | 47 |
| Paste Oil | | |
| wt % solids | 9 | 9 - 9.5 |
| asphalt in oil | 8 - 9 | 9 - 10 |

TABLE III (cont'd)

| | March 1944 15th - 31st | April 1944 1st - 30th |
|--|---------------------------|--------------------------|
| New formed Oil | | |
| wt % a.m.f. coal | 68 - 69 | 69 |
| Leistung tes/m ³ /hr | .27 - .24 | .24 |
| % petrol + MO - 330° in new formed oil | 85 - 100 | 100 |
| Gas make % a.m.f. coal | 25.5 - 25 | 24.5 |
| Stalls running | 6 | 6 |

TABLE IV

Throughput and operating times - Coal Stalls

(A) % Running time

| | 1941 | 1942 | 1943 | 1944 |
|----------------|---------------|------|------|------|
| | A | A | A | A |
| Jan. | | | | |
| Feb. | Oct. 1940 | 79 | 85 | 96 |
| March | to June 1941 | 76 | 90 | 72 |
| April | fell to 40% | 82 | 100 | 93 |
| May | rising to 90% | 86 | 85 | 97 |
| June | in June | 87 | 92 | 96 |
| July | 90 | 88 | 86 | |
| Aug. | 85 | 94 | 80 | |
| Sept. | 83 | 96 | 62 X | |
| Oct. | 80 | 97 | 82 | |
| Nov. | 78 | 100 | 83 | |
| Dec. | 70 | 88 | 73 | |
| | 74 | 90 | 91 | |
| Average | 80 | 88.6 | 84.1 | 91 |
| | June-Dec. | | | |

100% equals 6 stalls on line

X Air Raid damage

Flowsheet III gives the overall gas make and gas composition for coal and vapour phase stalls when making 320,000 tes/yr of aviation petrol.

FLWSHEET III

Gas Flowsheet dated 6.7.44
To make 39.7 te/hr. VT 707 Aviation petrol
320,000/yr (8,000 hrs)

| | | | |
|-----------------------------------|-------------------|---|--------|
| <u>Hydrocarbon Gas Production</u> | | <u>Hydrocarbon Gas - Usage</u> | |
| Coal Stalls | Te/hr. | Fuel Gas consumed | te/hr. |
| Gas phase | 21.345 | " exported | 5.341 |
| Heiz Gas | 10.467 | Buna Gas C ₂ H ₄ etc. | 1.041 |
| Gas Benzin | 31.812 | Liquefied Gas | 4.786 |
| Vergasung | 4.483 | Gas purged | 12.829 |
| | = 27.329 | Loss | 1.789 |
| | | | 1.543 |
| | | | 27.329 |
| 86% C - Vergasung | = 25.7 | <u>Composition of gas:</u> | |
| | = 39.4% Petrol + | | % |
| | Vergasung | | 15.4 |
| Liquefied gases. 1-C ₄ | - 2.374 t/hr. | | 19.3 |
| Treibgas | - 10.455 t/hr. | | 29.2 |
| | | | 17.3 |
| Total | = 12.829 te/hr. | | 1.7 |
| | = 32.6% of petrol | | 100 |

FLWSHEET III (cont'd)

Iso-butane separated 7007 m³/hr = 2.374 te
Composition: C₃ 2.5%, 1-C₄ 93.6%, n-C₄ 3.9%

Buna Gas 4828 m³/hr. (Linde Separation)

| | | | |
|----------------------------------|-----|----------------|------|
| H ₂ S-CO ₂ | 0.2 | C ₁ | 37.7 |
| CO | 0.8 | C ₂ | 25.3 |
| H ₂ | 9.2 | C ₃ | 14.8 |
| N ₂ | 5.4 | C ₄ | 6.6 |

Treibgas 4958 m³/hr. = 10.455 t/hr.
= 26.3% of petrol.

We were told that on the average they had five out of the six coal stalls on line. This represents 83% operating time for all stalls. Table IV shows the operating times achieved month by month, over a year they achieved:-

| | |
|-----------------|---------------|
| Up to June 1941 | as low as 40% |
| June- Dec. 1941 | 80% |
| 1942 | 88.6% |
| 1943 | 84.1 |
| Jan.- May 1944 | 91 |

The average coal (a.m.f.) throughputs were

| | | |
|------|-----------------------------|------------------------|
| 1942 | .384 tes/m ³ /hr | max .430 (for a month) |
| 1943 | .369 tes/m ³ /hr | max. .392 (") |
| 1944 | .360 tes/m ³ /hr | max .400 (") |

Flowsheet III gives the overall gas make and gas composition for coal and vapour phase stalls when making 320,000 tes/yr of aviation petrol.

FLOWSHEET III

Gas Flowsheet dated 6.7.44
To make 39.7 te/hr. VT 707 Aviation petrol
320,000/yr (8,000 hrs)

Hydrocarbon Gas Production

| | |
|-------------|----------|
| Coal Stalls | Te/hr. |
| Gas phase | 21.345 |
| Heiz Gas | 10.467 |
| Gas Benzin | 31.812 |
| | - 4.483 |
| Vergasung | = 27.329 |

86% C - Vergasung = 25.7
= 39.4% Petrol +
Vergasung

Liquefied gases. 1-C₄ - 2.374 t/hr.
Treibgas - 10.455 t/hr.

Total = 12.829 te/hr.
= 32.6% of petrol

Hydrocarbon Gas - Usage

| | |
|---|--------|
| Fuel Gas consumed | te/hr. |
| " " exported | 5.341 |
| Buna Gas C ₂ H ₄ etc. | 1.041 |
| Liquefied Gas | 4.786 |
| Gas purged | 12.829 |
| Loss | 1.739 |
| | 1.543 |
| | 27.329 |

Composition of gas:

| | | |
|------------------|---------|------|
| C ₁ | tes/hr. | % |
| C ₂ | 4.205 | 15.4 |
| C ₃ | 5.272 | 19.3 |
| 1-C ₄ | 7.977 | 29.2 |
| C ₅ | 4.751 | 17.3 |
| | 0.465 | 1.7 |
| | 27.329 | 100 |

| |
|-------|
| % gas |
| 19.6 |
| 5.8 |
| 17.5 |
| 47.0 |
| 6.5 |
| 5.6 |
| 100 |

FLOWSHEET III (cont'd)

Iso-butane separated 7007 m³/hr = 2.374 te
Composition: C₃ 2.5%, 1-C₄ 93.6%, n-C₄ 3.9%

Buna Gas 4828 m³/hr. (Linde Separation)

| | | | |
|----------------------------------|-----|----------------|------|
| H ₂ S-CO ₂ | 0.2 | C ₁ | 37.7 |
| CO | 0.8 | C ₂ | 25.3 |
| H ₂ | 9.2 | C ₃ | 14.8 |
| N ₂ | 5.4 | C ₄ | 6.6 |

Treibgas 4958 m³/hr. = 10.455 t/hr.
= 26.3% of petrol.

We were told that on the average they had five out of the six coal stalls on line. This represents 83% operating time for all stalls. Table IV shows the operating times achieved month by month, over a year they achieved:-

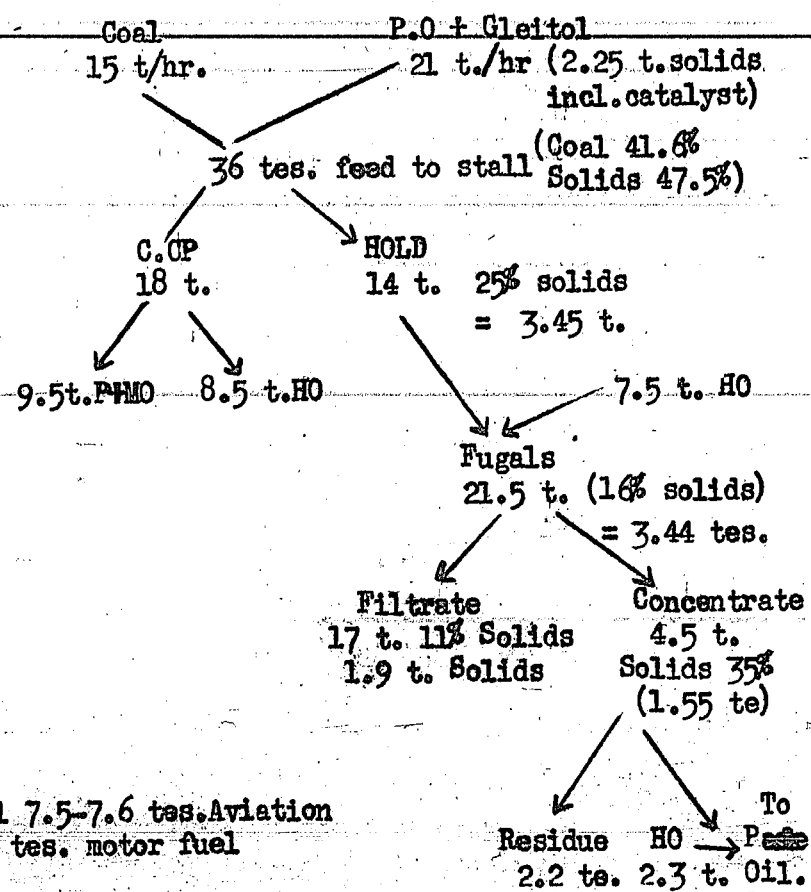
| | |
|------------------|---------------|
| Up to June 1941 | as low as 40% |
| June - Dec. 1941 | 80% |
| 1942 | 88.6% |
| 1943 | 84.1 |
| Jan. - May 1944 | 91 |

The average coal (a.m.f.) throughputs were

| | | |
|------|-----------------------------|------------------------|
| 1942 | .384 tes/m ³ /hr | max .430 (for a month) |
| 1943 | .369 tes/m ³ /hr | max .392(") |
| 1944 | .360 tes/m ³ /hr | max .400(") |

Three flowsheets are also given, Flowsheet I was prepared for us by Dr. Jacob and this represents recent operating conditions.

FLWSHEET I
Coal Stall Flowsheet (Jacob)



From this flowsheet it can be calculated that -

- (a) Oil recovery was 78% on oil + asphalt fed to the kilns.
- (b) Oil lost at sludge was 0.65 tes/hr. or assuming 6% ash + H₂O in coal, 4.6% on the a.m.f. coal.
- (c) Final petrol yield is

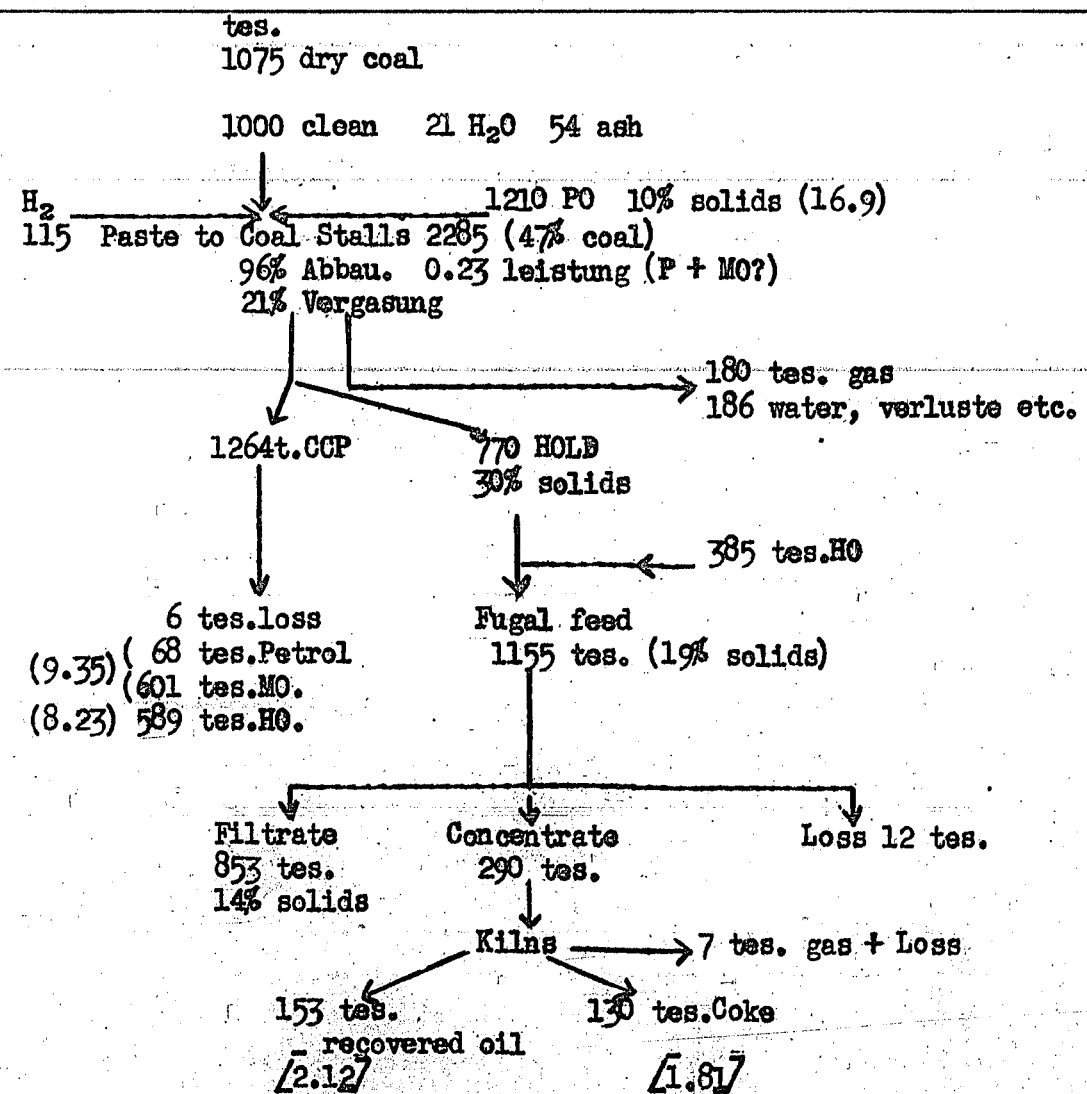
$$\frac{7.6}{.94 \times 15} = 53.8\% \text{ aviation petrol}$$

or

$$\frac{8.5}{.94 \times 15} = 60.3\% \text{ motor fuel}$$

Flowsheet II was prepared in 1939, it represents what they expected to achieve.

FLWSHEET II
Coal Flowsheet 1939, 700 ats. (From Records)



Note: Figures in brackets are scaled down to a basis of 15 t/hr. dry coal for comparison with Jacob's Flowsheet I.

H.O.L.D. (cont'd)

Vapours from HCP 435°C. H.O.L.D. cooled by addition of hydrogen to 375°C.

Fugal feed. All H.O.L.D. diluted with liquid phase heavy oil to 16% solids.

Fugal filtrate. 9 - 10% solids, 14% asphalt in oil. This is used for making up paste oil.

Fugal concentrate. 35-36% solids.

The temperatures in the converters were adjusted in relation to the coal throughput so that on the average the new formed heavy oil was equal to the heavy oil lost at the sludge plant i.e. 4-5% on the a.m.f. coal. The average make of new formed oil was 68-70% on the a.m.f. coal, of this 95-96% was petrol and middle oil to 330°C. The system was, therefore, balanced as regards heavy oil make and no separate heavy oil hydrogenation was carried out.

Detailed operating data taken from records found on the plant are set out in tables I, II & III below.

TABLE I

Coal Stall

Typical Operating Data from Records

Stall 702 :- Feb. 1944

| | 16.1 | 15.3 | Calculated coal in |
|----------------------------------|-------|-------|--------------------|
| Freshcoal/hr. | 16.1 | 15.3 | paste plus gletol |
| Throughput Kg/l/hr. (Paste) | 1.16 | 1.14 | 38.6 and 37.2% |
| m ³ /gas/t. feed | 2,100 | 2,110 | |
| m ³ /gas/t. coal | 5,490 | 5,740 | |
| Cold gas % of total gas | 60.3 | 56.2 | |
| H ₂ p. pressure ats. | 544 | 562 | |
| Middle temp. °C. | 478 | 475 | |
| C.C.P. t/hr. | 18.9 | 18.7 | |
| H.O.L.D. t/hr. | 18.0 | 17.2 | |
| % Solids in oil in H.O.L.D. | 22.9 | 21.6 | |
| % Asphalt in oil in H.O.L.D. | 17.1 | 14.0 | |
| H.O.L.D. % Coal | 112 | 113 | |
| " Sp.Gr. | 1.207 | 1.197 | |
| Ash in Solids | 64.7 | 64.1 | |
| < 325° | 7.6 | 8.8 | |
| Abbau | | 96 | |
| Vac. residue > 325 | 30.5 | 27.5 | |
| Cold catchpot product % to 330°C | 49 | 48.9 | |

TABLE I (cont'd)

At this time the stalls as a whole were taking the following rates:

| | | | |
|-------|-------------|---------|--|
| 701 | 10 - 12 | tes/hr. | |
| 702 | 12 - 14 | " | |
| 703 | 12 - 14 | " | |
| 704 | 14 | " | |
| 705 | 12.5 - 15 | " | |
| 706 | 12.5 - 15.5 | " | Stall 701 had run |
| Total | 73 - 74.5 | " | 480 days (April 1944), Stall 707 - 365 days. |

TABLE II

(Extracted from records)

| Stall : | 701 | 702 | 706 | 706 |
|--|---------|-----------|---------|----------|
| | 5.1.43. | 20.2.43. | 5.3.42. | 26.4.43. |
| Paste te/hr. (46% coal) | 32 | 32 | 34 | 35 |
| Paste oil te/hr. | 6.0 | 5.0 | 3.0 | 5.0 |
| Wash oil te/hr. | 1.7 | 1.7 | 1.5 | 1.7 |
| Inlet gas m ³ /hr | 25,000 | 31,000 | 27,000 | 27,000 |
| Brei gas | 3,000 | 3,000 | 3,000 | 3,000 |
| Cooling gas m ³ /hr converters: | | | | |
| 1 | 12,000 | 12,000 | 5,000 | 9,200 |
| 2 | 8,000 | 8,000 | 8,500 | 10,800 |
| 3 | 7,000 | 5,000 | 6,500 | 4,500 |
| 4 | 14,000 | 12,000 | 7,500 | 5,900 |
| Exit gas | 65,000 | 55,000 | 53,000 | 64,100 |
| Gas to H.C.P. | 6,600 | - | 6,000 | 6,000 |
| Pressure ats. inlet | 667 | 663 | 670 | 677 |
| exit | 640 | 636 | 638 | 638 |
| P.D. ats. | 27 | 27 | 32 | 39 |
| Converter temps. | | 475 - 470 | | |
| H.O.L.D. m ³ /hr. | 15.6 | 15.8 | 17.7 | 16.2 |
| te/hr. (approx) | 18.6 | 18.9 | 21.2 | 19.5 |
| Cold Catchpot (S.G.1.001) | 21.3 | 19.9 | 18.6 | 20.2 |

(b) Coal-Stall-Operating-Conditions

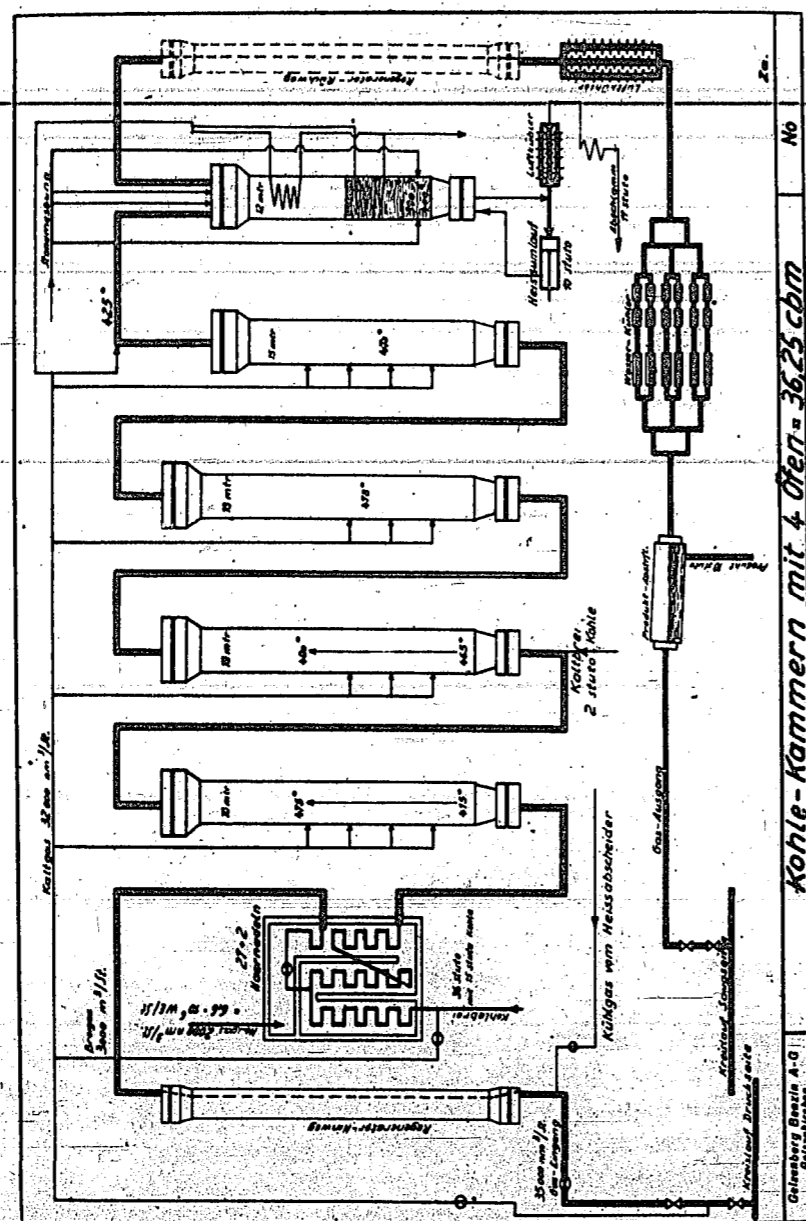
Coal paste was made up to 45-46% coal. The catalyst amounted to 1.2% Fe SO₄ (Kontakt I), 1.5% Bayer Masse (Kontakt II) a by product of the aluminium industry and 0.3% Na₂S (Kontakt III), all expressed on dry coal.

The sodium sulphide was added to remove the chlorine which appears as HCl during decomposition of the coal. When the coal stalls started up there was no paste interchange and the load on the preheaters was such that tube metal temperatures often exceeded 520°C and tubes bursts were frequent. The preheaters have proved to be a major operating difficulty at 700 ats. To alleviate this trouble paste interchange was introduced and it was then necessary to inject additional pasting oil with the 46% coal paste to dilute the paste to about 40% of coal in order to reduce pressure drop. An ordinary tube stack interchanger was employed and all the paste and additional paste oil (gleitöl and 13,000 m³/hr of gas were put through the shell. The pressure drop was at times as high as 45 ats. but this did not cause serious difficulty. The use of a paste interchanger enabled them to keep preheater tube metal temperatures below 520°C and this reduced the frequency of tube bursts. For further details concerning preheater tubes see section, C.

Consideration was given to reducing the preheater load by injecting coal paste in place of cooling gas to control temperatures in the converters but we do not know whether this was put into practice.

Summarised data from Dr. Jacob

- Stalls 6 + 1 under construction
- Converters 4 = 36 m³
- Coal Feed 13 to 15 tes/hr/stall (raw coal 94% a.m.f. coal)
- Coal in paste 45 - 46%. Total solids 53%
- Paste oil solids 9%
- Preheaters 27-29 hairpins, flue gas 590°C maximum, preheater metals 520°C maximum, Paste exit preheater 417-435°C.
- Converter temps. 480°C max. 476°C normal
- H.O.L.D. 14-15 tes/hr for 33 tes/hr paste 24% solids 18% asphalts in oil in H.O.L.D. (by Ludwigshafen method)



V. COAL HYDROGENATION

(a) Paste Preparation and Coal Stalls.

Coal was brought from Nordstern III & IV collieries, this was washed coal with 4 to 4.5% ash, 30% volatiles and 0.05 to 0.1% chlorine. Its carbon content was 83% on an ash and moisture free basis (a.m.f.) More recently a coal of 81% carbon content from Leopold colliery had been used. The washed "raw" coal usually contained 8 - 12% moisture. From the analyses available it is clear that a careful watch on quality was kept, in particular on the content of bright constituents. The coal was first crushed to < 8 mm in a hammer mill and then it was dried during grinding by circulation of hot flue gas to the ball mills. CO₂ was added to reduce the risk of explosion, in this operation the water content was reduced to 2% or less. The coal was swept out of the ball mill by the flue gas stream, through a classifier which returned oversize to the mill, to a cyclone and weighing hopper. The coal was ground so that 40-45% passed through a 10,000 D.I.N. sieve. The dry, ground coal was put to the Concentra rod mills together with the pasting oil and catalyst. Heavy oil pasting oil was invariably used. This was made up from fugal filtrate, recovered oil from the kugel ovens and surplus liquid phase heavy oil. The rate of pasting oil was measured by Eckhardt meters and it was adjusted to give 45-46% coal in the paste. The paste mills were jacketted to maintain a paste temperature of 120-130°C.

There were three ball mills each with its corresponding paste mill. Apart from the use of CO₂ in the coal mills, the layout and method of operation was similar to that at Scholven.

The coal paste was circulated under pressure around a ring main feeding the high pressure paste injectors. There were 38 injectors in all each rated at a nominal 10 m³/hr paste, they could do 15 m³/hr maximum. These injectors were horizontal machines with central hydraulic cylinder with paste cylinders one at each end of the machine. With six coal stalls on line the total volume of paste and pasting oil to be injected was about 200 m³/hr. The coal stalls operated at a total pressure of 700 ats. and a hydrogen partial pressure of 550 ats. The stalls were arranged in the same way as Welheim and Scholven. The

preheaters were in line with the converter bays. The convection coolers for heavy oil let down (H.O.L.D.) and product from the interchanger and the four parallel banks of water coolers for product were mounted on a gantry behind the converter bay. The cold catchpot and circulator catchpots were accommodated in the space between the back of the converter bay and the control room. The injectors and circulators were housed in four buildings behind the stall control rooms. A good idea of the layout can be obtained from the photograph of the stalls at Welheim in the Welheim report. There were six coal stalls built in pairs and a seventh had just been completed. The high pressure vessels, converters, interchangers and hot catchpot were enclosed in a concrete box with an opening at the front side for the purpose of removing vessels for maintenance work. The high pressure vessels were stood on stools which were protected from the heat of a fire by cement lagging. This construction made access to bottom joints very difficult as pipe joints had to be made and broken in the confined space inside the stools. Each stall had four converters making a total reaction volume of 36 m³ per stall; and one product - paste interchanger. For a short time five converters had been used in one stall. The converters which had V2A liners were insulated internally with cement asbestos lagging 65 mm thick the same as for 325 ats. vessels. They said that they had no trouble with coke building up in the converters. Balg-linsen, elastic lens rings, were fitted to the hot parts of all stalls. The preheaters were of the rectangular type with 3 or 4 flue gas distributing ports. The branches from the twin circulating fans were lagged externally with a very light insulating brick. The rate of flue gas circulation was stated to be 150,000 m³/hr. The preheaters had 27-29 hairpins.

On the coal stall interchangers the K-values were 200-150 kg cal./°C/hr/m² and these held for a considerable time so long as the exit paste temperature did not exceed 350°C. This temperature seems high and ought to be checked. The K-values for the preheaters were given as 5-6 in the cold pass, 3-4 in the middle pass 6-8 in the hot pass, all these values being expressed on the outer surface of the element.

A line diagram of a coal stall is shown in Fig.I. It will be noted that this represents the set up before the introduction of paste interchange. The hot recycle of H.O.L.D. in the hot catchpot and the cooling coil were originally used to stop settling in the catchpot, these were being abandoned in favour of the direct addition of hydrogen at the bottom of the catchpot. The liquid was cooled to about 375°C and the vapour left the catchpot at 435°C.