

Author and Title	C.I.O.S. File No.	Item No.
CHAFFEE, C. C. Wirtschaftliche Forschungsgesellschaft m.b.H. WIFO Fuel Blending Station. 1945. 7 p.	XXVI-68	30
CHAFFEE, C. C., ATWELL, H. V. I. G. Farbenindustrie Mainkur-Hoechst. (1945) 33 p.	XXVI-79	30
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COCKRAM, C. Visit to Gelsenberg hydrogenation plant. 1945. 41 p. diags.	XXX-105	30
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ELLIS, J. R., HIRST, L. L. Report on visit to Kukuck I plant at Niedersachswerfen. (1945) 5 p. diagr.	XXXII-15	30
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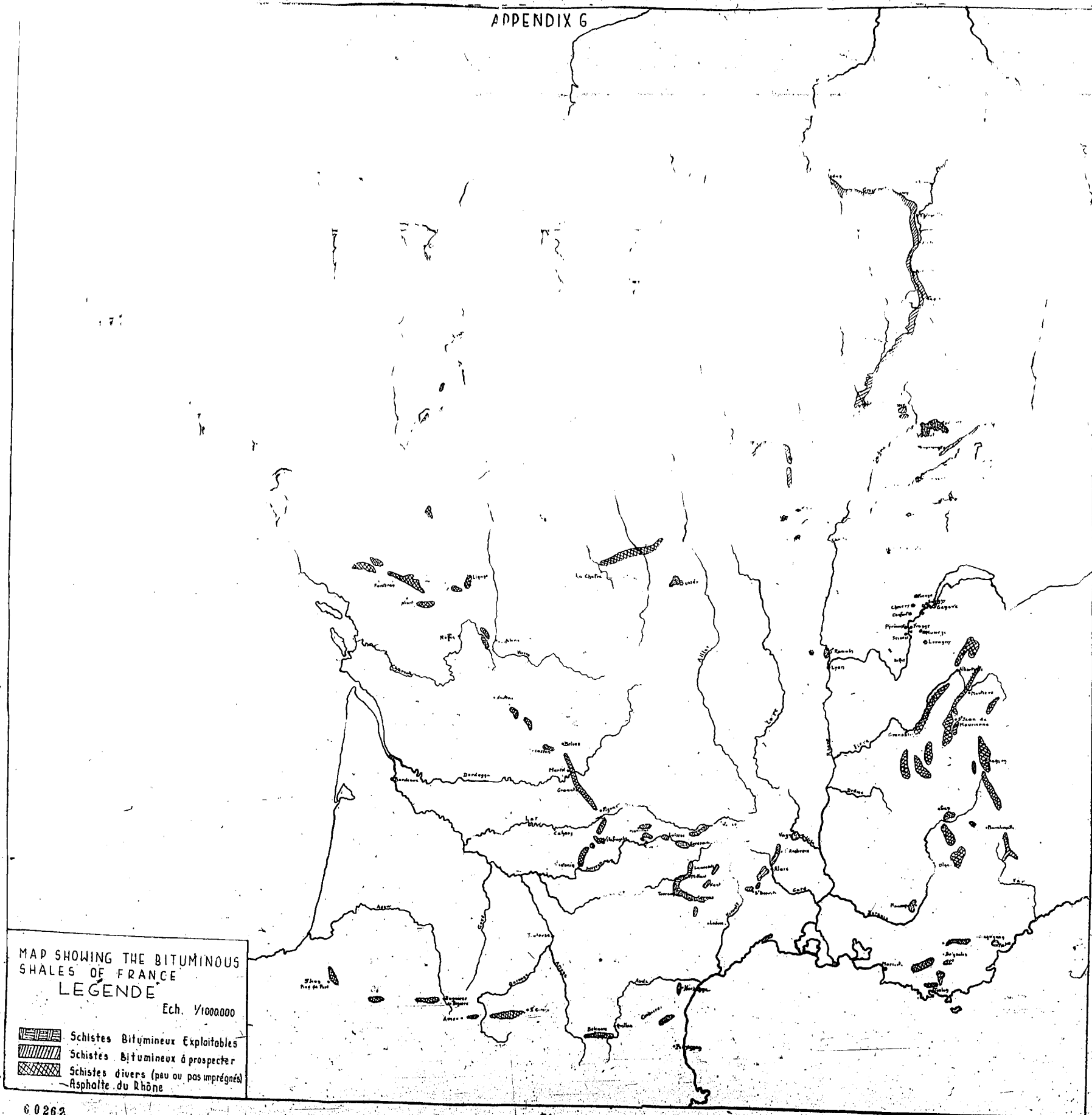
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The following items were written by members of the U. S. Government Technical Oil Mission during their visit to Germany in 1945. They are available on this microfilm.

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



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APPENDIX G

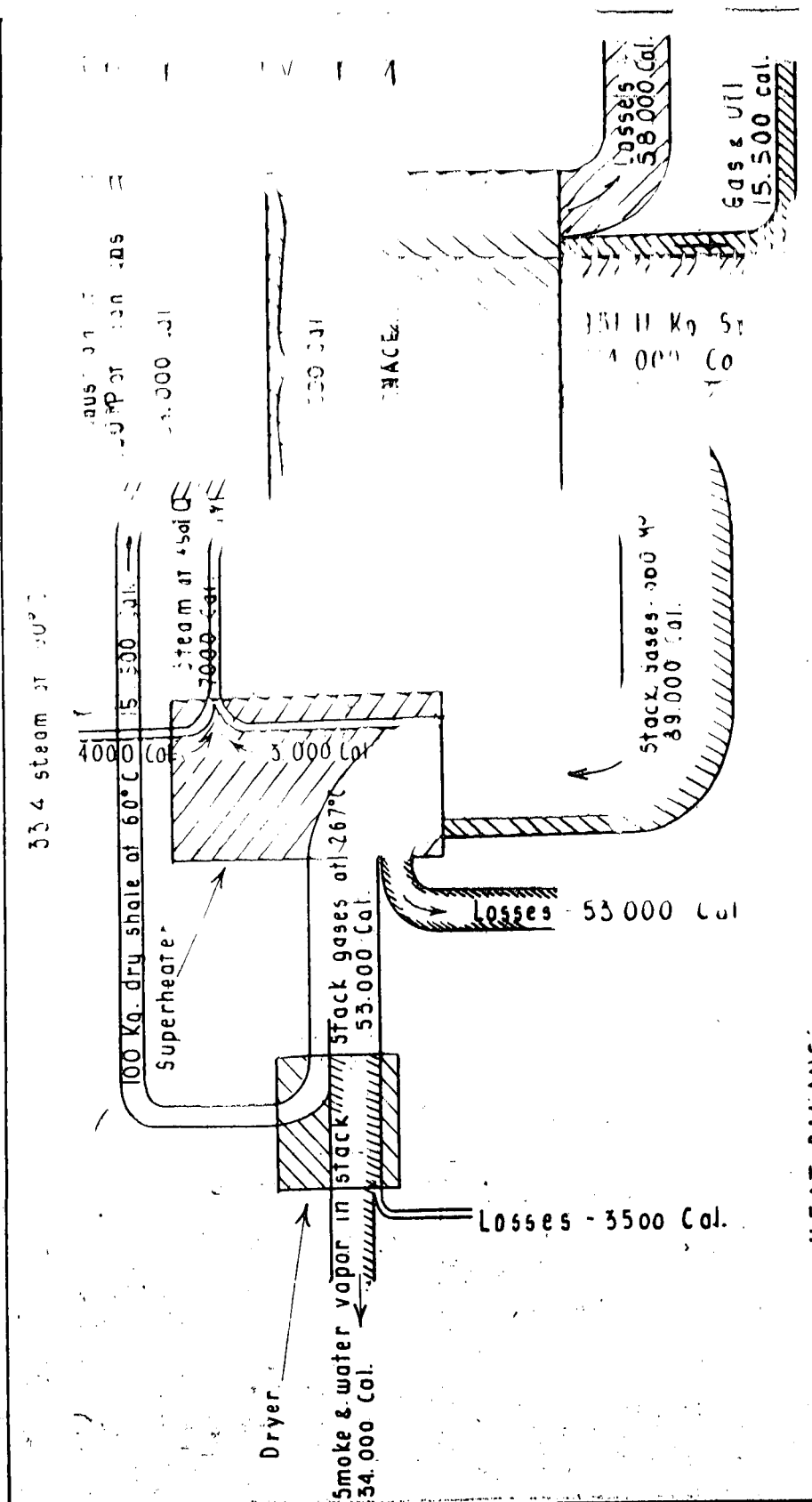


MAP SHOWING THE BITUMINOUS
SHALES OF FRANCE
LEGENDE

Ech. 1/1000000

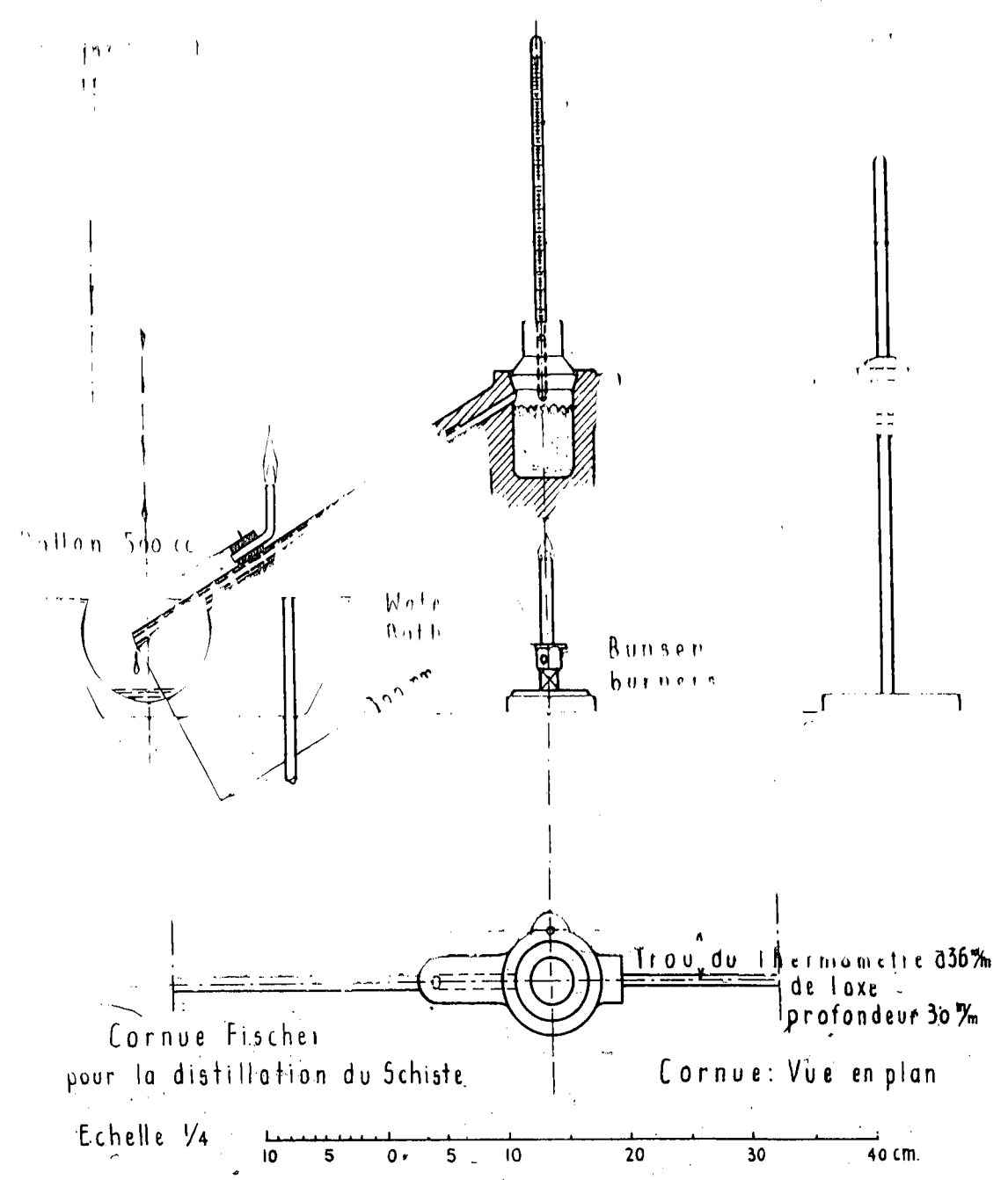
-  Schistes Bitumineux Exploitable
-  Schistes Bitumineux à prospecter
-  Schistes divers (peu ou pas imprégnés)
-  Asphalte du Rhône

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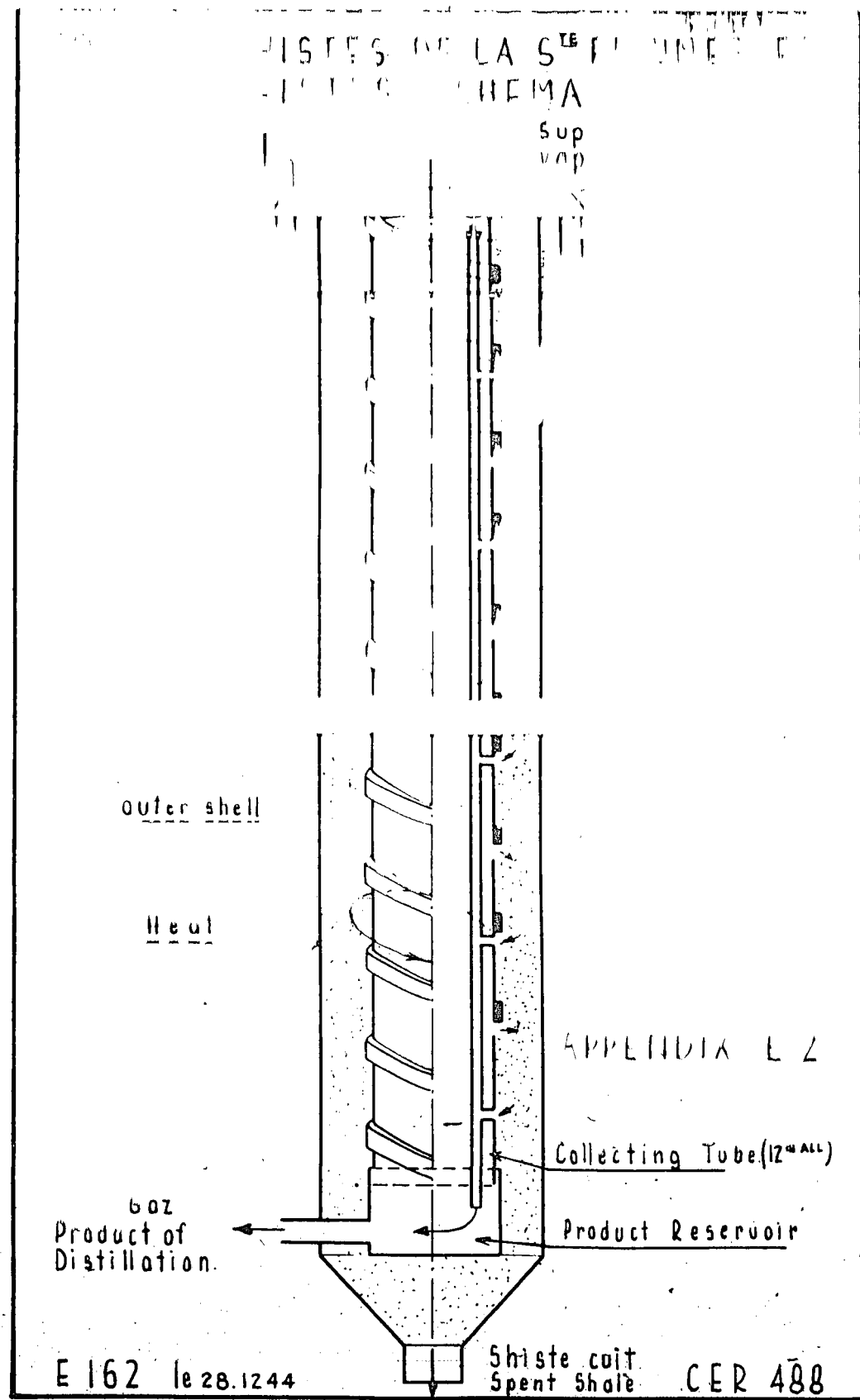


HEAT BALANCE
 Test run 22d May, 1944 on Shale Distillation Unit
 Societe Bitumes et Schistes, Lavernhe, France.

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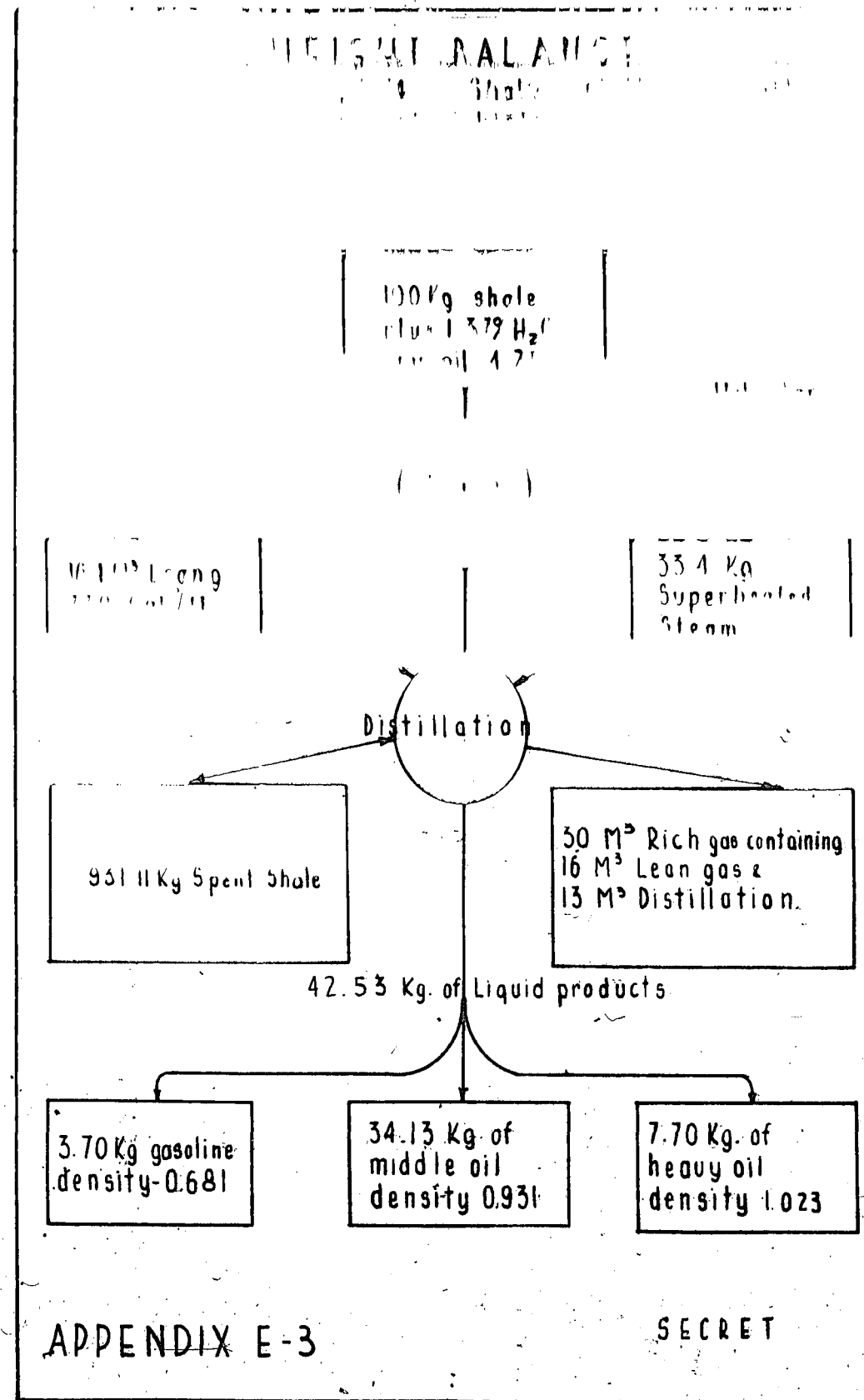


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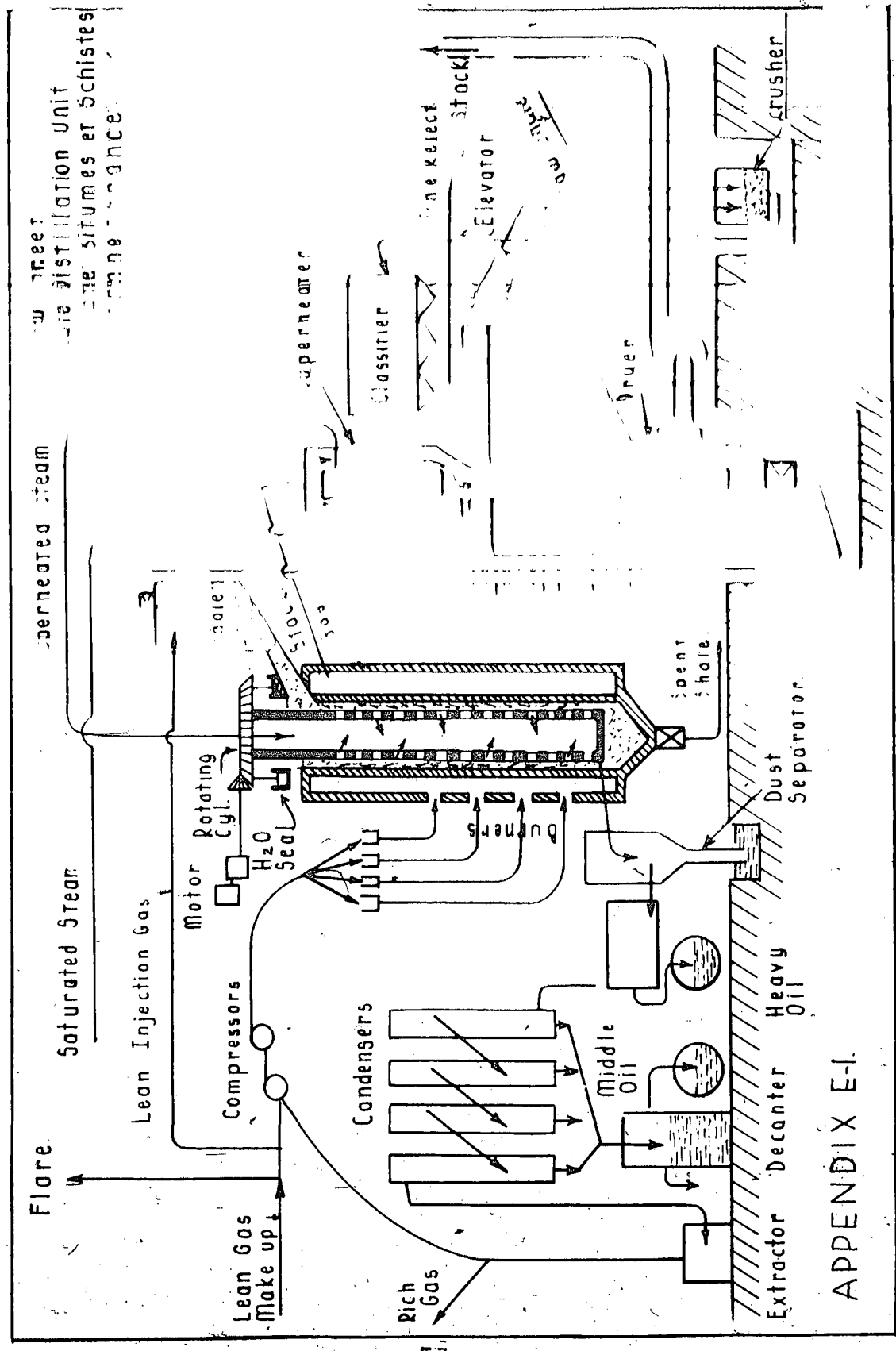
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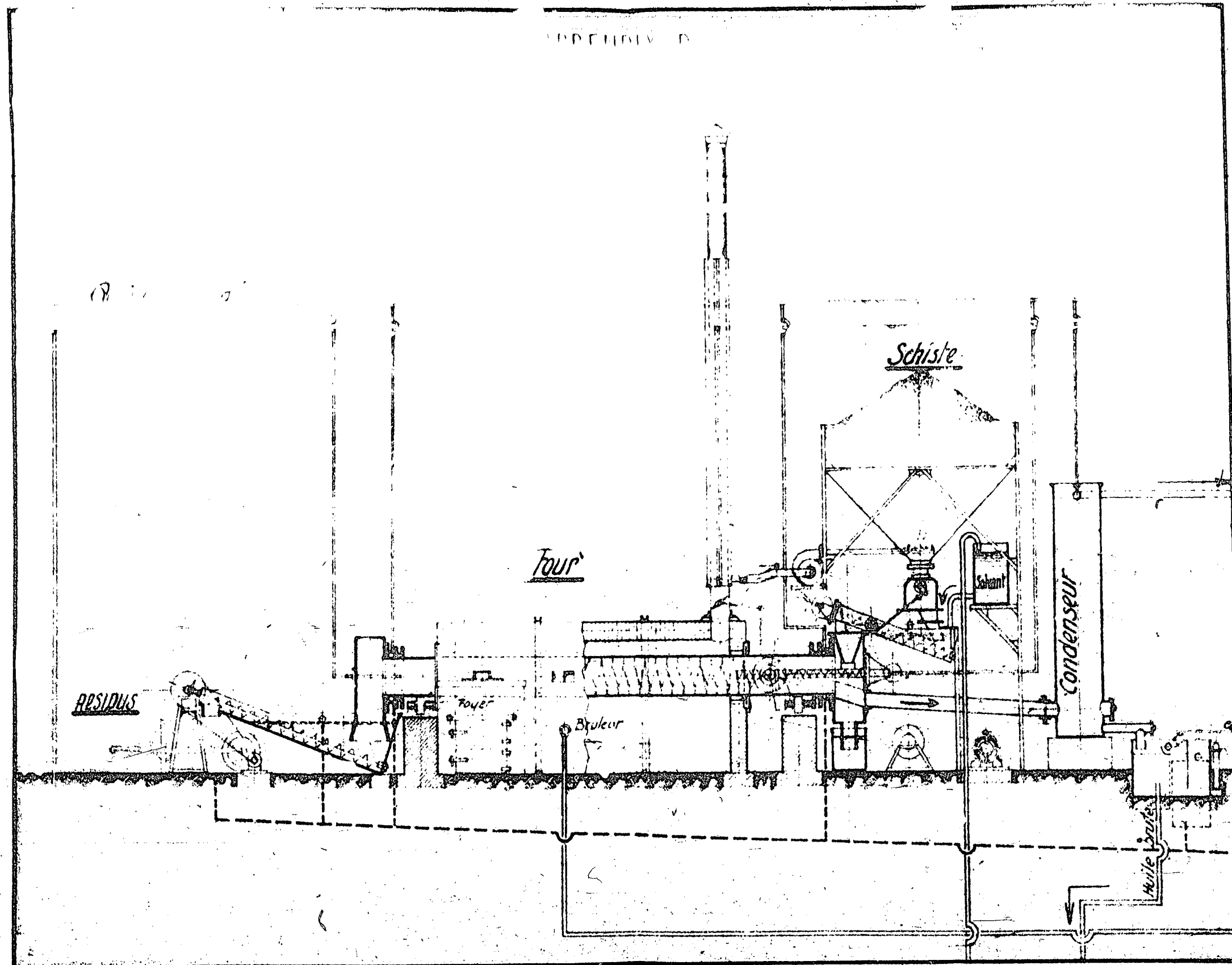
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Shale Distillation Unit
 Les Unités de Schistes
 de la Compagnie

APPENDIX E-I

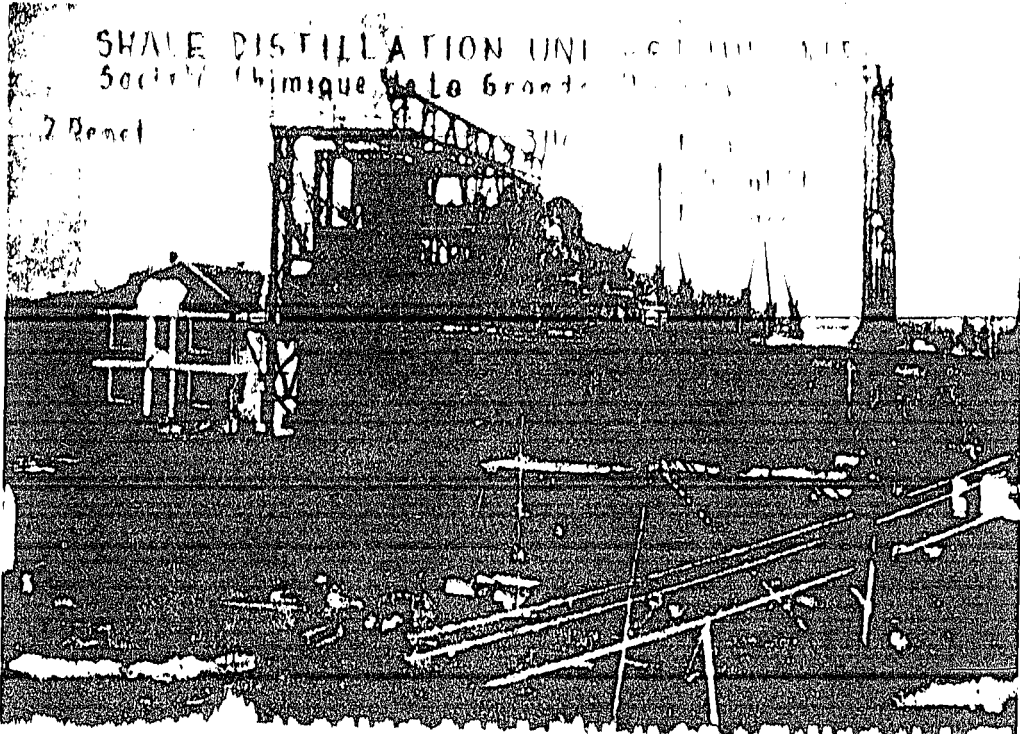


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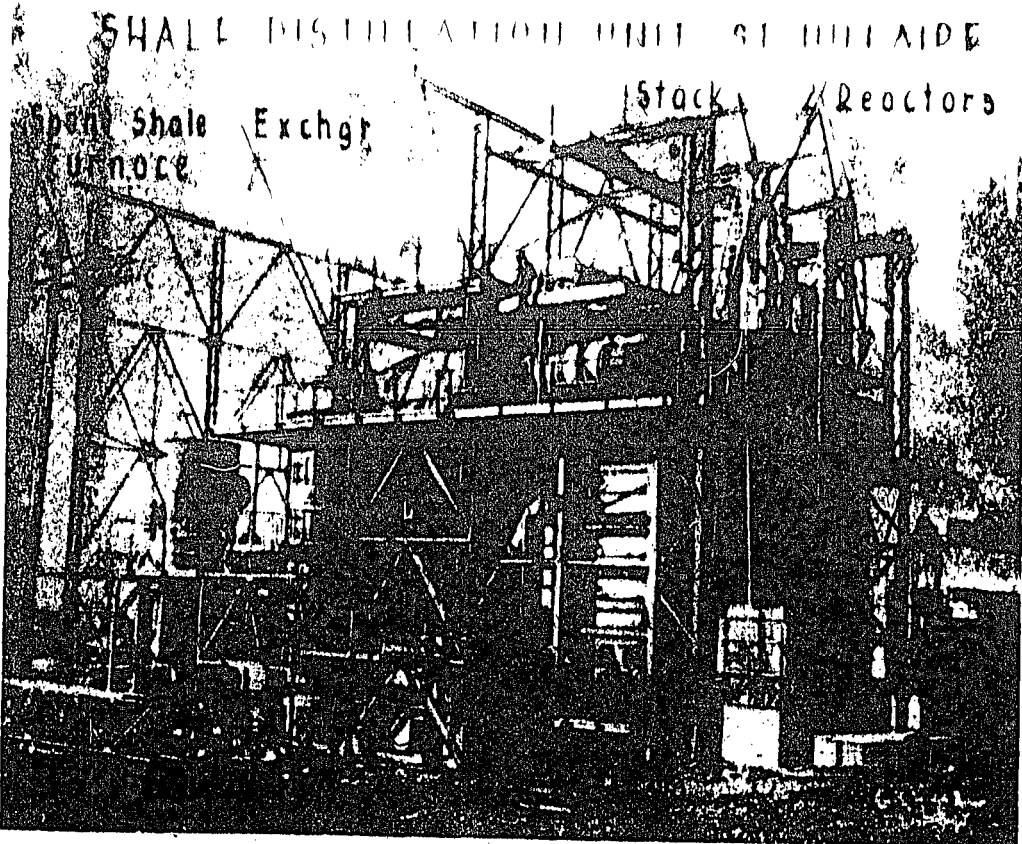
APPENDIX

SHALE DISTILLATION UNIT - ST. JULIA AIDE
Société Chimique de La Grande Pile



SHALE DISTILLATION UNIT - ST. JULIA AIDE

Spent Shale Exchgr Stack Reactors
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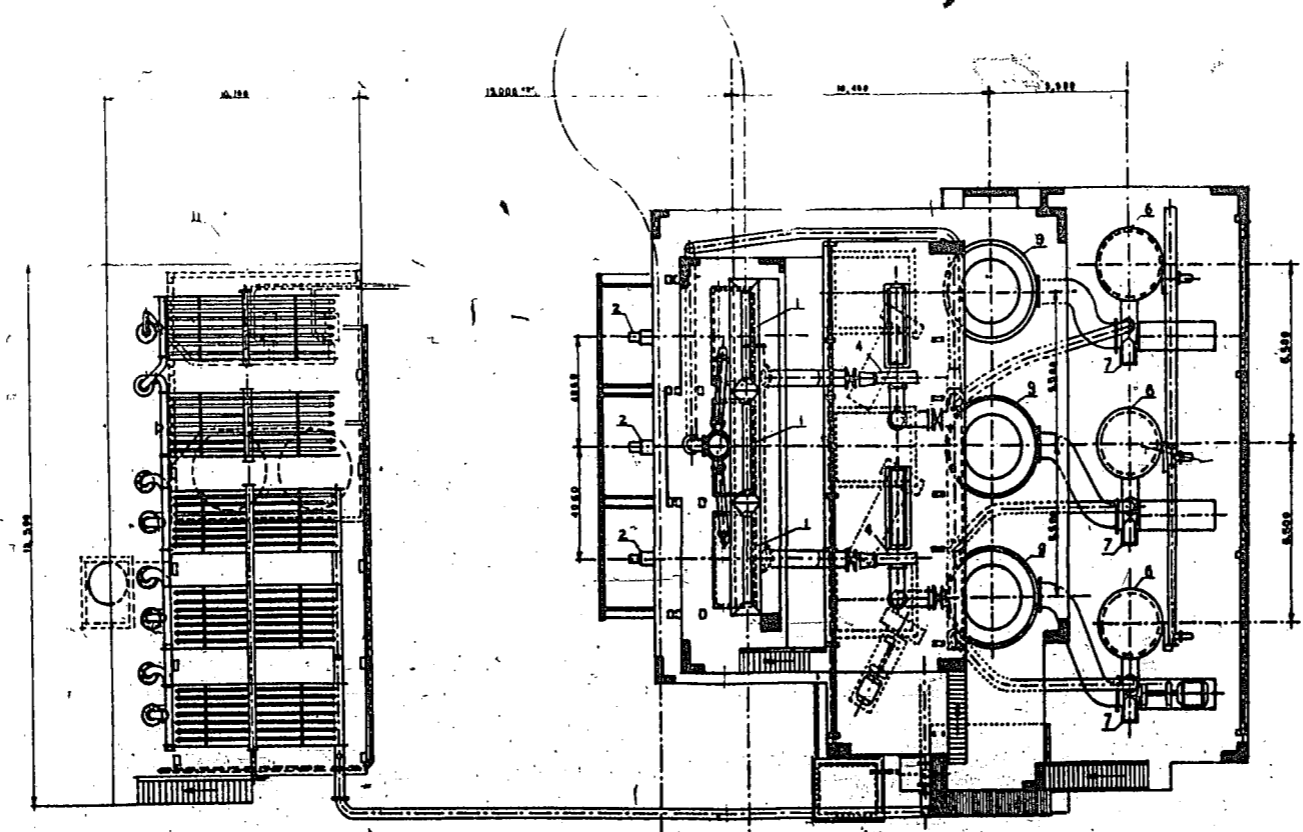
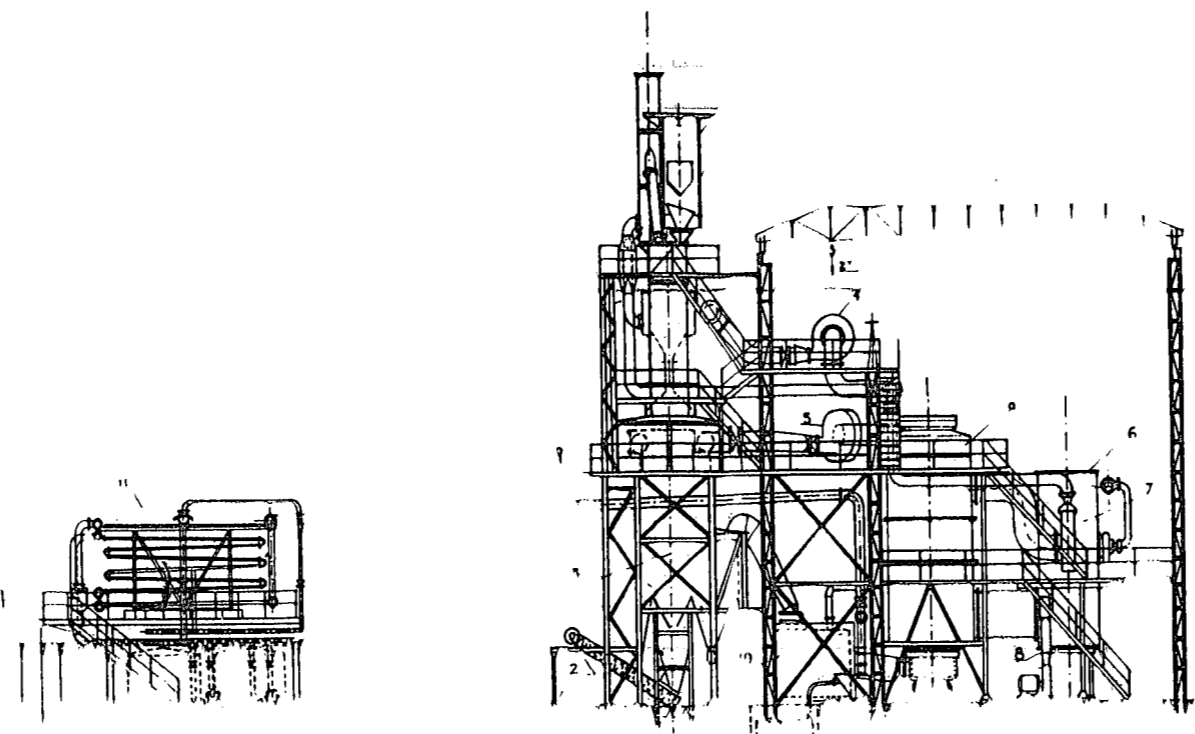


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TOUR DE DISTILLATION DE
SCHISTES BITUMINEUX
600 T/J

ENSEMBLE
DOCUMENT "C"
ALSOB MISSION

STATION CHIMIQUE DE LA GRANDE
PARISSE

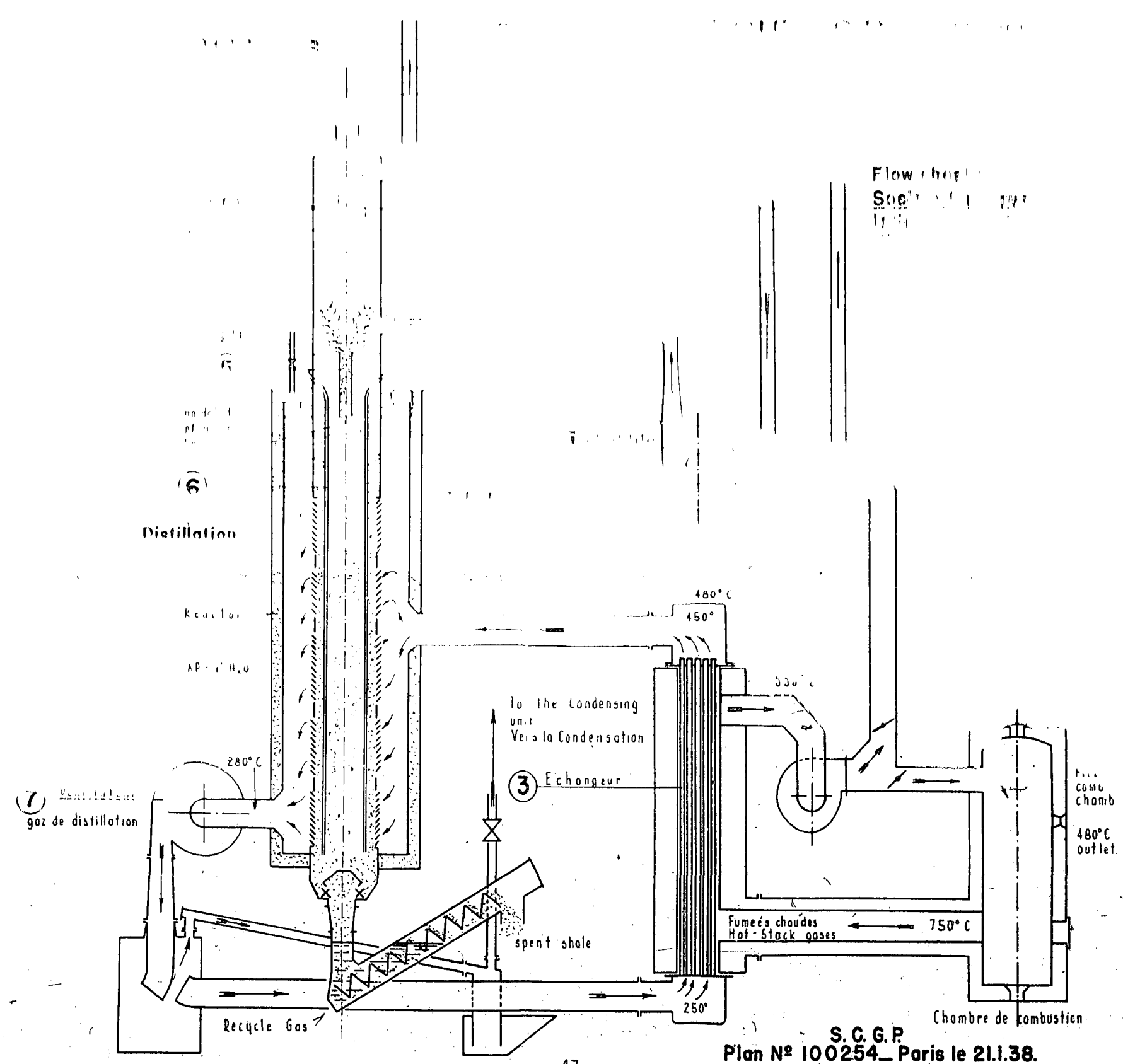


LEGENDE

- | | | | |
|----|-----------------------------------|----|--------------------|
| 1 | Arrivée du schiste | 1 | Shale inlet |
| 2 | Extraction de schiste sort | 2 | Spent shale outlet |
| 3 | Cornue | 3 | Reactor |
| 4 | Ventilateur de préchauffage | 4 | Preheat blower |
| 5 | ventilateur de circulation de gaz | 5 | Recycle gas blower |
| 6 | Chambre de combustion | 6 | Combustion chamber |
| 7 | Ventilateur de fumées | 7 | Exhaust gas blower |
| 8 | Ventilateur d'air | 8 | Air blower |
| 9 | Echangeur | 9 | Heat exchanger |
| 10 | Séparateur | 10 | Separator |
| 11 | Condensation | 11 | Condensing unit |

Collecteur gaz de chauffage

l'ingé. de base



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S. C. G. P.
Plan N° 100254 - Paris le 21.1.38.

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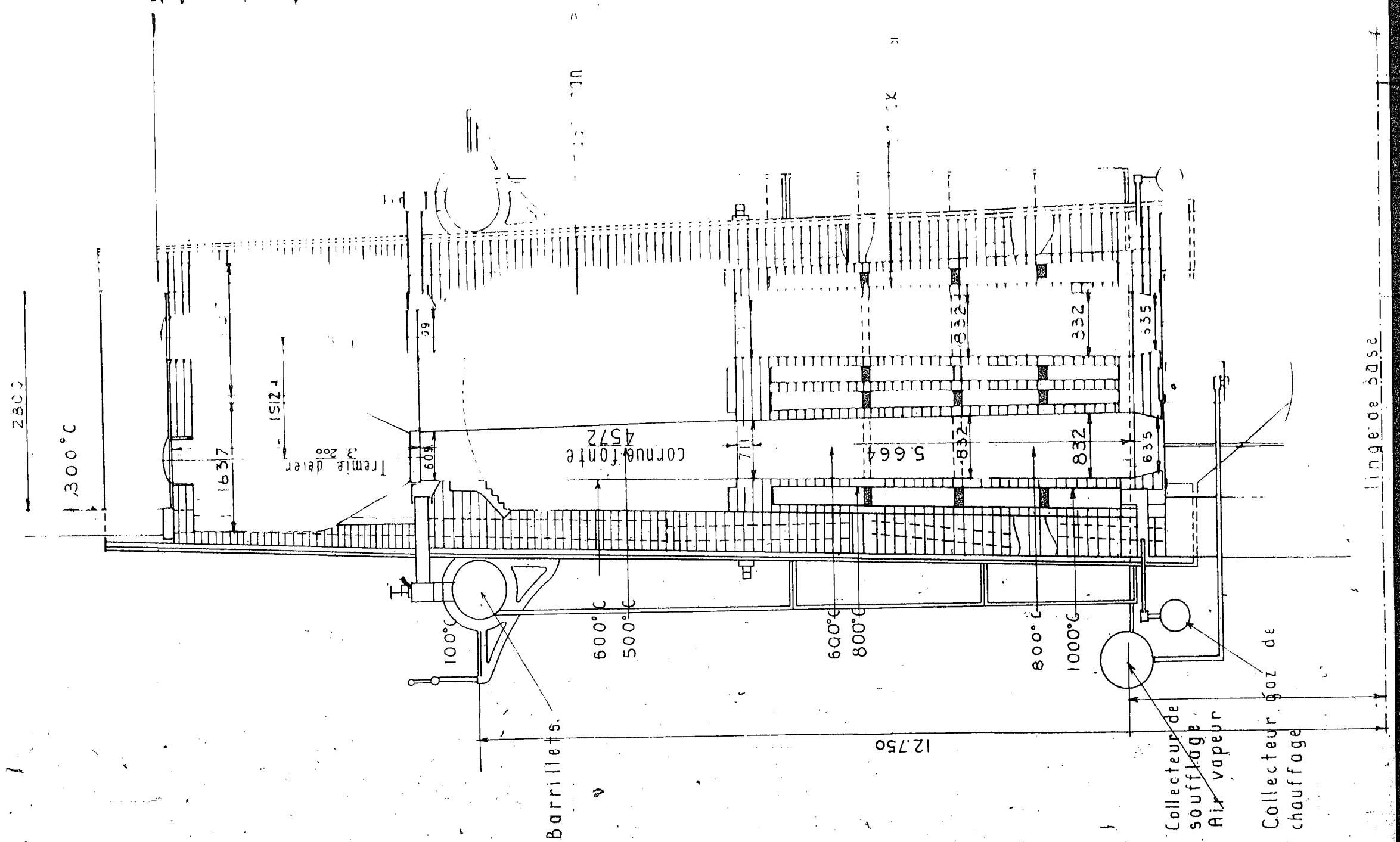


Chart No.1

Ore extraction and oil production at Autumn

Ore		Crude Oil		Gas		Total efficiency - crude oil and essence gas	
Year	Value	Year	Value	Year	Value	Year	Value
1931	74.541	1	4.831 6.48	452	0.61 9.3	5.283	7.09
1932	87.941	1	6.205 7.05	577	0.65 9.3	6.782	7.70
1933	84.576	1	5.992 7.08	597	0.71 10.0	6.589	7.79
1934	73.606	1	5.535 7.51	652	0.90 11.8	6.187	8.41
1935	88.473	1	6.956 7.85	694	0.80 10.0	7.650	8.65
1931 to 1935	409.137	1	29.519 7.21	2,972	0.73 10.1	32.191	7.91
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1936	108.200	111.638	8.034 7.19	858	0.78 10.7	8.892	7.97
1937	110.833	110.513	7.600 6.88	749	0.68 9.8	8.349	7.56
1938	128.519	122.988	8.338 6.79	813	0.66 9.8	9.131	7.45
1939	139.259	140.162	9.248 5.60	918	0.65 9.9	10.166	7.25
1940	133.828	132.935	7.877 5.93	653	0.49 8.3	8.530	6.42
1936 to 1940	617.639	618.236	41.097 6.65	3,991	0.64 9.7	45.088	7.29
-o-o-o-o-o-							
1931 to 1940	1,026.776	1,027.373	70.616 6.87	6,963	0.67 9.8	77.579	7.55

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V - POSSIBILITY OF PRODUCTION INCREASE.

These possibilities have been considered here not from the standpoint of a long range expansion of the plant, but from the standpoint of the best possible use made of the existing facilities, in their main parts and the strengthening of the lateral or auxiliary units, if such strengthening is necessary.

The main part of the plant is the distillery, the production of which governs the volume and the efficiency of all the other components.

As mentioned above, for the production of oil the quantity of ore can be raised to 1,550 tons of ore per day.

- a) the fine ore is screened
- b) all retorts are equipped with blowers.
- c) the new 25 t/day retorts are operating.

This requires a daily consumption of 1,760 t. of ore. The mine should therefore furnish yearly 640,000 t. or 2,100 t. per day, since the mine operates only 6 days a week. A suitably equipped mine should produce 200,000 tons yearly. It will be necessary therefore to acquire besides the mining equipment, all that is needed to increase the production, for instance tracks, w-gonnettes, diesel locomotives, compressors, hammer-piqueurs, sliding vibrators, etc., all of which could be found on the market.

Since it must be admitted that with the rationalization measures the efficiency of the 'piqueurs' will be improved, it will be indispensable to use for the production 350 'piqueurs' and 'foreurs' and about 210 auxiliaries, or a total of 560 men against 245 used at present.

Besides the improvements and expansion already planned by the managements for the distillery, there is nothing to be changed. The only addition should be the purchase of a blower installation for the 3rd Battery.

As to skips, which must have an efficiency of 93 t. per hour or 2,232 t/day, they should be operated loaded to 100 %, which will be barely satisfactory. A reserve should be provided, which should exist already now.

For the crushing, which should have a capacity equal to the skips, the same observation applies.

The screens will probably have to be reinforced.

It is not known whether the increase in efficiency of all the component units has already been planned by the management in connection with the installation of the new batteries, or whether eventually the needed machines have already been ordered, but this is possible.

The personnel will have to be increased. Probably from 450 to 500 men will be sufficient for the increased production. This will mean that 300 new men will have to be put to work, from which one must deduct the manpower released from cracking.

The refinery should suffice for the increased production because the annual production of crude essence will reach 4,000 t. and the present plant has already handled 4,000 t. of crude essence in 1939.

It appears therefore that there are no insurmountable difficulties in the immediate program up to 41,000 tons of crude oil in 1940. The basic problem to be solved is the increase of 600,000 tons of oil.

- a) crude essence, density 0.785 56.6%
- b) residue oil, density 1.000 7%
- c) gas 16.25%
- d) coke 20.25%

After usual refining with sulphuric acid and rinsing with soda

	by volu	by weight
Pure essence	5	49.2
Liquid	5	5.2
Losses		5.6

	by weight
Pure essence	50.40
Residue oil	7
Liquid	2.95
Gas	16.25
Coke	20.25
	103.15

The efficiency in hydro carbons is therefore of 60% at the best, while the rest is almost entirely lost for the petroleum economy.

A portion of this rest can be reclaimed. Cracking gases, which are being burnt in the plant, produce a very rich gas, the disposal of which in bottled form, if one can avoid polymerization, can be profitable. It is all the more possible, because the distilling plant furnishes a surplus of a gas, 2000 to 2500 poorer in calories, which could be used for heating during the cracking operation.

As a whole the cracking unit makes a good impression. Its personnel together with the personnel of the refinery consists of 500 persons, which represents 50 people per 1000 tons of oil per year, a figure which can be considerably reduced by increased production and decrease in personnel.

Faced by dearth of oil and the necessity of utilizing as liquid fuel every drop of oil, the problem is whether it is advisable to lose at present 40% of oil for the production of refined fuels. A negative answer needs elaboration. Thus it is necessary to interrupt the oil cracking at Autun and add the crude oil as such in a certain percentage to the oil reserves of the country. This would considerably strengthen the oil reserves of the country and would be perfectly justified, since this crude oil can be put to good use, as Diesel oil, as Referat Mineralol has shown during tests. Even with an addition of 25% it does not lower the quality of the oil from the economic standpoint. Here again the research of Referat Mineralol has shown that the enterprise would be more advantageous than with cracking of crude oil. One must consider therefore that neither from the technical, nor economical standpoints are there any objections to the direct use of crude oil with the exclusion of the cracking.

The personnel released from the cracking could certainly be assigned to other duties in the same plant.

Chart No. 2

Processing of the crude oil at Autun.

YEAR	Production of crude oil in tons, with essence of gas		Products obtained by processing		Other Products	
	Essence	Gas	Liquid Products	Semi-solid and solid products	Ammonia sulphate	Various sulphate Products
1931	5.283	300	1.638	2.777	4.0	856
1932	6.782	489	910	1.876	32	1.078
1933	6.589	460	1.399	2.449	12	1.074
1934	6.187	603	1.133	2.304	226	1.106
1935	7.650	534	1.301	2.802	276	1.224
1931 & 1935	32.491	2,366	6,381	12,208	335	5,338
1936	8.892	1,290	687	1,629	40	1,398
1937	8.349	4,215	106	579	19	1,320
1938	9.151	3,741	970	1,038	20	1,478
1939	10.166	4,175	253	1,233	496	1,335
1940	8.530	3,824	168	383	332	1,640
1936 & 1940	45.088	17,255	2,184	4,862	1,289	7,071

Anxious to use as a reinforcement for the oil reserves of the country, the 'posidonian' ores of Wurtemberg, which are poor in oil (4 to 5% of oil) the Lurgi of Frankfurt on Main, has developed a new method and has built a furnace, the Lurgi-Schweitzer, which is being experimented with. The first small furnace has given good results, and a larger test furnace has been built, of 3m diameter and 70 t. handling capacity.

This furnace should be operating soon. Operating results are expected. Faced with the possibility, or even necessity of developing the ore distillation in France, it may be useful to establish a contact with the Lurgi firm, for the beginning of tests of distillation of French ore poor in oil. In this case not only Autun ores should be considered but also 'basic' ores from Gievey (Franche Comte) the exploitation of which from 1933 to 1935 was hampered by a to inadequate handling of the furnace.

CONDENSATION

The oil vapors and the gases extracted from the furnace are channelled to the condenser. There they are cooled and condensed in air and water coolers. From the obtained mixture, oil and ammonia water, the latter is isolated and taken for ulterior handling as a unit of ammonia sulphate. Non-condensed gases are directed through water washers, in which the ammonia gas is retained. The essence of gas is reclaimed starting from the gas liberated from ammonia through oil washing in oil washers. For the oil washing one uses the light oil yielded by cracking.

Permanent gas leaving the condenser still contains 15 to 20 gr of essence per m³. For a total quantity of 300 m³ of permanent gas per ton of ore, it means that about 6 kg of essence per ton of ore are consumed during the heating of the retorts, which is not permissible, due to the deficiency of oil in the ore.

The production of the gas essence should be any way improved through careful washing in special apparatus. It appears entirely feasible to reclaim at least 50% of the essence which is at present wasted. With the present production it would give a surplus of about 400 t. of essence per year, or 45% of the actual produced essence, and the total efficiency in oil per ton of ore would improve to 0,3%. For a production increased to 41,000 tons per year, it would make 1,700 t. of essence per year, saved for the economy.

The total number of personnel of the distillery and the condensing plant is 180 people, of the 203 registered. Therefore there is a need of 18 men per 1,000 tons per year, which is very high, but which could be explained and partially excused by the small production. It is admissible that with a higher production this figure can be considerably reduced.

IV - PROCESSING OF THE OILS

Oil resulting from distillation has a specific weight of 0,895 to 0,900, a definitely saturated character, a sulphur strength of 0,5% asphalt strength of 0,3% paraffin strength of 3 to 6%, phenol strength of 5 to 6%, azote base strength, of which pyridine is of 2 to 3%.

Two periods are distinguishable in the handling of the crude oil; one which extends up to the establishment of the cracking unit in 1935-38 and the following one. Until 1935 the crude oil was processed only through distillation. From 1936 on, one started to crack the whole oil.

1. Distillation

Products resulting from distillation and the consecutive refining varied greatly. One obtained from the essence, besides the primary essences and distillants, 'lampant', engine fuel, diesel oil for diesels, paraffin, lubricating oil, lubricants, 'brai', and coke. On Chart No. 2 are shown the obtained quantities. Using these figures, which are of official sources, one can determine exactly all the losses relative to this process of handling crude oil; since the efficiency in liquid hydro-carbons was only of 64%, the losses must have been considerable, and the lubricants mentioned in the chart can hardly be considered. As a matter of fact it is probable that a portion of the heavy oils have been set aside for their production.

2. Cracking

The unit established in 1936 totally changed the processing of the wholly cracked crude oil as well as the one converted into essence and coke. Cracking can also be applied to 'brai' and to residue oils, but though 'brai' is sometimes cracked (fusion point 90 degrees) one very rarely cracks residue oils because their efficiency is not advantageous.

See Chart 2.

The plant was designed by the U.O.P. for the use of the DUBBS method, built in France out of French materials, under the direction of the licensing Society, and put into operation. Its capacity is 63m³ per day. Initial pressure is 40 atmospheres, the pressure in the reaction chamber is 20 atm. The working temperature goes from 480 to 519 degrees, depending on whether coke or residues are handled. There are two coke chambers, one being cleaned, the other in operation. The capacity of each chamber is sufficient for a 24 hours duration of work.

Here are the results of cracking coke:

The proportion of essence of gas to the crude oil does show any inexactitude.

Aside from the regression of the quantity of essence of gaz relative to the crude oil, the chart shows a continuous decrease in efficiency from year to year without any apparent reason. It is possible that over a period of years the quality of ore has deteriorated. It is also possible that the distilling in the retorts was not as well carried out.

The factory management considers the expansion of the distillery. It has already placed an order for 10 Pumpherton retorts with the Cie de Constructions de Four Paris-Montrouge, but these are of the type handling 3 tons daily. The new battery, formed by groups of two retorts is expected to be delivered ready for operation in November 1941. The management doubts that this date will be respected since according to its opinion the retorts are not needed before February-March 1942. When they will be in operation the quantities handled will be increased to 250 t. daily for the non-screened ore and 350 t. daily for the screened ore. The quantity handled by all the facilities of the plant will be $1200 + 350 = 1550$ t. of screened ore which, with the average % of oil of 7.29 of the last few years would furnish 113 tons of oil per day or for 365 days 41,000 t. of oil, a considerable figure for the oil economy of the States, which would justify certain sacrifices. The sacrifice consisting of the elimination of the fine ore, which should not be distilled. In the daily 1,550 tons of ore the eliminated quantity represents 210 t. or 13%.

How can this be utilised, so as not to increase the cost of crude oil? With a 7.29% for every ton of oil, 1,86 t. of fine ore would be thrown away, and the cost price of the oil will be increased 55 F. per ton, considering that the ore price is 30 Frs a ton. With the present prices of crude oil this added expense is possible.

But there are ways of using the fine ore.

In principle, three means are available:

- a) Distill the fine ore in special furnaces.
- b) Make briquettes out of the fine ore, which could be then distilled in the Pumpherton retorts.
- c) Use the fine ore as fuel.

In France, Mr Ab Der Helder has built a furnace for the distilling of the fine ores. According to the literature, the Societe Chimique de la Grande Paroisse, at St-Bilaire has been experimenting with two types of 25t and 50 t per day capacities. There has been no reports on the results. There is no way of estimating the functioning and the economic qualities of these furnaces. There have been negotiations in 1938/1939 between the 'Lyonnaise des Schistes' and 'La Grande Paroisse', which would make the latter firm equip and test

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at Autun on an industrial scale, two Ab Der Helder, and one Lantz furnaces. In case of favorable results La Lyonnaise reserved the right of exercising the purchase option. It is thought that the Ab Der Helder furnaces are liable to be operating.

The making of briquettes from the fine ores would not be a practical solution because the production costs would make the ore too expensive. The test has provided no concrete results on this subject.

The use of the fine ores as fuel in the plant has certain advantages. According to plant statistics the coal consumption reaches 0.5 tons per ton of produced gasoline and may even be higher. In relation to 1 ton of crude oil, this represents 0.3 t. of coal. Taking into consideration that the ore has three times less calorific value than coal, it would be necessary to provide 1.5 t. of ore as fuel per 1 ton of crude oil. The fine ore used would constitute in addition 1,86-1.50-0.36 t. per ton of crude oil. For a production of 1,000 t. of crude oil 18,600 t. could be distilled by a 1000 t. Ab Der Helder furnace.

The use of fine ore as fuel in the plant has the obvious advantages of completely eliminating the necessity for the plant to use an imported costly fuel, the purchase of which presently is associated with serious difficulties and causes operational losses. Economically the heating with one's own fuel, when coal costs 500 francs per delivered ton, has serious advantages.

One must not lose sight of the fact that the combustion of the fine ores would necessitate a special heating and possibly special grates but this could be worked out.

As to the distillation system to be used, the Pumpherton furnace in this case appears as the suitable solution, particularly when the high operating costs are greatly amortised over long years of exploitation.

Obviously a distillery used for the handling of ores which are deficient in oil, should not be of high initial cost nor high exploitation cost. Among the known large distillation furnaces (Lurgi with circulating gaz, tunnelled furnaces of Estonia etc..) none could be suggested, because in this case they do not suit the conditions. A new type of furnace would be necessary, the construction and processing of which should be adapted to ores which are poor in oil.

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Little fore-thought is given to accidents. A number of unprotected gears is visible, transportation cables are moving freely in the galleries etc.. The numbers of accidents is bound to be high. It is obvious that remedies should be found for these cases, because it would be very difficult if not impossible.

OIL PRODUCTION

THE DISTILLERY

It is composed of 120 retorts, Pumpherston, scotch type, divided into 3 batteries of 40 retorts, externally heated. Each group of 4 retorts has common heating. The fuel is the permanent gas, extracted from the condenser after condensation of the oil and essence vapors, and the separation of ammonia. The quantity of the gas delivered by the retorts is sufficient, with the rich layers, like the Grande Coube which offers distillation residue with sufficient strength to heat the retorts.

The gases resulting from the heating of the two batteries are utilized for the production of steam in a Roubert 'ugue boiler'. The boiler produces 1000 kg of steam per hour.

The ore is crushed in roller machines to the size of 80 mm and then distilled, either with fine ore contained or after screening, eliminating pieces smaller than 10mm, 12% of the ore is thus eliminated.

The retort process is influenced by the presence of fine ore. The capacity is 2,5 to 2,7 tons per day without screening, or 4 tons per day with screening, or a 50% increase, in this case an important circumstance, if it is wished to increase production, since it makes possible to increase the efficiency of the distillery by 50% without any new investment.

Steam is added while distilling. 300 kg of steam are blown in per ton of ore through the lower portion of the retort, for to utilize on the one side the fixed carbon of the distilling residues, in the form of water gas, for the interior heating and addition of air in the retort and on the other side for to reclaim the ammonia from the ore.

Considering the size of the retorts and their high production costs they handle a very small quantity of material. The scotch experts have attempted to increase the efficiency by raising the heating temperature which is ineffective since the distillation residues melt when the temperature at the bottom of the retort changes from the normal heating of 1.100 degrees, tends to form joints and ceases to be removable. It then becomes necessary to stop the operation for cleaning. The study of these phenomena in the retort

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bottom has proved that it was necessary to blow air which gave most favorable results. Efficiency is considerably improved. Following the scotch experiments air was blown in at Autumn, with such good results, that already 32 retorts are working with blowers. The same equipment has been ordered for the remaining 40. By Autumn it will be in operation. Eight other retorts of the second battery will also soon be blown.

For every ton of ore 70m³ of air are added and 300 kg of steam. The increase of treated quantities is considerable; one treats:

- 7 to 8 tons per day, when the ore is not screened
- 10 to 11 tons per day when the ore is screened.

The large scale test made with several retorts, showed that with a strength of 13% of the fine one could produce 9,13 tons per day, and 8,10 tons per day with a strength of 22.5% of the fine. Thus the above figures can be considered as cautious, notably the ones referring to the screened ore, because they are the result of actual exploitation and not test.

One can thus definitely admit that blown air can triple the efficiency of the retorts. One must also consider that with the use of screened ore a surplus of 40% passing through the retorts.

Here is what the blown process would give in 120 retorts: Treatment with the fine without blown air: $120 \times 2.7 = 324$
Treatment with the fine with blown air: $120 \times 7.5 = 900$
Treatment without the fine with blown air: $120 \times 16 = 1920$

The introduction of the blown air, and the segregation of the fine would almost quadruple the treated quantities by the present plant. This is the cheapest and fastest way of increasing production.

As far as energy consumption and maintenance of the Pumpherston are concerned, they are very favorable, since the first is infinitesimal. There are 2 electric motors per battery, one of 6 HP for the smoke gases, the other one of 10 HP for the injection of steam and air. The repairs are negligible. The retorts function for years without repairs; some are still operating after 10 years of uninterrupted functioning. It therefore appears safe to assume a functioning during 265 days of the year.

As a contrast the efficiency in oil is not good. It reaches 85% of the efficiency of the Fischer retorts. It is supposed that it could be increased due to an amelioration of Condensation. It is confirmed by the efficiency of the gas essence, which was between 1931 and 1935 at 10.1% of the crude oil and fell to 9.7% from 1935 to 1940.

On the following chart No 1 is given the oil production of the last 10 years. Note that from 1931 to 1935 no exact figures of distilled ore were available. For to figure out the efficiency in oil, the extracted ores must be considered as distilled. As shown in the period from 1935 to 1940 this has not resulted in serious inexactitudes.

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It would be suitable to use electric drilling with Wydia-steel bits.

At Grande Couche, owing to the danger of mine-gas explosions one uses the electric spark and dynamite; at Thelot one prefers the blasting string and explosives as compared to 'chicrete'. Grande Couche requires 175 gr of explosives per ton, and elsewhere due to the rill-rock formations located between the layers of ore - 247-474 per ton, a really considerable figure.

Extraction costs are infinitesimal. On the spot one uses little of the 'stancons' and only the durable galleries, the slopes and the transversal benches have been bored-in. In spots, iron crossbeams. The lumber used is chestnut and oak. This material is not processed or treated against rotting and therefore does not last over four years.

Water service is more or less in order. A better drainage of the extraction galleries could be obtained. The water secretion is of 120m³ per hour normally, or at high tide 180m³. The water is channelled to the distillery and the refinery. This quantity is sufficient for the treatment of 500 tons of ore per day during the filling and cracking operations.

Ventilation is good. The installation includes an electric fan with 10w³ per man's capacity. At Grande Couche one also uses for the ventilation of the shafts small devices powered by compressed air.

The underground exploitation is carried out in spots by means of 'sliding vibrators' and the use of mine wagonettes of 500 liters capacity. The loading spots are serviced by Diesel locomotives. From there the ore is carried directly to the distillery crushers by means of two skips.

3 - EFFICIENCY AND LABOR CONDITIONS

As of today the total production reaches 500 tons a day. Two or three crews work a shift of 8h.45m including the entry and exit time. Net working time is therefore 8 hours. The mine personnel consists of 95 'piqueurs' and 'foreurs' (drillers) and 150 auxiliaries. Of the latter 32 are used in blind shafts and crossways. Therefore, there remains for the current work 113 men.

The 'piqueurs' and 'foreurs' are divided into variously composed groups, depending on the veins. At Grande Couche and at Veins 6 and 8, 4 piqueurs and 2 foreurs form a group. At the vein No 10 there 4 piqueurs and 4 foreurs. More foreurs are needed here because the vein has an intermediate zone of 1m thickness, which must be moved by the foreur. Exceptionally in shafts equipped with the 'sliding vibrators' the groups are formed of 3 piqueurs and 2 foreurs.

The piqueurs (including foreurs) pull down an average of 550 tons per crew or 30% more than in 1935. Figuring in cubic meters of rocks if the ore has a specific weight of 2.05, it would make 2.70 m³ per day, or a fairly good figure.

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The number of the auxiliaries, 12% of the producing laborers is overwhelming and burdens the production costs by sharply increasing the non-productive expenses which is evident from the fact that the efficiency per head of the personnel total mining is only 2,34 tons per day or 1,3m³. It is evident that it is necessary to raise the efficiency to the 1935 level, at least through technical or social measures.

One would notably facilitate the plugging work by using heavy hammers to reduce the chunks detached by explosion. These chunks constitute up to 20-25% of the total mass. Thus one would eliminate the time lost while sectioning the blocks with other hammers.

It is inconceivable that forks are being used for sweeping the ore, because this leaves on the spot not only small pieces but also medium sized ones, which decrease the miners' efficiency and the exploitation of the mine. It is obvious that a coal-broom would bring better results, but it remains to be seen whether the management desires it, since these measures would increase the quantity of ore which interferes with the distillation.

The question of the complete mechanization of the mine will be treated up here having been already discussed elsewhere.

Among the social measures, one could first revise the salaries. The present earnings of the mine appear to be definitely below the normal. The average earning of the piqueur, 62 frs per day with a maximum of 100 frs for the best, is insufficient as an incitement for the full deployment of capacity for labor. It is possible that the lack of supervision and direction on the part of the subaltern employees of the mine, contribute to the decrease in efficiency. There is a large number of shafts and veins. Still only nine ladders are to be found. It is definitely insufficient. Unfortunately it is noticeable that the management of the mine has done little to ease the working conditions of the personnel, avoid unnecessary power losses and make the work more pleasant. Welfare organizations are totally absent. The accident prevention is considered defective. Here are some examples.

The entrance for the personnel to the mine is a sloping way, on which is placed the track, used to bring lumber to the mine. The laborers have to walk the 100 m, without finding steps, rails, landings or other facilities. They walk with difficulty on a slippery soil, which from the very entrance takes all the joy out of the work and only adds sadness to tiredness at the exit.

There are neither latrines or cloakrooms near the entrance to the mine. There is one washroom near the distillery; I suppose it is reserved for distillery workers, and is three km from the mine. For this reason the miners could not use it though it is available.

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during ... has been the following:

Years	Ore extraction
1931	74.541
1932	87.941
1933	84.575
1934	73.606
1935	88.473
1936	108.200
1937	110.833
1938	126.519
1939	138.259
1940	133.828

The ascending curve of the ore extraction since 1936 is due to the fact that the cracking installation at Autun has begun to function.

The ore is extracted from these mines from an average depth of 100 meters. The extracting procedure used in "scoutement amovible" which is used in the various layers and is adapted to exploitation conditions. In the Grande Couche exploitation the mobile installations have a width of 5 meters, the pillars are 6.25 meters wide. Between the pillars, every 1.4m, figuring from center to center, there should be a 4m wide vent. Thus the pillars have a base surface of 6.25x10m. In the veins No 6 and 8 the removable installations have a width of 10m, the long pillars are 5m wide. Lengthwise, between the pillars, every 25m, a suitable large vent allows sufficient ventilation. In the galleries safety pillars are installed. When necessary they are also equipped with vents. This exploitation procedure is due to the fact that there has been no preliminary work in the mine, nor preparatory tests of the deposits. The fairly uninterrupted deposit incited the management to proceed blindly. The result of this extraction procedure is an unbelievably inefficient mine exploitation.

In the Grande Couche alone the loss due to pillars reaches 40%, in the veins 8 and 10 30%.

These losses are still increased by the following losses:

1. due to safety pillars of the exploitation galleries, slopes, blind galleries, marecages" approximately 13% of the surface exploitable is usable for extraction.
2. Land slides who appear to be a rarity in the mine, cause considerable losses of exploitable surfaces, because following a land slide. A 100, width of ore has to be abandoned. This loss amounts to 10% of the present possible extraction of 40% and can therefore be estimated at 40% of the deposit.
3. Careless work and inadequate tools cause loss of extracted ore, which can be estimated based on statistical figures, to reach 4% of the deposit. After adding up all losses, one can establish the

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efficiency of the Grande Couche deposit as of only 40%; at the other layers it may reach a maximum of 50%. As an average the efficiency of the deposit can be estimated at 45%.

Extraction methods showing such results can be only branded as non rational and proper measures should be taken to replace them by more rational operation.

For this reason it is advisable to immediately and energetically initiate preparatory work - mainly the building of presently non-existing galleries. This work should not be an economic burden, since it will produce an amount of ore equivalent to the one presently extracted from existing spots, and the extraction proper would not add additional duties.

With a prepared mine it will be possible to completely change the extraction procedure and in any case it will be recommended to try to increase the exploitation of the Grande Couche, with its old-fashioned pillars, because the present losses could be reduced by half. In low powered exploitations up to about 2m. a largely exposed exploitation would have certain advantages, because besides the increase in the extraction there is a possibility of mechanising the exploitation by the utilising among other 'haveuags', 'des glissieres a vibration' (sliding vibrators), conveyers, and scrapers.

The introduction of the new rational method of extraction, permitting a sizeable reduction of operating losses, should be imposed as an immediate duty on the management of the company. The drilling of the shafts is done with pneumatic drills (about 15kg). The depth of the exploitation's drill hole is 2m. The drilling is an independent operation which the team executes through the whole deposit, which keeps 2-4 men on the spot, depending on the operating conditions. The efficiency of the drilling is astonishingly low. There are 20 drilling holes per layer made by 2 men, which partially explained by the lack of enthusiasm in the accomplishment. Without considering this, the efficiency could be increased as well as economy of air obtained, if the diameter of the drilling hole was better adapted to the diameter of the 'cartouche' (cartridge). For a 'cartridge' of 26 mm in diameter one drills a hole the final diameter of which is 20 mm instead of 30-32 mm, which would be sufficient. A diameter of really necessary dimensions would mean a time gain of 40%. The profile of the drill as well as the square hammer connection do not appear to suit the conditions of this work, and should be determined through systematic tests, which should not cause too much loss of time.

So as to facilitate the hard work of the driller and increase the efficiency it would be advisable to try the use of 'forage'

Since the various layers appear to be of uniform hardness, one is inclined to think that for greater efficiency of the drilling

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II - ORE EXTRACTION.

1 - Ore Deposits.

The site of the Autun deposit is in the shape of an irregular triangle, with a base of 32 km and a surface of about 240 km². The deposits, which geologically belong to Permian, have a very large number of layers which reach a total power of 1000m. All the layers contain some BITUMEN in very varying degrees, though the strength of contents of Bitumen varies not only according to different layers between 4-12% of oil per ton of ore but diminishes from the center towards the edges of the territory. The position of the layers is not disturbed and in general appears unaffected. From the viewpoint of the oil contents, the only layers considered for extraction are the one apt to yield 60 liters of oil per ton, or containing 5% of oil. From this standpoint only the central part of the site of 3.4 km is considered as exploitable. Actually only the "Complex" are exploited:

- 1) The veins "Grande Couche" of the "floor" Comaille
 - 2) Veines 6, 8 and 10 of the "Floor" Thelot.
- On the "floor" Comaille the veins "Petite Couche" can be considered as exploitable on the "Floor" Thelot 7 out of ten layers are exploitable.

It is difficult to determine the extent of reserves in the deposit, because indications found in the literature on this subject vary from 21 to 200 millions of tons. There has been no extensive drilling to determine the location of veins and the possible value of their exploitation; same applies to the drillings which were to determine the condition in neighboring fields; the work has been started only a few months ago.

Confining the study to the central part of the site, and there only to the exploitation in existence, down the depth of 300 meters, according to Prof. J. C. Armanet, the ore reserve can be estimated at 54 million tons. In the light of Mr. Grangjean's, Director of the French Geological Map Service, estimate for this site of 60 million tons up to 400 meters depth, Prof. Armanet's figures appear dependable. This does not mean that the reserve deposits can be considered as 100% raw distillation material. The present exploitation methods at Autun allow under the most favorable conditions to obtain 50% of ore. Admitting that due to amelioration of operating methods 60% of ore could be directed to the distillery the reserve would amount to 0.6x54,000,000 = 32,400 tons. Thus as it will be shown below, the actual exploitation, provided the improvements and expansion plans for which have already been laid, are realized, could use 1,760 per day or 640,000 tons per year. With such a yearly extraction the calculated reserve could suffice for 50 years, which period would amply justify increase in production to the limits which can presently be reached without inordinate difficulties. It must be added that very probably the ore stock exceeds largely the adopted figure, and

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that it is in the interest of the State and the Company to examine the full extent of the Autun deposits by making a suitable number of tests, in view of determining even approximately the oil reserves of the site based on objective elements.

MINING EXPLOITATION.

The actual exploitation is located at Thelot, 3 km north of Autun. Two exploitations are being operated, separated by an unexploitable terrain of about 80 meters. One of the exploitations is located on the middle "floor" of the deposit La Comaille and consists of two exploitable layers: "Grande Couche" and "Petite Couche".

The second exploitation belongs to the top "floor" of the Trillery and includes the Thelot "Complexe" of 10 layers. These two exploitations are connected by a transversal layer of 800 meters length. Ore veins of the Grande Couche and veins No. 8 and 10 of the Thelot "Complexe" are exploited at present; veins No. 6 is being prepared; exploitation of the Veine No. 4 of the same ore. "Complexe" has been discontinued. The estimate of the deposit exploitation value has been made by the management of the company based on the strength of oil contents of the ore, which is determined in liters per ton of ore.

Here is the oil strength of different layers exploited in exploitation:

Name of the layer	Strength M.	Oil Strength Lit/Ton	Oil Strength % of ore	Remarks
Grande Couche	3.20	95	8.5	Pure veine
Petite Couche	1.00	70	6.3	" "
Veine 4 Thelot	3.00	60	5.4	" "
Veine 6 Thelot	0.60	110	9.9	Intermediate avge:
Veine 8 Thelot	0.80	85	7.7	" 1m/0.5
Veine 10 Thelot	2.00	70	6.3	" 1m

The exploitation of the Grande Couche yields 60% of the total extracted ore. Taking this into consideration and the fact that the veins 8 contributes 15% and the veins 10 - 25%, the average oil strength of the ore extracted could be actually estimated at 87 liters per ton or 7.84% of the weight of the ore. As shown on the given statistics this figure can be considered as an arbitrary one. Independently of the exploited layers the average oil strength of the ore varies between 84 litres (1940) and 113 litres (1935).

At present 500 tons of ore are extracted daily, which corresponds to a yearly production of 150,000 tons. The ore ex-

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(use of extraction hammers, coal-shovels instead of forks, mechanisation of the exploitation, "have uses" etc...) The management is not sufficiently concerned with the welfare and the comfort of the personnel. Safety precautions are defective: it is necessary to build an entrance corridor along the normal exploitation with, among others, an electric lighting system. It is desirable to examine this possibility seriously.

II OIL EXTRACTION

1. Distillation.

The distillation installation consists of 120 retorts of 25 t/day capacity, arranged in two batteries of three.

- a) screened ore 7/8 t/day.
- b) non-screened ore 10/11 t/day.

These favorable results have incited the management to equip 32 retorts with blowers and to order another set of this equipment for the second battery. The average normal daily amount of non-screened ore passing through 120 retorts without addition of air amounts to 3 t/day; 10-day ore uses about 400 t/day. When both screened ore and blowers are used, the daily average could be increased to 1,500 tons.

The management had ordered 10 large retorts with capacity of 25 t/day each, which are to be delivered in November of this year. For the screened ore the possible capacity of the retorts is 35 t/day, or a daily average of 350 tons. When the new retorts begin to function the distillation can thus, using the screened ore, be increased to a daily average of 1,500 tons, which corresponds to yearly crude oil output of 41,000 tons.

The fine ore coming from screening in a daily quantity of 210 tons for the large production can be:

- a) either distilled in special blast furnaces (Arder-Holder Furnaces or
- b) be used in the plant as fuel.

These two possibilities should be considered and decided upon through the preliminary work and tests.

The Fumpherston retort should be considered as completely suitable for the distillation of the Autun ore. Due to the possibility of the possible development of distillation of ores which are poor in French oil, it appears advisable to make tests in the furnaces Lurgi-Schweitzer recently installed by the firm Lurgi, using the Autun ore as well as the Greveney ore, considering that this furnace is economical both in regard to initial price and exploitation.

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C) Condensation-

The extraction of essence of gas from the gas resulting from condensation is below what it could be. The permanent gas issued by the condensation, still contains 15-20 gr. of essence per M³ of gas. From this essence one could obtain, through additional washing and special installation, with the present production - 400 tons of essence - the increased production 1,700 tons a year. It is desirable to examine this possibility seriously.

III - PROCESSING OF THE OIL.

Until 1935 the crude oil has been processed exclusively through distillation and refining of certain functions. A number of products were manufactured, starting with essence and down to dark grease. The extraction of liquid hydro-carbon amounted to 64%, the total loss being estimated at 16%.

From 1936 on all crude oil has been submitted to cracking in a Dubbe installation. Due to cracking and following refining one obtains from the cracked crude oil 50% of gasoline, 7% of residue, 30% of liquid tar - a total of 60% of liquid hydrocarbons. What remains is 16% gas, 20% coke and 3-4% of losses.

The cracking gases can in a certain measure be transformed into compressed gases. Due to the limited extraction of liquid products and the dearth of gasoline it would be appropriate to discontinue the cracking installations and to assign the non-refined oil to the country's reserves. In the light of the studies of the Mineral Oil Services such a utilization of the crude oil will not meet any opposition either from the technical or economical view points.

IV - POSSIBILITIES OF AN INCREASE IN PRODUCTION.

An expansion of the present installation is possible due to the fact that the management has done much of the preliminary work.

Provided:

- a) all the retorts are equipped with blower installations;
- b) that the 10 new ordered retorts are functioning;
- c) that only screened ores are distilled;

a daily average of 1,500 tons of ore could be used as a production figure. This corresponds to a daily production of 40/41,000 tons.

The costs of the investment, which already have exceeded the amount anticipated by the Company management, should not exceed reasonable limits; the purchase of the necessary material is within the present possibilities.

The personnel should be increased by 600 to 650 men.

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SCHIST OIL OUTPUT AT AUTUN

JUNE 1941

MINÉRIE SCHISTEUSE

by

INGENIEUR, Ingénieur

MINÉRIE SCHISTEUSE

A V P S K

I - SUMMARY

II - Extraction of schists

- 1- Deposits of schists
- 2- Mining of the deposits
- 3- Output and labor conditions

III - Extraction of Oil.

- 1- Distilleries.
- 2- Condensation.

IV - The processing of Oil.

- 1- Distillation
- 2- Cracking

V - Possibilities of Increase in Output.

Chart No. 1 - Schist extraction and oil production

Chart No. 2 - Processing of raw oil.

I - SUMMARY

I - ORE EXTRACTION

A) Ore deposits.

Ore reserves in the center of the Autun deposit and down to a depth of 300 meters can be safely estimated at 54 million tons. Considering the expansion of the enterprise within the limits of the existing facilities, to a yearly output of 41,000 tons of crude oil, based on extraction of ore amounting to 640,000 tons, the ore reserves would be sufficient for at least 50 years.

B) Mining of the deposits.

At present three layers are being exploited; the Grande Couche (The Main Layer) and the Veines No 8 and 10 of the Thelot unit. The exploitation of the No 6 is being prepared. Only layers apt to yield at least 60 liters of oil per ton of ore are considered exploitable. The Vein Petite Couche (Small Layer) and four Veins of the Thelot unit are therefore qualified for consideration. The oil yield of the exploited layers varied between 60-113 litres per ton and produced over the last five years an average of 95 litres per ton. The present extraction amounts to 150,000 tons a year. The exploitation is depthwise. Owing to inadequate methods employed the operating losses are very high with the efficiency not exceeding 45%. The exploitation is carried on without a preliminary survey and without any preparation of the mine. It is urgently needed to change the exploitation methods; including, first, extensive preparatory work combined with preliminary tests (carrotages). The pillared mines of the Grande Couche and the exploitation of the exposed layers of the Thelot mine should be assessed to obtain better exploitation of the deposits.

The efficiency of the drilling in the mine-shafts is very low. It is necessary to try to increase the efficiency of the drilling through rationalising. (Adaptation of the diameter of the mine-shafts to the diameter of the "cartridge", facilitating the work by supporting the drilling, eventually the introduction of rotary electric Drills, etc.)

C) Efficiency and labor conditions.

The total personnel is 245 men, 45 of which "piqueurs" and "foreurs" are the producing workers, 118 laborers for the extraction and 32 men for the preparatory work. The output of the "piqueurs" is 5.5 tons/layer, the total output of the team is 2,34 tons per man per layer. This output is too low, the number of non-productive operations being too high. The efficiency should be increased by rationalisation measures and the adjustment of salary levels to the minimum needs level.

Initial boiling point 240
 Final boiling point 325
 Diesel index 50
 Initial boiling point 250
 Final boiling point 300
 Diesel index 50

treated and yields kerosene, gas-oil and premium lubricating oil. The aluminic compound is recirculated back into the crude oil undergoing its first Al Cl₃ treatment. In so doing, the Al Cl₃ required is greatly reduced and is from 3.5 to 4.5% by weight of the charge treated.

The secondary oil is given an acid and clay treatment to produce a lube oil inferior to the premium oil but suitable for use in industry.

The semi-industrial plant then is as follows:

- First cut (A) Refining - 1st treatment with Al Cl₃
 (B) Polymerization - 2nd treatment with Al Cl₃

- Second cut (A) Clay treatment.
 (B) Acid and clay treatment of secondary oil

Distillation consisting of a double furnace with two stills. The object of the distillation is to realize:

- (A) The intermediate distillation between two treatments with Al Cl₃.
 (B) The fractionation of the polymerized product and clay treatment of kerosene, Diesel oil and premium oils.

The heavy original cut: 50-100, is still under study. However, it is hoped to realize a good yield of lube stocks from the same. Apparently this cut can yield 50-55% lube oil with a 70 V.I.

The Vacuum Oil Company is also working on this problem but have no results to report.

The yields and inspections of the products from the 0-50 cut are given herewith in the following table.

Efficiency and average characteristics of the products obtained after treatment of shale crude oil light fractions (fraction 0-50) by the refining and polymerization process of the Standard Pac. see Petroles.

	Kerosene	Gas Oil	Premium Oil	Secondary Oil	Phenol & Pyridine	Losses
Efficiency by weight relating to the fraction 0-50 without phenols or pyridine..... %	24.6	32	3	3	3	3.3
Efficiency by volume relating to the crude shale oil	12	15	25	3	3	3.55
Characteristics						
Specific gravity at 15° C	0.808	0.843	0.815	0.825	0.860	
Saybolt visco. at 210° F	-	-	-	-	-	
Engler visco. at 50° F	-	-	-	-	-	
V.I.....	-	-12°	-	-	-	
Four point	-	Luchoaire	90°	75°	-	
Flash open cup	-	110°	-	-	-	
Conradson carbon	-	-	4.42	3.9	-	
Acidity	-	-	0.02	-	-	
Sulphur	0.05	0.12	-	-	-	
N.P.A. Color	-	-	-	-	-	
A.S.T.M.:						
Initial boiling point °C	250	240	-	-	-	
Final boiling point °C	-	325	-	-	-	
Diesel index	-	50	-	-	-	

FRENCH CRUDE OIL PROCESSING

Name of Company	Societe Minerale des Schistes Bitumineux	Geo. Location in France, Province	Oil	Use, Refinery or Schistes
Location	Autun	St. Hillaire (Allier)	Oil	Lubricating (Lubricant)
Capacity of unit observed	150-200 ton/day reactor	2-30 ton/day (bulkline)	30 ton/day	
Capacity of unit designed	150-200 ton/day reactor	2-30 ton/day (bulkline)	30 ton/day	
Working time factor	Unobserved carbon	Size of gas blowers and ducts	2. shales/ton, 4. cycle	Use of rotating reactor
Potential yield of shale processed	60-100 liters/ton	60-80 liters/ton	2.5 liters/ton	
Recovery basis Fischer test	60	1/2		
Carbon in spent shale	Rich only	1/2		
Normal rate of process	0-10 ml.	1/2		
Working time factor	1/2	1/2		
Max. shale temperature of	500	50 less than 1 cm. /10 this 1 cm.		
Plus gas outlet temperature of	300	1/2		
Time of residence thru reactor	25 hours	2 hours		
Type of reactor	Vertical stationary-heating by direct combustion of spent shale in reactor & combustion of gases around reactor	Vertical stationary-heating by recycling products of distillation in exchanger	Vertical rotating-decanting - gas burner - from distillation	
Gas produced	1750 cu.ft./ton shale	Not available	4.71/ton shale	30 cu. ft./ton shale
Quantity recycle gas/ton shale	None	None	None	None
Quantity recycle oil/ton shale	None	None	None	None
Steam required/ton shale	350 Btu. 0.5 psi	None	None	None
Electricity required Btu/ton shale	4.0	None	None	None
Man/shift	5	5	5	5
Extraneous heat required	None	None	None	None
Materials of const. ton/ton shale-day	0.4	None	None	None
Cast iron	0.4	None	None	None
Steel	0.0	None	None	None
18-g	0.0	None	None	None
Fields & on crude oil	None	None	None	None
Gasoline	None	None	None	None
Diesel oil	None	None	None	None
Other oil	None	None	None	None
Residue	None	None	None	None
Gas analysis-CO & Hg	24	None	None	None
Olefins	1.5	None	None	None
CO	1.0	None	None	None
H ₂	16.0	None	None	None
CH ₄	24.0	None	None	None
H ₂ O	22.0	None	None	None
Respectively - Gasoline	12% St. run-50% cracked. Octane No. 65 after acid wash. Product considered 50% saturated & 50% unsat.	None	None	None
Diesel oil	42 Octane No.	None	None	None

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As already mentioned, present trends in the French industry are toward converting the crude shale oil into lubricating oil. Actually, the products made today are converted into gasoline, Diesel oil and heavy lubricating oil, but it is expected that in the future nearly all of the crude will be converted into lubricating oil. At Autun, a Dubbs unit presently cracks their product into gasoline and heavy fuel oil. A semi-industrial lube plant is now being built which will shut down the Dubbs unit entirely, according to future plans. At St. Hillaire, the gasoline and Diesel oil are fractionated out and used directly as local fuel. The heavy cut is sent to Fomen for further processing. Details on these are not available. The Petit furnace is so small as to be of no interest at this time from the point of view of processing. The same is also true of the Lavoye unit.

The most advanced lube oil development has been done by Standard Francaise des Petroles (a subsidiary of the Standard Development Co.), in conjunction with the Societe Minerale des Schistes Bitumineux.

The crude is first split into two parts, the lighter cut of 315° C. and the heavier cut.

Specific Gravity at 15° C.	0.814
Iodine Number	70
Olefinic Compounds	55%
ASTM	I.B.P. - 130° C.
	F.B.P. - 335° C.
Sulphur % by wt.	0.39
Phenols and Pyridic alkaloids	8%

The cut is first washed with dilute sulfuric acid and caustic soda to remove the phenols and pyridic compounds. It is then cracked in the presence of AlCl₃ at 90° C. to remove compounds interfering with polymerization. The aluminic compound, on decomposition, yields fuel oil. The oil itself is now distilled to remove heavy polymers and then is polymerized in the presence of 3-4% AlCl₃. The aluminic compound from this operation is decomposed and re-distilled to form the second part of the secondary oil to be discussed later. The polymerized oil itself (after the aluminic compound has been removed) is clay

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standard sizing grid and skip hoist, to raise the shale to the top of the furnace, complete the auxiliary equipment.

The products after leaving the furnace pass through a separating chamber and go thence to the condenser. There is nothing of outstanding note with the latter.

The weight of the furnace proper handling 30 tons/day, is 18 tons or 0.6 tons of steel/ton shale-day. The weight of the present dryery, crusher, gasogene (coke oven) and condensera is unknown, but as they were brought from scrap piles to serve the experimental unit (and hence oversized in every case), their weights would be of little use for comparative purposes. The weight of the 100-ton furnace - wt. of steel per ton of shale charged, will be less than the present 30-ton unit, so that the total weight of the 100-ton unit with auxiliaries will be less than the present 30-ton unit with auxiliaries. The weight of the 100-ton furnace is 18 tons or 0.6 tons of steel/ton shale-day.

The 7 H.P. motor, driving the inside cylinder, is the largest power consumer in the plant. This plus the blower elevators and pusher will bring the power consumption to 0.5 KW/ton shale/day. The steam and water consumptions are negligible.

The liquid product yield from this plant is 45 liters/ton shale, which is over 100% of the Fischer assay test. This means that, despite losses, the rotary vertical furnace is more efficient than the 100 gram aluminum Fischer reactor. The gas quantity reported is 700 cu. ft./ton, but this is high because no attempt has been made to remove the light gasoline fraction and butane. No analysis of the gas is available.

It should be noted here that if the shale were coated with a layer of the liquid product (as in the Petit Furnace), the gas quantity should be greatly reduced in proportion to the olefines present in the gas. This step cannot be discussed here in France at this time, due to legal technicalities, but is one that should be considered for future applications in the U.S. (See section of this report dealing with the Petit Furnace).

The inspections on the liquid fractions are meager and incomplete. The gasoline, boiling between 40 - 180° C. is said to have an octane No. C.F.R.M. (?) of 80. It is said to contain

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aromatics and olefines in fair quantity. The former are undoubtedly due to the nature of the shale processed here. There is some wax present in the crude, but no inspection on this. The distillation on the

Below 100°	12%
200°	20%
300°	55%
360°	75%

The spent shale contains 4% unburned carbon. This is sufficient to burn the spent shale in a recovery furnace, such as the one seen at St. Billivro.

In conclusion, this unit appears to be the best seen, so far, for future development. It has the best heat transfer to the shale and product removal after distillation, allowing for low temperatures with the resulting absence of cracking. Its construction is simple, and the weights of steel and iron required are very combined with the free ash. American

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IV. Société Bitumes et Schistes, Laverne (Aveyron).

The plant is currently handling 30 metric tons per 24 hours. It is a very interesting unit, and in most respects shows the best promise of all the shale plants seen so far. A larger unit to handle 100 tons per day is under construction and should be under construction

The furnace consists essentially of two concentric vertical cylinders, the smaller one rotating and the outer one fixed. The outer furnace has a diameter of 60 cm. and the inner one 48 cm. Both cylinders are 6 meters long and 15 mm. thick. Crushed shale passes down the annulus between the two, and is constantly turned over in its travel by a cast iron helix 3 cm. wide bolted to the inner rotating shell. The helix is inclined to give the shale an upward thrust by turning it over and keeping it from binding and packing. (If the helix were reversed, the shale would pack up solidly.)

Heat is applied to the outer cylinder by 12 gas burners, 120° apart, staggered vertically to give even heat distribution over the cylinder's surface. The heating gas presently comes from a coke oven, but in the larger unit, now contemplated, it will probably come from a spent shale burning furnace.

The maximum flame temperature is held down to 700° C. while the maximum temperature of the outer cylinder is 575° C. Due to good heat transfer (short heat travel in annulus - 6 cm.) the maximum shale temperature is only 450° C. slightly above the cracking temperature. This is the lowest shale distillation temperature met so far in shale furnaces studies. It is possibly only because of the short travel and the constant agitation of the shale.

It should be mentioned here that the shale is sized by crushing from 0 - 25 mm. Hence this plant is not handicapped by fines or dust as are the Antun and St. Hilaire units. In fact the fines greatly aid in heat transfer of this type and do not interfere with the removal of distilled gases. This is a very important fact when considering the relative economic values of the several processes.

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To remove the products of distillation, superheated steam is introduced at the top of the furnace. It is produced by waste heat from the spent shale and is superheated by the spent combustion gases to 400° C in a heat exchange. It passes down into the inner revolving cylinder and thence laterally into the shale-filled annulus, through holes in the sides of the cylinder. In this manner it immediately contracts the products as they are distilled and sweeps them back into 12 vertical tubes, fastened to the inside of the inner shell leading to a product draw-off. A vacuum of 20 mm. water is maintained at the base of these collecting tubes. The quantity of superheated steam used is 30 - 50 kg/ton of shale or roughly 1 liter of water (steam) per liter of crude oil produced. This water can not be re-used in ammonia, phenols, etc. and is highly corrosive.

The inner shell is suspended from roller bearings at the top of the furnace. This construction is simple as the suspension is at a cold zone and needs no special materials. A guiding trunion at the base keeps the cylinder from getting out of line but carries no load. Rotary motion is imparted to the inner cylinder by bevel gears driven by a speed reducing unit. A 7 H.P. motor is sufficient to turn the unit 3/4 of a revolution per minute.

Another interesting feature of this plant is the shale feed control to the unit, which consists of three hydraulically-operated gate valves, one above the other. The first and third open simultaneously, allowing in the first case shale to enter the seal chamber. The first gate closes, the second opens, allowing this measured quantity to enter the lower chamber. The second gate then closes and the third opens, dumping the shale into the furnace proper. In this way, the shale enters the furnace in the absence of air, and in a steady measured flow. A small quantity of superheated steam is constantly rising into the shale feed, aiding in preventing air leakage. The spent shale leaves the lower end of the furnace in a similar fashion.

Prior to entering the furnace, the shale is crushed by standard rotary crushers. Any type of crusher can be used which will break the shale up into pieces 25 mm. or smaller, since dust and fines are not a drawback. After crushing, the shale is dried by the combustion gases that have left the steam superheater, exchanger and are exhausted by a boiler to the atmosphere at 500° C. Hence the shale is heated up to 150° C before entering the furnace. A

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III. Petit Furnace (Grenoble - Hts. Savoie)

This unit has successfully operated as a pilot plant of 7 tons/day capacity. The yields and quality of its products are good. Its simple design and low material requirements should make it an economical full scale unit. An industrial plant of 600 tons/day has been designed.

The unit consists of a horizontal rotary cylinder, 6 meters long and 50 cm. in diameter surrounded by a square brick furnace with 2 gas burners. The cylinder rotates 1 1/2 turns/minute and is driven by a 3/4 H.P. motor. The helix is welded to the inside of the cylinder, 6 m. long, and the shale is moved from one end to the other.

Shale, crushed to 15-20 mm. size (but containing fines), is fed into a hopper. Stack gases are passed up through the hopper to remove moisture. The shale is then fed into a tank containing an oil which is the primary product of distillation. The oil-moistened shale is conveyed by an inclined worm in an airtight cylinder to another worm which feeds directly into the rotating kiln, also sealed off from the air. The shale is heated up to distillation temperature - 600° C - by contact with the hot cylinder walls. The front end of the kiln is held at 500° C and the discharge end 700° C. The shale remains in the kiln approximately 15 minutes. The spent shale falls into a water quench tank and is conveyed to the dump cars by an inclined screw conveyor. It contains 4% unburned carbon.

Between 80 - 100 kg. of steam is formed per ton of shale processed. This, plus a quantity of non-condensable gases equal to 5 times the volume of distilled products, is injected at the discharge end of the kiln and sweeps the products of distillation back through the kiln to the product duct. Here the dust is settled out and the products taken to the condensing unit. Water is injected here to keep the tar and dust from coagulating in the duct. With this recycling of gas, the products distilled are immediately removed from the hot zone and are not cracked into undesirable components.

The yields, based on the Fischer assay test are very high - 98 to 100%. The shale processed contains 30 to 40 liters per ton of liquid product. The gas made is of the lowest quantity seen so far - 210 cu.ft. per ton of shale. The designer of the

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furnace claims that this low figure is due to two reasons. One, the distilled products are immediately removed on leaving the shale, and two, the saturating of the shale with the liquid product, predominantly olefinic, polymerizes with the unsaturated gases the instant they are formed. It is interesting to note that if a heavy oil is used as a covering medium, a final product is much heavier in boiling range.

The individual yields are as follows, based on crude: Gasoline 27%, Diesel oil 5%, Lube oil 50% and residue and loss 18%. The gas analyzes 33% CO₂ and H₂S, 19% olefines and 56% H₂ and CO. No instructions are available on the gasoline, Diesel oil or lube stock.

The total weight of steel and cast iron in the rotating furnace is 3 tons which equals 0.4 tons of steel and iron/ton shale day. The power consumption is approximately 1 Kw/ton of shale processed. Since no recovery furnace exists at this unit to burn the spent shale, coal in a gas producer is used to supply fuel gas amounting to 35 Kw/ton of shale processed. This is in excess of the gas produced in the distillation which is also burned in the furnace. The ratio of coal gas to distilled gas is approximately 3.5 to 1.

The designer feels that a large industrial plant to handle 600 tons/day could be built. This would have 4 cylinders in 1 furnace, each 1 1/2 meters in diameter and 20 meters long. The limiting factor is the surface area per ton of shale which is 70 kg/sq. meter of cylinder surface.

The unit has a definite economic advantage in that it can take fines in the shale feed, a feature not available in the Autun and St. Hillaire units. It does not have the advantage of continuous contact of the shale with the total heated surface as in the vertical rotating cylinder at Lavernhe. Its gas yield is considerably lower than the latter, but this may be due to the oil saturation of the fresh shale, a feature patented by M. Petit and hence not available at Lavernhe.

In conclusion, this unit is definitely a potential type for development in the U.S. For trials, an ordinary cement kiln, with the helix and other features added, could quickly be built at the mine site. The unit probably is not as efficient as the Lavernhe unit if the latter is equipped with the shale oil saturation equipment.

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The utility requirements are not large. 23 Kg. of steam at 15 p.s.i. pressure per ton of shale are needed. Also 0.4 K.w. electricity per ton of shale. The latter is used mostly in the form required by the shale. The shale used is very low in ash.

The total quantity of materials required to construct the distillation unit is 500 tons per day. The following is a list of the materials required:

	Mat'l/Day	
Steel	60	1.0
Cast Iron	40	0.1
18 - 8	3	0.0
Cast	100	0.2

The advantages of this process over the older one used at Fumpherston and Antun are obvious. The immediate removal of the hydrocarbon product from the shale the instant they are released and the elimination of high temperatures, thereby reducing the danger of cracking, make for a high liquid yield. The whole process is easily controlled. Furthermore, no large iron castings are needed. All the metal (except for structural steel) is of simple black iron sheet.

The main disadvantage lies in the large gas-handling equipment required, which calls for big valves, motors, blowers, etc; and a fairly large structural steel layout. Secondly, there are other large shale refining processes, which allow for greater amounts of fines, thereby eliminating a large potential loss of mined material. The relatively large amount of unburned carbon in the spent shale leaving the distillation reactor cannot be considered a loss, as it is all recovered later in the furnace. Economic conditions could well call for the use of the recovered dry hydrocarbon product gas or other processes, in which case the spent shale could supply all the necessary heat.

The crude oil product from this unit is put through a simple distillation process, resulting in vol. % yields as follows: 15% gasoline, 40% Diesel oil and 45% residue. The Societe Chimique de la Grande Paroisse does not process this residue but sends it on to a refinery at Rouen where it is said to yield 23%

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Lubricating oil, 12%
not available
1 oil
10% resin.
ion. Th
follow
ter produc
on the

	sol	sol
Sp. Gr.	0.7	0.9
Trav	70	24
Eng	29	95°
5	41	144
10	47	173
20	54	217
30	60	256
40	67	294
50	74	327
60	82	360
70	92	
80	103	
90	118	
95	128	
M.P	141	
C%		85.8
H ₂		11 "
Vol. %	200	30

It is understood that the company intends to hydrogenate some of these products after the war, but at this time, no data on this subject is available.

The analysis of the non-condensable gases from the retort are:

	Vol. %
CO ₂	8.1
H ₂ S	7.7
N ₂	12.0
H ₂	22.0
CH ₄	31.0
C ₂ H ₆	4.8
C ₃ H ₈	13.0
CO	1.4

In conclusion it should be pointed out that this unit does not have the advantages of the Petit or Lavernhe Units. Inability to handle fines, large gas handling equipment and lower yields make this unit less attractive as a possible one for further development in the United States.

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II. Societe Chimique de la Grande Paroisse St.Hillaire (Allair)

This process has successfully completed the pilot plant stage and is in process of construction on a full scale basis. Present difficulties in French transport have prevented the delivery of the shale loading equipment and blowers. The pilot plant will handle 80 metric/tons/day and the full scale plant 500 metric tons/day. The guaranteed yield by wt., based on the Fischer assay test, is 95%, although 98% is expected.

The process differs considerably from the Dumpherson unit in that it depends entirely on the recirculation of gas for the heating of the shale. This is done in two steps. The first drying step brings flue gas at 550° C through the preheater, which allows the hot gases to pass through the crushed shale heating the latter up to 150° C and thence to the stack. The actual distillation is carried out in a much bigger chamber by recirculating a gas, consisting of the products of distillation, through a heat exchanger where they are heated to 510° C and thence into the distillation chamber. During this recirculation, 1/10 of the total gas quantity is removed and sent to the condenser for product recovery.

The reactor is of rectangular shape with sides consisting of inclined metal slats. These allow for the easy passage of hot gas from the surrounding annulus into the shale without the danger of the latter backing up into the gas ducts. The whole construction is of light sheet metal. The distilled products in the recycle stream are drawn from the base of the reactor into a blower and pass then into a tubular heat exchanger where they are heated up from 280° C to 510° C before reentering the reactor. Hot stack gases enter the shell of the reactor at 750° C and leave at 525° C. Part of these then enter the pre-heating chamber as already mentioned. A separate and smaller blower is used to transfer the latter stack gases. All blower speeds remain constant as well as the inlet stack gas temperature. The reactor temperature is varied by diverting some of the in-going stack gases to the exchanger.

Shale, broken up to 4 - 50 mm size, enters the top of the pre-heater by a belt conveyer. The time of contact in this chamber is 1 hour in the 80-ton pilot plant and 2 1/2 hours in the full scale unit. It then passes into the distillation chamber where it remains 3 hours in the pilot plant and 5 1/2 in the full scale unit. As there is a 1 hour "dead" period in each unit (passing through

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connecting ducts, etc.) the total times are 5 and 8 hours respectively. The spent shale is taken by a worm conveyer, through a water bath to a special type furnace where it is burned to supply the necessary stack gases for the exchanger. The carbon content of the spent shale entering the furnace is 12%, and on leaving is nil. The water cooling step is necessary to hold down the combustion temperatures in the furnace to 800° C and thereby prevent the fusion of the ash or cinders. If all the dry gas, recovered from the oil refining, is used as extra fuel, only 50% of the spent shale need be burned, and the rest is available for process steam supply.

The spent shale burning furnace is of unique design. It is fed continuously, but dumped four times an hour. The grate is operated by an electric drive and is rotated 180° at the end of each period. The temperature of combustion is controlled by the air intake required.

In the full scale plant, another furnace to make flue gas is installed operating either on dry hydrocarbon product gas or an oil residue. The latter fuel would only be used in emergency. It is normally expected, however, here in France to burn all the dry product gas, as its commercial value is not high due to its predominantly saturated components.

The quantity of fines, with the present crushing equipment is 20%. It is expected that this figure will be reduced to 12%. The allowable fine content is a maximum of 2% less than 4 mm., of which 10% must be 1 mm. The allowable moisture content (to meet the 95% by Fischer assay test) is 4%.

The exchanger is tubular, having 120 tubes in the 80-ton unit and 160 tubes in the large unit. The tubes are 60 mm. I.D. and 70 mm. O.D. and 6 meters long. Since the sulfur content is fairly high in the shale, sulfide deposits tend to reduce the heat transfer in the tubes, entailing frequent cleaning. To allow for continuous operation, the full scale plant is designed to have 2 reactors of 250 tons/day each capacity and 3 sets of exchangers, blowers and spent shale burning furnaces. Hence, the two reactors will operate at all times, while one exchanger will be down for cleaning. This is not so in the pilot plant, where only one exchanger exists, thereby frequently shutting down the unit.

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successful solution would be considered as an accomplishment of the greatest significance. Its realization now at hand, or assured, will no doubt present some important new aspects to the postwar petroleum industry in Europe. Also included in the Appendix, in a V each prepared map indicating the known areas of exploitable prospective deposits of oil shale in France.

With reference to the areas on the map indicated as being prospective, it should be stated that the shale deposits so marked are known to be oil bearing, although precise determinations of their content have not yet been made. They are however, believed to have a content averaging 10 litres per ton, and as such are considered to be of marginal quality. Owing to the billions of tons of shale of this quality readily available on the surface their potentialities may be expected to be thoroughly exploited in the near or immediate future. Also, the fact that a simple process of distillation at temperatures of the order of 500°C. is all that is required to extract the crude liquid from ordinary oil shale is an assurance that a process of fuels and lubricants such as these, will acquire a certain degree of importance as a matter of national necessity.

Summary of Distillation Units

The most promising unit seen so far is that at Severac (Societe Bitumes et Schistes). Its process features, actual yields and simple design make it outstanding for use either on low oil bearing shales or rich ones.

If this unit can also be built to combine the Petit oil saturating step of the fresh shale, its overall performance should exceed anything built so far. This should not be difficult to do technically. The patent rights will have to be carefully reviewed and arranged for.

It must be borne in mind that still other improvements and designs exist here in France probably based on German practice, which are unavailable at this time. As these come to light a detailed report of the same will be submitted. There is a strong possibility that an improved horizontal Petit type furnace can be built which will operate better than the vertical Lavernne type.

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Description of Processes

I. Societe Miniere des Schistes, Autun (Saone et Loire)

This is the oldest and largest shale oil plant in France. It is currently processing 800 metric tons of shale per day. It has operated continuously throughout the war and escaped damage from bombing or sabotage.

The retorts used here are almost identical with the Scotch units at Pumphreston. There are 120 in all, each processing 7 tons/day. The temperatures used and general operating procedure is very much like the Scotch practice, except that the Autun unit injects extraneous steam at the base of the reactors as well as using the steam made by quenching the spent shale. This latter step cuts down the throughput, but greatly improves the per cent yield. It naturally alters considerably the analysis of the liquid gas. Much less acetylene is present, and the amount of ethylene and methane are to be found.

The reactors are large and cumbersome. They require considerable quantities of cast iron and masonry, as well as large and complicated loading equipment. The latter accounts for the relatively high electric power requirements. (For a complete description of this process, see report on Scottish Shale Oil Industry, June 1944, by Lt. R.C. Alarich, USNR).

The final products from this process are 60% gasoline with an octane number 65, and 40% Diesel oil with a cetane number of 45. The gasoline is 12% straight run and 88% cracked, the latter being processed in a Dubbs unit. The total products (when added together) are said to be 50% saturated and 50% olefinic. The shale itself contains much less wax and is generally much less paraffinic in nature than the shales at Pumphreston - a point which must be kept in mind when comparing the two units. The relatively high importance of gasoline in France at this time as compared to other products, is also a reason for deeper cracking and less lubes and Diesel fuel. Ammonia is also recovered, as at Pumphreston.

The ultimate yield of this process, based on the Fischer assay test (see appendix) is 80%, resulting in yields of crude oil equal to 75 liters/ton. The spent shale contains approximately 3% unrecovered carbon.

This process is suitable only for rich shales and is not of interest for future development either in France or the U.S.A.

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known of the great low grade, surface exposed shale deposits in many countries of Europe. They therefore may be dismissed as being unable at this time to contribute directly to the solution of the many problems facing the shale industry on the continent.

Prior to 1941, the great low grade shale deposits of continental Europe, such as were known to exist in the Severac area in southern France, the Crevenay area in eastern France, the Wurttemberg and other areas in Germany, and the Kinne-Kleva area in Sweden, were considered only as remotely potential sources of fuels and lubricants. However, recent personal observation of the French effort, together with a brief study of data and report just released by the French, has disclosed important advancements in the development of large scale low grade shale operations. These include large scale commercial operations in Germany and Sweden since early 1943, advanced pilot plant practice in France in 1944, and the introduction in France of new surface electrical exploring methods of delimiting shallow oil shale deposits, which surface outcrops are hidden by alluvium or other spalled cover.

The importance of the low grade deposits in France lies in their large tonnage reserve and their amenability to low cost operations. In general, shales having a content of less than 50 litres per metric ton may be considered as of low grade quality. Recent surveys (1943-44) by the French have focussed attention on two such surface exposed deposits of rather large tonnage.

One of these known as the Severac deposit in the department of Aveyron, is located about 135 miles northwest of Marseilles. Its ultimate extent has not yet been determined but its known surface exposures are sufficient to indicate reserves of from 500,000,000 to 1,000,000,000 tons, the oil content varying from 40 to 45 litres per ton. This in turn indicates a recoverable oil reserve of from 125,000,000 to 250,000,000 barrels. Pilot plant operations on a 30 ton a day basis have been conducted by the French on the site of this deposit throughout the last half of 1944. Preparations have been made for commercial scale operations in 1945. The description and details of the pilot plant operations at Severac are given in other parts of this report.

The largest of the presently known surface exposed oil shale deposits in France is located near Crevenay, about 90 miles southwest of Strasbourg. Its content is said to average 40 litres of oil

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per ton and its thickness and areal extent indicates a shale reserve of from 2,000,000,000 to 3,000,000,000 tons, or a recoverable oil content of from 500,000,000 to 750,000,000 barrels. Current French pilot plant practice on shales of this content and character show recoveries of 97 to 100% of the content as determined by the "Fischer assay". This is equivalent to a recovery of 650 barrels of crude shale oil per acre foot of shale. It therefore became apparent that shale beds such as these near Crevenay, whose thickness varies from 30 to 50 feet may be expected to yield from 20,000 to 30,000 barrels of shale oil per acre.

Although the prime motive factor in drawing French attention to the immediate potentialities of the low grade shale deposits was their urgent necessity of petroleum fuels, there is little doubt that the successes attained in low grade shale operations in both Germany and Sweden in 1943 also played an important part. Likewise, German interest in the necessity of increasing French production as early as 1941 was also a very important factor. A good example of this latter factor is set out in a report by Mr. Keltzer, one of the leading petroleum engineers of the German oil shale industry. Mr. Keltzer was sent by the German government to France in June 1941 to study and report on the possibility of increasing French shale oil production in the Autun area. A copy of the French translation of his report is included in the appendix of this report. The Germans, however, were not successful in their efforts toward increasing the French production primarily due to a shortage of experienced mining labor. A further attempt on the part of the Germans to increase French production was made in 1943, when two of the leading shale oil technicians of France were conducted on a visit to the shale centers of Germany and Sweden by officials and technicians of the German shale oil industry. A copy of the French technicians' report of this survey of German and Swedish operations in 1943 was not made available for inclusion in this report, but the French have permitted a study of it which will serve as a basis of a report on the German plans and situation in 1943. A separate report on this subject is now in process of formulation and will immediately follow the receipt of this report.

Included in the appendix of the current report is a copy of a French report entitled "Les Schistes Bitumineux Français" which was presented before one of the technical societies in April 1941. It is interesting to note the statement on Page 9 of this report, that prior to 1941, both French and German efforts to treat low grade shales had so far, ended in failure, but that their ultimate

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in general are richer than the low grade shales of southern France so that if a process can be developed to economically process the latter, it should prove feasible at home. The U.S. Army is also interested in shale as a possible quick source of fuels in such countries as China, where local mining labor is plentiful and oil transport from the sea is very difficult. A distillation unit capable of producing 100 BPD of crude shale oil might be strategically important. For these reasons, an inspection trip of the French units and pilot plants was made by Lt. Col. V. V. Brown, USA, Lt. Col. G. A. D. by U. S. Army, and Lt. Col. G. A. D. by U. S. Army, on 12 October 1944.

Scope of Report

This report deals primarily with the methods used in distilling the shale. Time did not permit a study of French shale mining methods, but these are in places of little interest at this time, as practice in the U.S. is well advanced in this production phase.

Some attention has been given to the processing of the crude oil obtained, but this again is secondary to the main question of crude oil recovery. Each French refinery has its own unique problem to meet. Furthermore, the composition of the shale oil itself will have considerable effect on the quality of the final product. The question of cracking, reforming, polymerizing or hydrogenation is a relative one and in every case and the application of which would be difficult to correlate with U.S. shale developments.

It must be mentioned here that much of this material was received and accepted as confidential information on the part of the ALSOS Mission. Its distribution therefore, to competitive French or other interests must be scrupulously avoided as the patents on some of these processes have not yet been awarded.

Oil Shale Resources of France.

The only developed areas of oil shale production in France at the present time are confined to two localities in central France. The deposits of both areas are of Permian age. The most important, the Autun area, in the department of Saone et Loire, has been a commercial producer of oil shale for the past seventy-

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five years. The shale deposits of this area occur at depths of from 100 to 180 meters below the surface and have a recoverable oil content of from 80 to 100 litres per metric ton. Daily production at present varies from 600 to 700 tons of shale per day the mining methods requiring mostly hand labor. In view of the almost prohibitive costs of mining the shale, the practice as observed during the visit at Autun could not be considered as potentially applicable to contemplated American operations.

The shale beds proper vary in thickness from 2 to 4 metres. The character of the shale is such that it yields readily to the crushing operations, prior to being fed to the distillation furnaces. After passing through the coarse roll crushers, the only crushing stage required, the maximum size of the shale pieces is reduced to 60 millimeters in diameter. About 80% of the fines (15% of total tonnage mined) are rejected and conveyed to the dump, the remaining shale being fed to the furnaces. The available reserves in this area are believed to approximate 40,000,000 tons, of which 25,000,000 tons are considered as probable reserves.

Operations in the St. Hillaire area in the department of Allier are very similar to those of the Autun area. The shales are of the same character and are mined from a depth of approximately 180 meters. They are not quite as rich as the Autun shales, the recoverable oil content varying from 60 to 80 litres per ton. Commercial production of shale since 1937 has been limited to 30 tons a day which is treated in a single furnace. This furnace has served as a pilot plant and is the forerunner of a modern 500 ton unit, now under construction at St. Hillaire. The operation and details of this plant are described in another part of this report. The proven reserves of the St. Hillaire region are given as approximately 20,000,000 tons and the additional probable reserves at 10,000,000 tons.

The only two areas of current shale production in France therefore, may be said to contain combined reserves of some 70,000,000 tons, the content of which averages about 80 litres per ton. This represents an ultimate recoverable reserve of crude shale oil in the amount of 5,600,000,000 litres or 35,000,000 barrels. Certainly, both the mining technique and the shale distillation operations as practiced at Autun and St. Hillaire are not to be considered as economically applicable to the more well

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ALSOS-59

ALSOS INTELLIGENCE MISSION
UNITED STATES NAVY

The French Shale Oil Industry

Introduction.

This report describes briefly the Shale Oil Industry of France and the technical methods used in obtaining the oil from the shale. Since France has only a small quantity of petroleum, the exploitation of shale is of vital importance. Present trend point toward using the shale oil obtained for further processing into lubricating oil rather than motor fuels, and it is hoped thereby that the country can be made self-sufficient in this vital product. Gasoline, Diesel oil and fuel oil will always have to be imported but if the lubricating oil is made can meet a long and vital need.

Furthermore, the French shale industry had numerous contacts and visits with German technicians during the occupation. It is now definitely established that Germany is exploiting her shale industry as much as possible, especially in Wurttemberg. From studying the French industry, contacts, names of persons and problems discussed by the Germans were obtained, which can now more easily be developed when the latter country is available for investigation.

The French shale deposits can be roughly divided into two categories, the rich deposits in the central section of France (Autun - St. Hillaire) and the low grade oil-bearing deposits in the south and east. The latter are presently of much greater importance due to their extensive occurrence, and offer opportunity for large-scale exploitation provided an efficient process of distillation can be developed. It is to this end that the French industry is now devoting its efforts.

Fuel technicians in the U.S., especially those of the U.S. Navy, have for some time been considering shale as a future source of crude oil. To this end, the Bureau of Ships has set up a group of technical officers to investigate and select the best distillation methods for processing U.S. shales. American shales

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1941

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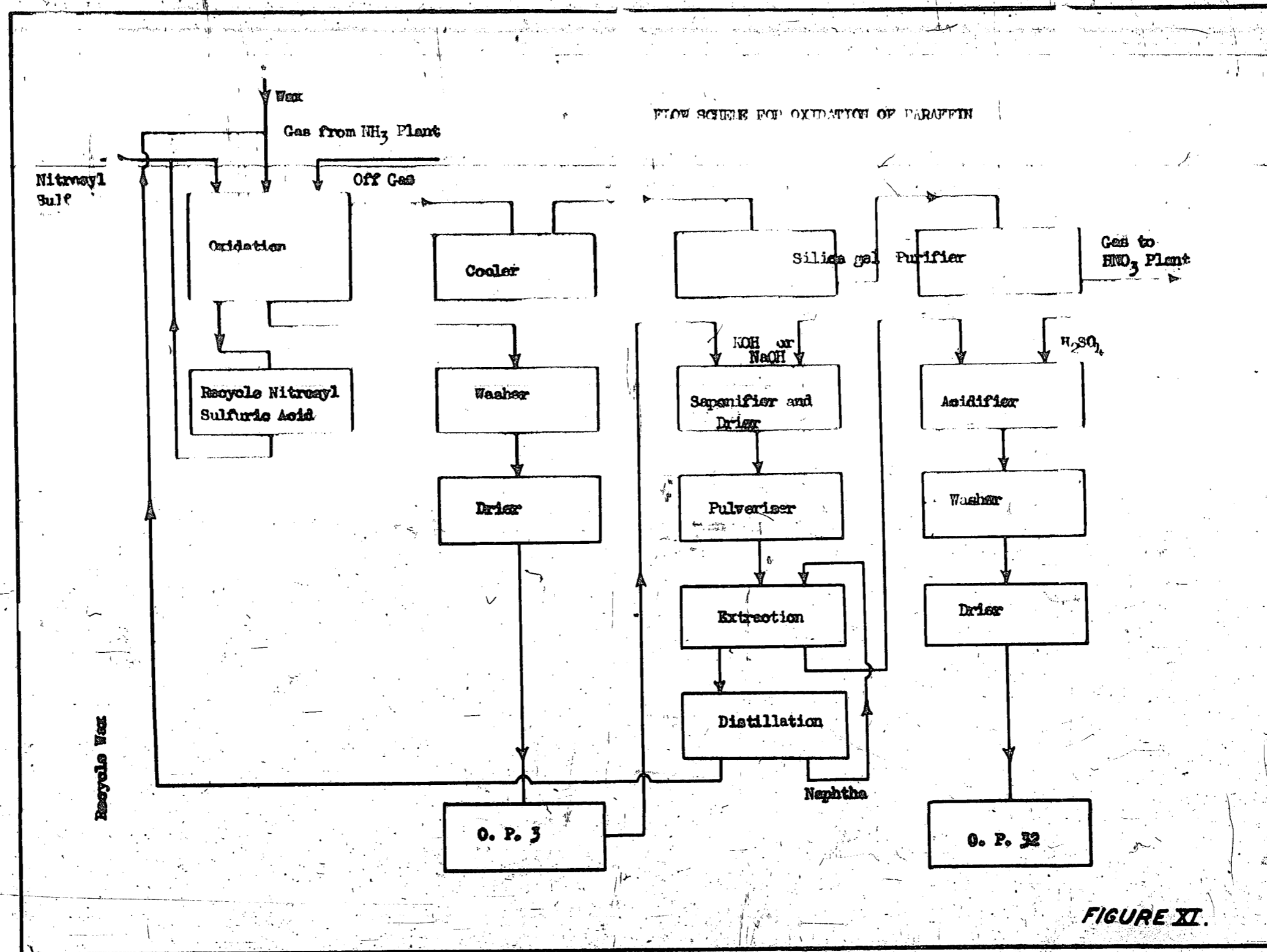
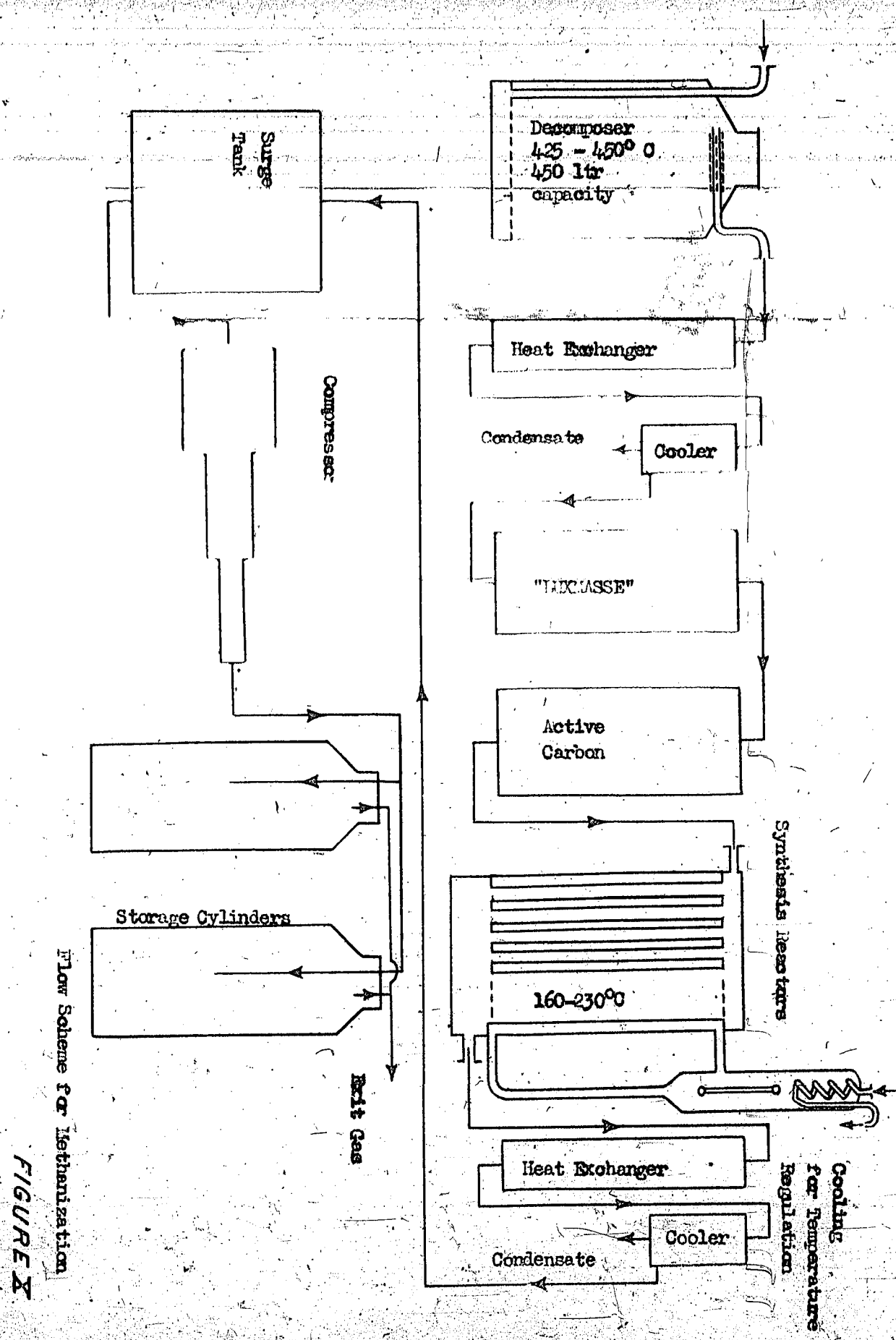


FIGURE XI.



Flow Scheme for Methanization
FIGURE X

FLOW SCHEME FOR NICKEL CATALYST PREPARATION

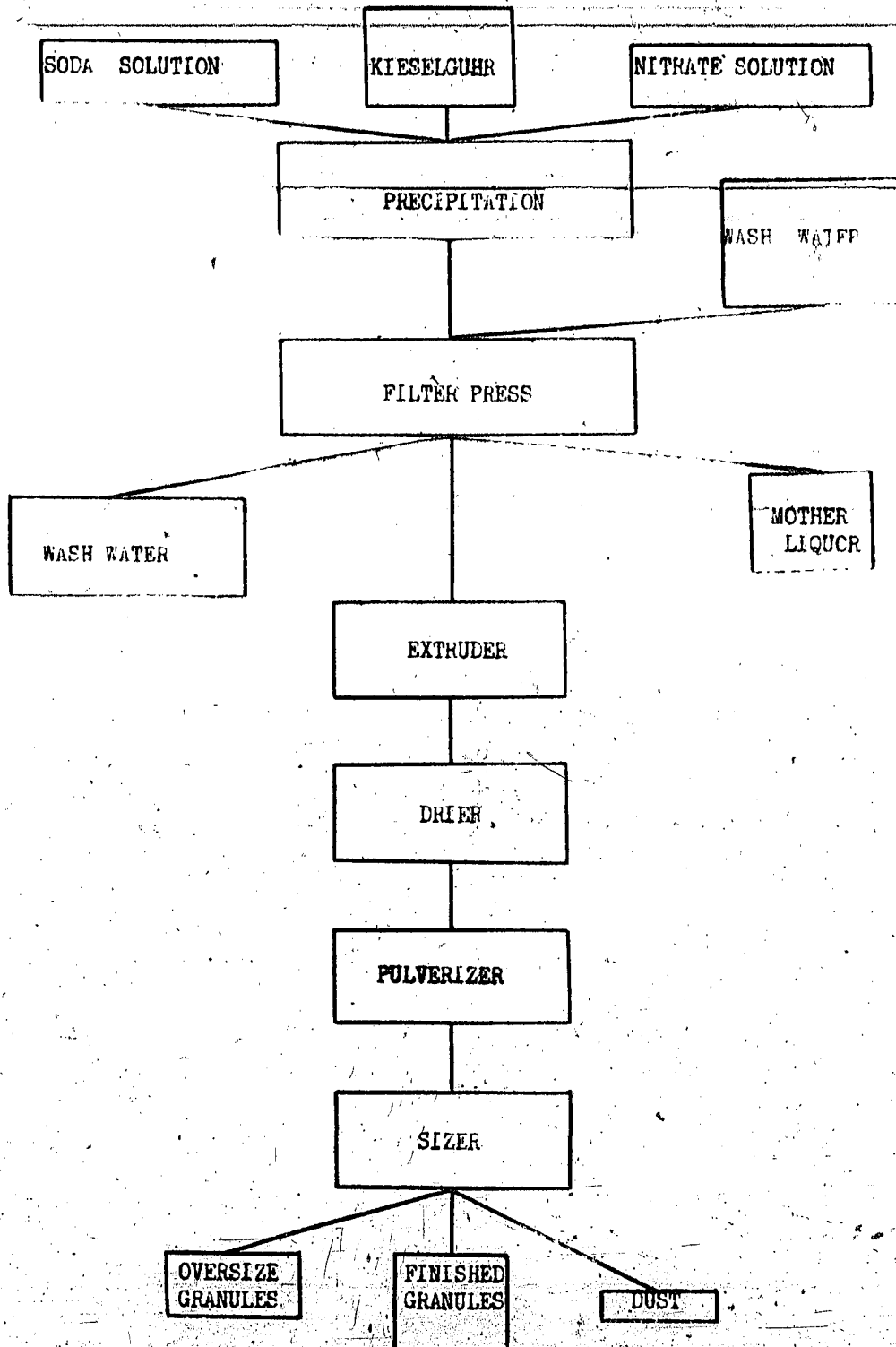
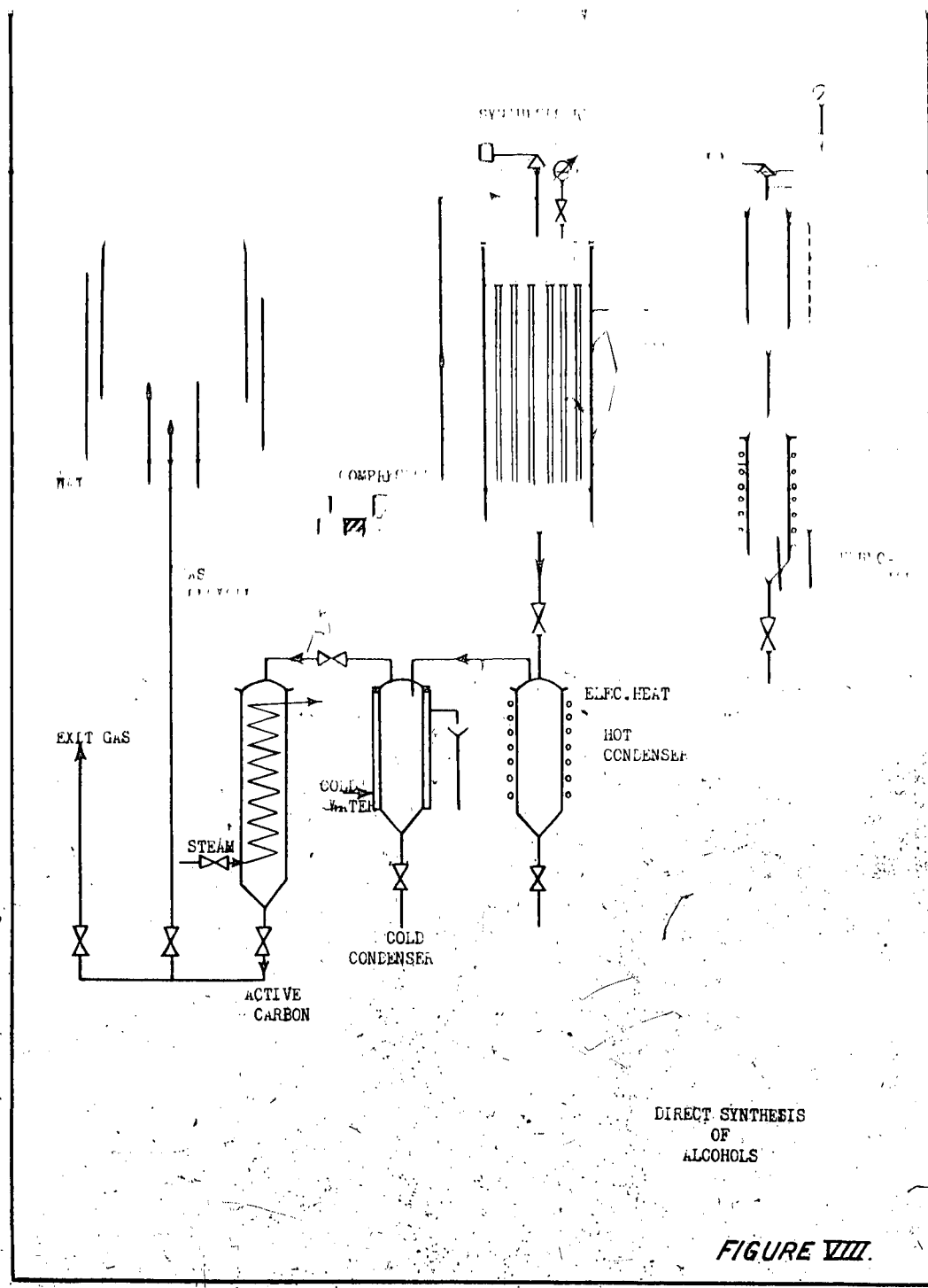
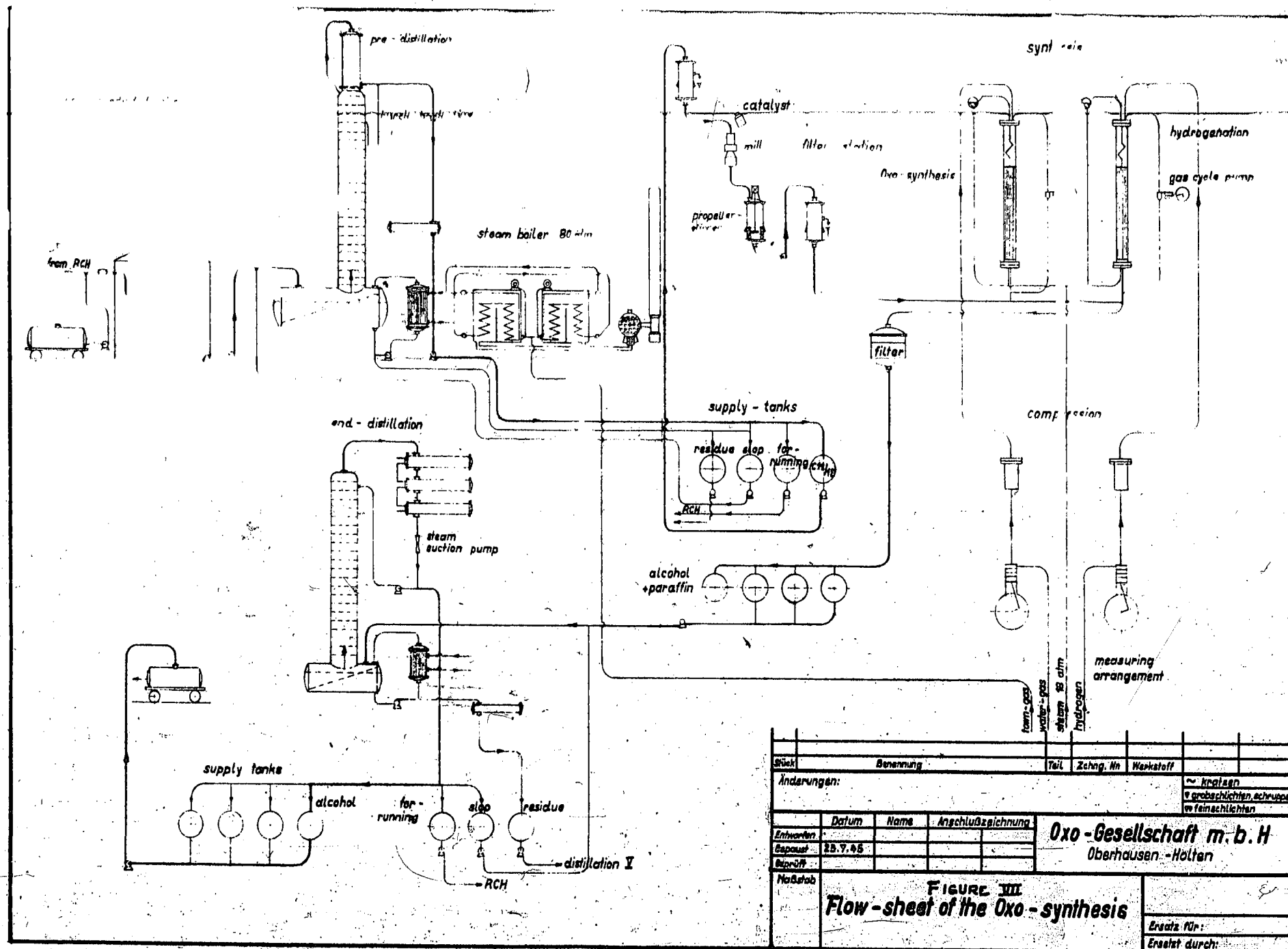


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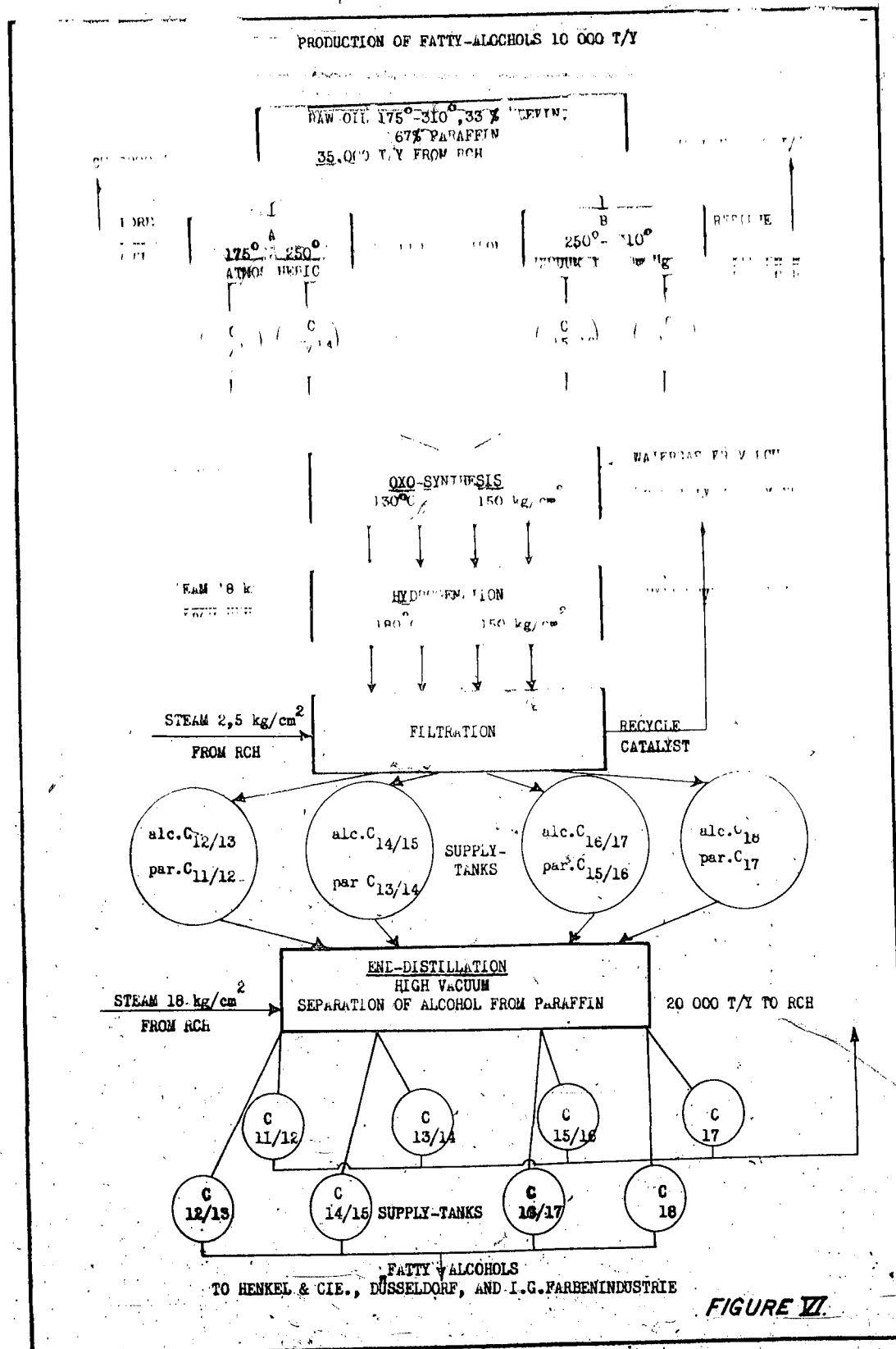


DIRECT SYNTHESIS OF ALCOHOLS

FIGURE VIII.



Blatt	Benennung	Teil	Zehng. Nr.	Werkstoff
Änderungen:				
Entworfen	Datum	Name	Anschlusszeichnung	
Begutachtet	25.7.65			
Gezeichnet				
Notabeb.				
Oxo-Gesellschaft m. b. H.				
Oberhausen-Holtan				
FIGURE III				
Flow-sheet of the Oxo-synthesis				
Erstellt durch:				
Erstellt durch:				



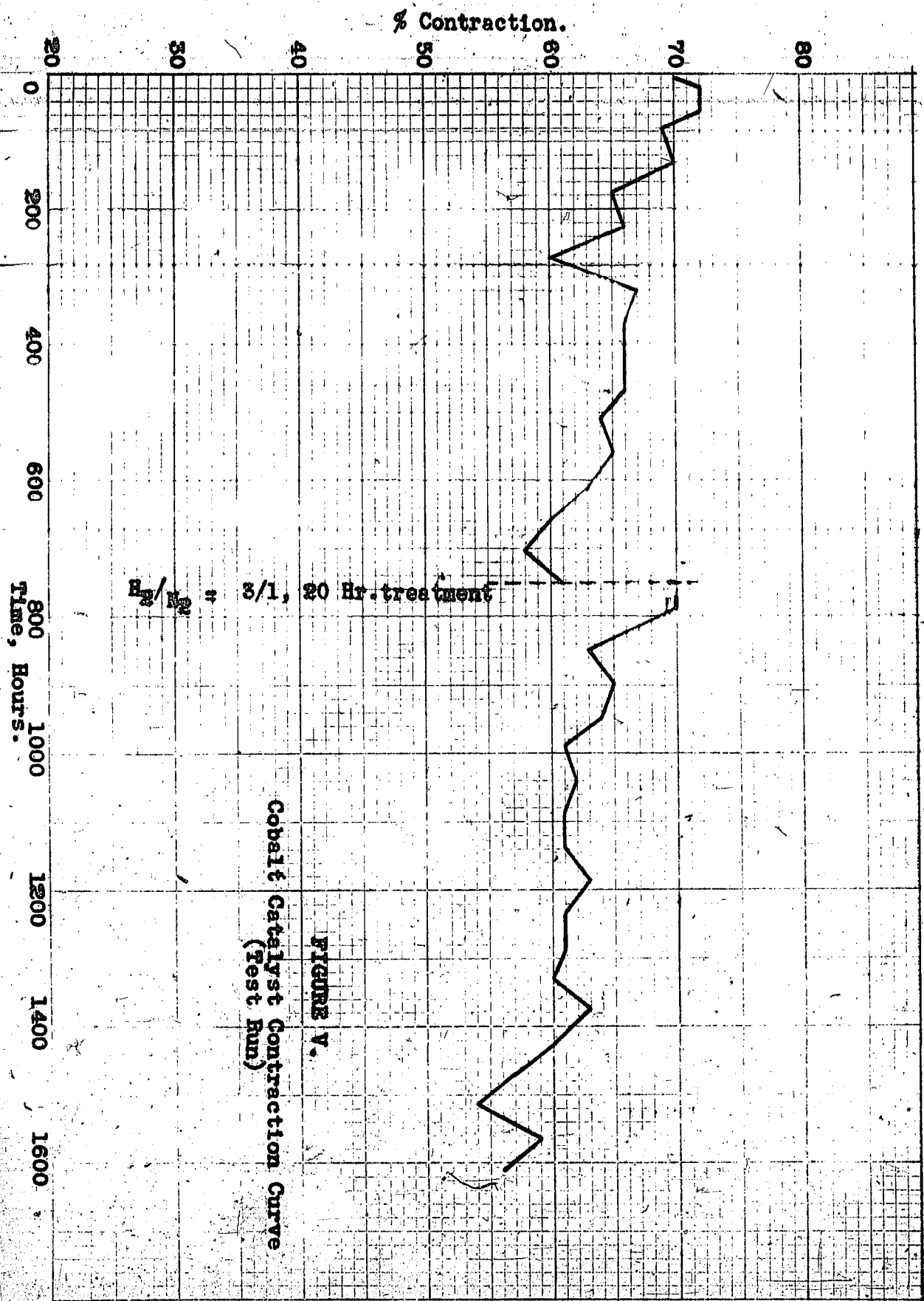


FIGURE V.
Cobalt Catalyst Contraction Curve
(Test Run)

APPARATUS FOR CATALYST TESTING

2 way valves

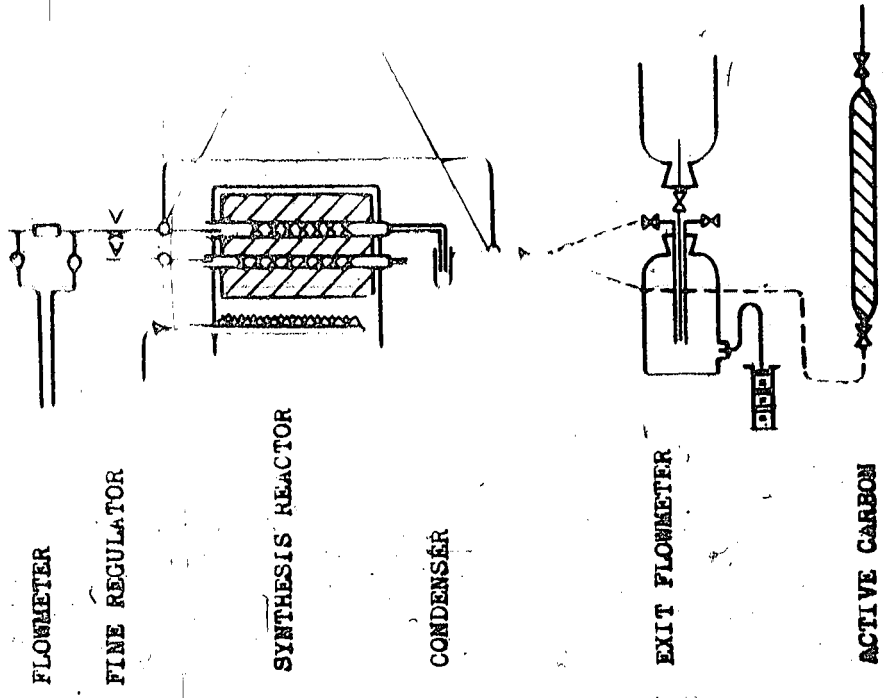


FIGURE IV.

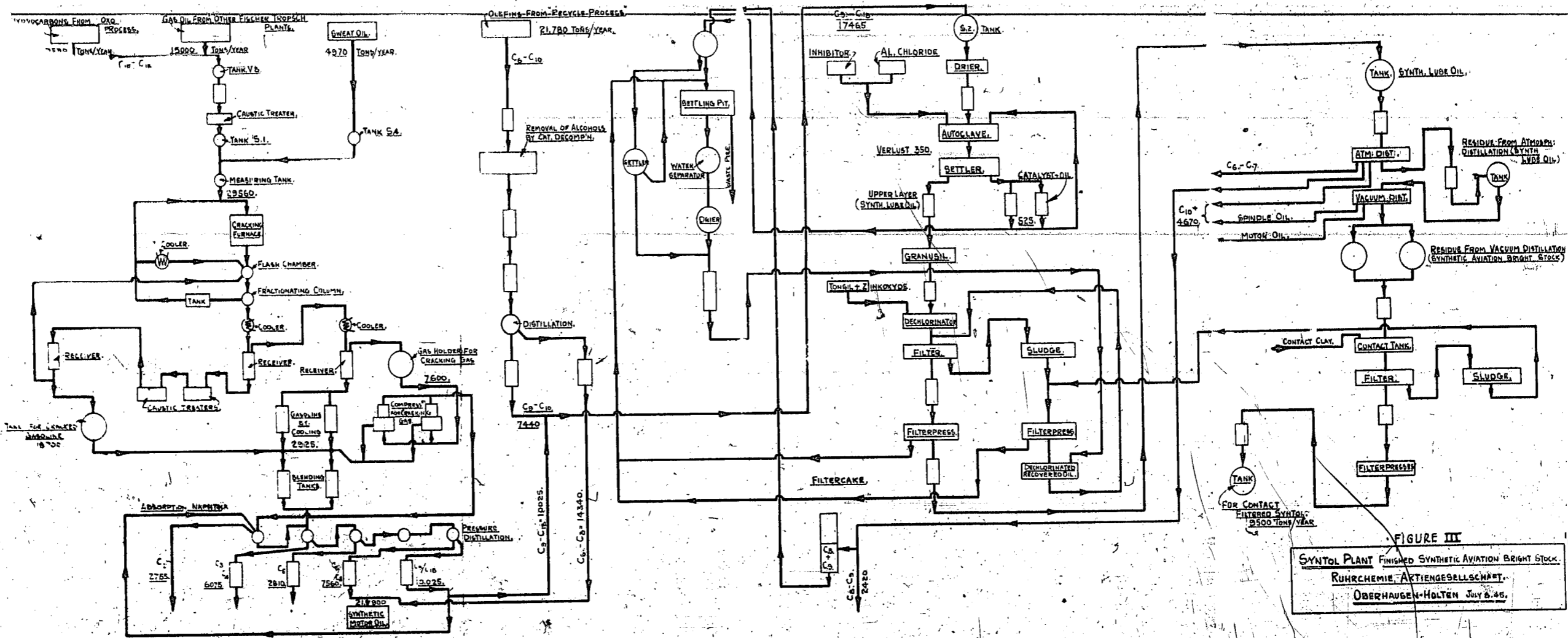
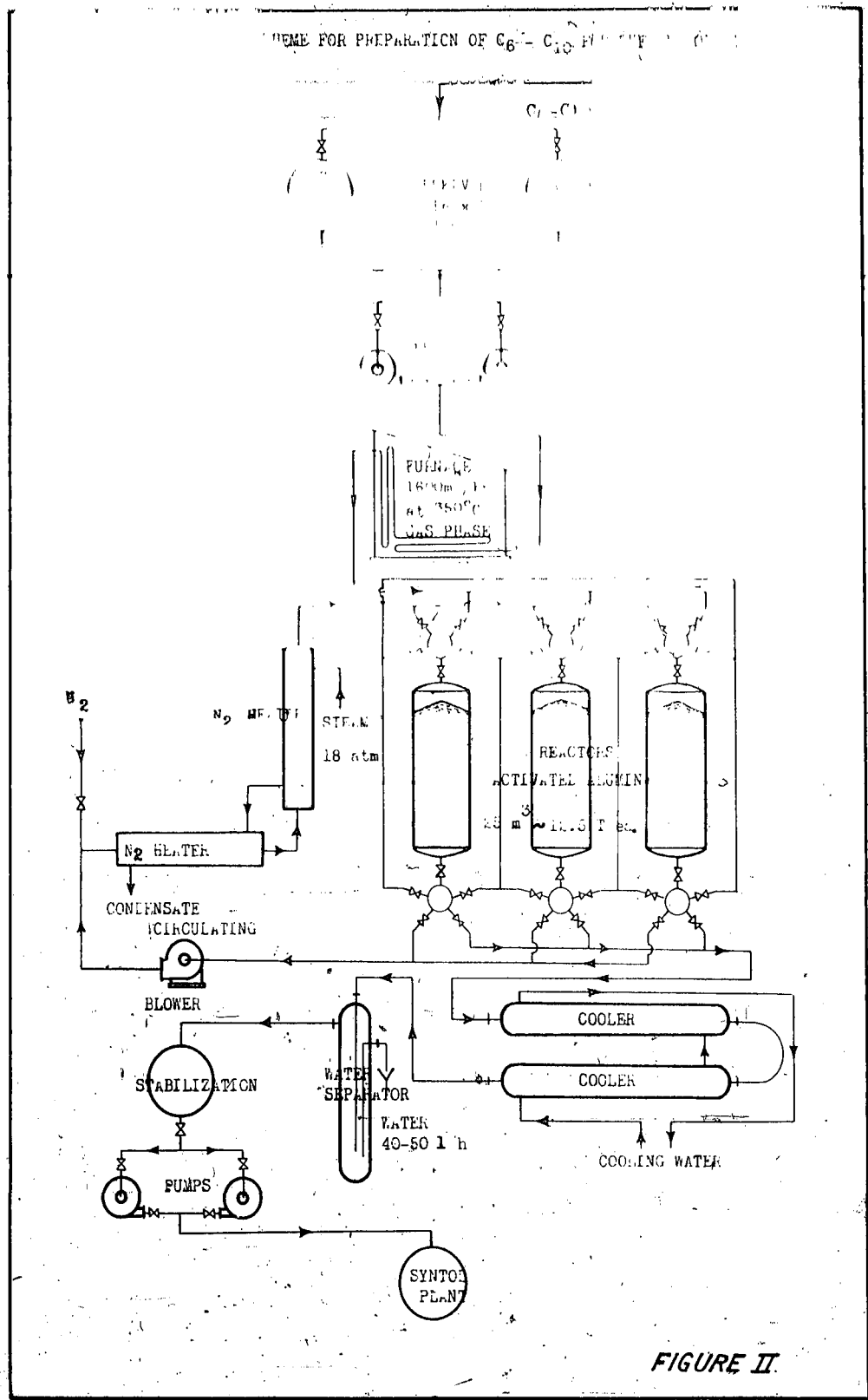


FIGURE III
 SYNTOL PLANT FINISHED SYNTHETIC AVIATION BRIGHT STOCK.
 RUHRCHEMIE-AG/GESELLSCHAFT.
 OBERHAUSEN-HOLTEN July 2, 46.



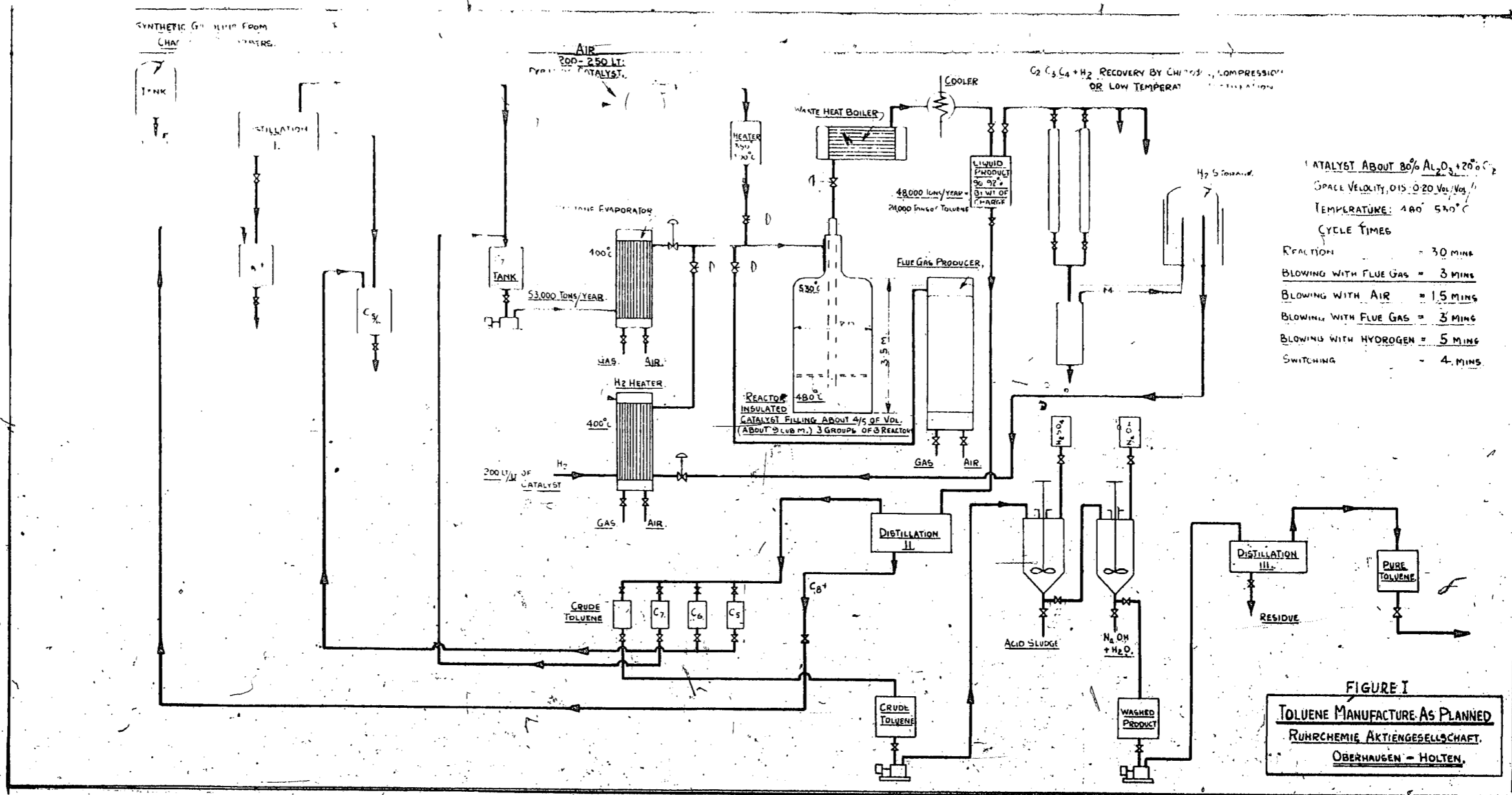


FIGURE I
TOLUENE MANUFACTURE AS PLANNED
 RUHRCHEMIE AKTIENGESELLSCHAFT
 OBERHAUSEN - HOLTEN

Question 5. What work has been done on micro-crystalline waxes?

Answer. No information is available.

RUST PREVENTIVES

What rust preventives of the solvent type, suitable for spray application, are used, and what is their composition. With particular reference to corrosion inhibitors?

What corrosion preventives of the petrolatum type brush or swab application, of the transparent hard drying type, and of the permanent film paint type are used? Are any special types of solvent solvents employed?

Are "fly-away" corrosion preventives used, and what is their composition?

Have any special compounds been prepared for cold protection in stored engines?

Are any corrosion preventive additives proposed for addition to turbine oils, transformer oils, etc.?

5. CABLE OILS.

Question 1. To what extent are oil filled cables used; what oil specifications are laid down; what are the best sources for oils of this type?

2. Are any blending agents or additives employed (e.g. resins)?

3. To what extent are oil impregnated insulated types used? What oils are used, and what specifications have been laid down?

4. What methods of test are used to determine dielectric strength, and s.i.c. for such oils?

Answer. No precise information is available.

6. COOLANTS AND DE-ICING FLUIDS.

Question 1. What coolants are used in automotive engines, both

for normal operation and low temperature, and in aviation engines? Are corrosion inhibitors added? Are soluble oil emulsions ever employed?

Answer. Water and water-Glysantin mixtures. The use of oil emulsions is unknown to Ruhrchemie.

Question 2. What fluids or compounds have been developed for de-icing of aircraft, and what additives are used? For what purposes are lithium and potassium compounds used in these compounds?

Answer. No information is available.

7. MISCELLANEOUS.

Question 1. Have detergents of the ester salts type been developed and how are they made?

Answer. No precise information is available.

Question 2. What developments have been made in products used for pest control?

Answer. No precise information is available.

Are they being used for any other purposes?
What are the user requirements?

2. To what use are the phenol extracts from solvent extraction or lubrication oils put?
3. How are olefine polymers such as those of ethylene, propylene and particularly of butene and butadiene being utilised?
4. To what extent are polymers used in the manufacture of lubricants?

Answer: No information is available.

3. WAXES.

Question 1: To what uses are waxes produced from petroleum, brown coal oils, and Fischer Tropsch processes being applied, and what particular properties are demanded by the user?

Answer: Paraffin waxes from petroleum, brown coal and the Fischer Tropsch process are used in the following industries, among others:

- (a) In the manufacture of floor polishes etc.
- (b) " " " " shoe creams etc.
- (c) " " " " cable insulation materials.
- (d) " " " " candles.
- (e) " " " " artificial flowers.
- (f) For sizing paper.

As well as these direct uses paraffins are worked up on a large scale in the cerisin industry.

Three types of paraffin wax resulted from the Fischer Tropsch synthesis namely slab paraffin wax, R.B. hard wax, catalytic paraffin wax (Kontakt-paraffin).

Their properties are as follows:-

	Slab Paraffin Wax	RB Hard Wax	Catalytic Paraffin Wax
Colour	White	White to ivory	Ivory to yellowish
Solidification Pt.	50/52°C	90°C	90°C (Brown)
Melting Point	50/52°C	100°C	100°C
Acid & Sap. values.	0	0	0
Components boiling below 450°C.	-	10	-

Question 2: What types of waxes (with specifications) are used in candle manufacture, paper impregnation, insulating?

Answer: In candle manufacture slab paraffin wax to a great extent, RB Hard Wax are used.

Question 3: To what extent is slack wax from solvent dewaxing processed into a usable wax, and how is this? What proportion is used as a substitute?

Answer: No information is available.

Question 4: What results have been obtained with the use of montan wax, and synthetic substitutes in grease manufacture?

Answer: Wax acids, obtained by the oxidation of RB hard wax, were tried as synthetic substitutes in grease manufacture. Two types were investigated, designated as OP3 and OP32. They have the properties shown below:

	OP3	OP32
Colour	White to ivory	Yellow to ivory
Solidification Pt.	80-82°C	80-82°C
Unsaponifiables	50	3-5
Acid value	70-80	140-150
Sap. value.	75-85	145-155

The wax acids were tried out in the manufacture of sodium soap greases as well as with industrial greases, e.g. hot-bearing greases. They were also tried in the standard army chassis grease (Abschmierfett). Greases developed from these wax acids fulfilled all that was required of them and were remarkable for their specially high water resistance.

Answer. Fats saponified with NaOH were in use, as far as is known to Ruhrchemie.

Question 5. For what reason and to what extent are greases used in shock absorbers and aeroplane landing gear?

Answer. No information is available.

Question. What material is employed in the manufacture of Voltol?

ANSWER.

To what extent are aqueous emulsions used in industrial lubrication?

Has this type of lubricant ever been employed in engines of any type?

They have been repeatedly used in industrial lubrication in recent years and with good results, as far as is known (e.g. with air compressors).

Question 3. By what processes is Voltol made?

Answer. No information is available.

QUESTIONNAIRE No. 3.

Special Products

1. BITUMENS.

Question 1. To what extent are bitumens of petroleum origin used in road construction, and to what extent are hot application, cut-back, and bituminous emulsion application used?

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Question 2. What advances, if any, have been made in bitumen emulsion manufacture, and what crude sources are preferred for this purpose?

3. Are emulsions of any other pitch residues used for any purpose?

4. What work has been done on soil stabilisation, and to what extent has this process been used for airfield runways, etc.? What types of emulsions have been found most suitable, and how are they made?

To what extent are bitumens and emulsions used for:

- (a) Paper impregnation?
- (b) Roofing?
- (c) Insulation?
- (d) Paint applications?

What filters, if any, are used?

Answer. No information is available.

2. EXTRACTS AND POLYMERS.

Question 1. Are lubricating oil extracts produced by either acid treatment or solvent extraction being used as substitutes for:

- (a) Linseed oil in paints and putties?
- (b) Plasticisers in rubbers and P.V.C. compositions?

- 88 -

- Question 3. What solvents such as diacetone, alcohol, methyl carbinol, etc. are used ?
4. To what extent are water and glycerol used in hydraulic fluids ?
5. What work has been done on non-inflammatory hydraulic oils ?
6. What oils seals are used, and has any trouble been experienced as a result of interaction between seals and fluids ?
- To what extent are compounded mineral oils used, and why ?

Answer. No exact information is available.

6. RAILROAD LUBRICANTS.

1. What oils are used for steam cylinder lubrication (satd. and superheat) valves, axle boxes, etc., and what compounding agents are used, and why ?
2. What type of crudes are preferred for this purpose ? What asphaltene content is usual in such oils ?
3. Are any special oils used in the lubrication of diesel locomotives, and what additives, if any, are employed ?

Answer. No information is available.

7. INDUSTRIAL LUBRICANTS.

- Question 1. What types of oil are used in steam turbine lubrication ? Are any troubles experienced with corrosion, emulsification, and if so, how are they combatted ? What additives are used, if any ? What is the average life of a steam turbine oil ?
2. What types of oils are used for transformers ? What electrical tests, if any, are carried out ? Are any additives employed ?
3. To what extent are mineral, non-mineral, compounded oils, and aqueous emulsions used in quenching ? What types are preferred for small and large objects ?

- Question 4. What types of cutting oils are used ? To what extent are sulphurised and compounded oils used ? Are corrosion inhibitors added, and if so, what are they ?
5. What types of slushing oils are used ? What additives are employed to give anti-corrosion properties ?
6. From what sources are textile oils refined ? What research has been done on carcinogenicity ?
7. What oils are used for clock and instrument lubrication, especially for aviation ? What compounding agents are used ?
8. What is "Radiol", and for what uses is it employed ?

What is the composition of machine oils ?

Answer. No information is available.

8. GREASES.

- Question 1. To what extent are barium, lithium and aluminium soap greases used ?
- Answer. No precise information is available.
- Question 2. What fats are used for greases, and what substitutes have been employed ?
- Answer. Fatty acids for the first runnings in the air-oxidation of paraffins are used as substitutes.
- Question 3. To what extent are waxes, especially montan wax, used in grease manufacture ?
- Answer. Montan wax is used as the standard army chassis grease (Abschmierfett). Experiments aiming at substitution by the synthetic Ruhrchemie wax acids OP3 and OP32 were in progress.
- Question 4. What types of greases are used for high temperature applications ?

Question 11. What additives are used as pour point depressors, V.I. improvers, anti-oxidants, antiwear additives, anti-corrosion additives, and film strength improvers?

Answer. Parafflow was used to lower the pour point and Oppanol to improve V.I. Other additives are unknown.

Question 12. To what extent are fatty oils used in motor lubrication? What kind of fatty oils are used, and what advantages have they?

Answer. Fatty oils were not used.

Were any lubrication difficulties experienced with producer gas equipment (both open and coal type) bottled gas, or acetylene in the engine?

3. MARINE

1. What lubricating oils are specified for use with marine diesel equipment?

2. What oils are used in steam turbine driven vessels? Are additives of any kind incorporated (anti-corrosion etc.)?

3. To what extent are compounded cylinder oils used, and what are the compounding agents employed?

4. To what specifications are stern-tube greases manufactured, and what compounding agents are employed?

5. For what uses are aqueous emulsions of oil, with the addition of montan wax, employed?

Answers. No precise information is available.

4. TRANSMISSION LUBRICANTS.

Question 1. What lubricants are recommended and specified for tank gear boxes, and car and truck gear boxes of normal and synchromesh types?

Answer. The use of a standard gear oil (mineral oil raffinate with a high pressure additive) was stipulated by the HWA for all gears and rear-axes.

Question 2. To what extent are voltolised oils and compounded oils used for tank gear boxes? What compounding agents are employed?

Answer. Voltolised and compounded oils have not been in recent use.

Question 3. To what extent are additives of the nitrogen type, chlorine type, etc., used in oils for gear boxes?

Answer. The nature of the high pressure additive is unknown to RCH.

Question 4. In what proportion are straight tooth, spiral hypoid, and worm gears used in rear axles, and what oils are specified in each type?

Answer. See answer to question (1). The gear manufacturers were permitted to use special (hypoid) oils for running-in purposes.

Question 5. What additives are used in extreme pressure oils?

Answer. No precise information is available. "Rtr 1" was used, among other additives.

Question 6. To what extent are compounded oils (fatty oil compounded) used for rear axle lubrication?

Answer. No information is available.

Question 7. What type of oil seals are used?

Answer. None.

Question 8. What temperature extremes are encountered in the different types of axle?

Answer. Reputedly 250°C. (in tanks).

5. HYDRAULIC, RECOIL and BUFFER OILS.

Question 1. To what extent are pure mineral oils used for this purpose? What specifications apply with particular reference to V.I., pour point, etc.? From what crudes are they made and by what refining methods?

2. To what extent are non-mineral oils used for this purpose? How much castor oil is used for this purpose?

Answer. No information is available.

Question 17. What lubrication system and lubricant are used on the flying bomb, including ancillary equipment, gyros, etc.?

Answer. No information is available.

Question 18. What type of aviation oils are used in jet propelled aircraft, L.G. turbines, rocket propelled missiles, etc., and what special properties are required? What temperature extremes are met in such devices? By what methods are low pour point oils obtained? Is more than one oil used?

Answer. No information is available.

Question 19. Are diluents employed with aviation oils, if so, to what extent, and why? What diluents are used?

Answer. Dilution, employing ordinary fuel, was used to improve the cold starting performance of some engines.

2. AUTOMOTIVE

Question 1. To what specifications are lubricants for petrol engines and high speed diesel engines produced, and what relative importance is assigned to the various properties?

Answer. They are produced according to the HWA delivery specifications (not available here at the moment). The viscosity and pour height were regarded as being specially important.

Question 2. What are the chief sources of motor oils? What crude oils are used, what refining methods, and to what extent are synthetic oils incorporated?

Answer. Since the highest grade components were placed at the disposal of the Luftwaffe, motor oil had to be mixed with 10-35% of synthetics to bring its quality to an adequate level.

Question 3. Are engine tests carried out before approval, and if so, what are they?

Answer. When a new type of oil was put into use engine tests

were made, as far as was thought necessary. The following properties were examined: ring-sticking, wear, aging, sludge formation and piston seizure. A small scale engine was used as described in the accompanying report.

Question 4. What laboratory tests are carried out on the oils used in the flying bomb?

Answer. No particular laboratory tests were carried out from the usual inspection tests.

To what extent is the oil used in the flying bomb more commonly used in other types of engines, and if so, what special conditions are met in such cases?

Answer. No.

Question 5. (a) What type of engine is used in the flying bomb?

Answer. No.

Question 6. Is trouble experienced with ring sticking in the engine, and if so, what is the cause?

Answer. No.

Question 8. What type of filters are used in mechanically propelled vehicles? If chemical type filters are used, is any trouble experienced with them?

Answer. Mainly slit-filters are used. Chemical filters were not employed.

Question 9. What mileage is normally recommended between sump drainings?

Answer. ca. 3000 km. (not known exactly)

Question 10. What inspections are carried out on used oils, and how do the various types of oil compare on used oil conditions?

Answer. No special inspection techniques are known to Ruhrochemie.

Question 4. To what extent is Voltol used in aviation oils? What are the reasons why it is or is not extensively used? What effect has Voltol on V.L. or pole height.

As far as is known to Ruhchemie, voltolised oils were only permitted for use in certain engines (aero-diesel). They could not be used in Otto engines because of a pronounced tendency to cause ring-sticking. The advantage was not so apparent in aero diesels.

Are the advantages of the incorporation of detergent type additives recognized in aviation oils?

These detergents are not used by Ruhchemie. No other precise information is available.

Are viscosity-temperature susceptibility curves of the Otto engine oils, etc., available?

No information is available.

Have additives of any other type been used in aviation oils, if so, what are they, and what results are obtained by their use?

Answer. No exact information is available.

Question 8. What trouble have been experienced with (a) oil frothing, (b) ring sticking, (c) bearing corrosion, and what remedies have been found?

Answer. No constructive measures against frothing and ring sticking are known. There is an unmistakable difference in the ring-sticking behaviour of different oils. RCH synthetic oil was better here than mineral or voltolised oil. Our investigations indicated that synthetic oils behaved well as regards frothing. This field has not been thoroughly investigated yet, however.

Question 9. Have any particular combinations of bearing metals given trouble with any particular oils, if so, how were the troubles overcome?

Answer. No information is available.

Question 10. What engine tests are carried out on an oil before it is accepted for aviation use, and by what methods are the merits of a given oil assessed? If small scale engines are used for oil testing, how do they correlate with main engine performance.

Small scale engine trials only were used by Ruhchemie. These gave a good evaluation (see accompanying report). The official tests in the BMW single cylinder and in different full size engines included general behaviour especially ring-sticking, sludge formation and thickening. The agreement of these tests with the RCH small scale engine tests has been very good.

Has any connection been found between condition of engine and type of oil?

Answer. No.

What laboratory tests are considered most important and has any laboratory procedure been evolved which ties in closely with actual behaviour in an engine?

No laboratory tests are known which give clear and certain indications of actual running behaviour. The most important laboratory tests are considered to be viscosity, pour point, acid value and saponification value.

Question 13. How many hours running are normally carried out between overhauls?

Answer. No information is available.

Question 14. To what extent are oil filters and/or centrifuges used on aircraft engines, and what types are used?

Answer. No precise information is available.

Question 15. Has any trouble been experienced with sludge deposition in oilways, etc., and if so, what remedies have been applied?

Answer. According to the RLM, difficulties did arise. Remedy measures are not known. Synthetic oils had only a small tendency to sludge formation.

Question 16. Has any trouble been experienced with scuffing of reduction gears and what remedies have been applied?

Question 11. What types of diesel fuels are used in (a) submarines?
(b) motor ships
of all types
(c) land vehicles?

Answer. No information is available.

Question 12. What is the composition of the submarine fuel heavier than water?

Answer. No information is available.

Question 13. What is the reason for use of very light diesel fuels almost in the kerosene range, for some types of small motor vessels?

Answer. The shortage of heavy diesel fuels and a relative surplus of Otto fuels.

Question 14. What importance is attached to boiling range, and has any relationship been worked out between specific gravity, aniline point and boiling range, with cetane number? What importance is attached to aniline point?

Answer. Diesel fuels in the lower boiling range have a relatively good starting performance. A sufficiently accurate estimate of cetane number can be made, in the case of fuels of similar origin, from the specific gravity and boiling range. Aniline point estimation is thus no longer considered necessary and has not been examined during recent years.

Question 15. To what extent are diesel fuels from petroleum, brown coal, shale, and Fischer Tropsch processes used?

Answer. Suitable fractions from mineral oil, brown coal, shale and the Fischer Tropsch synthesis were all used.

Question 16. What is the significance of the phenol content present in some diesel oils?

Answer. Phenol is present in brown coal diesel fuel. The proportion allowed to remain caused no running troubles. Removal of the last traces would be tedious and would involve the use of special processes

8. FUEL OILS

Question 1. What types and to what specifications are used for

- (a) heavy industrial?
- (b) light industrial?
- (c) Marine?

2. What experimental work has been carried out on combustion of fuels under boilers?

3. What main types of fuel oil burners are used?

4. To what extent are fuels of non-petroleum origin used?

5. Are coal suspensions used to any extent?

6. Has any difficulty been experienced on the compatibility of the various fuels in the boiler at various loads?

7. Has any trouble been experienced on the formation of gases in fuel oils?

Answer. Respondents have no information on this subject.

QUESTIONNAIRE NO. 2

LUBRICANTS.

1. AVIATION.

Question 1. What general types of aviation oils are used, from what sources are they obtained, and why were these sources chosen?

Answer. No precise information is available.

Question 2. What refining methods are used for aviation oils? To what extent are solvent-refined oils incorporated in aviation oils?

Answer. No precise information is available.

Question 3. To what extent are synthetic oils used in the blending of aviation oils, and what advantage, if any, result?

Answer. Highly viscous synthetic products (V 100°C = 60E.) were blended 50/50 with a mineral oil component of low viscosity. The synthetic component gave a decided improvement in ring-sticking behaviour according to statements by RLM.

Question 10. What methods, if any, are used to determine Road Octane Numbers, and how do the results correlate with test engine data?

Answer. Measurements of road octane number have not been carried out in Germany in recent years.

Question 11. What bench tests are carried out on fuels and lubricants performance, and what type of operation (e.g. full power, continuous, or intermittent) is used in the tests with a test engine?

Knock rating tests were carried out with numerous auto-engines at the request of the BVA. They led to the introduction of the Motor O.N. for motor-fuels because of its better agreement with test-results. The accompanying report contains details of the various tests used at the station for fuels and lubricating oil. Continuous drive was generally preferred in the trials. Comparisons with actual running trials seemed too difficult and costly and were only carried out in very special cases, e.g. at the DST higher science test.

Question 12. How is wear in an engine measured?

Answer. Wear was measured by the loss in weight of the piston rings. Other methods, e.g. cylinder measurement and iron-content of the lubricating oil were not so accurate.

7. DIESEL FUELS (AUTOMOTIVE, AVIATION AND MARINE)

Question 1. What is the status of research on aircraft diesel engines?

Answer. No precise information is available.

Question 2. What specifications are laid down for aviation diesel fuels, and why?

Answer. Cetane number 50, pour point -35°C . (because of use at high altitudes). The other requirements were normal.

Question 3. What work is in hand on special fuels for aviation diesels?

Answer. An aviation diesel fuel corresponding to the above specifications was made by blending a naphthenic German gas oil (Reitbrook) with a low-boiling synthetic component made by RGH.

Question 4. From what components and how is K.1 fuel made?

Answer. No information is available.

Question 5. To what extent is the cetane number considered an adequate measure of ignition qualities? How are they measured? What are the cetane number requirements of average aviation, automotive, and marine diesel engines?

Answer. Recent developments have limited the application of Cetane number. In particular, the cold starting behaviour of fuels, having different boiling ranges and with additives to accelerate ignition, is often quite different from what the cetane number would indicate. The specified cetane numbers are:-

Motor engines	:	Ca 40
Aero "	:	ca 50
Marine "	:	unknown

Question 6. What work has been done on combustion and ignition accelerators, and to what extent are they used?

Answer. No special investigations have been made by Ruhrchemie. The effect of ignition accelerators on cold starting behaviour was small.

Question 7. What work has been done on cold starting aids, and what is the normal cold starting procedure?

Answer. Measures taken to improve cold starting behaviour varied according to the type of combustion process employed in the engine. Pre-heating of the inlet-air, cooling water and oil was employed as well as glow-plugs (Glühkerzen) and lubricating oil dilution.

Question 8. What filtering systems are used for diesel fuels?

Answer. Felt-filters to a large extent.

Question 9. How is low pour point obtained, especially in conjunction with high cetane number?

Answer. By blending as under (3)

Question 10. What general research has been carried out on combustion in diesel engines?

Answer. None has been done by Ruhrchemie.

6. MOTOR FUELS.

Question 1. What specifications were adhered to for military purposes, particularly in respect of knock rating, gum content, stability and vapour pressure, and what methods of test were applied. What changes in specification have occurred during the war, with dates of such changes?

Answer. The army specification was:- Research O.N. 74, later motor O.N. 72; permissible gum-content - 10 mg./ml. (evaporation method); Reid vapour pressure - 0.65 atm. (Summer), 0.75 atm. (Winter).

Question 2. Were any difficulties experienced when vehicles were operated on fuels with the higher lead concentration, such as the new tank fuel which was introduced towards the end of 1944 (2.5 - 2.75 ml./l.G.)? Was there any reason for eliminating entirely added aromatics from some of these blends? Was any change made either in composition or proportion of lead evaquants added to the Ethyl mix?

Answer. RCH has no experience in this connection.

Question 3. What were the compositions of the various types of automotive gasolines, and of the main basic components? What happened to the Fischer-Tropsch gasoline, and what was the source of the large quantities of unsaturates normally included in the blended gasolines?

Answer. The composition depended on the availability of the following blending components: hydrogenation benzine, synthetic benzine, lime and foreign mineral oil benzine, benzol, alcohol. No fixed blending proportions were maintained.

Question 4. What effect did the cessation of Roumanian supplies have on the general position for motor spirit, and to what extent was Roumanian gasoline used in motor and aviation fuels?

Answer. The loss of Roumanian benzine caused a big increase in the proportion of benzol used in fuels. Large quantities of straight run and cracked benzine blends were used as motor fuels with the addition of TEL or benzol. An asphalt base straight run benzine component was worked up into an aviation fuel.

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Question 5. Was the general idea to work to a given octane number in blending these fuels, using Benzol and/or TEL as the adjusting agents, reducing the latter when more Benzol was available?

Answer. Yes.

To what extent has Methanol and Ethanol been used as a blending agent for internal civilian consumption, for use in non-military vehicles in occupied territories, and for military purposes? What factors have led to the small use made of these blending agents for the latter purpose?

Ethanol production was severely restricted by food requirements. Since methanol was only used in conjunction with ethanol this limited the availability alcohol blends. Alcohol was used only for civilian purposes and for army vehicles on the home front.

Question 6. What quality of Aromatic blended component was used, i.e. was all the toluene extracted from the benzol and what happened to the higher aromatics?

Answer. Toluol was removed from motor-benzol by careful fractionation. The higher aromatics remained in the fuel.

Question 8. What disposal arrangements were made for Estonian shale gasoline, and was any difficulty experienced in its use as a motor gasoline constituent? What tests, if any, were made to establish its suitability as a flying bomb fuel, particularly from the point of view of corrosion?

Answer. No special regulations for Estonian shale gasoline are known. RCH has no information as to any difficulties attending its use as a motor gasoline constituent or its suitability as a flying-bomb-fuel.

Question 9. To what extent have alternative fuels such as producer gas, acetylene, etc. been used for civilian and military purposes? What technique has been employed with these fuels?

Answer. No precise information is available.

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Question 12. To what extent was "run-out" fuel used in an effort to combat cold corrosion?

Answer. No information is available.

3. ANTI-KNOCK ADDITIVES

Question 13. What factors decided the amount of TFL to be incorporated in aviation fuels?

No precise information is available. Probably exhaust valve corrosion and sparking plug trouble are deciding factors.

Question 14. What attempts have been made to TFL beyond the present amount?

Answer. No information is available.

What experience has been obtained on spark plug fouling, exhaust valve corrosion, etc? What effect has TFL on overhaul periods? Has any connection been found between type of oil, and cylinder head deposits?

Answer. No information is available as regards aero engines.

Question 15. What difficulties have been experienced with lead deposition in storage and what inhibitors, if any, were developed to cure this? Was any special test devised to evaluate lead stability?

Answer. No information is available at Ruhrchemie.

Question 16. What laboratory methods are used to determine lead content of a fuel?

Answer. Ruhrchemie use the ZB method due to Ullrich.

Question 17. What work has been carried out on alternative anti-knock agents?

Answer. No information is available.

Question 18. What work has been done on the development of new lead evacuants, and in what percentage of theoretical are they used?

Answer. Ruhrchemie has no experience in this matter. Experiments by the RIM and HWA using increased addition of ethylene

bromide or chloride were, as far as is known to RCH, unsuccessful in practice.

Question 19. What experience has been obtained with water injection, or other supplementary booster fuel?

Answer. No information is available.

Question 20. Has any trouble been experienced due to cold corrosion as a result of the use of leaded fuels, and what steps were taken to combat such effects?

Answer. No information is available.

4. DETONATION RESEARCH

Question 1. What work has been done, and is in hand, on the rationalisation of factors controlling detonation, and on the fundamentals of combustion, e.g. Muhlner experiments?

Answer. See the accompanying research report.

Question 2. What experimental work has been carried out on pure hydrocarbons?

Answer. None by Ruhrchemie.

Question 3. What work has been done to investigate pre-ignition, and on the development of pre-ignition ratings of fuels?

Answer. See the accompanying research report.

5. GAS TURBINE FUELS

Question 1. What specifications are laid down for (a) Gas Turbine Fuels, (b) Flying bomb fuels, (c) Rocket fuels?

Answer. No information is available.

Question 2. What special requirements are necessary for these fuels and how have these requirements been met?

Answer. No information is available.

Question 3. What developments are under way in connection with fuels for these types of engine?

Answer. No information is available.

Question 6. To what extent is the C.F.R. Research, or similar method used, and why?

Answer. This test is used as a preliminary evaluation of knock rating. Final evaluation was by the BMW single cylinder or other such tests.

Question 7. What degree of inter-correlation is obtained between small scale engines, single cylinder full scale aero engines, and multicylinder main engines?

Even aero engine single cylinders do not agree on full evaluation so that the agreement between small scale engines and aero engines is not good although it is considered satisfactory. We have no first hand knowledge of tests in multicylinder aero engines.

Question 8. What flight tests have been carried out to determine the antiknock requirements of C-3s?

Answer. No information available.

Question 9. What reference fuels are used (a) under 100 octane number, or corresponding rating?
(b) over 100 octane number?

Answer. (a) I.G. Test benzine and Technical iso-octane.
(b) I.G. Test benzine and Technical iso-octane - leaded.

2. GERMAN AVIATION FUELS.

Question 1. To what specifications are C-3, B-4, and A-3 fuels produced?

Answer. Not known.

Question 2. What is the reason for the high aromatic content of the German C-3 fuel, and for its excessive margin of rich mixture performance? For what planes is this type of fuel specified?

Answer. Of the fuels originally developed and made available in Germany, the aromatic type hydrogenation products from bituminous coal had the best anti-knock value, particularly at rich mixtures, consequently this type of fuel was adopted by the Luftwaffe.

Question 3. From what components are C-3 fuels blended at the blending points?

Answer. Not known.

Question 4. What trouble has been experienced with C-3 fuels containing small amounts of water?

Answer. Not known.

Question 5. What difficulties have been experienced with a high aromatic fuels? (Effect on synthetic rubber, etc.)

Answer. No exact knowledge available. Believed to have some behaviour at weak mixtures.

Question 6. How important is gum stability considered? What gum inhibitors are used?

Answer. What trouble has been experienced with vapour lock to excessive fuel volatility, and also to handling of air? How were these troubles overcome?

Question 7. To what extent are cracked fuels used for aviation purposes?

Question 8. What work has been done on the development of safety type fuels?

Answers. No information is available.

Question 10. What engine studies have been carried out on the effect of engine factors, such as valve overlap, etc., on fuel performance, particularly at weak mixtures?

Answer. The results of tests are contained in a lecture by Dr. Schaub entitled "The Supercharged test in the N.S.U. engine of the Ruhrbenzin A.G., Oberhausen-Holten" given at a D.V.L. meeting in June 1941. Apart from this we have no other experience.

Question 11. What general methods are used for cold starting, and to what extent were special priming fuels used, and of what components did these consist?

Answer. Easily vapourised fuel was used and also lubricating oil dilution. Engine preheating was used at very low temperatures.

Research reports: P125a, P133, P135,
P136, P139, P142,

12. A few experiments were carried out in connection with foaming. They showed the poor reproducibility of the results without indicating the explanation of this behaviour. None the less, differences between individual oils could be recognised. RCH-oils behaved well as regards foaming.

Research report: P150.

13. Gear Oils.

Work on the development of a synthetic RCH gear oil was in the preparatory stage. Some endurance tests for the examination of the heat stability of normal gear oils were carried out at a large gear testing station of the ZF firm, Friedrichshafen. Further, investigations were made using well-known lubricating oil testing machine (the Almen-machine and four ball apparatus). The development of a practical test in a motor-cycle back axle cone drive was also under way.

Investigation: 1944 (2nd half)
Research reports: P148, P149.
Requested by: O.K.H.

14. Various.

Mechanical methods for the improvement of cold starting with auto engines at very low temperatures were tested in the small cold chamber. Benzine injection was used in this work.

Under investigation from: 1943- 1944.
Research report: P137, P147.

(Signed) ... Schaub.

Date: 23.8.45.

In the above report the following abbreviations are employed:-

BMW	Bayerische Motoren-Werke.
DVL	Deutsche Versuchsanstalt für Luftfahrt, Berlin, Aldershof.
RLM	Reichsluftfahrtministerium.
HWA	Heereswaffenamt.
E-Stelle	Erprobungstelle.
V1	Viskositätsindex.
ATZ	Automobiltechnische Zeitschrift.
ZF Friedrichshafen.	Zahnradfabrik Friedrichshafen.
RCH	Ruhrchemie.

APPENDIX 3.

ANSWERS TO QUESTIONS BY HACHELANN AND

SCHAUB DATED 23.8.45.

AVIATION FUELS.

Fuel Rating Methods

- (a) small scale engines
- (b) full scale aero engines?
- (c) multicylinder engines.

Ruhrchemie used only small scale engines for testing aviation fuels (See accompanying report on work of the RCH research station). These engines have the advantages of cheapness and simplicity and in addition only require small test samples. Agreement with the official BMW 132 single cylinder engine was satisfactory, especially when bearing in mind the differences which exist between different aero engines of the same size in fuel evaluation.

- Question 2. What relative importance is attached to rich mixture and weak mixture performance?
3. How is rich mixture performance measured?
4. How is weak mixture performance measured?

Answers. The use of rich mixtures is of especial importance with the aromatic aviation fuels common in Germany for they gave a much better performance with rich mixtures than with weak mixtures. The use of weak mixtures is important in reducing fuel consumptions. Fuel behaviour at rich and weak mixtures is measured by determining a performance curve.

- Question 5. What work is in hand for the development of fuels having improved weak mixture characteristics?

Answer. Ruhrchemie always stressed the importance of iso-paraffinic fuels because of their favourable behaviour at weak mixtures. We therefore aimed at the manufacture of such a fuel by the Fischer-Tropsch synthesis and some success was achieved as instanced by catalytic cracking of Fischer-Tropsch products and polymerisation and hydrogenation of C₄ and C₅ fractions.

Under investigation during: 1939 - 1944.
Research reports: P106, P107, P108,
P109, P111, P119.

The effect of the addition of RCH synthetic
oil to mineral oil was investigated.

Investigation: 1941, P110, P117.

The effect of the addition of RCH synthetic
oil to mineral oil was investigated in connection with the question
of the effect of the addition of RCH synthetic oil to mineral oil
on the cold starting behaviour of the engine. The comparison with residual oils
of different origin (RCH synthetic). The results of the investigation
showed that the addition of RCH synthetic oil to mineral oil does not have any fundamental
effect on the cold starting behaviour of the engine. The results of the investigation
showed that the addition of RCH synthetic oil to mineral oil does not have any fundamental
effect on the cold starting behaviour of the engine.

Investigation: 1944.
Research reports: P110

The Behaviour of Oils from Various Sources in the RCH Piston Seizure Test (see above)

The synthetic RCH lubricating oils behaved remarkably well, even better than fatted mineral aviation oils. Viscosity had no apparent influence, and in any case it could not be large. The usual mineral motor oils lie close together in a comparatively small range and their quality is adequate, except in high rating two stroke engines.

Research reports: P110, P112, P115,
P117, P122, P129.

5. General research was carried out on the relation between wear and oil consumption on the one hand and viscosity on the other hand. This was done to ascertain what disadvantages might arise on lowering the viscosity to improve cold starting behaviour. As expected, it was shown that viscosity influenced consumption but not wear, which appeared to be practically independent of it.

Investigations: 1942.
Research reports: P126, P127.
Requested by: HMA.

6. The action of Oppanol was examined in connection with wear, oil consumption and cold behaviour (effect of viscosity index). The use of Oppanol is advantageous, at least with oil which is not too strongly used.

Investigation: Inter: 1942/1943.
Research reports: P130, P131.
Requested by: HMA.

7. The effect of lubricating oil dilution was investigated, also in connection with cold behaviour. Investigations were made under practical running conditions at the testing station. It was demonstrated that the use of motor fuels need not cause leakage, because they rapidly volatilise, while the use of diesel oil can be dangerous in certain circumstances. Oil dilution is not favourable for the piston seizure.

Investigation: 1942 (first half)
Research reports: P130, P131.
Requested by: HMA.

8. Extensive work was done on the cold behaviour of oils from various sources (Rumanian oil, oil with added Oppanol). The cold chamber was used and at the same time an attempt was made to find a connection between practical results and laboratory tests (Schwinger viscometer). The straying of results at low temperatures (near the setting pt.) was too great to allow definite conclusions to be drawn.

Under investigation during: 1943 - 1944.
Research reports: P130, P141, P143.
Requested by: HMA.

9. Fundamental research on the low temperature pumping behaviour of oils from various sources, was carried out in the apparatus developed by RCH and described above.

Investigation: 1943.
Research report: P141.

10. A complete record of work on engine-wear done at the station over a period of years is collected in Research Report P151.

11. At the request of the OKM, various oils were examined as to their general behaviour. The origin of these oils is unknown to us.

As far as comparison is possible, there is no fundamental difference from the DVL results (experiments of Krieger on a 1000 cc engine).

Under investigation from: 1942-1944.
Research reports: P105, P106, P107,
P108, P118, P119,
P121, P122, P123,
P124

(v) Piston Seizure.

This test was developed using a high speed two stroke engine (Triumph BD 250 ml. air cooled). Evaluation followed from an intermediate series of results where the unknown oil was compared with certain standard oils. Comparison of different oils gave good agreement and the tests carried out by another station for this test is also comparatively reproducible by experienced personnel.

Under investigation from: 1940 - 1942.
Research reports: P110 May 1941,
P115 Jan 1942
Requested by: RWA.

(v) Oil Consumption.

Measurement of oil consumption was combined with the ageing and wear tests by weighing the lubricant used during the test.

(vi) Cold Behaviour.

(a) Cranking Resistance. For cold tests a large cold chamber (base area about 6 x 9 m) and a small one (base area about 2 x 3 m) were erected. Cooling was brought about by a constant blast of cold air (-55°C) from a Linde plant. The small chamber was especially suitable for engine tests. It was possible to cool an engine from room temperature to -40° in 5 hours. Engine behaviour could be observed from outside (measurement of r.p.m. engine temperatures actuation of the ignition adjustment and of the injection-quantity etc.). The engine could be started from outside by dynamometer. The insulating cover consisted of a bell shaped box, which could easily be removed or put into place during engine assembly.

Under investigation from: Nov. '42 - Summer '43.
Research reports: P137 June 1943.

(b) Pumping Behaviour. To examine this a special installation was constructed. The delivery rate, delivery pressure, suction and other quantities of interest were observed for an oil-pump driven at constant r.p.m. Observations could be made at different temperatures (down to -40°C).

Investigation: 1943
Research reports: P141.
Requested by: RWA.

The following problems were investigated using the methods described above:

1. The testing and development of a synthetic aviation engine oil possessing good ageing and cold behaviour as well as the outstanding performance of synthetic oils with respect to ring-sticking wear, consumption and piston-seizure. In the course of this work an oil was made which possessed the following properties. These results agreed with those of R. and Traventude (BMW 130 cc engine and Callisto Argus engine):

Ring-sticking behaviour was more favourable than with well-known mineral or fatted oils (Rotring, ASM) and as good as with the synthetic aero oils used in Germany. Wear behaviour was better than with the mineral reference oil (Rotring) and as good as with the voltolised ASM aviation oil. As regards piston seizure, this RCH synthetic lubricating oil was better than any other product tested. The setting point was below -40°C. Good cold behaviour (cranking resistance and "pumpability") was confirmed by trials at Rechlin. The original weak point of synthetic oils, namely, a tendency to thicken during running was so far overcome that, during the RLM ring-sticking tests, absolutely no increase in viscosity was observed, in spite of the length of run. The latter was very long because of the good ring-sticking behaviour (tests at Rechlin and Traventude). As regards sludge formation and engine-deposits, the oil also showed outstanding qualities.

Between the years 1939 and 1944, the pole-height was decreased from about 1.8 to 1.5 (1.52). At the same time there was a general tendency of the viscosity (at 50°) to decrease (from 22°E to 10°E).

Complete agreement, however, was shown to be too much
correct.

Under investigation during: 1939-1941
(development of the
lubricating installation)

P102 Ser. 1940)
P113 " 1941)

The development of this installation permitted
the examination and further development of synthetic
aviation fuels obtained by catalytic cracking and
polymerization of Fischer Tropsch products. Fuels were
obtained with a performance curve of the technical type
of one type fuel with little difference in performance
in the work mixture.

Diesel Fuels.

The station co-operated in work on the improvement
in cetane number measurement methods with the HWA engine
(throttle method). Work on the special action of
ignitable Ruhrchemie diesel fuel on diesel engine
combustion did not give any special results.

D. Lubricating Oils - Engine Lubricating Oil.

The development of suitable engine test methods
was a prerequisite for further development in the quality
of Ruhrchemie synthetic tubes. In order to give as
comprehensive a picture as possible, lubricating oil
testing covered the following points:

- (i) Ageing
 - (a) Increase in viscosity.
 - (b) Other chemical changes.
 - (c) Sludge formation.
- (ii) Ring sticking.
- (iii) wear and lubricating properties.
- (iv) Piston seizure.
- (v) Oil consumption.
- (vi) Cold behaviour.
 - (a) Cranking resistance.
 - (b) Pumping behaviour.

* A lecture by Dr. Schaub at the DVL meeting of June 1941
"The Performance Test in the NSU Engine of the RCH-
Oberhausen-Höltzen".

(i) Oil Testing.

To decrease the expense and time involved
and also because of the unavoidable straying in the
results of individual runs, it was decided to work with
short runs in small scale engines. Thus the ageing
behaviour was examined in a ten hour run with a NSU-501-
OSL engine (air-cooled) at high and constant temperature.
The thermal demands made on the oil were higher in this
run than with the RLM ring sticking test in the
run as can be seen from the following table:

Under investigation	1939-1941
Research report	P105, P106, P107, P108, P109, P110, P111, P112, P113
Conducted by:	H. Schaub

Chemical Change and Sludge Formation.

These were examined as under (i). The
quantity of sludge was observed by centrifuging out i to
the crank-web. The repeatability of result was not
There are no comparison tests with full size engines
apart from those with the RCH synthetic oil.

(ii) Ring-sticking Tests.

A test corresponding to that with the
BMW engine was developed for the NSU-501-OSL engine.
Although straying was still considerable, sufficiently
good agreement was achieved with the BMW engine for our
purpose. This is confirmed by results from different
Luftwaffe testing stations (Rechlin & Travemünde).

(iii) Wear Test.

This test was combined with the ageing and
sludge tests in a short 10 hour run. Only after a large number
of trials was it possible to get more or less
reproducible results from which conclusions could be
drawn as to lubricating oil behaviour. Wear was
estimated by weighing the piston rings. It was measured
with run-in as well as new rings. With the latter,
the absolute value is many times higher, as might be
expected. The evaluation of the oil is the same, however.
Experiments with a separately driven engine gave only
a slightly lower wear than with a machine under load.

APPENDIX 2.

Part-time Activities of the Ruhrchemie Engine Laboratories at Sterkrade-Holtien.

Report by Dr. WALTER KESSEL

Auto-Engine Fuels

1. Knock Rating.

The knock properties of Fischer Tropsch product were continually under examination. The station took part in collective work on the improvement of the precision and repeatability of knock rating methods with the I.G. and C.F.R. engines. Special investigations of the varying behaviour of synthetic benzine in I.G. and C.F.R. engines was instituted after the engine method had been adopted. This work has not been completed. It has been shown, however, that synthetic benzine is less sensitive to change in the ignition conditions than the reference fuels.

Under investigation during: 1939 - 1944
Research Reports: P119, P120, P121, P122, P123, P124, P125, P126, P127, P128, P129, P130, P131, P132, P133, P134, P135, P136, P137, P138, P139, P140, P141, P142, P143, P144, P145, P146, P147, P148, P149, P150, P151, P152, P153, P154, P155, P156, P157, P158, P159, P160, P161, P162, P163, P164, P165, P166, P167, P168, P169, P170, P171, P172, P173, P174, P175, P176, P177, P178, P179, P180, P181, P182, P183, P184, P185, P186, P187, P188, P189, P190, P191, P192, P193, P194, P195, P196, P197, P198, P199, P200, P201, P202, P203, P204, P205, P206, P207, P208, P209, P210, P211, P212, P213, P214, P215, P216, P217, P218, P219, P220, P221, P222, P223, P224, P225, P226, P227, P228, P229, P230, P231, P232, P233, P234, P235, P236, P237, P238, P239, P240, P241, P242, P243, P244, P245, P246, P247, P248, P249, P250, P251, P252, P253, P254, P255, P256, P257, P258, P259, P260, P261, P262, P263, P264, P265, P266, P267, P268, P269, P270, P271, P272, P273, P274, P275, P276, P277, P278, P279, P280, P281, P282, P283, P284, P285, P286, P287, P288, P289, P290, P291, P292, P293, P294, P295, P296, P297, P298, P299, P300, P301, P302, P303, P304, P305, P306, P307, P308, P309, P310, P311, P312, P313, P314, P315, P316, P317, P318, P319, P320, P321, P322, P323, P324, P325, P326, P327, P328, P329, P330, P331, P332, P333, P334, P335, P336, P337, P338, P339, P340, P341, P342, P343, P344, P345, P346, P347, P348, P349, P350, P351, P352, P353, P354, P355, P356, P357, P358, P359, P360, P361, P362, P363, P364, P365, P366, P367, P368, P369, P370, P371, P372, P373, P374, P375, P376, P377, P378, P379, P380, P381, P382, P383, P384, P385, P386, P387, P388, P389, P390, P391, P392, P393, P394, P395, P396, P397, P398, P399, P400, P401, P402, P403, P404, P405, P406, P407, P408, P409, P410, P411, P412, P413, P414, P415, P416, P417, P418, P419, P420, P421, P422, P423, P424, P425, P426, P427, P428, P429, P430, P431, P432, P433, P434, P435, P436, P437, P438, P439, P440, P441, P442, P443, P444, P445, P446, P447, 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2. Vapor Locking.

Little is known about the relation between the formation of bubbles of vapor (vapor locking), under actual running conditions and the usual laboratory test methods (Reid Vapor Pressure and 15% evaporated temperature). Research was, therefore, carried out at the station on vapor locking using commercial automobile engines under various working conditions. Based on this work, apparatus was evolved which duplicated practical conditions as exactly as possible. It is described in MTZ, 1941, Number 22. In the same connection, various engine factors were examined, e.g., fuel consumption, delivery-pump performance, etc. The research showed that fuel behaviour cannot be wholly necessary to construct a curve corresponding to the various working conditions. Under certain conditions a satisfactory enough agreement with the earlier evaluations can be obtained, i.e., according to the Reid V.P. or to a point on the boiling range curve. These comparison trials are not yet concluded.

Under investigation during: 1939 - 1944
Research Reports: P101 & P157

3. The Influence of Fuel Density on the Adjustment of the Fuel-Air Mixture in the Carburettor.

Brief trials were made bearing on the influence of fuel density on the adjustment of the fuel-air mixture in the carburettor. The results showed that the adjustment of the fuel-air mixture is not very sensitive to changes in fuel density. The influence of fuel density on the adjustment of the fuel-air mixture is not very sensitive to changes in fuel density.

Under investigation in: 1940
Research report: P104.

The Influence of Tetra Ethyl Lead on the Ageing of the Fuel-Air Mixture during Storage.

This was examined in a small number of trials. The results showed that lead compounds have a beneficial influence on the ageing of the fuel-air mixture during storage. The influence of tetra ethyl lead on the ageing of the fuel-air mixture during storage is beneficial.

The Influence of Boiling Range on Fuel Performance.

At the request of the D.V.L. was carried out to determine whether raising the F.B.P. would entail any considerable disadvantages as regards wear and oil-dilution. The work is still incomplete. Results up to now show no appreciable differences on increasing the F.B.P. from about 180° to 215°.

Under investigation in: 1944.
Research report: P158 of 26/11/44.

B. Aviation Fuels.

For the examination of synthetic fuels a small scale engine installation was developed. An MSU four-stroke engine of 500 ml. swept volume was used. Mixture formation was by injection of the benzine into the inlet manifold. It was attempted to get an evaluation corresponding to that with the BMW 132 N-engine of the RLM, as regards performance, using the smallest possible quantity of fuel. Numerous comparison trials with the DVL and the I.G. Oppau Technical Station showed that an adequate agreement had in fact been attained.

RHEINANIA - OSSAG
(continued)

- 137.7. Vorversuche für Pumpfähigkeit von Getriebeölen in der Kälte. Zogbaum & Weberitz 30.4.43.
- Prüfung von Getriebeölen der Wehrmacht auf Pumpfähigkeit in der Kälte. Zogbaum
- Richtung der abgeänderten Pumpapparatur und Anlagung einer Kältepumpe. Zogbaum & Zander
- Prüfung der als Eichöl zur Verfügung gestellten Getriebeöle der Wehrmacht auf Pumpfähigkeit. Zogbaum
- Vergleich der Pumpapparatur alter und neuer Bauart.

I.G.FARBEN, OPPAU.

- Der I.G. Prüfzylinder zur Messung der Pumpfähigkeit von Kraftstoffen.
- Entstehung und Vermeidungszweck des Versagensmotors K.
135. - Drawings from I.G. Oppau Rept. No. 487. - -
134. - " " " " " " 542. - -
133. - " " " " " " 518. - -
- 136.474. Geheime Ein Beitrag zur Prüfung des Klopfverhaltens von Flugkraftstoffen in Kleinmotor. Witschakowski. 25.8.41.
- 137.489. Die Durchführung von Oktanzahlbestimmungen nach dem Oppauer Verfahren. Singer. 22.1.42.
- JUNKERS(?)
- 138.2037. Einfluss von Schmierstoff und Kraftstoff auf Rückstandsbildung und Feuerringverschleiss (im Einzylinder-Diesel-Motor Jume 205). Sauermilch. 12.5.43.

DAIMLER-BENZ.

139. - Durchdrehversuche mit neuen Winterölen der Wehrmacht. Hohensee. 21.9.42.

DAIMLER-BENZ
(Continued)

140. Durchdrehversuche mit neuen Winterölen der Wehrmacht bei -20°C.

Nichtlinien für Durchdrehversuche der Wehrmacht.

ADOLF OETL A.G.

Kaltstart Versuche mit Einheitsöl der Wehrmacht (Mittelklasse, Motoröl, Norm. 526).

Kaltstart Versuche mit Einheitsöl der Wehrmacht (Hochdruck, Motoröl, Norm. 526).

Kaltstart Versuche mit Einheitsöl der Wehrmacht (Hochdruck, Motoröl, Norm. 526).

REINIGUNG BORSIG.

Technischer Bericht über die Reinigung von Dieselmotoren.

MISCELLANEOUS REPORTS.

146. - Niederschrift über die Sitzung des Arbeitsausschusses "Klopfmessungen im I.G. - und C.F.R. Motor". - 23. 6.44.
147. - Sonderausschuss zur Normung der motorischen Prüfung von Dieselmotoren beim DVM. - 22. 9.42.
148. - Einheitliches Verfahren für Dieselmotorenstoffe. - 18. 1.41.
149. - Heizölbeschaffenheit. - 16. 9.38.
150. - Technischer Bericht zur Normung der motorischen Prüfung von Dieselmotoren (Klockner-Humboldt-Deutz). - 11. 4.42.
151. - Arbeitsvorschrift zur Bestimmung der Pumpfähigkeit von Heizölen. - -
152. - Gebrauchsanweisung für den Kathoden-Doppelstrahl-Ossillographen (Quarz-Indikator). Nier. - -

DEUTSCHE KRAFTFAHRT FORSCHUNG
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106 Heft 7a. Zündverzögerung und Bewertung des Kraftstoff-
Zündverzugsmessungen an Diesel- und
Ottokraftstoffen. Ernst. 1944

Einfluss der Luftbewegung auf die
Ausbildung des Kälteeffektes
der Zylinderkammer.

REPORTS BY THE RESEARCH INSTITUTES

Verfahren zur Getauz...
Diesel... Stoffe.

Geheim. Verhalten...
bei...
Hochdruck.

Verfahren zur Getauz...
Diesel... Stoffe.

Schmierstoffförderung im Motor bei
Kälte bei ständigen Flugmotorenölen.

Getauzbestimmung von Dieselkraftstoff...
(mit... nach Dr. Neumann)

REPORTS BY THE PHYSIKALISCH-TECHNISCHEN REICHSANSTALT.

- 107 - Entwicklung und Prüfung eines
Kälteviskosimeters. Willenberg. 1944
DVL REPORTS.
- 108 - Vorschläge für die Beschlussfassung. Phillipovich. 13.5.44.
- 109 - Aussprache über Kraftstoffprobleme der
Luftfahrt. 17.6.41.

REPORTS FROM TECHNISCHE HOCHSCHULE.

- 110 - Kraftstoffbewertung hinsichtlich
Dampfblasenstörung (Dresden) Hager &
v. Eberan. 30.3.44.
- 111 - Gemischbildung im Otto-Motor beim Anlassen, Kraftstoff-Dampfspannung und Anlass-
verfahren bei Tieftemperaturen (Dresden) Werminghoff,
& Hans. 1.12.43 v. Schieszl
- 112 - Versuche zur Bestimmung der Bruchbestandig-
keit von Schmierölen (Doktor-Arbeit: Berlin) de Jong. -
- 113 - Untersuchung der Schmierfähigkeit von
Ölen (München) Kédmer. May 1944

REPORTS FROM TECHNISCHE HOCHSCHULE
(continued)

114. - Zahnradversuche bei Oelsumpftemperaturen
von 150°C. (Stuttgart) Welling. 5.8.44

Untersuchungen zur Entwicklung der
Selbstzündung betriebs in gemisch
betriebsbetriebs (Stuttgart).

Versuche über die Hitzebeständigkeit der
Schmiermittel unter Einbeziehung
von...
Welling.

REPORTS BY OTHER COMMERCIAL COMPANIES.

DEUTSCHE ZÄHLEWERKE.

Herstellung der...
Leit...

Entwicklung von Versuchseinrichtungen
und Verfahren zur Bestimmung des
Kälteverhaltens von...
13.3.44

Untersuchung zur Hitzebeständigkeit
von Schmiermitteln...
13.3.44

120. VR542a. Hochdruck Öle (Hypoidöle für Hoch-
belastete Antriebe). Richter,
Paul & Ullrich. 14.3.44

ITAVA

121. 10. Prüfung von Flugmotorölen in DKM-Motor. Wenzel. 20.3.41.

122. 30. Auswertung der Versuche im BMW-Ölprüf-
motor hinsichtlich Ölkohlebildung. Wenzel. 30.9.43

RHENANIA - OSSAG

123 - Pumpapparatur zur Kennzeichnung des Kälte-
verhaltens von Motoren- und Getriebeölen. Rossig. 25.4.44

124 3 Untersuchung von Getriebeölen auf Pump-
fähigkeit in der Kälte. Zogbaum
& Deberitz. 1.7.42

125 5 Umpumpversuche in der Kälte (Getriebeöl
der Wehrmacht-Winter). Hofmann. 25.9.42

BUHNCHEMIE REPORTS
(continued)

Erprobung verschiedener Hochdruckzusätze zu normalen synthetischen Kraftstoffen mit Unterversuch über verändertes Verhalten	Schaub	2.11.41
Versuche zur Verwertung von Dieselkraftstoffen	Schaub	16.11.41
Versuche zur Verwertung von Dieselkraftstoffen	Schaub	
DEUTSCHE KRAFTFAHRT FORSCHUNG		
herausgegeben von der Kraftfahrtechnischen Kommission	Schubert	
Motoren und Kraftstoffe		
herausgegeben von der Kraftfahrtechnischen Kommission		
Stand der Forschung über Gemischbildung		
Versuche zur Verwertung von Dieselkraftstoffen	Dreyhaupt	
75.94/1941. Bombenversuche über Gemischbildung und Verbrennung bei Benzineinspritzung (T.H.Graz).	Bianchi.	
76. 58. Klopfvorgänge an Motorzylindern (T.H.München)	Schmidt & Regel.	
77.99/1941. Versuche an einem Vergasermotor mit Selbstzündung (T.H.Stuttgart).	Ernst & Dorr.	
78. 74. Versuche über das motorische Verhalten synthetischer Ott-Kraftstoffe (T.H.Stuttgart).	Kamm.	
79. 54. Motorisches Verfahren zur Prüfung von Dieselmotoren (T.H.Stuttgart).	Ernst & Gross.	
80. 86. Der Stand der Zweitaktforschung.	(Various)	6. 6.40.
81. 91. Gemischbildung und Verbrennung (Diesel).	"	1.10.40.
82.103/1941. 2. Tagung des Arbeitskreises für Zweitaktfragen.	"	20. 5.41.
83.111/1941. 2. Tagung des Arbeitskreises für Fragen der Motorischen Verbrennung.	"	10.10.41.
84.Heft 3. Leistung und Wirtschaftlichkeit gasgetriebener Fahrzeugmotoren.	Rixmann.	1938

DEUTSCHE KRAFTFAHRT FORSCHUNG
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4. Frischschmierung beim Pleuel-Gleitlager (Huber & Eiberger)	Huber & Eiberger	
Untersuchungen an Fahrzeugdieselmotoren.		
Untersuchung der Ausstrahlung des Verbrennungsraumes schnelllaufender Diesel- und Ottmotoren	Pietsch	
86.Heft 5. Beitrag zur Erforschung des Verbrennungsvorganges im schnelllaufenden Dieselmotor	Knecht	1938
87.Heft 29. Zylinder und Kolbenringverschleiß	Reck.	1939
88.Heft 31. Messung der Klopfestigkeit an Otto Motoren.	Schütz.	1939
89.Heft 33. Untersuchung der Klopfgeräusche von Ottomotoren mit elektroakustischen Messgeräten.	Schmidt & Dorr.	
90.Heft 34. Die mechanischen Verluste des schnelllaufenden Dieselmotors und ihre Ermittlung mit dem Schleppversuch.	Wilmann.	1939
91.Heft 52. Vergleichende Untersuchungen an Lagergehäusenwerkstoffen.	Heidebrock & Döring.	1941
92.Heft 53. Das Aufspritzen des Kraftstoffes im Dieselmotor. Zündversugemessung mittels Photosellen in verschiedenen Wellengebieten.	Blume.	1941
93.Heft 54. Nachweis der Schmierfilmdurchbrechung durch Messen des elektrischen Übergangswiderstandes zwischen Kolbenring und Zylinder.	Poppinga.	1941
94.Heft 55. Kraftstoff und Motor beim Anlassen von Fahrzeugdieselmotoren.	Rixmann, Schaub & Conrad.	1941
95.Heft 57. Kraftstoffaufbereitung durch die Einspritzdüse.	Oschatz.	1941
96.Heft 59. Das Schmiermittel im Zahnradgetriebe unter besonderer Berücksichtigung der Grenzreibung.	Pietsch.	1941
97.Heft 60. Der Betrieb gemisch-Gespülter Zweitaktmotoren mit Flüssiggas.	Schmidt.	1941
98.Heft 61. Der Spülvorgang auf Grund einer neuen Auffassung der Expansionsströmung.	Schultz-Grunow & Weighardt.	1941
99.Heft 62. Aufladbergang von Viertakt Dieselmotoren der Spülvorgang.	Riedel.	1941

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33. F121.	Zusarbeitung eines Verfahrens zur Prüfung von Motorenölen hinsichtlich Ringstöcken, Alterung und Verschleiss.	Schaub	9.2.42
	Das Verhalten verschiedener handelsüblicher Flug- und Kraftwagenmotorenöl in Bezug auf das Kolbenfressen.	Schaub	13.6.42.
34. F122.	Verdünnung der Motorenöle für Winterbetrieb und Einfluss auf das Kolbenfressen.	Schaub	13.7.42
35. F123.	Flugölmischung E1951 aus RCH-Brightstock und einer niedrig viskosen mineralischen Komponente.	Schaub	10.9.42
36. F124.	Erprobung des Versuchsöles der MWA 337 (M96 Rest v. N.1 der I.G.).	Schaub	11.2.42
37. F125.	Vorbericht Motorenöl Nr. 3370 (M96 Esteröl).	Schaub	1.7.42
38. F126.	Analytische Bearbeitung eines Motorenöles der I.G. Farben.	Reitig	28.8.42
39. F125a.	Erprobung des Motorenöles 3370.	Schaub	8.4.43.
40. F126.	Der Einfluss der Zähigkeit auf den Ölverbrauch.	Schaub	19.9.42.
41. F127.	Über die Schmierwirkung von Dünflussigen Motorenölen (Winteröle).	Schaub	21.9.42.
42. F128.	Flugöl 3344 von der Grossanlage.	Schaub	6.10.42.
43. F129.	Flugöl 1979 von Hauptlabor.	Schaub	4.1.43.
44. F130.	Die Wirkung von Oppanolzusatz auf das Verhalten von Motorenöl.	Schaub	27.2.43.
45. F131.	Der Einfluss von Oppanolzusatz auf den Verschleiss.	Schaub	11.3.43.
46. F132.	Flugölmischung K1951/2 aus inhibiertem RCH-Brightstock und Neragöl.	Schaub	8.4.43.
47. F133.	Untersuchung des Motorenöles 3698.	Schaub	12.4.43.
48. F134.	Untersuchung des Motorenöles der Nerag (N.1)	Schaub	12.4.43.
49. F135.	Untersuchung eines normalen Wehrmacht-Winteröles der Nerag (RCH-Kurzbezeichnung: N.2)	Schaub	4.5.43

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51. F136.	Untersuchung von 3 Winterölen der I.G. Farbenindustrie A.G. Ludwigshafen Ma46, Ma48 und Ma49	Schaub	5.5.43
52. F137.	Anlassen von Otto Motoren bei tiefen Temperaturen.	Schaub	29.6.43.
53. F138.	Untersuchung des Motorenöles Nr. 3370 von Versuchslabor RCH.	Schaub	6.7.43
54. F139.	Prüfung des Motorenöles 3370 in Bezug auf Kolbenfressen.	Schaub	7.9.43
55. F140.	Die Klopfmessung von Synthes-Benzinen Abhängig von der Zündstellung.	Schaub	1.11.43
56. F141.	Über das Pumpverhalten von Schmierstoffen bei tiefen Temperaturen.	Schaub	29.11.43.
57. F142.	Durchdrehversuche mit Rumaniensöl.	Schaub	29.12.43
58. F143.	Prüfung eines Flugmotoren-Schmierstoffes SS1060 auf Kolbenfressen.	Schaub	7.3.44.
59. F144.	Die Schaumbildung bei Schmierölen.	Schaub	17.3.44.
60. F145.	Prüfung der Flugölprobe K2015 der Molaj.	Schaub	17.6.44.
61. F146.	Anlassen von Otto-Motoren bei tiefen Temperaturen 2. Zwischenbericht.	Schaub	27.6.44.
62. F147.	Prüfung von Getriebeölen auf Hitzebeständigkeit.	Schaub	29.8.44.
63. F148.	Vorversuche im Vierkugelapparat.	Becker & Schaub	26.9.44.
64. F149.	Kalteprüfung von Getriebeölen im Schaltgetriebe AK 7-200.	Schaub	29.9.44.
65. F150.	Die Beurteilung von Schmierstoffen nach dem Verschleiss im Motor.	Schaub	15.11.44. /
66. F151.	Alterungsverhalten der Flugölmischung K2025 (mit RCH-Brightstock, nicht inhibiert).	Schaub	19.10.44.
67. F152.	Vergleich von synth.Rückstans- und Destillatöl gleicher Zähigkeit zur Klärung der Wirkung des Brightstock-anteils.	Schaub	21.10.44.

ORIGINAL GERMAN TITLES OF DOCUMENTS EVACUATED

FROM THE RESIDENCE OF DR. SCHAUB OF RUHRCHEMIE, A.G.

	Bericht über Messergebnisse der Ruhrbenzin am HWA-Prüfmotor.	Schaub	7.11.40
	Bericht über Versuche zur Klärung von Umsetzungen bei der Oktanzahl-Bestimmung von PK-Kraftstoffen.	Schaub	11.11.38
	Nachtrag zum Bericht über die Versuche mit PK von 10.8.39.		
	Zwischenbericht über die bisherigen Versuche am Prüfmotor des Forschungsinstitutes für Kraftfahrwesen und Flugzeugmotoren Stuttgart.	Schaub	6.2.40
	Zwischenbericht Nr. 2. über die Versuche am Prüfmotor.	Schaub	6.5.40
	Bericht über Versuche mit Mischungen von Öl und Benzin.	Schaub	1.10.40
	Zwischenbericht Nr. 3. Die Bedeutung der motorischen Bedingungen bei der Erprobung von Schmierölen.	Schaub	19.1.40.
6. P101	Ein Gerät zur Messung der Dampfblasenbildung von Benzin.	Velde Schaub	20.6.40.
7. P102.	Überladeprüfung am NSU 501 OSL - Motor.	Schaub	5.9.40.
10. P103.	Vergleich von SS-Stoff mit anderen Diesellohlen in Bezug auf Düsenverkoken.	Schaub	5.10.40.
11. P104.	Versuche mit Kraftstoffen verschiedener Dichte.	Schaub	9.12.40.
12. P105.	Die Entwicklung eines motorischen Prüfverfahrens für Flugmotorschmieröle im NSU 501 OSL-Motor.	Schaub	14.12.40.
13. P106.	Motorische Prüfung einiger Flugöle im NSU-Flugol-Prüfmotor.	Schaub	20.12.40.
14. P107.	Motorische Prüfung synthetischer Flugöle mit niedriger Polhöhe im NSU - Motor.	Schaub	24.12.40.
15. P108.	Geheim. Bericht über die motorische Prüfung synthetischer Flugmotorschmieröle der Ruhrchemie.	Schaub	10.1.41.

16.	Zur Beurteilung von Kraftstoffen hinsichtlich der Dampfblasenstörung am Motor.	Schaub & Velde	19.2.41.
17. P109.	Bericht über die motorische Prüfung des synthetischen Flugöls "Flugol".	Schaub	1.11.41.
18. P110.	Die Entwicklung einer motorischen Prüfung für Schmieröle hinsichtlich der Dampfblasenbildung.	Schaub	6.11.41.
19. P111.	Geheim. Bericht über die motorische Prüfung des synthetischen Flugöls "Flugol".	Schaub	1.11.41.
20. P112.	Prüfung verschiedener Einheitsöle Wehrmacht im Triumph Motor hinsichtlich der Dampfblasenbildung.	Schaub	1.11.41.
21. P113.	Überlade-Vergleichversuche am NSU-Motor der Ruhrchemie A.G. und im BMW 131 Prüfmotor der Ruhrchemie A.G.	Schaub	1.11.41.
22. P114.	Auszug aus einem Bericht der Ruhrchemie A.G. über die Versuche mit "Flugol".	Schaub	1.11.41.
23. P115.	Motorische Prüfung synthetischer Öle zur Beobachtung des Einflusses der Viskosität und verschiedener Zusätze auf das Verhalten beim Kolbenfressen.	Schaub	12.1.42.
25. P115.	Entwicklung eines Verfahrens zur Prüfung von Motorenölen hinsichtlich des Kolbenfressens.	Schaub	15.1.42.
26. P117.	Prüfung von 4 Motorenölen auf Kolbenfressen.	Schaub	29.1.42.
27. P118.	Zündwilligkeit von RCH-Cetan.	Schaub	30.3.42.
28. P119.	Geheim. Ölprüfung am NSU-Motor.	Schaub	25.4.42.
29. P120.	Verhännung des Motorenöles für Winterbetrieb.	Schaub	22.5.42.
<u>RUHRCHEMIE REPORTS.</u>			
30.	Bericht über die Schmierfähigkeitsuntersuchungen (Lediney)	Lediney	3.8.38.
31.	Humboldt - Deutzmotoren-Versuche.	?	20.1.39.
32.	Zur Frage der Bestimmung des Asphaltgehaltes in gealterten Schmierölen.	?	30.4.42.

CIOS NO.	REPT. NO.	TITLE.	AUTHOR(S)	DATE
		<u>I.G.FARBEN, OPPAU.</u>		
		SECRET. A contribution to the Testing of Knock Behaviour of Aviation Fuels in Small Engines.	Shaker	25.8.41
		The carrying out of Octane Number Determination according to the Oppau Method.		
		<u>JUNKERS (?)</u>		
		Influence of Lubricant and Fuel on Deposit Formation and Gas Ring Wear (in a 4-cylinder Diesel Engine, Jun 2045).	General	12.5.41
		<u>DAIMLER BENZ.</u>		
		Cranking Tests with Wehrmacht Winter Oils.		
		Cranking Tests at -20° with the Wehrmacht Winter Oils.	H	
		Standards for Cranking Tests for Evaluation of the Startability of Engine Oils at Low Temperatures.		22.6.41
		<u>ADAM OPEL A.G.</u>		
142.	S.713.	Cold Starting Tests with Wehrmacht all-the-year-round Oil. (Standard Diesel of the Wehrmacht, HWAS26).	Gorissen.	15.1.43.
143.	Z.804.	Cold Starting Tests with Wehrmacht all-the-year-round Oil. (Mayback Engine HL62TR).	"	31.1.41.
144.	-	Cold Starting Tests with Wehrmacht all-the-year-round Oil. (BMW 2 Ltr. Engine Type 326).	"	14.7.41.
		<u>RHEINMETALL BORSIG.</u>		
145.	-	Testing 14 Uncompounded Gear Oils.	Heimann.	15.2.42.
		<u>MISCELLANEOUS REPORTS.</u>		
146.	-	Notes on the Meeting of the Working Committee (Knock Measurement in the I.G. and CFR Engines)	-	23.6.44.
147.	-	Special Committee for Standardising Engine Testing of Diesel Fuels by DVM.	-	22.9.42.
148.	-	Standard Method for Diesel Fuels.	-	18.1.41.
149.	-	Heating Oil Quality.	-	16.9.38.

CIOS REPT. NO. NO. TITLE. AUTHOR(S) DATE

MISCELLANEOUS REPORTS.

150 Technical Report on Standardizing Engine Testing of Diesel Fuels (Klockner-Humboldt-Deutz).

Instruction for Determining Pumpability of Diesel Oils

Instruction for

REPORTS BY THE PHYSIKALISCHE-TECHNISCHEN REICHSANSTALT

IOI NO	TITLE	AUTHOR(S)	DATE
	Development of ultrameter		
	<u>DVL REPORTS.</u> Reports for (Clarification of) Terminology on Aviation Fuel Problems.		
	<u>REPORTS FROM TECHNISCHE HOCHSCHULEN</u>		
	Evaluation of Fuels in relation to Vapor Lock Problems (Dresden).	Hager & v. Ebdorn.	
	Mixture Formation in Otto Engines at High Fuel Vapor Tension and Starting Process at Low Temperatures (Dresden).	Werminghof & Schiess.	
	Tests to Determine the Pressure Stability of Lubricating Oils (Dortmund, Berlin)		
111	Investigation of the Lubricating Ability of Oils (Munich).	Kedrov.	May 1944.
114.	Gear Tests at 150°C. Oil Sump Temperature (Stuttgart).	Wellinger.	5. 8.44.
115. 392.	Investigations on the Development of Self Ignition-Operation in a mixture Compressing Engine (Stuttgart).	Ernst & Dorr.	29. 3.41.
116.	Tests on the Heat Stability of Gear Lubricants in respect of the new Uncompounded Gear Oils.	Wellinger.	9. 5.44.

REPORTS BY OTHER COMMERCIAL COMPANIES.

DEUTSCHE VACUUM OEL.

117. VB532b.	Establishing the Limiting Flow Temperature of Lubricating Oils.	Paul & Richter.	30. 6.43.
118. VB540a.	Development of a Test Method for Determining the Low Temperature Behaviour of Lubricating Oils.	"	13. 3.44.
119. VB541a.	Investigation of the Heat Stability of Wehrmacht 8E Gear Oil.	"	13. 3.44.
120. VB542a.	E.P.Oil (Hypoid Oil for Highly Loaded Operation).	Richter, Paul & Urluss.	14. 3.44.

CIOB NO.	REPT. NO.	TITLE.	AUTHOR	DATE
121		Testing Aero Engine Oils in the DKW Eng'g		
		Evaluation of Tests for the BMW Oil Test		
		in relation to Oil Gels		
		<u>RHENANIA - OSSAG.</u>		
		Pump Type Apparatus for Indicating the In		
		comparative Behaviour of Oil at Low Temper		
122		Investigation of Ge... at Low Temperatures	Kep...	
		Pump Circulation Test at Low		
		(Wehrmacht Gear Oil)		
126		Preliminary Test for... at Low Temperatures	Zogbaum	
127		Testing Wehrmacht Gear Oil &... ability at Low Temperatures.	Zogbaum	
128	11.	Standardizing the Modified Pump Type Apparatus and Construction of a Curve for Pumpability at Low Temperature.	Zogbaum & Zander.	1. 4.43.
129.	14.	Testing the Reference Batch of Wehrmacht 8E Gear Oil for Pumpability.	Zogbaum & Schauer.	1. 4.44.
130.	18.	Comparison of the Old Model Pump Type Apparatus with the new Model.	Zogbaum.	1. 4.44.
		<u>I.G.FARBEN, OPPAU.</u>		
131.	-	The I.G.Prüfdiesel for Measuring the Ease of Ignition of Fuels.		1. 4.42.
132.	-	Origination and Object of Use of the Test Engine K.		1. 4.42.
133.	-	Drawings from I.G. Oppau Rept. No.478.		
134.	-	" " " " " " " 542.		
135.	-	" " " " " " " 518.		

DEUTSCHE KRAFTFAHRT FORSCHUNG.

CYOS NO.	REPT. NO.	TITLE.	AUTHOR(S)	DATE
79	74.	Tests on the Engine Behaviour of Syst I.C. Engine (V. H. Hultgen).	Kamm	
		Engine Method of ... (V. H. Hultgen)	Ernst	
80	86.	The Position of 2 Stroke Engines.		6. 6. 41
		2 Meeting of the ... 2 Stroke Engines		
		2. Meeting of the Working Group on 2 Stroke Engines		
		Power ... Engines	Neumann	1940
		New Oil ... Bearing	Huber & ...	1940
		Investigation of the Exhausting of the Combustion Chamber of High Speed Diesel and I.C. Engines.	Bisang.	
86.	Vol.5.	Contribution to the Exploration of the Combustion Process in High Speed Diesel Engines.	Kneule.	1938
87.	Vol.29.	Cylinder and Piston Ring Wear.	Beck.	1939
88.	Vol.31.	Measurement of Knock Resistance in I.C.Engines.	Schutz.	1939
89.	Vol.33.	Investigation of Knock Clatter of I.C.Engines with an Electro-Acoustic Measuring Apparatus.	Schmidt & Generlich.	1939
90.	Vol.34.	Mechanical Losses of the High Speed Diesel Engine and their Determination with the Towing Test (Schleppversuch).	Ullman.	1939
91.	Vol.52.	Comparative Investigation of Bearing Shell Materials.	Heidebrook & Doring.	1941.

DEUTSCHE KRAFTFAHRT FORSCHUNG.

CYOS NO.	REPT. NO.	TITLE.	AUTHOR(S)	DATE
97	Vol.43	Injection of Fuels in the Diesel Engine Ignition Delay Measurement by means Photo Cells in various ...		
98	Vol.54.	Detection of Lubricating Film Break by Measuring the Electrical Resistance Between Electrodes ...		
		Fuel and Engine ... Automotive ...		
99	Vol.57.	Fuel Properties ...		
		The Lubricant ... Special ...		
97	Vol.60	The Operation with Liquid ... Flushed ...		
98	Vol.61.	The Scavenging Process on the Basis of a new Conception of Expansion ...	Schultz ...	
99.	Vol.62.	Increased Loading of 4-stroke Diesel Engines. The Scavenging Process.	Kiesel.	1941
100.	Vol.63.	Ignition Delay and the Evaluation of Fuels Ignition Delay Measurement of Diesel and I.C. Engine Fuels.	Widmaier.	1941
101.	Vol.76.	Influence of Air Swirl on the formation of Fuel Stream in the Swirl Chamber.	Sauberlich.	
<u>REPORTS FROM THE ERPROBUNGSSTELLE; RECHLIN.</u>				
102.	2363.	Method for Cetane Number Determination of Diesel Fuels.	Lange.	18.11.41.
103.	2337.	SECRET. Behaviour of Aviation Fuels at High Altitude.	Cinsamann.	18. 4.42.
104.	2485.	Sludge Formation in Aero Engine Oils.	Baier et al.	20.7.42.
105.	2525.	Circulation of Lubricating Oil in an Engine with Low Temperature Resistant Aero Engine Oil.	Baier.	12. 2.43.
106.	2363.	Determination of Cetane Number of Diesel Fuels (with the Inertia Indicator according to Dr. Neumann).	Starke.	1. 4.44.

RUHRCHEMIE REPORTS.
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NO.	REPT. NO.	TITLE	AUTHOR(S)	DATE
		aviation oil 'leod K1951/2 made from	Schaub	8. 4. 43
48	P132	Investigation of Engine Oil 3698	Schaub	12. 6. 43
		Investigation of Engine Oil from Weraag (N...)	Schaub	
		Investigation of a Weraag "super" where the	Schaub	
		winter oil (non reference oil)	Schaub	
		Investigation of 3 (...)	Schaub	
50	P137	Starting I.C. Engines at low temperatures	Schaub	2. 11. 43
		Investigation of the Ester oil from the	Schaub	
		new Research Laboratory	Schaub	
54	P138	Testing Engine Oil 3993 in relation to	Schaub	17. 9. 43
		Piston Seizure.		
55	P140	Knock measurement of Synthesis-Benzin	Schaub	1. 11. 43.
		dependent on the Ignition system.		
56	P141	On the pumping behaviour of lubricants at	Schaub	29. 11. 43.
		low temperatures.		
57	P143	Cranking Test with Rumanian Oil.	Schaub	29. 12. 43.
58	P144	Testing an Aero Engine Lubricant SS1060 for	Schaub	7. 3. 44.
		Piston seizure.		
59	P145	The foaming of Lubricating Oils.	Schaub	17. 3. 44.
60	P146	Testing the Aviation Oil sample K2015 of	Schaub	17. 6. 44.
		"Molaj".		
61	P147	Starting I.C. Engines at low temperatures 2.	Schaub	27. 6. 44.
		Interim Report.		
62	P148	Testing Gear Oils for heat stability.	Schaub	29. 8. 44.
63	P149	Preliminary Tests with the 4 Ball Apparatus.	Becker & Schaub	26. 9. 44.

RUHRCHEMIE REPORTS.
(continued)

CIOS NO.	REPT. NO.	TITLE	AUTHOR(S)	DATE
64	P150	Cold Testing of ... AK7-200.		
65	P151	The Evaluation of ... Wear in an Engine.		15. 11. 44
66	P152	Ageing Behaviour of the Aviation Oil (with non bright stock components)		
67	P153	Comparison of Synthetic Residue and Distill Oil of similar Viscosity for Clarification the effect of Bright Stock Components		
68	P154	Testing various E.F. additions to current Synthesis oil, combined with investigation on various methods for determining stability.	Schaub	2. 11. 44.
69	P155	Tests with fuels of different properties	Schaub	16. 11. 44.
70	P156	Report on the incomplete Combustion Test	Schaub	11. 1. 45.
71	P157	Report on the Vapour Lock Correlation Test of the OKH.	Schaub	14. 2. 45.
<u>DEUTSCHE KRAFTFAHRT FORSCHUNG.</u>				
72	75	On the attack of Anti-Freeze materials on Metal and Rubber. (Staatlichen Materialprufungsamt, Berlin-Dahlem).	Schikorr & Alex.	
73	52	The State of Knowledge on Mixture Formation in I.C. and Diesel Engines. (T.H. Dresden).	Zinner.	
74	96/1941	Test on the Use of Power Gas in the Pure Diesel Process (T.H. Dresden).	Dreyhaupt.	
75	94/1941	Bomb Tests on Mixture Formation and Combustion with Gasoline Injection (T.H. Graz).	Bianchi	
76	58	Knock Processes in Multi-Cylinder Engines (T.H. Munich).	Schmidt & Regel.	
77	99/1941	Tests with a Carburettor Engine with Self Ignition (T.H. Stuttgart).	Ernst & Dorr.	

RUHR-BENZIN REPORTS
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REPORT NO.	TITLE.	AUTHOR(S)	DATE
SECRET	Report on Engine Testing of chemically synthetic Aero Engine Oils.	Schaub	10. 1.41
	Evaluation of Fuels for Various Lubrication troubles in an engine.	Schaub Veldner	15. 1.41
	Report on the Engine testing of the synthetic Aviation Oil K1860 (Japanese).	Schaub	15. 5.41
	The Development of an Engine Test for Aviation Oils in relation to Piston Seizure.	Schaub	6. 5.41
SECRET.	Report on Engine Tests of Synthetic Aviation Oil K.1880.	Schaub	7. 7.41
	Testing various Wehrmacht Standard Oils for Piston seizure in the Triumph Engine.	Schaub	1. 10.41
	Comparison of Supercharge Tests in the NSU Engine of Ruhrbenzin and the BMW 132 Engine of the Tec.Prufstand, Oppau.	Schaub	6. 9.41.
22.	Extract from a report on Tests with Gear Oils by the Rheinmetall-Borsig A.G.	Schaub	30.10.41.
23.	P114. Engine Tests of the Aviation Oil K.1929.	Schaub	7. 1.42.
24.	P116. Influence of viscosity and various additives on the behaviour of synthetic Oils in Engine Tests in relation to Piston seizure.	Schaub	12. 1.42.
25.	P115. Development of a Test for Engine Oils relative to Piston Seizure.	Schaub	15. 1.42.
26.	P117. Testing 4 Engine Oils for Piston Seizure.	Schaub	29. 1.42.
27.	P118. Ease of Ignition of R.C.H.Cetane.	Schaub	30. 3.42.
28.	P119. SECRET. Oil Testing in a NSU Engine.	Schaub	25. 4.42.
29.	P120. Dilution of Engine Oils for Winter Operation.	Schaub	22. 5.42.

RUHRCHEMIE REPORTS.

REPORT NO.	TITLE.	AUTHOR	DATE
	Report on Lubrication ability.		8. 1.43
	On the Question of the Determination of Asphalt Contents in Aged Lub. Oils.		
	Working out a method for testing of lubricating oils.		
	The behaviour of a different type of Aviation and automotive Engine Oils in relation to Piston seizure.		
	Aviation Oil Blend P1951 from RCH Bldg 8 and a low viscosity mineral oil base.		
	Evaluation of the "WA" Test (ME96 - Motor Oil from 10).		
38.	Preliminary Report on Engine Oil Test No. 3370 (ME96 - Ester Oil).	Schaub	1. 7.42.
39.	Analytical Investigation of an Ester oil from I.G.Farben.	Rottig	28. 8.42.
40.	P125a. Testing Engine Oil 3370.	Schaub	8. 4.43.
41.	P126. Influence of viscosity on Oil Consumption.	Schaub	19. 9.42.
42.	P127. On the lubrication effect of thin Engine Oils (Winter Oils).	Schaub	21. 9.42.
43.	P128. Aviation Oil 3344 from the Main Plant.	Schaub	6.10. 42.
44.	P129. Aviation Oil 1979 from the Main Laboratory.	Schaub	4. 1.43.
45.	P130. The Effect of Addition of Oppanol on the behaviour of Engine Oil.	Schaub	27. 2.43.
46.	P131. The Influence of Oppanol addition on wear.	Schaub	11. 3.43.

certain tanks with Maybach engines did require a special fuel of 78 O.N. C.F.R.M.M. as against the more usual supplies of 72 O.N.

Schaub claimed to have developed a small super-charged, fuel tank engine based on a 500 cc. 4 stroke motor cycle engine (the N.S.U.) which was alleged to correlate well with the B.M.W. engine after initial difficulties with the piston and bearings due to the increased temperatures and loads. Other engines developed and tested by the N.S.U. were the T.G. and

The direct fuel injection engine was used mainly for

A very small amount of work had been carried out on the engine to determine the only fuel which could be used

(c) Lubricating Oils. In the early part of the Army had tried to use the same oils summer and winter (Einheitsöl) both for engines and gears, but this had to be dropped due to experience on the Russian front and summer and winter qualities were introduced. Schaub was, however, of the opinion that the original programme could have been carried through. On the Russian front a system of fuel dilution had been developed for cold starting, using either Kerosene or gasoline, the latter being preferred. There was, however, no definite programme. Tramm developed an air bubble viscometer and every driver was supposed to measure the viscosity of his oil and dilute with fuel accordingly. This was not successful and was superseded by instructions, with tables, which gave the quantity of fuel to be added (% by vol. of gasoline) for a given outside temperature.

Questioned on synthetic lubricating oils, Schaub explained that the various synthetic aviation oils produced by Ruhrchemie had been given code numbers of SS.2000 to SS.2010. All of these oils had viscosities of about 3⁰E at 100°C. and 10 to 12⁰E at 50°C. With increasing code number the Pole Height had decreased from 1.8 to 1.5. Schaub mentioned that all the Ruhrchemie synthetic oils increased in viscosity in use - much more so than mineral oils - due to further polymerisation. This he gave as the reason for mixing mineral oils with the synthetic products in, for instance, the aviation oil S3. He also claimed that the latest Ruhrchemie production was superior to any other synthetic oils and superior to natural products, having overcome the disadvantage of increased viscosity in use. It was presumably for producing this product that the plant at Willingen was intended. Schaub also

mentioned that he felt that if they had had the influence of the I.G. they could have been able to get into production much more easily than had been the case.

Questioned on the use of synthetic oils by the Army Schaub stated that no I.G. synthetics were used for this purpose; Ruhrchemie production did, however, find a use in this field. Deurag, Rhenania and D.A.P.G. blended synthetic oils with natural oils and possibly used Oppanol (a V.I. improver) to give the Pole Height of 1.8 required by the Army. Schaub did not, however, favour the use of Oppanol because its efficacy disappeared with use due to depolymerisation. He had heard by hearsay that Oppanol was used in gear oils; more data on this subject could be obtained from Dr. Tramm. Schaub had no knowledge of any synthetic oils used by the Army.

(d) Engine Tests for Lubricating Oils. Schaub knew of the Intava DKW 2-stroke ring sticking test but he did not see how an engine operating with petrol lubrication could be expected to correlate with the BMW 132 single cylinder test with full scale engines. Of the engine tests developed by Ruhrchemie other than the N.S.U. fuel testing unit mentioned by Schaub, he mentioned the following work:

(1) Triumph Motorcycle Engine.

This engine is a two-cylinder side-by-side two stroke engine with a common combustion chamber, similar to the "Puch" engine but with a modification in the arrangement of the crankshaft. The manufacturers claimed that this engine needed a special lubricant due to piston seizure or scuffing in the arduous use in Army motor cycles. In the test set-up the engine was run with reduced cooling air so that it overheated and gave seizure or scuffing after about 10-15 mins. Instead of using the normal lubrication system, the oil was fed directly by an adjustable supply to the cylinder, the connecting rod roller bearing being lubricated by excess. By introducing the oil near the top of the piston at bottom centre, and by careful regulation of the feed, it was claimed that reproducible results could be obtained in a short time. Using this method Schaub claimed that he was able to prove that Ruhrchemie synthetic oils were the best of a series of about 6 oils circulated, under code numbers, for test. These same oils were also subjected to road tests in motor cycles fitted with the Triumph engine, the route including some steep hill climbing in Alpine country. It was found that the laboratory test agreed with the road tests.

dealing with fuels and lubricants, and these were removed. A complete list of these documents, with titles given in English and German is reproduced in Appendix I.

In the second visit to Nuttlar it was learned that it was intended to install most of the test engines evacuated from Ruhrchemie at Sterkrade-Holten in a slate quarry at Nuttlar but this was not actually done because of the speed of the Allied advance on the Western Front and the engines were being returned to Sterkrade-Holten. Dr. Schaub was located at Sterkrade-Holten.

4. Interrogation of Dr. Schaub July 21st, 1945.

Dr. Schaub was in charge of the engine test laboratory at Sterkrade-Holten, having been engaged in 1938 for the express purpose of developing engine testing of Ruhrchemie products. Born on April 22nd, 1911, he obtained his Doctor's degree at the Technische Hochschule, Berlin, having worked on the development of diesel engines, using, for example, gasoline. Previous to being employed by Ruhrchemie, he had been engaged in engine testing by Daimler-Benz.

Dr. Schaub claimed that he had never been a Party Member and proved very co-operative. However, throughout the interrogation he complained of not being able to refer to his reports and documents which had been removed by the previous team. From memory, Schaub gave the following details of the laboratory of which he was the leader.

Equipment. The equipment of the engine laboratory at Sterkrade-Holten consisted of 12 test engines, as follows:-

- 2 I.G. Pruf Motors for gasoline testing.
- 1 C.F.R. engine.
- 1 Supercharged 500 cc. 4-stroke N.S.U. (NECKARSULM) engine.
- 2 N.S.U. engines (not supercharged).
- 2 Triumph 2-stroke engines (similar to the Puch but with a different crankshaft).
- 2 4-cylinder Opel and Daimler-Benz engines.
- 1 H.V.A. (Deutz) diesel engine for cetane ratings.
- 1 F.K.F. Stuttgart diesel engine for fuel testing (developed by Kamm of Stuttgart but not considered by Schaub to be very good).
- 1 Four-ball machine.
- 1 Almen machine.

- 1 Z.F. (Zahnradfabrik - Friedrichshafen) gear test machine (bombed).
- 1 large cold chamber 5 x 9 metres (no chassis dynamometer).
- 1 small cold chamber for engine testing, with external brake. This used a Lude plant for cooling and a temperature of -40°C. could be obtained. A small cold chamber cooled by dry ice for gear oil tests.

Personnel. The total staff in the laboratory of Ruhrchemie was about 500, Dr. Tramm being the chief chemist. A large staff was due, in part, to the fact that the Ruhrchemie plant was a large concern and licensed their products to other firms engaged in the same industry.

Operation.

(a) **General.** It had been learned previously that Dr. Hagemann, one of the directors of Ruhrchemie, had been intimately concerned with the OKH. Questioned on this, Schaub stated that Hagemann was the leader of a group of the OKH which dealt with fuels, lubricants and rubber. Because of this connection, the Ruhrchemie engine test laboratory was one of the main places for engine testing for the OKH, other places being the Erprobungsstelle, Rechlin and the Army Test Station at Kummerow. The latter being the chief place for testing vehicles, having a large cold chamber. Nearly all the engine test facilities, except the supercharged engine, were used for work on lubricants and fuels for the OKH.

Schaub claimed that he did not have any detailed knowledge of the changes in quality of Army products and suggested that Tramm would be better informed.

(b) **Fuels and Fuel Testing.** The I.C. and C.F.R. engines were used to test Ruhrchemie gasolines. Questioned on the change made during the war in the method of test, namely from the Research Method to the Motor Method, Schaub considered that the reason for the original adoption of the Research Method was due to a certain amount of influence or pressure by the Benzol Verband, and it was eventually found, by correlation tests, that the Motor Method under-rated benzol blends less than the Research Method over-rated them. The Air Ministry had always used the Motor Method and its adoption by the Army allowed greater uniformity.

Alcohol blends were stated to have been mainly used for civilian purposes and not to any great extent by the Army.

EXTRACT TESTING AND UTILIZATION

Reference is made to the report of the investigation of the extraction of the products of the synthesis of the Fischer-Tropsch process.

Dr. Velde was interviewed on July 8th, 1945 at the Central Bureau of Research, Delft, Netherlands.

Dr. Hageman and Dr. Schaub were interviewed at Sterkrade-Holten on August 23rd, 1945 by:

Mr. H.L. West, British, Ministry of Fuel and Power.
Major L. Rosenfeld, British, " " " " " "

3. Dr. Hageman and Dr. Schaub were interviewed at Sterkrade-Holten on August 23rd, 1945 by:

W.F. Faragher, U.S., Petroleum Administration for War.
W.A. Horne, Junr., " " " " " "

2. Ruhrchemie A.G., Fischer Tropsch Products.

Dr. Velde - July 8th, 1945.

Motor Gasolines boiling up to 150-160°C. (302-320°F.) were shipped by Ruhrchemie to the Zentralburo as blending agents. The only specification was for Reid Vapour Pressure which was:

Summer 0.55 Kg/cm² max. (7.8 lb/sq. inch)
Winter 0.80 Kg/cm² " (11.4 " " ")

Octane numbers (C.F.R. Research) for atmospheric pressure products were 60-61 and for 10 atm. pressure products about 45-50. The latter products had lower Octane Numbers due to their lower olefine contents.

These products were manufactured as follows:

	Aero Diesel Oil for Junkers Engines	Normal Diesel Oil (known as Special Diesel Oil-N)
Sp. Gr.	0.770	0.775
Pour Point, °F	40	-4
Octane Number	70	80+
Viscosity at 20°C. (68°F)	ca. 1.06 centi- stokes.	1.1 centi-
Flash Point Boiling Range	170-230°C. (338-446°F)	180-270°C. (356-518°F)
Production tonnes/month	7	100-15

It was stated that the cetane numbers of diesel oil fractions in the same boiling range from atmospheric pressure and medium pressure (10 atm.) synthesis are almost identical.

Waxes. Two grades were produced for industrial use separated by vacuum distillation at atmospheric pressure:

(a) Melting Point 50-52°C. (122-125.6°F.) - Acid Free,
Colour - White.

(b) " " 90-92°C. (194-197.6°F.) - Acid Free,
Colour - Light Yellow.

3. Ruhrchemie A.G., Engine Test Laboratory at Nüttlar.

On the occasion of the first visit to Nüttlar, Dr. Schaub, who was in charge of the engine test laboratory at Sterkrade-Holten and who had been evacuated to Nüttlar, was not located and the Nüttlar laboratory was examined in his absence. It was a small wooden hut in which was installed a 4-cylinder automobile engine which was in running order for lubricating oil tests and a Four Ball Testing machine. No other equipment was installed but a number of crates containing various engine components were located. No documents of any value were located in the laboratory. A visit to Schaub's house, however, revealed a number of reports and documents

The mass is discharged and broken up in any desired machine. Extraction of the paraffin wax is then effected in an extractor, the best solvent being a gasoline fraction from the Fischer-Tropsch plant (b.p. 80-100 C). The extracted wax is recharged to the process. The soap is treated with dilute sulfuric acid, washed and dried. A simplified flowsheet is attached as Fig. X.

The finished wax is designated as OP 32. It is a yellow to brown hard material, solidifying at 80°C. and melting (clear) at 90-100°C. Its iodine number is 145-150. Saponification number is 190-200. The following number is 100000.

If desired, OP 32 can be bleached with nitric and sulfuric acid and nitric oxide gas, or may be bleached with sulfuric acid and nitric oxide gas.

METHANE ACTIVATION.

A Roumanian by the name of Slatineanu worked for two years at Ruhrchemie, Sterkrade-Holten, on the activation of methane at high pressure. He claimed to be able to combine methane and water to produce alcohols, as well as to produce ammonia and fatty acids from methane gas and nitrogen. The catalyst tried was the usual Fischer-Tropsch cobalt catalyst prepared at Sterkrade operating at 5-60°C and about 1500 atm. No useful products were ever obtained.

After being released by Ruhrchemie, Slatineanu was employed by the Concordia Company, Oberhausen. Here he duplicated the equipment used at Ruhrchemie, but obtained no positive results. He claimed that he was unable to reproduce the catalyst used at Sterkrade for his failure.

Dr. Saxner of the Concordia Company will give details on the subject, but they seem

has fallen below 100°C., then is purged with nitrogen and saturated with carbon dioxide. The catalyst should now contain 60% of metallic nickel (based on total nickel).

Process Operation

The flow scheme for this process is shown in Fig. X. The preheated gas is introduced into the decomposing vessel (kept at 425-450°C), where the organic sulfur compounds are decomposed, and the oxides of nitrogen reduced. After cooling, the decomposition products (H_2S , NH_3 , etc) are removed by luxmasse and activated carbon. The purified gas passes to the synthesis reactor where carbon monoxide, the carbon dioxide and the heavy hydrocarbons are converted to methane at 160-230°C. It is important that the operation be started at as low a temperature as possible. During the process, the temperature must be controlled to within 1-2°C. Increase of temperature is made only to compensate for decrease in activity of the catalyst. The water formed contains some ammonia and is removed from a separator beyond the condenser.

The decomposer is a cylindrical vessel (heated externally); the converter, a well-constructed tubular boiler. The catalyst fills the tubes (20-28 mm diameter), the cooling water being around the tubes. Control of the pressure of the generated steam provides good temperature control.

OXIDATION OF PARAFFIN WAX WITH NITROGEN OXIDES.

The oxidation of paraffin wax was carried out with the intention of producing high molecular weight fatty acids, retaining as much as possible the chain length of the hydrocarbon. The acid will serve as a substitute for stearic acid, after further processing.

The raw material was paraffin wax, refined or "pre-refined", which is a mixture of hydrocarbons with an initial boiling point at 120°C., with less than 10% over at 450°C. The average molecular weight is 600, corresponding to an average chain length of 40-45 carbon atoms. The solidifying point is about 90°C, and the melting point about 120°C. The wax is treated in an enamelled iron kettle with nitrosyl sulfuric acid at 120-125°C. Oxides of nitrogen (NO , NO_2 , drawn from the ammonia oxidation plant) are bubbled through the wax while it is continuously stirred. The gas flow is 65 cu.m. per hour. For a quantity of wax, 70 kg of nitrosyl sulfuric acid (1.2 g. gr.) is used. The nitrosyl sulfuric acid is made by saturating concentrated sulfuric acid with the gas from the ammonia oxidizer. The oxidation time depends upon the acidity desired, but is usually 10-12 hours. The acid number is then 70-75, and the saponification number 80-85. The oxidation is on the average, 50%, at these values. Stirring is discontinued; the greater part of the acid settles is withdrawn and re-used. The product is withdrawn to a washing vessel and washed with boiling water with as little agitation as possible, until the water is free from sulfates. The dried product, at this stage, is called OP3, and is suitable directly for use in a number of ways. The material is nearly white and is very hard. The solidifying point is 80° and the clear melting point about 90°C.

The paraffin wax is separated from the acids by treating melted OP3 in a kneading machine with concentrated caustic potash (30% minimum) or caustic soda, being sure to use at least 50% excess of caustic. The kneading machine must be provided with a means for heating for the temperature is maintained at 100°, until all water has been evaporated. The mass is cooled while kneading to effect a disintegration.

Impregnation follows the washing. Potassium hydroxide is used to give the desired content of alkali. Repeated experiments proved that impregnation in the filter by pumping through the cake a potassium hydroxide solution of the proper concentration does not give a uniform product. The reason is the formation of cracks in the cake during the operation.

To obtain a satisfactory impregnation, it is therefore necessary to paste the cake and to add the caustic to this mass. The quantity of potassium hydroxide is so gauged that the filter cake (moist) contains 3.0-3.5% of potassium hydroxide, based on the iron. This result is in general reached if the solution used in the suspending has a concentration of 6.0 g. KOH per liter. After impregnation, the catalyst is again put in the iron filter press. The cake obtained is then dried at 110°C. and subsequently shaped.

Reduction

Reduction with hydrogen or with a mixture of hydrogen and nitrogen follows at a temperature of 300°C (maximum). Higher temperatures cause over reduction and an inactive catalyst. Time for reduction is usually 30 minutes after the temperature is reached. This statement applies when H₂ and N₂ are used in the proportion of 3:1. Times are much shorter when hydrogen only is used.

Reduction is most satisfactory when the mass is disposed in layers of 25 cm. depth and when the gas flow is high (about 2000 cu.m. per hour per square meter of cross section). A content of more than 5-8% of iron (metallic) in the finished reduced catalyst must be avoided. Metallic iron is determined by the use of mercuric chloride by the normal procedure. Higher contents of iron give an inactive catalyst. Further, the iron soluble in 2% solution of acetic acid shall be 60-70% of the total iron. This determination is made by boiling the reduced catalyst for about two hours with reflux, using a protective inert gas.

After reduction, the catalyst is treated in the usual manner with nitrogen (cold) and then saturated with carbon dioxide. Contrary to the behavior of cobalt catalysts, much

heat is liberated when the iron catalyst is saturated with carbon dioxide. The saturation must, therefore, be conducted slowly.

METHANIZATION CATALYST AND PROCESS

Introduction

The methanization of coke oven gas (CO, H₂, CH₄, H₂O, N₂)

- 1) CO : 3H₂ = CH₄ + H₂O
- 2) CO₂ + 4H₂ = CH₄ + 2H₂O

Volume contraction is considerable due to the water formation and condensation. The catalyst contains nickel, magnesium oxide and kieselguhr.

Catalyst Preparation

For the preparation of 1 kg of nickel containing catalyst, 24 liters of a solution of nitrates containing 42.0 g/l. of nickel and 6.2 g/l. of magnesium oxide and 40 liters of a solution of sodium carbonate (80.0 g/l.) are separately heated to boiling. The hot soda solution is added slowly to the nitrate solution with efficient stirring (centrifugal stirrer). When precipitation is complete, 0.5 kg. of kieselguhr is added and mixed well by stirring.

The hot mixture is then filtered rapidly in a filter press and washed with 120 liters of hot water.

The moist filter cake is shaped in an extruder and dried on a belt heated by an open flame. The catalyst is next brought to proper size (3-5 mm) in a special mill (Kornmühle) and a vibrator. This preparation is shown in Figure IX.

The granular mass (called Grünkorn) is reduced for one hour at 350°C. with a mixture of hydrogen and nitrogen (3:1) at a space velocity of 6000-8000 (calculated at operating conditions). The reduced catalyst is swept by the stream of hydrogen and nitrogen until the temperature

Kieselguhr - A light, voluminous product is best, calcined at 700°C.

Potassium Nitrate - A light, voluminous product.

Nitric Acid - A light, voluminous product.

Solution Preparation

The preparation of the solution of mixed nitrates is carried out in an acid resistant vessel provided with heating and cooling coils and with an effective stirrer. The components are dissolved in the nitric acid. The copper is dissolved first, then the iron, and finally the calcium oxide. The quantity of the quantities of the components is determined by the heating.

2 Fe requiring 8 HNO₃
3 Cu requiring 8 HNO₃
CaO requiring 8 HNO₃

The dissolving of the copper is begun in the cold. As the metal dissolves with strong evolution of oxides of nitrogen, the temperature rises (high heat of reaction). By heating, the temperature is brought toward the end to 80°C.

Next, the dissolving of the iron is effected by adding the turnings gradually. The rate of addition is determined by the rate of evolution of oxides of nitrogen. The temperature of the solution rises to about 80°C., and as the reaction slackens toward the end, is held at this level by heating.

The calculated amount of calcium oxide in the form of calcium carbonate, quick lime or hydrated lime, is then added. This component should in all cases be finely divided. It must be added slowly in order to avoid local over-neutralization.

The solution is next heated to boiling and held at that temperature for several hours before cooling to room temperature. Prepared in this way, the solution contains little free nitric acid. The solution is stable and no deposit forms even if it is boiled for a long time. The content of iron, copper and calcium oxide is within the following ranges:-

Iron 115.0-125 g. per liter.
Copper 5.0-7.0 g. " "
Calcium oxide 11.0-13.0 g. " "

Total nitric acid is between 510-450 grams. If the iron and lime are free from insoluble impurities, the solution can be used directly. Otherwise, the solution must be filtered, a difficult operation because of the gelatinous character of the usual solid material. Filters if used, must be of material not attacked by the solvent. Before precipitation, the concentration of the solution is brought to 50 g/l. per liter.

The sodium carbonate solution is prepared by dissolving soda ash to a concentration of 100 g/l. The solution is not filtered.

Precipitation and Impregnation

Precipitation is effected by introducing, as quickly as possible, the nitrate solution, heated to 90°C., into the boiling soda ash solution while stirring intensively.

The quantity of soda ash used is such that, at the end of the precipitation, the pH is 6.8, determined with indicator strips (Folienkolorimeter). If necessary, either nitrate solution or soda-ash solution is added to produce the desired pH. Total time of precipitation must not exceed five minutes.

The contents of the vessel are then stirred for half a minute; evolution of carbon dioxide is over in this time. Then the calculated amount of kieselguhr is stirred into the mixture.

For the purpose of separating rapidly the mother liquor from the catalyst, the suspension is filtered by using pumps of large capacity. Washing with hot condensate (70-80°C), is continued until the cake will yield a finished catalyst containing 0.4-0.6% of sodium nitrate, based on the iron content. More complete washing is unnecessary and in fact lowers the desired content of calcium oxide. In general, 200-220 cu.m. of wash water per ton of iron in the cake is sufficient to give the desired result.

The alcohols formed are all primary and predominantly of iso-structure. By caustic fusion of the 200-220°C. fraction, soaps can be directly prepared.

Experiments had been discontinued when the laboratory was completely destroyed by bombing. Dr. Fischer, the chief technical expert, supplied the

IRON FISCHER CATALYST.

Introduction.

The dense sintered iron catalyst used by others was objectionable to Ethylchem for the reasons:

The temperature of favorable action was too high to be satisfactory in the standard converter, because of the high steam pressure necessary for the control of temperature; and the catalyst charge for the converter was too heavy for the converters in use. It was desired that no change of converter design be included at the time, so research was started to obtain an iron catalyst free from the two objections mentioned. It was not expected that a catalyst superior to the cobalt catalyst would be discovered, but the shortage of cobalt made necessary to attempt to equal the results obtained by taking advantage of the

The iron catalyst contained also copper, calcium oxide and potassium hydroxide. Kieselguhr is used as support.

The solution employed contains iron, copper and calcium oxide in the proportions 100:5:10.

Since the precipitation of the calcium oxide is not complete, the finished catalyst has the following proportions: iron: copper: calcium oxide: kieselguhr, 100:5:8:30.

The dissolved nitrates are precipitated by adding a solution of sodium carbonate.

Materials.

Iron - Iron Turnings. They must be free from such metals as chromium, molybdenum, nickel, vanadium, etc., and also be clean of oil and dirt.

Copper - Waste metal in the form of clippings of sheets, wire, etc. is used. Copper oxide of corresponding purity can be used.

Lime - Calcium carbonate, quick lime or hydrated lime are equally good.

TABLE IV (continued)

	RV.	RW
Repairs & Maintenance		
1% of the Plant cost of 11,000,000 RM	110,000	
Utility Operating Costs		
Insurance, Taxes & other costs	10,000	
Laboratory Costs		
General Operating Expenses including Management	100,000	2,661,070
Sum of Operating and Material costs.		7,926,070.
<u>Interest on Capital.</u>		
13.5% of 11,000,000 RM.		1,485,000.
<u>General Expenses.</u>		
Taxes		120,000.
		9,531,070.
Credit for by-product		144,000.
		<u>9,387,070</u>

100 kg. Fatty Alcohol costs therefore 78.23 RM.

4 March 1943.

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(Dr. Landgraff,
Oberhausen-Holten)

THE DIRECT SYNTHESIS OF HIGHER ALCOHOLS FROM WATER GAS.

The direct synthesis of alcohols was being studied in the small unit shown schematically in Fig. VIII. A mixture of fresh water gas and recycle gas is compressed in two stages to 10 to 100 atmospheres. The gas then passes downflow at a space velocity of 100-150 N liters per liter of catalyst per hour, over 5 liters of a promoted iron catalyst. The catalyst is contained in an electrically heated tubular reactor, the tubes being 12 mm. inside diameter. The product from the reactor passes through an electrically heated hot separator at 110°C, thence to a cold water condenser. After the pressure on the remaining gas is released, it passes through an activated-carbon adsorber. Part of the dry gas is discharged from the adsorber, the remainder passing to the generator for recycle.

The gas flow is started with the reactor temperature at 200°C. The temperature is then raised degree by degree until the desired conversion is obtained. This temperature is in the range of 220-240°C.

The catalyst is a cerium or vanadium promoted iron catalyst which is prepared in a similar manner to the Ruhr chemie precipitated iron catalyst. The composition of the cerium catalyst was stated to be:-

100 parts by weight of Iron	
5 "	Copper
10 "	Cerium
50 "	Kieselguhr

At the higher pressure (100 atm), the olefin content of the reaction product is small and the oxygen-containing products are of shorter chain length. Medium pressure (10 atm) favors the formation of higher molecular compounds with a higher proportion of olefins.

From one normal cubic meter of gas, 80-100 gms. of a mixture of alcohols, esters and olefins (to about C₁₈) are obtained in a single pass operation. The product (C₇) from the separator, condenser and active carbon adsorber is combined and cut into fractions by distillation. The alcohol content of the total product averages 50-60%. The alcohol content of the benzene fraction (80-200°C) was lower than that of the gas-oil fraction (200-320°C).

OXO PROCESS.

Some additional information on the Oxo- process developed at Ruhrchemie, has been obtained from the design engineer of the Oxo Plant.

The economic estimate attached as Table II, as well as the design data for the commercial unit were calculated from a pilot unit in which the reactor is 5 meters length and 35 cm. internal diameter. The design of this pilot unit is ultimately related to the commercial plant.

The pilot unit had a charge capacity of 10 kg. of olefin mixture.

The Oxo stage charge gas was water gas $\text{CO:H}_2 = 1:1.1$ to $1:1.5$ and was recycled until the composition reached $\text{CO:H}_2 = 1:2$.

There are also attached two schematic flow sheets of the process, Figures VI and VII.

TABLE II.

ESTIMATE OF THE WORKING COSTS FOR 12,000 TONS PER YEAR OF FATTY ALCOHOLS.

(Based on a supply of 12,000 tons per year from the Ruhrchemie, Westphalia Plant.)

<u>Material Input:</u>	RM	
12,000 T. olefin at 0.40 RM/Kg.		4,800,000
6,000,000 Nm ³ water gas, 10 atm. at 0.04 RM/Nm ³		240,000
3,000,000 Nm ³ hydrogen, 10 atm. at 0.075 RM/Nm ³		225,000
<u>Operating Costs:</u>		
Operating wages & salaries incl. social security tax (150 men x 2000 RM/year)		300,000
<u>Energy:</u>		
<u>Steam.</u>		
78,000 T/Y, 80 atm. at 4.00 RM/T.	312,000	
97,000 T/Y, 18 atm. at 3.50 RM/T.	339,500	
100,000 T/Y, 2.5 atm. at 2.55 RM/T (vacuum and heating)	255,000	908,500.
<u>Electricity.</u>		
10,160,000 KWH @ 0.03 RM/KWH (gas compressors, pumps)		304,800.
<u>Water.</u>		
1,344,000 m ³ at 0.075 RM/m ³		100,800.
<u>Accessory Material.</u>		
Catalyst Cost		72,000.
<u>Operating Material.</u>		
Oil etc.		24,000
		<u>1,889,900.</u>

No accelerated test that could be used to predict catalyst life had been developed, nor was any standard test used for evaluating experimental catalysts.

TABLE I.

TEST DATA FOR COMMERCIAL BALT CATALYST

Hours from start	Temp	Pressure	Condensed oil cc	Total $\frac{5}{100}$
4	1	70	-	-
16	"	72	98	-
56	"	72	89	-
80	"	69	94	14
128	"	70	70	-
176	"	65	77	-
223	"	66	76	9K
272	"	60	75	-
319	"	67	69	120
368	"	66	62	-
416	"	66	73	13K
464	"	66	67	-
511	"	64	56	10K
559	"	65	60	-
607	"	63	63	11
655	"	60	66	-
703	"	58	62	11
752	"	61	64	-

Catalyst treated for 20 hours with $H_2/N_2 = 3/1$.

767	185-186	70	-	-
785	"	70	75	-
849	"	63	68	-
896	"	65	65	119
945	"	64	60	-
992	"	61	63	104
1041	"	62	59	-
1089	"	61	62	115
1137	"	61	63	-
1185	"	63	63	117
1233	"	61	60	-
1287	"	61	60	100
1339	"	60	57	-
1376	"	63	58	108
1425	"	60	52	-
1469	"	57	58	94
1517	"	54	49	-
1565	"	59	49	88
1613	"	56	47	-

(1) Condensed oil and C_5 + from activated carbon adsorber.

the polymerization time. In accordance with the laboratory work, it was intended to use 4-6% by wt. of $AlCl_3$, react the olefins for ten hours with the catalyst at 150°C and finish the reaction by heating for 2 hours at 600°C. Phentiazin was to be added to the olefins before polymerization. Dechlorination, distillation and finishing were planned analogously to the previous work.

LABORATORY TEST FOR ACTIVITY OF FISCHER CATALYST.

The catalyst produced in the factory as well as the reduced catalyst, is tested continually for activity. The factory product is first reduced during two hours at 450°C with a mixture of hydrogen and nitrogen (3:1) at the rate of 300 liters per hour for each 4 g. of cobalt. The catalyst can also be reduced at 400°C with relative pure hydrogen. By either method the final catalyst contains 40-50% of the cobalt in the oxide.

The test for activity is made in an aluminum-block furnace, designed especially for the purpose. The furnace is heated electrically or by gas flame to 185°C and controlled at this temperature within 0.1°C by regulator. Flow meter and gas adsorber are connected to the furnace.

The catalyst is put into a glass tube of 15 mm. inside diameter to a bed length of about 30 mm., corresponding to a weight of cobalt of about 4 g. After reduction the tube is put into the furnace, carefully avoiding oxidation, and treated with synthesis gas (4 liters per hour; $CO/H_2 = 1.0/1.8-1.9$). During the 500-hour test, the activity of the catalyst is measured each 48 hours (approximately). Contraction of the gas volume and yield of liquid products are measured and serve as measures of activity. The liquid products are expressed as cubic centimeters of liquid per normal cubic meter of synthesis gas. Liquid products comprise oil that condenses at room temperature in the receiver and the liquid that is removed from the activated carbon adsorber by steaming at 150°C and 15-20 mm. Hg. Water formation can also be used in appraising the catalyst activity.

As is shown in the drawing (Fig. IV) the contraction of the gas volume can be determined by turning the three-way cock so that the feed passes through the by-pass to the flow meter.

$$\% \text{ contraction} = \frac{c}{a} \times 100$$

a = feed gas volume
b = tail-gas volume
and a-b = c

Development work on the control of viscosity of the finished oil by changing conditions during the polymerization step was also carried out by Dr. Clar. By using a small amount of $AlCl_3$ and a comparatively high temperature, an oil of low viscosity is obtained, whereas reversal of these conditions leads to the production of oils of high viscosity. Using 1.5% by wt. of $AlCl_3$ and keeping the polymerization temperature at $100^\circ C$. from the start of the reaction (by preheating the olefinic charge) results in the production of an oil with a viscosity of 4⁰⁰E at $50^\circ C$. When 4-6% $AlCl_3$ is used and the polymerization temperature is kept at $150^\circ C$. for 12-20 hours, followed by heating to $60^\circ C$ for 2 hours, an oil of 38⁰⁰E at $50^\circ C$ is obtained. These latter conditions were selected for the planned manufacture of aviation-grade bright stock, except that the total reaction was limited to 12 hours, based on the experience that the plant process generally works smoother than the corresponding laboratory procedure. Some work has been done on the polymerization of pure heptene-1; it was found that polymerization to lubricating oil can be effected by 1% of $AlCl_3$ without formation of any catalyst complex; the $AlCl_3$ remains granular and no loss by heavy polymers is experienced.

Experimental work also showed that an improvement in the viscosity index resulted when olefins with longer chains were polymerized, as compared to those with short chains or a mixed feed stock containing both long and short chains. Oils containing long chains (above C_{12}) were also found to be more resistant to oxidation.

IV. Planned Manufacture of Aviation-Grade Bright Stock (Syntol Process).

a) Preparation of Feed Stock.

Since the viscosity index of synthetic oils by the Ruhrchemie process depends on the chain length of the olefins polymerized, and since a higher viscosity index than that of the regularly manufactured synthetic motor oil was required for the aviation-grade oil, certain modifications in the process had to be made. It was intended to polymerize only olefins ranging from C_9 to C_{18}

and to supplement the material obtained by cracking with the olefins obtained directly by the "Recycle Process". The charge stock to the cracking plant was furthermore to be increased by C_{15} to C_{18} paraffins which remained unreacted in the OKO process.

The "Recycle Process" has been designed for the manufacture of olefins. It consists in using water gas as primary synthesis gas to which is subsequently added recycle gas from the synthesis. The water gas contains 87% of CO and H_2 (ratio of $CO:H_2$ 1:1.23-1.25) and one volume of water-gas is mixed with three volumes of recycle gas so that the ratio of $CO:H_2$ in the actual synthesis is 1:0.8. The catalyst used contains 100 parts cobalt, 15 parts MgO and 200 parts kieselguhr; the temperature of the synthesis is $200-220^\circ C$, and the pressure 10-15 atm.; the space velocity is $800 m^3$ gas/ $10 m^3$ catalyst/hour. The total yield (liquid products and liquefiable gas) amounts to $160 g/m^3$ of "ideal synthesis gas" (CO and H_2). This yield includes a final conversion step, for previously unreacted gas, using an aliphatic Fischer-Tropsch unit.

The C_6 to C_{10} hydrocarbons from the "Recycle Process" contain about 20% of alcohols that must be removed before use in the Syntol process. The alcohols are converted to olefins and water by passing the hydrocarbon-alcohol vapors over activated alumina at $340-350^\circ C$ ($644-662^\circ F$) at a space velocity of 1 volume hydrocarbon-alcohol (calculated as liquid) per volume of catalyst per hour. (See Fig. II). The catalyst is prepared by precipitating a 5% Na aluminate solution with CO_2 , removing the alkali by repeated washing, predrying, extruding and drying at $400^\circ C$ ($752^\circ F$). The C_9 , C_{10} olefins are then separated by distillation and fed into the polymerization process together with the C_9-C_{18} olefins from the cracking process. The preparation is indicated on the attached flow sheet (Fig. III).

b) Polymerization and Finishing.

The polymerization procedure planned for the manufacture of aviation-grade bright stock does not vary from the practice followed in the manufacture of lubricating oil with respect to equipment and operations. The principal difference is the planned addition of phentiazin to the olefinic feed stock, the temperature conditions and

The oxidation test consists in passing 15 liters of oxygen per hour through 200 g. of oil held at 160°C. (320°F); the test is continued for 6 hours. Increase in viscosity, expressed as percentage of the original viscosity, and saponification number are used as criteria of the oil quality. When lubricating oils from natural sources are subjected to this test, they do not show any increase in viscosity, whereas the Ruhrchemie oil increases its viscosity by 150% of the initial viscosity.

Another test used is termed the "heat of reaction" test. The oil is brought to 140°C (284°F) under N_2 ; oxygen is passed into the oil and the temperature rise during short time intervals is noted. Uninhibited synthetic oils show an abrupt rise in temperature shortly after oxygen introduction has started. Natural oils show a gradual "heat of reaction" under these conditions.

A third test is used by Dr. Clar to estimate the thermal stability of synthetic oils. It consists in heating 500 g. of oil to 300°C (572°F) for three hours and determining the weight loss at the end of the test. The test is highly empirical and results are dependent on the shape of the distillation flask and other details. The following comparative results have been obtained:

Temperature.	Sample.	% Loss.
300°C.	Automobile oil from Natural oil (Greenring)	No cracking
300°C.	Synthetic oil (6-80E at 50°C)	5%
330°C.	Automobile oil from Natural oil (Greenring)	5%
330°C.	Synthetic oil (6-80E at 50°C)	40%

Since unsaturation appeared to be the main cause of the lack of stability of the synthetic oil, it was attempted to improve the stability by subjecting the polymerized oil to a second $AlCl_3$ treatment immediately following the polymerization. For this purpose, the oil was treated with 1% $AlCl_3$ at 180°C (350°F) for three hours. This process was, for a time, used in actual production, but was subsequently abandoned since the stability improvement was only temporary and since it was stated that the oil was satisfactory for its purpose without the additional treatment.

A permanent improvement in the stability of the synthetic oil was obtained by Dr. Clar by adding 0.2-0.5% by weight of phenthiazin

H
N
S

to the olefins before polymerization. The compound enters the reaction in an unknown manner and undesirable products formed by the addition of phenthiazin are removed during the treatment to which the polymerized oil is subjected in the course of the regular manufacturing procedure. Addition of phenthiazin to the finished oil is unsatisfactory, since it leads to the formation of gel-like material in the oil. The additive used by Ruhrchemie is the crude product obtained by melting 1 mol of diphenylamine with 2 mols of sulfur in the presence of $AlCl_3$ (2% by weight), stirring the mixture for 8 hours during which the temperature is raised from 200°C to 150°C and decomposing with water.

Other compounds which have been used successfully in the same way as phenthiazin, but which were not considered for commercial production, are 8-thiophthal and anthraquinonethiol.

The improvement in stability obtained by the phenthiazin treatment was indicated by the results of the laboratory tests. The viscosity increase in the oxidation test amounted to only 20% of that of the original oil, as compared to 150% increase for the uninhibited oil.

Another process designed to improve the stability of the synthetic oil is the addition of 0.3-0.5% by wt. of sulfur to the feed stock before polymerization and treating the polymerized oil with 1% by wt. of $AlCl_3$ at 250°C. (482°F) for 5 hours. It is stated that the sulfur is removed completely in the form of H_2S during the latter treatment. The finished oil does not contain sulfur, but it is claimed that its stability is greatly improved nevertheless.

c) Finishing of Synthetic Lubricating Oil.

The dechlorinated oil is distilled under atmospheric pressure yielding some gasoline, naphtha and Diesel oil. The residue is distilled under vacuum giving Diesel oil and spindle oil as distillates, whereas the residue is the synthetic lubricating oil.

The total yield of gasoline and Diesel oil resulting from both distillations is about 33% by weight of the olefinic feed stock; the yield of synthetic lubricating oil is 68% by weight and the loss in distilling the oil is about 1%.

To prepare finished automotive lubricating oil, the residue from the vacuum distillation is combined with a certain amount of collected clay (Tonell) at 150°C.

Disposal of Catalyst-Complex Oil

The lower layer obtained when the autoclave content, after polymerization, is allowed to settle consists of a catalyst-complex-oil mixture. To recover additional synthetic oil, the heavy oily layer is blended with Diesel oil in an autoclave and heated to 200°C (392°F) for 3 hours. The treatment results in the formation of an asphaltic mass which separates while hot from the solution of synthetic lubricating oil in Diesel oil. The lubricating oil solution must be dechlorinated in the same way as the original synthetic oil. The dechlorinated oil is then fractionated to recover the lubricating oil. This oil differs from the main product in its less favorable viscosity-temperature curve (pole height about 2) and high Conradson carbon residue (2-3%).

e) Inspection Data of Synthetic Lubricating Oils.

Inspection data of the spindle oil and automotive lubricating oil are given as follows; also included is a bright-stock which is obtained when the polymerization is carried out with 4-6% $AlCl_3$ at 150°C., followed by heating to 60°C.

	Spindle Oil.	Motor oil.	Brightstock
Density at 20°C.	0.845	0.855	0.865
Viscosity at 50°C.	2.3	7	38
Pole Height	1.75	1.75	1.82
Neut. No.	0.01	0.05	0.05
Sap. No.	0.04	0.10	0.10
Flash Point °C.	195	220	320
Pour Point °C.	-50	-45	-25
Loss by Evaporation (Hess Test 1 hr. 100°C)	45%	12%	2%
Conradson Carbon	0.01	0.04	0.20
Petroleum Residue	2%	3%	5%
Hard Asphalt	0%	0%	0%
Iodine No.	ut 5	bt 5	abt 30
CI	0.01	0.01	0.01
CI	0.10	0.15	0.30

Records of engine tests on the synthetic oils have been obtained from Dr. Schaub at Nuttley and will be available for study after microfilming.

The lubricating oil was delivered to the German Army (probably through the WIFO at Heiligenstadt) under the name of "Wehrmachtseinheitsöl" (Army All-Purpose Oil). The oil was apparently used as such for summer grade automotive lubricating and no inhibitor or other additive was used.

III. Improvement of Stability and Viscosity Index.

Due to the unsaturated character of the synthetic lubricating oil made by the Ruhrchemie process, the oil is not stable with respect to oxidation. Development work to improve the oil in this respect was carried out under the direction of Dr. Clar, who was interrogated on the subject.

The work was carried out entirely under laboratory conditions, using a number of oxidation and "cracking" tests as criteria for the quality of the oil and to evaluate the effect of inhibitors.

* All tests used appear to be rather arbitrary and are mentioned mainly to permit a certain comparison of natural and synthetic oils of this type and to gauge the effect of improving treatments.

carried out in a Dubbs unit at 500-520°C (932-968°F) at a throughput rate of 4.5 tons of oil per hour. Steam, at a rate of 1 ton per hour, is added to the vapors leaving the furnace and prior to entering the reaction chamber. The furnace outlet pressure is 4.5 atm. (67.3 psi).

In addition to gas oil, the cracking of "sweat oil" from the manufacture of wax is practised. This material is obtained as follows: The primary synthetic product from the Fischer-Tropsch synthesis boiling above 320°C. (608°F) is vacuum-distilled, yielding a distillate (boiling from 220-380°C (608-716°F)) and a residue. The residue is again distilled under vacuum and the resulting distillate is packed up for the manufacture of wax.

The oil obtained by sweating during the wax manufacture represents the charge stock for the cracking. Cracking conditions are the same as for gas oil except that the temperature is kept at 480-500°C (896-932°F). The products from this source are blended with those obtained from gas oil, but cracking of the two charges is carried out separately.

The following cracking products result from both procedures:

Liquid olefins in gasoline boiling range	68% by wt.
Cracking Gas	30% " "
Loss	2% " "

The olefinic gasoline contains about 70% olefins. The boiling range of the material from the cracking of gas oil lies between 30° and 200°C (86-392°F), whereas the end-point of the corresponding olefins from "sweat oil" lies at 220°C. (428°F).

The cracking gas has the following composition:

C ₃ +C ₄	18% by wt. of charge.
C ₂ H ₄	4% by wt. of charge.
C ₁ +C ₂ +inert gas	8% by wt. of charge.

b) Polymerization.

The feed stock is dried in a CaCl₂ tower to ensure that the water content does not exceed 0.015%. Polymerization is carried out in 7 autoclaves, each of which has a capacity of 21 m³ and is filled with 18 m³ of olefinic gasoline; the autoclaves are equipped with a closed coil for cooling and heating. The autoclaves are charged at room temperature and 1.2-1.5% (by weight of the charge) of AlCl₃ is added; the mixture is then stirred and the temperature is raised to the following values:

40°C (104°F)	2 hours
60°C (140°F)	3 hours
80°C (176°F)	3 hours
100°C (212°F)	3 hours

The first three steps are reached by control of the amount of cooling applied, whereas the last step is reached by heating by steam.

A technical grade of AlCl₃ with an iron content of about 5% FeCl₃ is used, and no advantages were found to result from using a catalyst of higher purity.

The reaction product is transferred to settlers where the synthetic oil separates as upper layer from the catalyst-complex-oil lower layer. A settling time of 2-3 hours is required. A certain amount of the catalyst-complex-oil mixture is left in the autoclave as solvent for the catalyst for the next charge.

The synthetic oil obtained in the above way contains some chlorinated hydrocarbons which are decomposed catalytically. For this purpose, the oil is treated with 1.5% of Tonsil (HCl activated clay) and 1.5% by wt. of ZnO at 180°C. (356°F) for 3 hours; the materials are added separately. The reaction mixture is subsequently cooled to 80°C (176°F) and filtered in a Kelly filter. The oily filter cake is extracted with gasoline to recover additional oil; and an extract oil containing 0.003% Cl and having a neutralization number of 0.04 is obtained.

III. Development Work.

Laboratory work has been done on the aromatization of hexane, octane and nonane.

Preparation of benzene from hexane over the above-mentioned catalyst requires a reaction temperature of 480-500°C. (896-932°F) and gives a yield of 85% by weight of liquid product, of which 30% is benzene. Octane and nonane give the same yields of aromatic hydrocarbon but require lower temperatures since,

Synthetic Lubricating Oil Manufacture.

I. Introduction.

Experimental laboratories for research in the field of synthetic lubricating-oil production were completed in 1937. During the same year, the construction of a plant for the manufacture of 1000 tons/month of automotive lubricating oil of 6-8°E. at 50°C. was started. The plant began production in 1938; due to good operating practices, it was able eventually to reach a capacity of 1500 tons/month. Primary charge stock included gas oil from other Fischer-Tropsch plants in addition to the feed stock made at the Ruhrchemie plant.

Development work was later carried out on the manufacture of aviation-grade lubricating oil (380°F. at 50°C.) and the construction of a plant was started in 1943. Due to repeated severe air attacks, it was decided to erect the plant underground near Willingen, west of Kassel, but this change was not accomplished. The plant was designed for a capacity of about 1000 tons of (finished product).

II. Lubricating Oil (Automotive Grade)

a) Preparation of Feed Stock.

[Information on the subject was secured mainly by the interrogation of Dr. Walter Schuff, manager of manufacturing of hydrocarbons by the Fischer-Tropsch process at the Holten plant. Dr. Schuff had held this position since 1939.

The starting material for the manufacture of synthetic lubricating oil by the Ruhrchemie process consists of olefins in the gasoline boiling range having one double bond at the end of the chain. The olefins are obtained by separately cracking the Fischer-Tropsch gas oil and "sweat oil" from the manufacture of wax derived from the Fischer-Tropsch synthesis. Gas oil, both from plants operating at atmospheric pressure and at 10-15 atm., is suitable, and the practice at Ruhrchemie consisted in supplementing their own production (10-15 atm) with purchased material from other Fischer-Tropsch plants. The gas oil has a boiling range of about 230-320°C. (446-608°F.). Cracking is

RUHRCHEMIE AKTIENGESELLSCHAFT
STERKRADE-HOLTEN.

CIOS No. 30/5.01.

I Summary

A process has been developed by Ruhrchemie to convert heptane into toluene, using a $\text{Cr}_2\text{O}_3 - \text{Al}_2\text{O}_3$ catalyst. The development work was finished in October 1944 and a plant for the production of 24,000 tons of toluene per year was designed. Plant construction was abandoned however, on order of the German government when the plant was about 20% complete.

Information on the process was obtained by interviewing Dr. Walter Müller, who was in charge of the development work.

II Process Details

The charge stock consists of the C_7 fraction (boiling range 90-100°C) (194-212°F), from the Fischer-Tropsch synthesis of hydrocarbons (15-20% olefins) or a C_7 cut from crude oil. The vaporized and preheated (400°C) feed stock is passed into the reaction chamber where it is converted, yielding 90-92% by weight of liquid product which contains about 50% by weight of toluene. The reaction takes place at about 480-530°C (896-986°F). No external heating of the catalyst container is required, since the heat of combustion released during the regeneration of the catalyst is "stored" in the catalyst bed and is utilized during the aromatization reaction. The maximum temperature during regeneration is 550°C. The reaction chamber has a total volume of about 11 m^3 , of which 8.8 m^3 is filled with catalyst. The on-stream time of each reactor was intended to be 30 minutes, and the design provided for 3 groups of 3 reactors. The space velocity is 0.15-0.20 (volume of liquid feed stock per volume of catalyst per hour). Details of the time cycle are given on the attached flow sheet (Figure I).

The catalyst consists of activated alumina with about 20% Cr_2O_3 . (*)

Coke formation during the reaction period amounts to 2.0-2.5 g. of coke per liter of catalyst per hour, corresponding to 1.5-1.8% by weight of the feed stock; these details are given on an hourly basis since they were obtained in the pilot plant, the operating cycle which was 60 minutes instead of 30 minutes, as planned for the full-scale plant. The life of the catalyst is about two years. By that time, the reaction temperature has to be raised by about 100°C and the yield of liquid product, as ascertained in an actual test carried out two years, decreases to 84.8% by weight of the feed stock, the toluene content falling to 1.1%.

Regeneration of the catalyst comprises two steps separated by a purging step with flue gas. The first regeneration step consists in burning off the carbon deposit with air, whereas the second step serves to reduce the six-valent chromium to three-valent chromium by passing hydrogen, preheated to 400°C, over the catalyst. Hydrogen is obtained from the process itself by separating the hydrogen liberated during the aromatization from the C_2 , C_3 and C_4 hydrocarbons.

The total liquid product, containing 50% of toluene, is fractionated in a 60-plate column (45 theoretical plates). The crude toluene, which contains 3-5% of olefins, is acid-treated and rerun to produce nitration-grade toluene.

(*) The activated alumina is prepared by precipitation of a 5% Na aluminate solution with CO_2 , as described in the section on synthetic lube oil at Ruhrchemie.

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SUPPLEMENTAL REPORT
ON RUHRCHEMIE, A.G.
STERKRADE-HOLTEN, RUHR.

Introduction

Examination of documents evacuated from this plant as well as the information obtained by interrogation of certain key personnel, indicated that additional information was desirable on certain phases of research, plant operation and product testing and utilization.

The present account is in addition to and supplements that previously reported in the C.I.O.S. report dated 5 July 1945, "The Fischer-Tropsch Plant of Ruhrchemie, G. M. B. H. Sterkrade-Holtten, Ruhr", compiled and edited by Carlotta M. Hall (M. F. 100) and Dr. W. Hagemann (U. A. 100).

The process information reported was secured by interrogation of key personnel of Ruhrchemie by the following parties:

1. At Sterkrade, 5-7 July 1945, by:

Dr. W.F. Faragher, (U.S., P.A.W.), Leader
Dr. W.A. Horne, (U.S., P.A.W.)
Dr. H. Schindler, (U.S., P.A.W.)
Capt. C.C. Chaffee, (U.S., Ordnance)

2. At Sterkrade, 19-23 August 1945, by:

Dr. W.F. Faragher, (U.S., P.A.W.) Leader.
Dr. W.A. Horne, (U.S., P.A.W.)
Mr. J.G. Allen, (U.S., P.A.W.)
Dr. G.S. Bays, Jr., (U.S., P.A.W.)
Mr. B.L. MacKusick, (U.S., P.A.W.)

The personnel securing information on product testing and utilization are listed on page 31.

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SUPPLEMENTAL REPORT

RUHRCHEMIE-A.G.,
STYRKRADE-HOLTEN
Hansen-H
RUHR

Reported by:

DR. W. V. FARAGHER, (U.S., P.A.W.)
DR. W. A. HORNE, (U.S., P.A.W.)
MAJOR D. A. HOWES, (BRITISH, M.F.F.)
DR. H. H. F. HINDLER, (U.S., P.A.W.)
CAPT. C. H. CHAFFER, (U.S. ORDNANCE)

MR. H. I. WEST, (BRITISH, M.F.F.)
MAJOR T. ROSENFELD, (PHILIPPINE M.F.F.)

On behalf of:

BRITISH MINISTRY OF FUEL AND POWER

and

U.S. TECHNICAL INDUSTRIAL INTELLIGENCE COMMITTEE

22 October 1945 (from t.p.c.)
CIOS TARGET NO. 30/5.01.

FUELS AND LUBRICANTS

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

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Dates of Trips: 5-8 July, 1945
21 July, 1945

19-23 August, 1945

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STERKRADE - HOLTEN

Farragher, W. J.

REC'D. MAR 11 1946

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

HISTORICAL - FROM 1944

In April 1944 they received instructions from the S.S. to start making further excavations but this time nearer the north face of the hill. This work was given the code numbers of B.11 and B.12. B.11 was later divided into two sections and given the names of KUKUCK and EBER. (B.11 and B.12, Nordwerke, entailed the excavation of 1,000,000 M³ of rock) The KUKUCK I plant was to be an Underground Hydrogenation plant and the EBER plant to be an Underground Liquid Oxygen plant. It was stated that the latter was 80% complete and was to be run by the JUNKERS firm.

The initial work on B.11 and B.12 was commenced with existing staff of the I.G. at Niedersachswerfen, i.e. about 400 men. In order to speed up the work and to provide men skilled in the art of excavation in tunnels and caverns, men were imported from the Saar, Ruhr and Aachen and foreign workers were used too to act as unskilled labour. To provide the skilled staff necessary for the work the Grossdeutscher Schachtbau G.m.b.H. were brought in to share the work with the I.G.

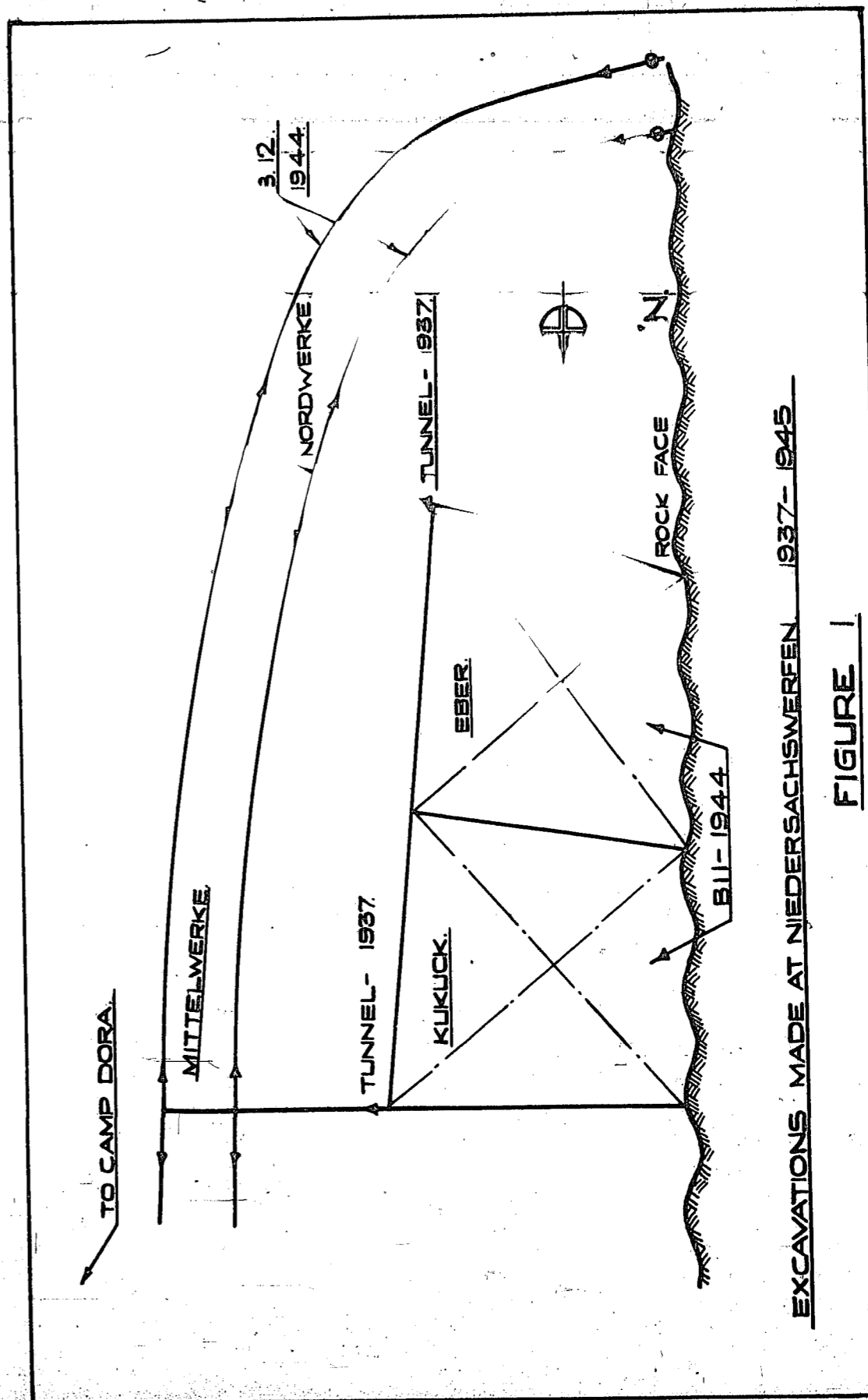
By March 1945 the working strength had reached 1,600 and about 75% of the excavation of KUKUCK I appeared to be complete.

The average output of the men was about 0.65 tonnes/man day.

The largest cavern was to be 47 feet wide and 37 feet high. Other caverns were to be deeper but not so wide. The main communication tunnels which ran along the east boundary of the plant and between the oil and gas sections were to be 38 feet wide and 26 feet high.

Very little mechanical erection had taken place the only signs being a half erected air compressor in the machine section for the D.H.D. plant.

The information was also given that the KUKUCK II plant was at ELLRICH to the west of Niedersachswerfen and was the oil storage and loading and offloading station for the KUKUCK I plant.



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KUKUCK PLANT AT NIEDERSACHSWERFEN

REASON FOR VISIT.

As a result of the disclosure of information at Leuna that an underground hydrogenation plant was to be built at Niedersachswerfen the site of the plant was visited on May 2nd, 1945, by Major I. L. Hirst and Dr. I. L. Hirst.

STAFF INTERVIEWED.

The following staff were interviewed:

Alfred Eichhorn	-	Technische Abteilung
Paul Grimmel	-	Works Manager
Hellmuth Leitweber	-	Berg Engineer

LOCATION OF SITE.

The site of the plant was not noticeable from the air or the ground because there were workings already in existence prior to 1944. The plant was being installed at the Anhydrite workings of the I.G. at Niedersachswerfen and thus it was possible to remove all excavation and other materials from the site and use them for fertiliser manufacture at Leuna, Wolfen and Oppau or tip them on the Dump at Leuna leaving little trace of the existence of any abnormal activities.

HISTORICAL - TO 1938.

Up to 1936 the whole of the Anhydrite for the I.G. had been obtained by the quarrying of the rock from the face of the hill, the average output of the men being of the order of 8.7 tonnes/man shift. In 1936 they received instructions from the Reich that they must make tunnels and chambers in the rock and so change from face working to tunneling to get the stones for the manufacture of fertiliser. This work consisted of a large tunnel being driven from the face of the rock a considerable distance in and then driving tunnels at right angles to the main tunnel, to make what was, and is still known as the Nittelwerk. (see Figure I). This work was completed in 1938, with no approach from the southern side of the hill, and therefore no visual sign of the operation would be there.

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REPORT ON VISIT TO KUKUCK I PLANT

AT NIEDERSACHSWERFEN

Reported By:

Major J.F. Ellis.
Ministry of Fuel and Power

Dr. L.L. Hirst.
U.S. Bureau of Mines

CIOS Target Number 30/70

Fuels and Lubricants

1945

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

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

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**KUKUCK I PLANT
NIEDERSACHSWERFEN**

Ellis + West

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**COMBINED INTELLIGENCE OBJECTIVES
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