CONFIDENTIAL

GERMAN PETROLEUM INDUSTRY
HAMBURG DISTRICT

REPORT No.2

RHENANIA-OSSAG MINERALOLWERKE, HAMBURG

HARBURG REFINERY

Reported By

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on behalf of the

BRITISH MINISTRY OF FUEL & POWER

AND THE

U.S. TECHNICAL INDUSTRIAL INTELLIGENCE COMMITTEE

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COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

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

HARBURG REFINERY

This Refinery was in operation before the war and was designed to handle imported topped Mexican and Venezuelan crudes for the manufacture of gasoline, lubricating oil base stocks and all grades of asphalt. The Refinery had a capacity of 450,000 tons/year crude input and comprised the following plants:-

1. One pipe still crude distillation unit.
2. One pipe still vacuum " "
3. One semi-continuous asphalt air blowing unit.
4. Edeleanu dewaxing plant.
5. One standard agitator type acid washery.

Water and steam requirements were supplied from the Refinery's own boiler house and water pumping station but power was taken from the mains through the refinery transformer station. Adequate laboratory facilities, office accommodation and workshops together with wharfage and rail sidings capable of handling some 1,000,000 tons/year of products were available. The asphalt production was handled in bulk and/or packed to suit market demands and the usual filling facilities for this purpose had been provided.

Except for asphalt, no finished products were manufactured at the Refinery, the crude white distillate being sent to Wilhelmsburg Refinery and the base lube cuts to Grasbrook Refinery for final treatments.

During the war indigenous crudes, mainly drawn from the Reitbrook, Neinhagen, Heide and Austrian fields have been processed and the refinery has also been extended by the erection of the following plants:

1. <u>Topping Plant.</u> Built by Wilke Werke, Brunswick.

   Commissioned 1940.
   Capacity 1,000 tons/day.

2. <u>No.2 Dewaxing Plant.</u> Built by Borsig and Edeleanu Construction Company

   Commissioned 1943.
   Capacity 7,500 tons/month.

This plant was originally designed to operate the SO_2/Bensol process but due to corrosion troubles, attributed to poor construction materials operation was changed to the Di-Chlor-Ethane process. Operation was conventional and calls for no comment.

3. Synthetic Lubricating Oil Manufacturing Plant.

Built by Bosaig and C.Peters, Hamburg.

Commissioned 1942.
Capacity 700 tons/months.

This plant was in commission only a short time before being dismantled and removed to Osterode for erection underground. Some details of the process and operating conditions are attached to this Report as Appendix "A" contributed by Messrs. D.Morten and W.H.Thomas.

4. Solvent Extraction Plant.

Built by Bosaig and Edeleanu Construction Co.

Capacity 10,000 tons/month.

This plant, like the above dewaxing plant was designed to operate on the SO_2/Benzol process, but again serious corrosion troubles were encountered which were attributed to the use of poor construction materials. Operation however, was conventional and calls for no special comment.

5. Clay Extraction Plant.

Local construction and design.

Commissioned 1942.
Capacity 6,000 tons/month

The process comprises extraction with gasoline followed by redistillation of the recovered oil with the lubricating oil fraction returned to the acid washery for further treatment.

A number of minor modifications to the existing plant were also carried out during the war, the most important of which was the extension of the vacuum unit to double capacity by the improvisation of a second pipe still.

At this point it is also convenient to refer to the use of stainless steel in place of homogeneous lead lining as practised at this Refinery for protection against corrosion in SO_2 plants. The Refinery Management were very satisfied with the service given by stainless steel and expressed a marked preference for stainless steel in place of lead for similar duties in future. Makers' catalogues describing the types of steel used, namely Mannesmannrohrenwerke, Düsseldorf, grades V.2A and V.4A, with other information and literature including some research on the use of lead are included with the collected documents.

The Refinery was heavily bombed during the last months of the war and the following plants are considered to have been practically destroyed. In any case no estimate of the extent of the repair work involved is possible until the sites have been cleared.

1. No. 1 Dewaxing plant
2. No. 2 Dewaxing plant.
3. Solvent extraction plant.
4. Clay extraction plant.

Fairly extensive damage was also done to the water pumphouse and boiler house, but sufficient plant is available, after repairs, to service the Refinery on a limited scale. A considerable proportion of the tankage has been destroyed and extensive improvisation will be necessary, including the use of barges as storage, to overcome the acute shortage.

The rail and wharf handling facilities will also require much attention to put them in order again.

The coal handling facilities are also badly damaged and it will be necessary to provide for oil firing in the initial stages.

However, the distillation equipment both atmospheric and vacuum has sustained comparatively little damage and the asphalt air blowing plant could be restored for operation without serious difficulty.

In addition the laboratory, which included extensive glass blowing facilities has been badly damaged, but the engine testing equipment was already dispersed to Hitzacker and escaped injury. (See Report No. 6.)

It is stated that ample spares are still available and the Management are confident that the refinery could be commissioned on a reduced programme after about 4 month's work.

From the appearance of the Refinery this estimate must seem excessively optimistic, but it is obvious that considerable experience in the rapid repair of bomb damage has been obtained by the refinery engineering staffs, for which due allowance must be made.

<u>Personnel Interrogated</u>

Dr. Stegemann	-	Refinery Manager	} Harburg
Mr. Titschack	-	" Engineer	}
Dr. Lutkemeyer	-	Technical Director	Shell House
Mr. Maercklin	-	Chief Engineer	" "
Mr. Becker	-	Asst. "	" "
Dr. Zeigs	-	Asphalt Department.	

<u>Date of Visit & Party</u>

<u>16th May, 1945.</u>

Mr. P. de H. Hall	-	Brit: Ministry of Fuel & Power
Mr. Donald S. Fraser	-	U.S. Petrol: Admin: for War
Mr. C.A. Harrison	-	Brit: Ministry of Fuel & Power

(Sgd.) C.A. HARRISON.

A P P E N D I X "A"

HARBURG REFINERY

Synthetic Lubricating Oil Plant

GENERAL

The synthetic lubricating oil plant of the Rhenania-Ossag was situated at Harburg, but after air-attack was removed to Osterode for re-erection. The plant was commissioned in 1941 and reached its maximum production in 1943.

The plant was designed for an output of about 400 tons/month, but actually reached 700 tons/month. The process was the normal one of wax cracking followed by aluminium chloride polymerisation of the resultant olefines.

DESCRIPTION

The process was not very selective as regards original wax and the actual material used, which was the wax from Austrian crude, contained from 25% up to 30% of oil.

The wax was cracked in the vapour phase at a temperature of 560-590°C. and gave 60% of cracked distillate of 30°-310°C. boiling range, which had a bromine number of 80-110. Some of the wax was recycled, and there was a continuous withdrawal of 10-15% calculated on the fresh feed.

The catalyst used for polymerisation was a commercial grade of sublimed $AlCl_3$. This was pasted with lubricating oil (q.v.) and pumped into the reactors which were of the batch type. The process was operated at about 10°C. and the reaction took about 4-5 hours. The reaction was, however, more rapid if the olefines were produced from oil-free wax.

It was not necessary to dry the olefine feed specially, a sufficiently dry material being obtained by ordinary settling. The process was, however, operated under slight pressure to prevent ingress of water vapours.

The basis of the plant was a reactor tank with a centrifugal pump circulating the material through a heat exchanger which would be used first for removing the heat of reaction and later for heating the material towards the end of the process. The main material of construction was wrought iron, not steel or cast iron. Nickel-chrome - mainly V_2A or V_2A was used for valve trimming, impellors, etc.

Various methods of operation had been tried, the original process being to operate a series of 7-ton batches and processing down to a bromine number of about 2. This test was carried out by taking a sample of the reaction mixture and separating the catalyst out by water washing.

It was later found that better yields were obtained in the following manner:- 3 x 7-ton batches were started and run to a bromine number of 40, and then transferred to a larger but similar arrangement of about 25 tons' capacity. Circulation was continued for another 6-7 hours, the temperature being gradually raised to about 80°C. At a bromine number of 5, the sludge was allowed to settle and was drawn off to another vessel. Here the sludge was washed with water and the separated oil was returned to the 25-ton tank and recirculated while further polymerisation occurred and the bromine number was finally reduced to about 2. This was an important stage of the process as it increased the yield of high viscosity oil.

It should be appreciated that the local complexities of operating conditions were designed to give the best oil to meet urgent demands and that a balance had always to be struck between speed of operation, viscosity of oil and viscosity index.

There was no attempt at catalyst recovery, the spent material being discharged through the main separator system of the refinery.

The polymerised material, at a bromine number of 2, was settled for 20 hours and then drawn off to larger tanks for still further settling. The next stage in the treatment was to add 5% of refining earth and 1%

of lime, passing this material through a distillation unit to remove light ends, and filtering the residue. This would normally be of a pale yellow colour, but, if the original wax had been oil-free, could at best have been almost white.

The various grades of oil required were separated by vacuum distillation, giving essentially spindle oil, machine oils and a heavy lubricating oil residue, which was the synthetic oil required and was known as Grade 1006. This had the following properties:-

Viscosity at	50°C.	- 47°E.
at	100°C.	- 5-6°E.
Viscosity Index		- 100-110
Pour Point - about		- 27°C.

The yield on wax was about 40%, and the rather high pour point is due to light waxes in the original feed-stock; this is why the olefines were cut at 310°C, else the pour point would have been higher. The oil gave quite good results in the oxidation stability test (see Report No.1, Section 3). It was stated that any oxidation products of the oil were fatty acids, and that this would help the detergency.

The oil, according to the Intava and Rhenania testing laboratories will function for 17 to 18 hours in a ring-sticking test compared with 8 hours for normally refined mineral oil. When the latter is mixed in 50/50 b blend with the synthetic oil a ring-sticking result of 10 10-11 hours is invariably obtained.

Part of the machine oil fraction was used for pasting the aluminium chloride catalyst as mentioned above, and the spindle oil cut was generally returned to the polymerisation process, as it was found that this considerably helped to increase the yield of high viscosity oils.

DOCUMENTS

Monthly and yearly Refinery reports and a collection of Technical reports dealing with the manufacture of Synthetic Lubricating Oil were collected from Shell Haus and put in the document bags.

Personnel Interrogated Dr. Stegemann, Refinery Manager.

(Sgd.) D. MORTEN
W.H. THOMAS