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GERMAN PETROLEUM INDUSTRY
HAMBURG DISTRICT

REPORT No.4

DEUTSCHE VACUUM OIL.A/G

including

D.V.O. REFINERY. SCHULAU - Nr. WEDEL.

Reported By

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on behalf of the

BRITISH MINISTRY OF FUEL & POWER

AND THE

U.S. TECHNICAL INDUSTRIAL INTELLIGENCE COMMITTEE

JUNE, 1945

C.I.O.S. Target No.30/3.09

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

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

44750-1 (61)

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DEUTSCHE VACUUM OEL, A.G.

INTRODUCTION

Though specifically calling attention to the Schulau Refinery and Laboratory, this target would seem best dealt with in a combined report, which covers the activities of the Deutsche Vacuum Oel, A.G. at their two refineries at Schulau and Oslebshausen, as well as the Intava Laboratories at Schulau and the Engine Laboratory which was evacuated from Schulau to Bad Oldesloe.

SUMMARY

Section I - Schulau Refinery

The refinery plant is all old and of no technical interest, but a description of some of the more important products manufactured is given in the body of the report.

Section II - Oslebshausen Refinery

This refinery is considerably more up-to-date than Schulau, and the most interesting features were a DuoSol Extraction Plant combined with a Benzol-Acetone Dewaxing Plant for the manufacture of high grade lubricating oil.

Section III - Intava Laboratories - Schulau

Besides a certain amount of chemical research, a considerable volume of work was carried out on the application testing of lubricants of all kinds. There was a considerable amount of good quality mechanical and motor testing equipment.

Section IV - Engine Test Laboratory at Bad Oldesloe

The equipment evacuated from the Schulau Laboratories had been re-erected here, and was essentially for the purpose of running performance tests on aero-engine fuels and lubricants.

SECTION I

SCHULAU REFINERY

Date of Investigation - May 15th, 1945.

Names of Investigators

Mr. P. de H. Hall, (Brit.)
Mr. D. Morten, (Brit.)

Personnel Interrogated

Dr. W.Knudsen - i/c refinery operations, Hamburg Office.
Dr. Orlass - Plant Manager.
~~Dr. H. Ewers - i/c Grease Plant.~~

Description

The equipment is all quite old and of no technical interest. The essential operation of the refinery was the manufacture of industrial lubricants and greases. The plant consists of a bench of 6 shell stills operating without any fractionating facilities, agitators for light distillate treating, Nobel centrifugal plant for lubricating oil acid treatment, earth treaters, dewaxing by ammonia refrigeration, wax sweating house and a grease plant.

There are the usual compounding and barrel filling facilities and a small plant for the re-refining of used oil by acid and earth treatment. Though considerably damaged and short of tankage, the plant is able to operate with the exception of the wax sweating process, which was completely destroyed. The crude oil normally used came from the Reitbrook field and amounted to approximately 2,500 tons/month. The refinery could operate at a somewhat higher throughput when other crude oils were available.

Products

The industrial lubricants produced range in viscosity from 2.5 - 4.5° Engler at 50°C. A fairly complete range of industrial lubricants and compounds, as well as Calcium, Sodium and Lithium base greases was made, the more interesting among these products being described in some detail as follows:-

(i) Lubricating Compounds

(a) Cutting and Soluble Oils

A satisfactory cutting oil was made by the simple process of dissolving ½% sulphur at 20°C. in a light machine oil. Soluble oils were the usual type compounded with naphthenic acids, naphtha sulphonates, rosin, etc. The naphthenic acids were obtained by alkali treatment of distillates up to the spindle oil range, and were also shipped from the Oslebshausen works. The average acid value was extremely low, a figure of 50-60 being given. Naphtha sulphonates were purchased from Schindler and Schliemann, both firms being situated in Hamburg.

(b) Emulsion Oils

As a war-time expedient, to reduce the consumption of steam cylinder and marine lubricating oils, a 50/50 oil/water emulsion was manufactured by blending from ¼-½% crude Montan Wax together with ½% of blown rape seed oil into suitable oil bases at about 60°C. The wax and rape oil are added to the oil first, and the water slowly added while stirring. It was also possible to make a substitute grease using 5-7% of crude Montan Wax which was said to be suitable for light duty. The only important point in manufacture was that the material must be stirred down and filled off at a temperature not over 20°C. It was also stated that a small addition of Montan Wax conferred considerable water resistance on sodium base greases.

(ii) Greases

The grease plant was of the open kettle type and no doubt, while operating as an art rather than a science under the control of Dr. Ewers, gave excellent results. There was no point in collecting a mass of formulae for greases, as the whole operation was based on what fats could be obtained from time to time, and there was no question of selecting materials except for the manufacture of lithium base grease, the composition and process of manufacture of which will now be described in some detail.

Intava Instrument Grease - (Colour - Rose)

Formula:
- 4.10% wt. Prime White Stearine (Neut. No. - 210)
- 0.77% wt. Montan Wax Special
- 93.39% wt. Refined Oil (1.6°E. @ 50°C.)
- 0.74% wt. LiOH (Solid 60%)
- 1.00% Glycerine or Glycerogen from I.G.

Method of Manufacture - to make a 1,000-kilogram batch.

About 20% of the oil is heated to 80-90°C. with the stearine and Montan wax, then add the LiOH and about 5% of water. Heat to 140°C. in the open kettle for 2-2½ hours, then add the rest of the oil and heat to 180°C. for 1½-2 hours. Add the Glycerine and run out hot into iron pans, giving a layer about 2½" thick. It will be noted that the loss of water would continue during the processing, and that the final heating would give the necessary complete dehydration. The material is then cooled for 16 hours till it reaches a temperature of about 30°C. and passed through a shredding machine on to a twin-roll homogeniser, which breaks the gel structure. The rolls were of steel and running practically touching at a differential speed of about 2 to 1. It was stated that the thousand-kilogram batch could be passed through in about 2 hours. The material was of excellent appearance, though this was helped by the addition of the dye, and had the following properties:-

Flow Point - 160°C.
Drop Point - 165°C.

Production was about 35 tons/month, and this was nearly all filled into one-kilo packages.

SECTION 2

OSLEBSHAUSEN REFINERY

Date of Investigation - May 22nd, 1945.

Names of Investigators

Mr. P. de H. Hall, (Brit.)
Mr. E.H. Boomer, (Cndn.)
Mr. Paul K. Kuhne, (U.S.)
Mr. C.H. Barton, (Brit.)
Mr. C.A. Harrison, (Brit.)
Mr. D. Morten, (Brit.)
Mr. W.H. Thomas, (Brit.)

Personnel Interrogated

Dr. H.C. Goetz - Director - Hamburg Office
Dr. W. Knudsen - i/c Refinery Operations - Hamburg Office
Herr H.D. Rueschmann - Works Supt.
Herr. H. Harms - Head Office representative in Bremen
Herr Kuchta - Works Chemist

Description

This plant was relatively little damaged, but was nevertheless prevented from operating by the destruction of the condensers and tail house of the primary distillation unit.

1. Tankage available, 100,000 cubic metres.

2. Foster Wheeler Atmospheric Still. About 15 years old. This had a capacity of about 10,000 tons per month and would take about six weeks to repair.

3. Vacuum Pipe Still. Capacity is about 3,500 tons per month. This was used for lubricating oil redistilling and was designed only to run to heavy fuel bottoms and not asphalt. Repairs could be completed in about 8 weeks.

4. DuoSol Unit. This had been damaged by bombing in 1943, but had been repaired and was only short of one propane storage tank. Also propane itself was

not available. The capacity of the plant was 180 tons a day, which, on the basis of a 26 day month, gives an intake of 4,700 tons per month. The unit was erected in 1938 and it was designed by Max D. Miller in the United States.

The chief interest in the unit lies in that it has a 2-stage extraction process, the first stage giving half Raffinate, this, in the second stage, being converted into high V.I. and low V.I. lubricating oils. There is a very sound technical reason for this method of operation in that certain of the crudes used are of such a hydrocarbon composition that, besides paraffinic oils, a good quality naphthenic oil can also be separated. The solvent ratios used were as follows:-

Stage 1 - 4 volumes propane, 1 volume Selecto.

Stage 2 - Add a further 1 volume propane and 4 volumes Selecto.

The Selecto is 50/50 phenol/cresol mixture.

The high V.I. oil from this type of 2-stage extraction process is so well refined that a hot contacting process was not required for reduction of colour. The oil was, however, given a low temperature contact treatment at 80/90°C. with about 1% of earth merely to satisfy official requirements which originally called for earth treatment.

During the war about 10% of used aviation oil was added to the Duosol feed. This appeared to be the method of operation which had given rise to a report that the DuoSol process was being used for the prime purpose of recovering aviation oils containing Voltol.

The plant is, as expected, well engineered, and largely fabricated in Germany. The instruments are of German, French and American origin, but patterned on well known types, such as Leeds-Northrup and Foxboro, and presumably made under licence. The extraction sections consisted of two vessels for each stage, each stage containing 7 sections.

An interesting operating detail is that the solvent recovery furnaces, of which, of course, three are necessary under this means of operation, are fired by pulverised coal and this was found to be very controllable, giving excellent heat distribution. Naturally it would be more economical to run on coal, but firing by this means was preferred by the plant operators and, on occasions when oil was being used, the operators would revert, at the first opportunity to coal firing.

5. Solvent Dewaxing Plant. This was in complete operating condition and had an intake capacity of 3,000 tons per month. The solvent used was Benzol 65%. Acetone 35%. The chillers were of the usual Carbondale type, 4 made by Borzig and 2 by Wegelin and Huebner. These latter had a rather elaborate gear method of driving the scrapers instead of the conventional chain.

The three filters, each of 33 sq. metres area, were made by Dorr Oliver and give 25% of oil in the wax cake. It was stated that the cake was formed 5/8" thick. By repulping the wax the oil content was reduced to 8%.

The plant worked very well and was said to be easy to operate, but they were developing some modifications to the filters, so far only on the pilot scale design, to give a much thinner wax cake. The wax was sent to the Schulau refinery for further processing.

The intention was to operate the filters at higher speed and using more solvent, thus considerably stepping up the filter rate which could only be done by a complete removal of the wax from the cloth. This was to be carried out by an ingenious device described below.

There were no scraper blades on the filter, but rotating close, but not necessarily touching the cloth, was a perforated pipe drilled with a large number of approximately ¼" holes. It was stated that the wax would stick to the pipe, thus removing itself completely from the cloth, passing through the perforations into the pipe which became completely full of wax. The wax could be removed from the centre of this pipe by any simple method.

This would seem to be an interesting development and would obviate any wear on the cloth or wires due to the action of the scraper blades and would appear to be able to operate with filters which were not truly circular.

6. Power Plant. This was in operating condition and consisted of three modern German built Babcock boilers operating at 600 p.s.i. Weser river water was used but was Permutit treated on account of its high dissolved salts content. Electric power was generated by 2 pass-out turbines, one of which had been temporarily removed for safe storage, the exhaust steam being used in the refinery.

7. Staff. The normal staff was given as 330 labour, 55 staff. It is now 150 labour, 30 staff.

8. Fuel Consumption. The usual fuel consumption at the refinery is 3,500 tons per month. As indicated above, coal is the normal fuel as it is very much less expensive than fuel oil and only refinery residues were normally burnt.

9. Crude Oil. The Vacuum Oil Co. had their own production of crude oil and would normally supply to Oslebshausen the following crudes:-

Nienhagen (Hadensdorf, Haenigsen) - 2,500/3,000 tons/month.

Eickelohe - 500 tons/month.
Oberg - 650 tons/month.

DOCUMENTS TAKEN

A series of flow sheets on the refinery process were collected. In addition, some documents on lubricating oil specifications, which will complete information on oil specifications collected at Hamburg, were taken.

INTAVA RESEARCH LABORATORIES
SCHULAU

SECTION 3.

Date of Investigation May 15th and 16th, 1945.

Names of Investigators

Mr. P. de H. Hall,	(Brit.)
Mr. Vladimir Haensel,	(U.S.)
Mr. Paul K. Kuhne,	(U.S.)
Mr. E.H. Boomer,	(Cndn.)
Mr. D. Morten	(Brit.)
Mr. W.H. Thomas	(Brit.)

Personnel Interrogated

Herr Erik Zyamken - Director of Deutsche Vacuum Oel and Manager of Intava.
Dr. Knudsen - Refinery Manager.
Dr. Orlass - Assistant Manager.
Dr. K. Keital - Chief Research Chemist.
Dr. E. Brandt - i/c Intava Laboratory.

Description

Besides the necessary laboratory accommodation for maintaining control over refinery production there were additional laboratories in which Intava carried out chemical research as well as work on the application and testing of lubricants of all kinds.

Industrial Lubricants, etc.

The laboratory contained a number of test rigs for examining the practical behaviour of lubricants in ball bearings and roller bearings, and plastic journal bearings. The essential feature of these rigs was a strong shaft fitted with three bearings. The outer bearings were normally the ones under test, the load being applied to these hydraulically by pressure on the much larger central bearing. When plastic bearings were being tested, all three bearings could be under test. Arrangements were made to heat or cool the bearings as necessary. Drilling and milling machinery - which was, incidentally, of a much better class than that available in the main refinery workshop - had been installed to test cutting and soluble oils.

Automobile Laboratory

This was a well-arranged building, complete with chassis dynamometer, which was used mainly for testing the smaller range of automobiles up to about 40 B.H.P. A number of rigs were installed for testing brakes, gear boxes, springs etc., all being liberally fitted with thermocouples. Work on cold starting of engines and friction loss in gear boxes and axles at low temperature had also been carried out.

Aero-Engine Test Laboratory

This had been evacuated to Bad Oldesloe and is reported in Section IV.

Products Research Laboratory

The bulk of the apparatus in these laboratories was of good, but not of outstanding type and the standard methods of test employed were those issued by D.I.N. and A.S.T.M.

Viscosity was determined by means of conventional Ubbelonde tubes but the control of the constant temperature baths was only by the Jucheim contact thermometer which does not give the control necessary for the determination of viscosity Index to one or two numbers Generally, "Pole Height" (see "Umwandlungs-Tabellen fur Viskositätszahlen" by Dr. L. Ubbelohde - 1943. Publisher - S. Herzel Leipzig.) was used as a means of expressing the temperature-viscosity relationship because it was considered to be a more suitable method of expression.

Elementary analysis appeared to be in considerable use and an ingenious mechanical means for controlling the rate and time of heating of the furnace was demonstrated.

Sulphur was estimated by the Ter-Meulen Quartz-tube method and tricresol inhibitor was determined by a procedure involving the use of p-nitroaniline and potassium nitrate.

The normal chromate method was employed for the estimation of lead tetraethyl but the actual estimation of lead was by titration using sodium acetate.

For the testing of greases, a fluidity test was employed in which a plunger of known weight was caused to bear on a sample of worked grease and to extrude it through an orifice. The weight was increased until a specified efflux rate was obtained, the result being expressed as a weight in grammes.

In order to evaluate greases with regard to their behaviour at low temperatures (down to -60°C.) in the inner race of the roller bearing lubricated with the grease under test was rotated at constant speed at successively lower temperatures until the grease seized and exerted a torque on the outer race. This torque is plotted mechanically on a chart.

Dr. Baader's test for the tendency for sludge formation in turbine oils in the presence of glass, lead, copper, zinc, iron etc., was demonstrated. The apparatus consisted of a constant temperature bath holding glass containers of the oil under test, together with mechanical means whereby spirals of glass or metal were caused to move up and down, in and out of the oil for 72 hours at 96°C. Sludge formed in the oil was estimated in the normal manner and the spirals were weighed before and after the test. By subtracting the figures obtained for glass from those yielded by the use of the metal spirals, the sludging effects of the various metals could be determined.

Apparatus for the measurement of lubricating power had been constructed in line with that of Prof. Wolff at Halle Hochschule but was apparently still in the experimental stage. It consisted of a flat brass plate each end of which rested on the outer races of two identical roller bearings which were rotated in the same direction and at the same speed with their lower halves immersed in the oil under test. Wires were attached to the ends of the plate and were led over pulleys to support balance pans while a third balance pan suspended from the centre of the plate enabled a known vertical load to be applied at the contact between the plate and the outer races of the bearings. Horizontal resistance to movement of the brass plate was controlled by weights placed on one pan and the lubricating power of the sample was measured by the weight required by the other pan to balance the system when working.

No temperature control was provided and this would appear to be essential if the apparatus is to yield repeatable results.

DOCUMENTS TAKEN

A list of the documents taken is given in Appendix 1 of this report.

INTAVA RESEARCH LABORATORIES
ENGINE TEST LABORATORY, BAD OLDESLOE

SECTION IV

Date of Investigation - May 18th, 1945.

Names of Investigators

Mr. Vladimir Haensel, (U.S.)
Mr. Donald, S. Fraser, (U.S.)
Mr. Rene J. Bender, (U.S.Navy)
Mr. D. Morten, (Ministry of Fuel & Power)

Personnel Interrogated

Dr. Hans Wenzel and his assistants.

Description

The laboratory was designed for testing aviation fuels and lubricants. An attack on April 24th 1945 destroyed a considerable part of the laboratory and equipment, includinga C.F.R. engine, an I.G. engine, a cold testing engine and two D.K.W. lubricating oil test engines.

At the present time the remaining equipment consists of one D.V.L. test unit fitted with a B.M.W. cylinder for fuel testing and a single cylinder B.M.W. unit for lubricating oil testing.

The D.V.L. test unit was of the normal type using the B.M.W. No. 132 cylinder. No detailed report on this will be made, as the operation of similar engines has been covered by other parties.

The ring-sticking tendencies of lubricating oil were assessed using the same type of cylinder rather differently mounted. The engine was operated to give 60 B.H.P. at 1920 r.p.m., the plug level temperature being raised to 275°C, by reducing the cooling air; ignition was timed at 30° B.T.C. There were 15 litres of oil in circulation, and this was maintained at 110°C. Samples could be withdrawn every hour. The test was stopped when the load dropped by 2%, blow-by being usually indicated at the same time. The fuel used was aviation reference B4, and the reference oil was Intava Red Band. This gave very reproducible results at about 8 hours. 100% synthetic oil would often operate up to 20 hours, but the 50/50 mixtures normally used by the German Air Force were considered good at 10-12 hours. In order to avoid running the main engine for such tests small engines had also been installed. These were single cylinder, 2 stroke liquid cooled D.K.W.s, type EL.462, 88 mms. bore, 76 mms. stroke, giving a capacity of 462 ccs. It did not appear that this engine gave very successful correction with the single cylinder B.M.W.

Documents and Samples Removed.

Documents on test procedures and miscellaneous reports have been found and removed. Results on fuel and lubricating oil tests are buried under débris and could not be removed. It is not believed that these inaccessible documents are of much value in view of the fact that the sources and methods of production of the fuels and lubricants are not given on the test sheets. The samples were submitted by numbers and the testing personnel had no idea of the composition of the fuel.

A 50 liter sample (2 - 25 liter cans) of synthetic lubricating oil from POLITZ (Settin), a small sample of ET-110 (iso-octane) and a 50 liter sample of reference fuel for supercharge tests were removed from the premises.

APPENDIX I

INTAVA AND D.V.O. DOCUMENTS FROM SCHULAU

1. Bauvorschriften für Flugmotoren
   a) March 1938
   b) October 1940 (2)

2. Automatische Elementaranalyse

3. Deutsche Luftfahrtforschung
   a) Tagung, Klopfverhalten und Lagerung von Kraftstoffen
   b) Einfluss des Bleigehaltes auf Flugmotoren - Bauteile
   c) Einfluss des Bleigehaltes von Kraftstoffen auf Bauteile von Flugmotoren
   d) Die Wirkung der Peroxyde im Motor
   e) Kolbenringverkleben im Siemens Ölprüfmotor

4. Berichte über die Schmierstofftagung
   a) 1941 Dezember
   b) 1942, Mai

5. Zur Viskosimetrie, Die "Polhöhe" Methode

6. Die Struktur von Gelfetten und deren Änderung

7. Organisation chart, Wedel refinery of Deutsche Vacuum-Oel

8. Luftwaffe Specifications
   a) B 4 and C 3 (Nov. 1944)
   b) V 2 and Einlauf V 2 (April 1944)
   c) Diesel fuels (Aug. 1944)
   d) Aviation lubricating oils (May 1943)

9. Automatic Elementary Analysis (reprints)
   by Reihlen & Weinbrenner
   by Reihlen

10. Methods of test of aviation oils fixed by the Luftwaffe (Nov. 1943)

11. Stability to temperature and metals of turbine oils.