C O N F I D E N T I A L

GERMAN PETROLEUM INDUSTRY

HAMBURG DISTRICT

REPORT No.13

REICHSINSTITUT FUR ERDÖLFORSCHUNG

DER TECHNISCHE HOCHSCHULE

HANNOVER

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

FUELS AND LUBRICANTS

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

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

44750-1(133)

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SECTION 1. Conclusions

The Reichsinstitut has carried out a considerable amount of valuable fundamental research. The studies on selective solvents for the separation of wax from oil in liquid systems at ordinary temperatures and pressures show promise. The studies on refining of diesel oils from brown coal tar by hydrogen fluoride appears to be a useful and valuable process. Much of the other work done was of a minor character dealing with problems submitted by Government agencies and are in many instances unfinished. The work on flame thrower fuels and torpedo lubricants is not important. The work on oil production practice does not appear to add anything to present knowledge. The documents collected contain the essential records of the work at the institute in detail. Not all documents are reviewed.

SECTION 2. Historical

The Reichsinstitut was created as a research organization attached to the Hochschule as a part of the Four Year Plan. There were 91 employees in Hannover and 20 at Holzminden. The Institut reported directly to officials of Dr. Speer's ministry and received funds and direction from that source. The field of activities was very broad and included all branches of pure and applied science excepting biological sciences. Many problems arising out of the war were referred to the Institut, particularly by the Kriegsmarine research centre, Chemisch-Physikalische Versuchs-Anstalt at Dänisch--Nienhof.

The Institute was badly damaged in air raids and little work was proceeding there prior to surrender. Part of the Institute had been transferred to Holzminden, Dr. Dornow with twenty men.

Dr. Schneider, Director, was interrogated at Wellie and documents secured from him. Dr. Moos, Chemical Engineer, and Dip. Ing. Gottschall were interrogated in Hannover. Dr. Moos spent 1934-36 in New York in the employ of the Edeleanu Company.

SECTION 3. Description of Institute Work

a) Solvent dewaxing-

This work is the initial part of a broad problem involving the development of a new dewaxing process and of new pour point depressors. It was planned to develop dewaxing to an end result of a few

percent of wax and then develop pour point depressors for such dewaxed pils. A systematic investigation on the solubility of pure hydrocarbons in a wide variety of solvents has been carried on together with solubility studies of wax and oil mixtures in the same solvents. All work has been in liquid phases and generally at temperatures above the melting point of the wax components. Considerable success has been achieved in developing selective solvents applicable to the systems investigated. Many of the solvents are new components synthesized at the Institute. Hydrozine derivatives are particularly good in separating the wax content of an oil. A unique solvent mixture of 90% dioxane and 10% formic acid was very successful in extracting asphalt from oils when used in equal proportions with the oil at 5° C. The number and variety of solvents studied is very great and some general solubility relationships involving structure, temperature and oil-solvent ratios were developed. Detailed reports were obtained and deposited with C.I.O.S. Secretariat.

b) Raffination of diesel oil from Brown coaltar with hydrogen fluoride-

This work was carried out with the objective of producing a good quality diesel fuel from a low quality diesel fuel obtained by fractional distillation of brown coal tar. The work was quite detailed and a complete report obtained. The results are of value and offer promise as a raffination method. The effect of time of contact and ratio of hydrogen fluoride to oil was examined. The temperature used was always below the boiling point of the fluoride, 21° C., and usually about 18° C. The fluoride was technical grade, 96% HF or better. The time of contact had no great effect after 60 minutes. At shorter times, the yield of diesel fuel increased and the quality did not change markedly. The HF: oil ratio affected the yield and quality, a ratio of 1:2 being found advantageous. An example of the results is as follows:

HF:Oil = 2, 60 minutes, 18°C.

	Original Diesel Oil	Refined Diesel Oil	Lubricating Oil	Fuel Oil
S.G. 20°C.	0.9087	0.859	0.920	1.012
Av. B. Pt.	293°C.	285	-	-
Cetane No.	40	48	-	-
Pour Point	-7.5°C.	-9	+ 10	- 10
Iodine No.	83	28.6	46.1	-
S	1.22%	0	0.52	2.7
Conradson C	0.15%	0	0.21	-
Ash	0.14%	0	0.0	-
Net Kg Cal/Kg	-	-	-	9560
Viscosity	-	-	22.5 E/20	-
	-	-	4.1 E/50	-
Polhöhe	-	-	2.4	-

The extract (fuel oil) amounted to 41% and the raffinate 59%. The raffinate is vacuum distilled at 12 mm. to an end point of 220°C. to yield 32% diesel oil, and further distilled at 1-2 mm. to an end point of 360°C. to yield 25% lubricant, the residue and loss being 2%, basis original crude diesel oil.

Exhaustive raffination with HF produced diesel oils of very low pour point but the cetane number was affected adversely. The process worked well on any diesel cut from brown coal tar yielding about the same improved quality raffinate in each case.

Mild steel can be used in equipment fabrication and no corrosion difficulties arose in this laboratory work. In separating the two layers by discharge from the bottom of the reactor, the

interface was detected precisely by the change in electrical conductivity. Removal of hydrogen fluoride from the extract was brought about simply by evaporation.

In addition to the main problem described, the use of hydrogen fluoride as a refining agent was of some value in a naval problem of producing torpedo lubricants having a gravity greater than sea water. The application is in lubrication of electric driven, trackless torpedos.

Hydrogen fluoride is an excellent desulfurizing agent and operates to extract sulfur compounds from all oils including gasoline.

c) The detection of tertiary carbon atom compounds in hydrocarbon mixtures-

A useful color reaction for detection of hydrocarbons containing tertiary carbon atoms sensitive to 0.01% was developed. Briefly, 0.5 ccm of a 0.5 solution in CS_2 of $AlBr_3$ and 0.075 chlorosulfonic acid ester in CS_2 were added to 1.0 ccm of the oil under test. After five minutes at room temperature, the oil layer is removed and methanol is added, drop by drop, to the residue. An orange to blue color indicates tertiary carbon atom hydrocarbons. A bi-product of this work was the preparation of hydrocarbon mixtures free from iso- and allicydic compounds.

d) Flame-thrower fuels-

Work was carried out at the direction of the German Navy on substitute materials for rubber and soap as gelation agents in the preparation of flame thrower fuels. A substitute was obtained but not put into practice.

A topped crude oil was hot blown to produce asphaltic and acid bodies and an increase in viscosity. From 1 to 1.5% of metal salts and some naphthenic acid from tar oils were added. The mixture was heated. The resulting mass was very viscous and thixotropic. It was mixed with gasoline in a 3:2 ratio and the mixture was a satisfactory flame thrower fuel having gel like properties and

adherence. Addition agents improving flammability and temperature were sought. Butanol peroxide was found to be very useful in this regard but too costly.

e) Lubricants for optical apparatus-

A problem referred to the institute required the development of lubricants for periscope mechanisms and the like that had no effect on the glass parts. It had been found that vapours from usual oils deposited on the glass surfaces with detrimental results. The desired result was not achieved by new oils but by careful and exhaustive evacuation of the usual oils.

f) Compass fluids and hydraulic fluids-

Work was carried out on the development of fluids of low freezing point and with zero temperature coefficient of viscosity.

One result reported, but not reproduced, showed a solution of 1% polyisobutylene of molecular weight, 100,000, in benzene to have a constant viscosity of 25 centistokes between 20 and 50°C. Another useful fluid was 5% polyisobutylene (M.Wt. 15000) in iso-octane. The final fluid used a narrow boiling kerosene fraction in place of iso-octane. The viscosity of these fluids changed relatively little with temperature.

A useful hydraulic fluid was developed. It was 47.5 parts glycerine, 50 parts water and 1 part sulfonated rizinusöl. (castor oil).

g) Improved performance of internal combustion motors by exhaust gas recycle-

Studies were made on improvement of efficiency of motors by recycle of part of the exhaust gas to the air intake. Results were good.

h) Miscellaneous-

A large number of other problems such as synthesis of alkylated cyclo-hexanes (1.1. 3.3. 5.5

Hexamethyl cyclohexane), vapour lock studies, steam engine lubricants, substitutes for phosphorus in incendiaries, coloration of incendiary flames, high pressure lubricants, paraffin emulsions, etc., were in progress or complete. The following list of documents collected from the staff of the Instituteindicates the range and type of work.

SECTION 4. Documents from Reichsinstitut für Erdölforschung der T.H., Hannover

1. Reichsberichte für Chemie, June and July, 1944.

2. Berichte über die Schmierstoff-Tagung, December, 1941.

3. a) Refining of diesel oils with Hydrogen fluoride.

   b) Treatment of pure hydrocarbons with hydrogen fluoride.

4. Marine lubricating oil specifications.

5. Patents:- liquid level in oil wells, used lubricants and greases, refining of oils with formic acid, rotary well drilling unit, production of halogenated ketones.

6. List of personnel of Reichsinstitut.

7. List of proposed researches at Reichsinstitut.

8. Recycle of exhaust gases in internal combustion motors.

9. Refining of oils by selective solvents for wax removal.

10. File containing the following items:

   a) Treatment of torpedo lubricating oils.
   b) Flame-thrower fuels.
   c) Determination of vapour lock temperatures in aircraft motors.
   d) Oil well drilling turbine details.
   e) Production of highly alkylated cyclo hexanes.
   f) Steam engine lubrication.
   g) Vapour lock.
   h) Emulsion lubricants for team engines.
   i) Fuels and lubricants: reports for Oct., Nov., Dec. '43, Jan.-July inclusive '44.

j) Vapour lock in fuels.
k) Short reports on refining with HF.
l) Solvent extraction of oils.
m) Coloration of flames.
n) Detection of iso and allicyclic cpds. in saturated hydrocarbon mixtures.
o) Flash-vaporization studies.
p) Ultrasonic waves in production of high pressure lubricants.
q) Preparation of hydrocarbon mixtures free from iso-paraffins.
r) Iso-therms and iso-valens in tube furnaces.
s) Critical pressure determination.
t) Crude oil-gas solutions.

1 file containing the following documents (M-2):

a) Correspondence on deparaffination, properties of diesel fuels, vapour lock, proposed researches on coke oven gas.
b) Emulsion lubricating oils.
c) Report on properties of lubricants.
d) Lubricants for steam engines.
e) Pure oil lubricants.
f) Compass fluids.
g) Torpedo lubricants.
h) Purification of used lubricants.
i) Hydraulic fluids, correspondence.
j) Hydraulic fluids, reports.
k) Wireless transmission of oil level measurements.
l) Flame-thrower fuels.
m) Oil-shale deposits.
n) Gas-oil ratios in producing formations.
o) Analysis of Pacura crude oil.
p) Use of topped crude oil as diesel fuel.
q) Rotary chains in drilling.
r) Crude oil as diesel fuel.
s) Lubrication of steam engines.
t) Production of greases suitable for optical equipment.
u) The utilization of coke oven gas.
v) Flow-meter measurements on oil.
w) Flow measurements at oil wells.
x) Four-ball lubricant testing machine.

y) Safety fuels.

Date of Visit & Party

23rd. to 26th. May 1945.

Mr. E.H.Boomer	(Can.)
Mr. V. Haensel	(U.S.)
Mr. Donald S.Fraser	(U.S.)
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