

FILM STUDY GROUP
REPORT ON MICROFILM REEL NO. 23

Prepared by

BUREAU OF MINES

ABSTRACTS OF REEL 23--A

Bag No. 3424

T.O.M. Reel No. 23

Item
No.

- 4 Operating sheets on coal hydrogenation. Years: 1940-1941. Month-to-month report on operation, figures on raw materials used, products obtained, utilities. Apparently of little value. (74 pages).
- 5 Same as Item 4. No technical information (74 pages).
- 6 Notes on Catalyst I.G. 6719 and 6434.

The feasibility of the use of a certain catalyst for coal hydrogenation is discussed. Catalyst 6719 is of the following composition:

75 percent	FeS
22 "	WS
3 "	NiS

For the time being (letter dated April 6, 1940) this catalyst gives the optimum results. Catalysts containing over 30 percent or less than 22 percent WS are inferior to the catalyst described. The limits for NiS are 2-4 percent. More unfavorable catalysts result when the Ni is replaced with Co, Cu, or Zn. The operating temperature is between 21-23 m.V. A detailed description of the catalyst production is given.

Catalyst 6434 is composed of Terrana (not known to abstractor) with about 10 percent WS₂ and approximately 8 percent HF.

The preparation of this catalyst is described in detail. The regeneration of the used catalysts is discussed. The used catalysts are crushed and roasted in a furnace at a maximum temperature of 550°C. The ground mass is then treated with 2 percent HF and ammonium sulfide, and treated from there on as the original mass used as starting material.

- 7 Automatic hydrogenation plant controllers. Total of 23 pages; several drawings and sketches are included, showing details of the mechanisms used.
- 8 25 pages of operating sheets and drawings. The first sketch represents the gas preheater (economizer-type) for chamber 15. The drawing gives several views of the apparatus; finned tubes are used as transfer agencies. Thermocouple locations are given. The second sketch shows a flow scheme of a hydrogenation unit (chamber 15) with thermocouple placements. Several other sketches are presented with thermocouple locations.
- 9 Six flowsheets of tar, coal, and benzene stalls.
- 10 Eight drawings. Four drawings refer to a separator unit. The exact details are not clearly discernable. The other four drawings pertain to a spring-loaded expansion (needle) valve.

11 Oil deposits, processing, and important oil companies in Bohemia, Moravia, Slovakia, and U.S.S.R. This information consists of 78 pages.

Many maps are presented showing sites of oil production, refineries, natural gas deposits, storage places, etc. Furthermore, geological data are given on the various oil fields and their environment. Production figures are presented too. There are many maps of the U.S.S.R. which also show production other than oil.

12 A pencil flowsheet of the Leuna alkylation plant.

13-15 A few flowsheets on phosphoric acid production and polymerization of isobutylenes; also a flowsheet of Marsol with SO_2 and flowsheet for production of Marcolat 30.

16 Fifteen pages on the subject of separation of aliphatic alcohols from associated hydrocarbons by extraction with aqueous CH_3OH . This is written in the form of a report paper covering some descriptive matter as for instance, apparatus construction, as well as physical data, such as the solubility of alcohols and hydrocarbons of the Synol fraction in aqueous CH_3OH .

17 Flowsheet of propane oxidation plant.

18 Construction details of DHD reactors.

19 Flowsheet of Leuna DHD plant.

20 A 4-page letter having to do with an agreement between Erabag and Ammonia-werke-Merseburg on the procurement of butane.

21 The constitution of Mesasin-sulfonates. A 12-page article written in report form. No descriptive matter.

Conclusions: When high-molecular aliphatic hydrocarbons are treated with sulfur chloride, all the possible isomers of monosulfochlorides are formed in approximately equimolar quantities.

The rule of Hass and Weber was found to apply to this operation.

By saponification of the sulfochlorides with alkalies, monosulfonates of the type Mesapon N are formed.

22 A 14-page paper on the Sulfo-chlorination of Isobutane. The following conclusions were reached:

1. When isobutane is chlorinated in CCl_4 solution, no matter what the conditions may be, only a monosulfochloride is always obtained. The formed compound is the primary isobutane monosulfochloride.

2. So far no disulfochloride was detected.

3. A subsequent sulfochlorination of the primary isobutane monosulfochloride in CCl_4 solution only yielded a mixture of chlorosulfochlorides. From this mixture no disulfochloride could be eliminated.

23 A new method of oxidation, using ozone. This is a 13-page report.

It is chiefly concerned with the conversion of olefines into carboxyl acids, also applying ozone treatment in order to locate the position of double bonds in olefinic structures. The report states: Of the methods described in the literature dealing with the conversion of olefines into carboxyl acids by ozone none are quantitative and they may not be used for the determination of the location of individual double bonds in mixtures of olefines.

It could be demonstrated that a quantitative oxidation with ozone is possible without any side reactions to occur, if the ozonite is treated with an alkaline suspension of silver oxide.

- 24 This is an 18-page report on the splitting of chlorine from high-molecular weight aliphatic alkyl chlorides without displacement of the double bond. A large number of hydrocarbons are considered in this paper.
- 25 A 9-page report on a special type Mesamoll. This paper apparently has to do with the processing of Mesamoll which, it appears, is a softening agent for "Agelit P.G.U."
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- 26 Constitution and distribution of products obtained by sulfochlorination of n-butane in CCl_4 solution. This is a 20-page report. It contains freezing point curves for the systems:

1.-- N-Cyclohexylbutane (1) sulfamide
N-Cyclohexylbutane (2) sulfamide

and 2.-- (1,3) Butanedisulfochloride
(1,4) Butanedisulfochloride

Under proper conditions for monosulfochloride formation, approximately 84 percent of monosulfochlorides form; 67 percent of these are butane-2-sulfochloride and 33 percent are butane-1-sulfochloride. In all cases and under any conditions, two, thus far unknown, isomeric disulfochlorides form - namely the 1-4 butane disulfochloride and 1-3 butane disulfochloride.

- 27 The formation of disulfochlorides by the sulfochlorination of high molecular hydrocarbons (semi-sulfochlorination and total sulfochlorination). This is a 15-page report, and is very similar to the work described under Item 26.

Semisulfochlorination of the high molecular hydrocarbons leads to about 85 percent monosulfochlorides and 15 percent disulfochlorides.

When total sulfochlorination is resorted to, the percentage of disulfochlorides formed increases sharply. As much as 40 percent of disulfochlorides are obtained.

- 28 A 20-page paper having to do with the preparation, properties, and comparative capillarity investigations with 8 hexadecanes derived from sulfonates and sulfates. This work deals with the production of new wetting agents which can be used as a substitute for laundry soap. They found that cetyl-alcohol-sulfate has good possibilities for such a job. They described results of various tests when different wetting agents were used on different kinds of material.

Item
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1. Inspections of knock ratings of typical DHD products. This is a paper of 15 pages, 9 of which are graphs. A total of 6 samples were investigated. The following main tests were made:

Specific gravity	Composition
Octane number, with and without lead.	Degree of saturation
Aromatics	ASTM boiling range, etc.

The graphs relate mean effective pressure (Nutzdruk) and fuel-air ratio.

2. Correspondence on catalyst requirements and manufacturing DHD. A series of 15 letters and office memoranda containing information apparently discussed in conferences between men at Ludwigshafen and Leuna. No particular technical information is presented.
3. Operating instructions for DHD plant, Leuna, 6 pages, giving a very complete description of starting up-operation of DHD chamber No. 1.
4. Report on visit to the hydrogenation plant at Poelitz. This report comprises 4 pages, giving information on the Poelitz set-up, its operation and production.
5. Report on visit to DHD plant, Ludwigshafen. Seven pages of very much the same type of information as was disclosed in Item 4.
6. Catalyst discussion. A 2-page memorandum on details having to do with policy and administration chiefly.
7. Conference on separation of aromatic gasoline from DHD bottoms. Four pages, giving the results apparently of a discussion on above subject. Four different methods of accomplishing the above mentioned operations are mentioned. Each method is supplemented by a rough flow sketch.
8. Work-up of DHD products. A total of 13 pages having to do with possible product evaluation; starting material, final material as well as details pertaining to local plant conditions.
9. Operating conditions for new reactor system at Poelitz. 14 pages giving information on operating cycle and cleaning cycle. Discusses starting-up operation, operation proper, new features of the apparatus, experiences, etc., as well as general chemical principles.
10. This 14-page report deals in the production of high antiknock gasoline by dehydrogenation of heavy naphthas. Among other things, such as file memos, the following points are brought out:

I. High efficiency fuel in general.	VI. Thermal conditions.
II. Order to Leuna.	VII. Scheme of the Leuna plant.
III. Chemistry of the dehydration.	VIII. Product flow.
IV. DHD supports for Leuna.	IX. Mixture with high-efficiency fuel.
V. Course of the process.	X. Properties in the engine.

A short elaboration follows each topic.

11. Four pages giving information on the DHD production. Deals with catalyst problem, the possibility of using sulfate and aluminate clays are discussed, and it also speaks about other catalyst features, such as stability, apparent density, pelleting, etc.

12. Thirty pages dealing with high antiknock fuel status at end of December 1940. Discusses the various ways which Germany used to get a hold of its fuel. Some of the information is difficult to read.
13. Experiments with 100-liter reactor. Report No. 6, 12 pages, some unintelligible. Graphs are included. Detailed operating data are given.

14. Same as Item 13, 15 pages.
15. Forty-five pages of patent applications on organic chemicals, such as production of antifoaming agents, high molecular sulfur-containing compounds, production of unsaturated compounds, polymerization of olefins, recovery of oxygen-containing organic compounds as well as non-oxygen containing compounds, sulfamido compounds, salt-deficient sulfonates, plastic masses, and sulfonic acid esters.
16. Patent applications in processes. Eight pages on process of separation of aromatic hydrocarbons from hydrocarbon mixtures. Process of purification of non-aqueous liquids.
17. Patent applications, apparatus. Twelve pages dealing with instruments designed to measure pressures and quantities depending upon pressure absolutely free of any errors possibly introduced by change in temperature.
18. Horizontal coal hydrogenation converters. Twenty-six pages of drawings and descriptions showing the arrangement of horizontally placed converters. The reason for placing the converters horizontally (actually inclined at a small angle) was to allow the equipment to be camouflaged below thickly-leaved trees.
19. Twenty-five pages of letters and some photographs of steel samples, apparently to be contracted from Krupp. No particular interest.
20. Some 120 pages of material dealing with lubricating oil specifications and properties. A lot of the information is sporadic and only contained in letters. A large section is difficult to read.
21. Fourteen pages dealing with procurement of material chiefly, and administrative details in relation to a new lube oil plant to be constructed.
22. Lube oil conservation. Five pages of some small miscellaneous matter pertaining to various cylinder oils.