FILM STUDY GROUP

REPORT

T.O.M. REEL NO. 70

Prepared by

SHELL DEVELOPMENT CO.

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Shell Development Co. Emeryville, Calif.

### Abstracts on Reel 70

## U. S. Technical Oil Mission Microfilm

Abstracts Bags 1477, 1478 and 1481

Item 4A p. 2-15 Production figures for primary Fischer products for 1943.

Item 6A p. 17-24 Production figures for primary Fischer products, January to July, 1944.

Item 70 p. 26-175 Synthesis catalyst shipments from Ruhrchemie, Oberhausen-Holten.

Item 80 p. 177-278 Raw material requirements and material balances for synthesis catalyst preparation from 1936 to 1944.

Item 12E p. 281-288 Construction schedules or programs, about 1942-3

P. 291-376 Monthly summaries of Fischer production, shipments and stocks are given for Steinkohlen-Bergwerk, Rheinpreussen, Homberg (Niederheln), 1944. Subjects: Gasoline production, gasoline shipments mixed diesel fuel production and shipments, light diesel fuel, gatsch, hard paraffin liquified gas production and shipments, lamp oil, Kogasin, butanol (Sektol II), propanol (Sektol I), Dilenol, propyl ether (teck.) fusel oil, crude acetone, methyl ethyl ketone, alcohol consumption, aluminum alcoholate plant, Plant IV (lub. oils). Plant V: Grease production, crude products and their disposal, alcohol plant, paraffin disposal, detergent production, motor alcohol, benzene tar-oil, wash oil, H2SO4 consumption, catalyst consumption, crude adsorbant consumption fatty acid concentrate, phenolate concentrate iron granules, glycol, aluminum hydroxide paste, syrup, H2SO4 consumption (by plants), finishing adsorbant consumption, 45% NaOH.

Item 16A

Daily reports on production, shipment, storage of gasoline, light diesel fuel, mixed diesel fuel, kogasin and other p. 380-447 Fischer products including alcohols and propyl ether, January to July 1944.

Item 17A p. 449-461 Operating summary for the motor fuel plant for 1942,3. some monthly reports from January to October, 1944.

Item 19C p. 463-468

Summary (in English) of the production of water gas and reformed oven gas at Homberg (licers).

p. 470-477

Production charts: throughput of synthesis gas, weight of liquid + "gasol" per volume synthesis gas, weight of liquid per volume synthesis gas.

p. 478-544

Miscellaneous daily summaries of material requirements for the generators, cracking plant, converters, gas purification. Also catalyst requirements and production of primary products. Also A-K plant, alcohol plant and products. April to October, 1942.

Item 24B p. 546

Detailed drawings of Rheinpreussen synthesis gas plant after rebuilding. July 28, 1938.

Item 25B

Description and detailed drawings of the construction of the p. 548-554 heat-recovery apparatus of the water gas generators at Rheinpreussen and geographical layout of plant.

Item 26D p. 556-564 A detailed description of the operation of the thermal cracking plant to be delivered by Wilke Werke AG to Rheinpreussen. Also a description of cracking furnace-3. Also an index to drawings which are however not included.

Item 42A p. 567-584

Monthly tabulations of yields and inspections of Fischer products and tabulation of equipment and capacities for: Essener Steinkohle. Hoesch Benzin GmbH (Mittledruck), Gewerkschaft Rheinpreussen, Krupp Treibstoff GmbH, Gewerkschaft Viktor.

Item 51A p. 587-594 Calculations of the cost of making Fischer products based on the experience during July, August, September, 1943

p. 595 600 (not legible)

p. 601-604 Cobalt losses in practice at Rheinpreussen.

Item 56E p. 606-607 Production of solid soap from liquid fatty acids and dry calcined sodium hydroxide.

p. 608 (See also page 714)
Preparation of water emulsion paint using Pantoxyl.

p. 610 Graph of acid and saponification number vs. time for "Parestol".

Item 60C p. 612-631 Directions for inspection of Fischer gasoline and  $C_3$ - $C_4$  fractions. 5/9/41.

Item 62E p. 633-634 Numbers and titles of patent applications filed by Rheinpreussen -Moers, September, 1943 to September 1944.

Item 66E (not legible)

Item 70E p. 637 Diagram methods of converting coal and possible products.

Item 75I
p. 639-665

(Stettin-Politz hydrogenation plant 12/1/1944)
Reconversion of reactors to thermal cracking of petroleum residue.
Stabilization stripped products under pressure. Independant operation of plant units. Use of sulfur containing fuel gas (Leuna 12/14/44). Rapid discharging of inflamables from plant components for fire prevention after air attacks. (Braunkohle-Benzin A.g. 12/5/44). Substituting canals for water and drainage piping to minimize difficulties after damage by air attack.

p. 666-669 (Administrative documents regarding secrecy and policy for emergency supply of fuel to military.)

Item 79H
p. 671-68l Technische Hochschule Stuttgart report on design of multipurpose test engine including description of design, photographs and cross-section drawings.

Item 84E
p. 682-683
Di-aluminum hydroxide as possible thickening agent to replace starch in printers ink for textiles.

p. 684 Fragmentary notes on utilization of C7-C12 alcohols. Homberg 11/16/44.

- Inquiry on "Symalin" (Rheimpreussen) as salve base. 1/23/45. p. 684
- Notes on contemplated delivery of Rheinpreussen fatty acid to p. 686 · Schering A.G. Krupp treibstoff, 8/15/44.
- Notes on defective paraffin shipment. Markische Seifen-Industrie. p. 688-689 11/15/44. 1/18/45.
- Conversion of pure alcohol plant for the production of higher p. 690-691 alcohols. 10/8/43
- Converting mixer for higher-alcohol production. 11/27/42. p. 692-693
- Note on DRP 680069 (not legible) 7/10/44 p. 694
- Conference of all synthesis plants at the Bergbauverein on p. 695-698 November 25/42 on the manufacture of soap from fatty acids from synthetic oil plants.
- Comment on high water content in a soft wax shipment. p. 700
- Item 85E
- (Difficultly legible) Oxidation of hard wax. p. 703-707
- Graphs for large-scale experiment oxidizing wax showing acid p. 708-713 and saponification numbers with time.
- Details of using Pantoxyl as paint binder. p. · 714-716
- Summary of the investigation of high-molecular-weight paraffin p. 717-718 oxidation products. Tables show acid number, saponification number, per cent fattt acid, molecular weight, per cent unsaponifiable material, per cent carbon, per cent hydrogen per cent oxygen for the oxidation products.
- DRP 554 621 K122 (I.G. 8/8/30) Method of producing aqueous p. 719-720 suspensions of pigments.

DRP 552 624 Kl22 (I.G. 12/14/28) Method of producing aqueous suspensions of pigments.

Belg. 379 395. Production of paint and impregnation materials.

DRP 564 922 KL 45 (FG. 11/10/32). Method of producing emulsification agents.

DRP 576 003 Kl 12 (IG. 4/13/33) Method of oxidizing hydrocarbons (using nitrogen oxides).

Item 86E

Item 86E

p. 723-736

Detailed report on the oil shale industry in Estonia, and recommending expansion giving data of Motor Fuel Plant 2/3/42

Item 88A

p. 738-740

Prospective and actual production of motor fuel at Scholven, Gelsenberg, Victor Rauxel, Rheinpreussen, Ruhrbenzin, Krupp, Essen. Steinkohle, Hoesch.

Item 92C p. 742-747

English notes of memo from Dr. Steinbrecher (Ruhland 1/5/38) on influence of synthesis gas composition on product yields, catalyst life etc.

p. 748-762 Original memos of above

Item 95C

p. 763-770

Regeneration of cobalt catalyst by hydrogenation of adhering hydrocarbons.

- 1. Catalyst paraffin was not completely removed.
- 2. Part of the catalyst paraffin was cracked.
- 3. Apparently new especially high molecular weight paraffin (wax) was formed.
- p. 771-780 Paraffin removal from Fischer catalyst by treatment with aqueous alkaline solutions at temperature above the wax melting point.
  - 1. Boiling with aqueous 5% Na<sub>2</sub>CO<sub>3</sub> at atmospheric pressure removed 65 to 75% of the paraffin extractable with benzene. NaOH was no more efficient and removed excessive quantities of SiO<sub>2</sub>.
  - 2. Fine particle-sizes were more difficult to clean.
  - 3. The most efficient results were obtained using 3 parts alkaline solution per part of catalyst.
- p. 781-782 Controversy between Rheinpreussen (Moers) and Ruhrchemie relative to the condition of a shipment of catalyst.
- p. 783 Testing Fischer catalyst by determining the relative absorptions of CO and H2 for a number of temperatures from 50 to 200°C.

  The details of the method of Hoesch-Benzin are given.
- p. 784-798 Unidentified curves of equilibria between some oxides, probably iron, and CO2 and H2O.
- p. 799-810 Miscellaneous analytical data for elementary composition of Fischer catalysts.

- p. 812-820 Disgussion of catalyst transportation problems.
- p. 823-825 Review of the research results of the chemists at the motor fuel plants for the year ending September 29, 1943.
- p. 827-830 Work Program of the Exploratory Laboratories.
  - 1. Dehydrogenation of hydrocarbons (butane to C<sub>3</sub> and C<sub>4</sub> olefins) over present chrom-alumina catalysts.
    - 2. Ketone formation from alcohols.
    - 3. Processing synthetic resins.
      - a) Production of ethyl methacrylate.

b) β-methyl-a-keto butanol.

- c) Synthesis and purification of methylisopropenyl ketone and its "plexigum"—like products of polymerization.
- 4. Development of the ketone resin "Emekal" from methyl ethyl ketone and formaldahyde to production of 4 tons/month.
- 5. Synthesis of sec-butyl stearate for use as plasticizers.
- 6. Synthesis of aluminum alkaholates plasticizers (Butal & Propal" and their utilization (pyrotechnical, lacquer and medical.
- 7. Development of CO<sub>2</sub> additive products of Al alcoholate, specifically of "Butal"
- 8. Production of active Al hydroxides and oxides from Al alcoholates and their technological and medical utilization as well as the development of an experimental plant for Al hydroxide and oxide production.
- 9. Development of a new gelation agent (Rh6) from Butal and our C6 to C9 fatty acids as well as the Wittener Process fatty acids.

Utilization of these salts of fatty acids for purposes of "Wa Pruf 5 Ic"

12. Development assignments of "OKH; Wa Pruf 5" (Various pyotechnical subjects are outlined).

# <u>Lab 2</u>

- Synthesis of the esters of acetic, propionic and butyic acid with propyl, butyl and amyl alcohols.
- 2. Separation of olefins and paraffins by treatment with selective solvents (particularly SO<sub>2</sub>).
- 3. Synthesis of esters of phthalic acid and other dicarboxylic acids. (Plasticizers, gelation agents, glyptals).
- 4. Reactions of Dicarboxylic acids, particularly phthalic acid and alcoholates.
- 5. Disposition of the oxidation products of "Parestol" and "Partoxyl" in the broadest sense, for example, fatty acids, from which resins (alkyl type) soaps, waxes, salve bases, linseed oil substitutes (from oxy acids) might be made. Hydrogenation of the oxidation products to alcohols and esters.
- 6. Introduction of the sulfo- and nitro- groups into paraffinic hydrocarbons.
- 7. Oxydation of the lower paraffins to dicarboxylic acids with nitric acid.
- 8. Dicarboxylic acids from "Parestol" or from paraffin or over the phenols of coking plants.

#### Lab 3

- 1. Completion of the theoretical works on sec-octyl acetate (Tricloroacetic acid and benzolsulfonic acid as catalysts, H<sub>2</sub>S-formation).
- 2. Placing the synthesis of sec-octyl acetate to semi scale production (corrossion problem, filtrations).
- 10. Removal of CO2 and H2S with aqua-ammonia.
- 11. Production of motor fuel for (?) from carbon monoxide.

Item 98E p. 831-834

## Condensation of methyl ethyl ketone and formaldehyde

A summary of two years developments, 1940 to January, 1942. 1) Dehydrogenation of hydrocarbons, i.e., propane to propylene, using chromium and aluminum oxide catalysts; 2) dehydrogenation of alcohols to ketones, i.e., sec-butyl alcohol to methyl ethyl ketone, with the aid of zine catalyst; 3) synthetic resin work: a) methyl ethacrylate b) β-methyl-γ-ketobutanol c) preparation and purification of methyl isopropenyl ketone and its plexigumlike polymers; 4) development of the ketone resin Emekal from methyl ethyl ketone and formaldehyde as a hydrocarbon-soluble resin for the lacquer industry; 5) development of sec-butyl stearate as a plasticizer; 6) uses and process for the preparation of aluminum and magnesium alcoholates; 7) development of carbon dioxide addition products of aluminum alcoholates especially of "Butal" i.e., "Carbutal"; 8) preparation of active aluminum hydroxides and oxides from aluminum alcoholates; 9) preparation of a new gelatinizing agent from Butal and & C6-C9 fatty acids.

- p. 835-837 Detailed programs for the exploratory laboratories (see pages 827-830).
- p. 838-844 Lengthy proposed scheme for assigning compensation for patents (unidentified, incomplete).
- p. 846-848 Types and numbers of personnel assigned to Drs. Haussen and Wiedemann.

Item 99H
p. 849-851 Discussion of octane number determination (July 15, 16, 1941 Motor Lab.)

6 .

- p. 852-854 (Identical to forgoing).
- p. 855 (Program)
- p. 856-858 (Identical to above).
- p. 859-863 Miscellaneous graphs of octane number versus compression ratio.
- p. 865-868 Memo on determination of octane numbers from the Motor Laboratory to the central office of petroleum in Berlin. 8/27/41.
- p. 869-870 Figures possibly for above.

Item 103

p. 871

Vol. I of "Chemisch-Technische Untersuchungs Methoden" edited by E. Merck covering the coke industry. Table of contents is reproduced.

Bag 1481 CIOS target C 30/1.61 - Group 3

Fuel Research and Technology at Rhenish-Westfälisches Kohlen-Syndikat, Essen.

p. 888-891 Two items describing facilities as extremely hampered by absence of personnel on military leave. Activity of the fuel research department was apparently limited to answering consumer problems of product utilization.

Bag 1481 ClOS Target of Opportunity Group 3

p. 892-895 Fuel Technology and the Reichsvereinigung Kohle, Essen-Bredenerz.

Item 1

p. 893

Chart showing in detail the organization of the "Reichskohlenrates" (Imperial Coal Council)

Item 2

p. 894

Chart showing organization of "Reichsvereinigung Kohle" (Imperial Coal association).

Item 3

p. 895

Chart showing organization of the "Abteilung Technik und Wirtschaft der Brennstoff verwendung" (Division of technological and commercial utilization of fuels).

Item 4

p. 895

Table showing composition of the "Energiesparaktion" (Board for conservation of energy) for industrial establishments.

Bag 1481 ClOS Target T 15/9 Group 3 30/4.11 C30/86

Coal extraction plant of Ruhrol GMBH at the Hugo Stinner Werke Welkeim near Bottrop, I.W.

Item 1

p. 895

Flowsheet for 26,000 tons per year Pott-Broche Plant.

Item 1 p. 895

Road tar resistant to weathering developed by Vf7 (Vereimgung fur Teerforschung?)

A road tar resistant to weathering is produced by blowing normal anthracene oil at 160°C (320°F) with air until a viscosity of about 3° Engler at 50°C is reached. This product is mixed with ordinary pitch (softening point 60-75°C Kraemer-Sarnon) - this "weathering tar" contains a maximum of 1% intermediate oils, a maximum of 5% heavy oils, no phenols and naphthalene. Free carbon content is less than 16%. Drop point is higher than 30°C, brittle point below -25°C. This tar is therefore more resistant to softening and to embrittlement than ordinary tars. This tar is not used for first, but for subsequent coats.

Item 2

Unidentified diagram of thermocouple positions (Drawing BK 16-2).

Bag 1481 ClOS Group 3 Target 30/7.06 C30/73

Fuel Research Activities of the Koblenwertsloft - Verbande.

Item 2 p. 895

Roasting Coal pyrites for sulfur removal. More heat is developed than in roasting ordinary pyrites on account of 5% carbon.

Item 3 p. 896

Use of aminophenol to eliminate the harmful effect of carbon disulfide on leaded gasoline.

Reciprocal action of sulfur compounds and tetraethyllead.

The german war machine changed the motor fuel during the African campaign to a benzine, benzol mixture compounded with tetraethyllead. The motor fuel was similar in composition to fuels previously used in other war theaters.

However, the motor fuel decomposed through the combination of tetraethyllead with sulfur compounds to precipitate lead sulfate.

The concern here is not about the known decomposition of tetraethyl lead as an antionidant, but a chemical conversion of tetraethyllead with sulfur compounds to insolubles, and to lead sulfur combinations.

It was decided that a sulfur compound was the cause of this decomposition. Research indicated, that carbon disulfide contained in small amounts in motor gasoline was the cause of the decomposition.

Pure sulfur-free hydrocarbons mixed with tetraethyllead, showed the same phenomena after the addition of carbon disulfide. It was further established that the reaction was strongly temperature sensative.

The trouble may be avoided wither through complete removal of the carbon disulfide or through the addition of materials to inhibit or retard the reaction. The latter method is possible through the addition of aminophenol in small amounts.

A test was worked out whereby the fuel was placed in a glass pressure flask and held in a drying oven at 110° and the time for turbidity to occur through precipitation of lead sulfate (ide) was determined.

Item 4 p. 895

Filterability of Fischer diesel fuels was improved at low temperatures by incorporation of crystalline wax which separated clearly together with small quantities of amorphous wax which originally plugged the filter.

Item 5 p. 895

Diagram of Permagas-Plant

Bag No. 1481

Target of opportunity. Catalogue of war-time types of German heating and cooking stoves etc., 16 items pages 896 to 1093.

Gives very many reproductions and drawings.