FIIM STUDY GROUP
SUBJECT INDEX AND REPORT

T.O.M. REEL NO. 67

Prepared by

GULF RESEARCH & DEVELOPMENT CO.

# GULF RESEARCH & DEVELOPMENT COMPANY Pittsburgh, Pennsylvania

# ABSTRACT AND INDEX OF TECHNICAL OIL MISSION

MICROFILM

REEL NO. 67

# INDEX OF REEL NO. 67

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#### SCANNING OF REEL NO. 67

#### BAG 3445 TARGET 30/5.01

# RUHRCHEMIE A. G. - STERKRADE-HOLTEN Frame No. 1 ITEM 32: Iron Catalyst for Low Pressure A short memorandum discussing an iron catalyst of special preparation suitable for a temperature of 200 to 225°C. and pressure of 15 ats. Nothing is said about the preparation of the catalyst. The experiments are reported in Reel 66, Item 25. ITEM 33: Preparation of Oil of High Olefin Content 5 This is a very short report of one page on experiments described in Reel 66, Item 25. ITEM 34: Report of 1941 of Recent K.W.I. Work (Outline) 7 This is a short report on polymerization of definite monoolefins to viscous oils and city gas production in connection with the Kogasin synthesis. 16 ITEM 35: Micro-Indine Number The Bromine number is not reliable especially for determination of olefins for the OXO synthesis. The micro-iodine number is said to be reliable. ITEM 36: Research Program for Medium Pressure Experimental Plant 18 These are only short program outlines for the experiments which are described in Reel 66, Item 25. ITEM 37: Conference with Krupp on Recirculation of Water Gas 39 This is a short report on a conference with Krupp concerning hydrocarbon synthesis directly from water gas describing the operation, gas analysis and yields (see Reel 66, Item 25). 43 ITEM 38: Petroleum Industry of Russia, 1941 This is a printed publication from the high command of the

army about occurrence of petroleum in Russia, the composition of petroleums from the various fields are shown and location indicated by maps. There is a further chapter on refineries and the products from petroleum, the difficulties of the Russian petroleum industry and pipe lines and other

meens of transportation.

A printed pamphlet from V.D.I. Berlin for 1941 discussing the drawbacks of gasolines to which were mixed ethyl and methyl alcohol as is customary in Europe. The main drawbacks are sensitivity against water which is miscible with alcohols and brings about stratification into two layers and limited miscibility of methyl alcohol in gasoline. Such gasolines are especially sensitive to low temperatures because they become cloudy and stratify more easily into two layers. The paper deals with additions to such gasolines to counteract these drawbacks. Addition of dimethyl acetal is mentioned. Many tables and graphs for ternary mixtures are given.

Another drawback of the addition of methyl and ethyl alcohol to gasoline is the formation of minimum azeotropes which bring about a lowering of the temperature at which the gasoline begins to distill and may cause vapor lock. The addition of higher alcohols as well as addition of other compounds which are already used for addition to gasolines such as benzol, toluol, xylol, acetone, and isopropyl ether counteract this satisfactorily. Many graphs and tables and 15 references are given.

# ITEM 40: Apparatus for Determining Absorption Isotherms

**7**9

This is a short discussion of the dynamic and static method for determining absorption isotherms and a description of the apparatus for the static method which is considered superior for gases of low molecular weight.

These pages give sketchs of the apparatus for the static method for determination of the absorption isotherm, and a graph of the ethylene isotherm.

# ITEM 41 Parts of Summary of Licensees Results 1938-40

84

This is a graphical and tabular representation for different works showing percentage CO conversion, percentage contraction and other data, such as space velocities,  $H_2/CO$ , etc.

# ITEM 42: Overhauling Oven 51

92

This is a lamella oven which was difficult to empty and therefore the oven was inspected and overhauled. The item is almost illegible.

# ITEM 43: Time required for Emptying the Oven

95

These are details showing time and working hours required for emptying an oven.

# ITEM 44: Detailed program for synthesis, Normal Pressure 1938-1940

98

There are many graphs showing the desired program of days of operation as abscissa and degrees C. as ordinate. Figures for space velocities, contraction, etc. are shown on the graphs.

ITEM 45:	Specifications for Treib Gas	105
cerbons wi	These are specifications for mixtures of C2 to C4 hydro- th respect to heating value, vapor pressure and impurities.	
ITEM 46:	Extraction - Oil	106
oil for Ov	This is a table giving the distillation curve for extraction on 31 and 32 of March 28, 1941.	•
ITEM 47:	Results of the Recirculation with an Injector	108
in the wat	This is a short plant report concerning preliminary results er gas recycling synthesis to produce more olefins by using r for the recycling of the off gas without removing the light	
ITEM 48:	CO/H <sub>2</sub> ratio for medium pressure synthesis	114
synthesis ; I, II, and and third	This is a memorandum concerning the composition of the gas, the gasoline and the CO/H <sub>2</sub> ratio in the off gas in stages III. It was found that gasification increases in the second stage if the CO/H <sub>2</sub> ratio increases about to 1.8, therefore the ld be reduced in the second stage.	
ITEL 49:	Water Gas Conversion	116
onolers, e in Novembe	These are two flow sheets showing the catalyst ovens, saturators, to. and a graph of data during the starting of the pressure oven r, 1938 showing the hours of operation as abscissa and space temperature and contraction as ordinates.	•
ITEM 50:	Medium Pressure Synthesis, 1938.	119
	This is the same graph shown in Item 49.	
ITEN 51:	Scheme for Working Up Products	121
and light	This gives the flow sheet for processing C <sub>3</sub> & C <sub>4</sub> hydrocarbons and heavy naphtha from the activated carbon. A flow sheet is the C <sub>3</sub> & C <sub>4</sub> hydrocarbon washer, extracting the CO <sub>2</sub> with K <sub>2</sub> CO <sub>3</sub> .	
ITEM 52:	Work In Block 9, Stage 1 (high Methane Formation)	125
space velo	Tables and a graph of results when starting Oven 92 at a city of 500 m <sup>3</sup> /hr. at 161°C. are given. The CH <sub>4</sub> content of is in the first 35 hours was up to 50 per cent and then decreased tow hours to 3 to 5%.	
ITEM 53:	Details of Ovens	129

The measurements of the catalyst chambers are given as follows:

2046 double tubes 4500 x 44 i.d./24 mm. c.d. Catalyst volume 10 m3 Water volume 12.25 m3 Water cooled catalyst surface 2200 m2 ITEM 54: Work Instructions for Solvent and Hydrogen Treatment 130 Instructions for the hydrogenation and extraction of low pressure ovens. 134 ITEM 55: Alteration in Method of Calculating Yields Methods for measuring the products of medium pressure; paraffin, low pressure oil, condensate, C3 & C4, etc. are given. 138 ITEM 56: Yields from Gas Analyses, April, 1940. This is a table giving gas analyses and balance for April, 1940. 139 Stepwise Degassing of the Pressure Wash Water ITEM 57: A short table of procedure. 140 Formation of Vapor Locks in Gasoline Lines ITEM 58: This is a long report (partly illegible), from the Institute for Automobile Internal Combustion Pachines of Dresden, regarding formation of vapor-lock by gasolines showing the laboratory apparatus used, in sketches and photographs, and giving many curves of vapor pressure. 220 ITEM 59: Loss of Cobalt at the Cotolyst Factory at Holten Several reports (partly illegible) describing recovery of cobalt and giving tables concerning losses. 243 Two Works Laboratory Notabooks ITEM 60: These are handwritten (partly very illegible) notebooks dealing with:

Low pressure ovens: .

Lamellas 5000 x 1525 x 2500 mm. high. 555 plates. 1.6 mm. thick

126 tubes, 42.5 mm. diameter on top. 504 tubes 35 mm. diameter at the bottom

7.4 mm. apart

Aggregates 6 and 7

Medium Pressure Ovens:

Aggregates 1 to 5 and 8 to 12

Catalyst volume 12.95 m3

-630 tubes 35 mm. diameter Catalyst volume 13 m3.

- 1. Influence of the purification of synthesis gas with activated carbon and FRN on catalyst activity.
- 2. Carbon determination in used catalysts.
- 3. CO<sub>2</sub> determination in life tests using different sealing liquids.
- 4. Absorption of CO2 on catalysts.
- 5. Synthesis gas samples. This is a test to determine whether a dry gas sample changes on long standing similar to the changes on storing in contact with water.
- 6. Poisoning tests with condensate of Chemische Werke Essener Steinkohle.
- 7. Testing of catalysts under pressure with consideration of conditions of the plant.
- 8. Tests on synthesis gas I over a period of 6 to 10 hours.
- 9. Determination of the activity of catalysts by testing the oxygen absorptivity.
- 10. Continuation of Number 7.
- 11. Clay catalysts. These are catalysts prepared from clay with cobalt and thorium exide. Treatment of clay with HCl is described.
- 12. Testing of catalysts from the plant.
- 13. Comparative reduction experiments.
- 14. The influence of adding water vapor to synthesis gas especially with respect to conversion under conditions of the synthesis.
- 15. Sulphide decomposition of spent Feinreinigen Mess.
- 16. Corrosion testing of several paints for resistance to gasoline, oil, heating oil and cracked gasoline.
- 17. Testing of the activity of catalysts, poisoning experiment with Rheinpreussen gasoline.
- 18. Testing of the activity of catalysts, addition of acetylene.
- 19. Experiments on desulphurization of waste water by absorption of the sulphur set free.
- 20. Concerning pyrophoric spent cobalt thorium catalyst.

- 21. Olefin determination on products. (Table).
- 22. Carbon monoxide conversion on cobalt thorium magnesium catalysts. (Short table).
- 23. Cobalt in paraffin.
- 24. Effect of sulphur purification in synthesis (table and graph).
- 25. Laboratory experiment concerning H2S from pressure cooling water.
- 26. Sampling of C3 and C4 hydrocarbon fraction.
- 27. Airing of Hoesch catalyst (cobalt thorium magnesium).
- 28. Aging experiments.
- 29. Impregnating experiments.
- 30. Testing of catalyst activity in Oven 2 (Impregnating experiment).
- 31. Experiments on reduction of catalyst.
- 32. Investigation of water gas before fine purification for C, H, and carbonyl.
- 33. Investigation of chlorinated lime for effective chlorine.
- 34. Continuation of experiments on working out a method to test catalysts at medium pressure.
- 35. Same as No. 32.
- 36. Testing of the activity of a cobalt catalyst with a mixed gas containing ammonia.
- 37. Absorption of oxygen on cobelt thorium catalysts.
- 38. Short newspaper clipping concerning separation of oil emulsions from Rundschau deutscher Technik No. 41, Oct. 1940.
- 39. Dismulgan (this is the compound described in the news-paper clipping).
- 40. A. S. O., a crude purification mass (Japanese).
- 41. Interferometric experiments on mixtures of nitrogen and hydrogerbon.
- 42. Experiments concerning spontaneous inflammability of paraffin in insulating materials.

### ITEM 61: Stability of Paraffin Waxes

635

A graph showing the chemical stability (Oct. 19, 1942) of normal cake paraffin (liquefying point/52), RB hard wax (liquefying point 100°C), and catalyst paraffin (liquefying point 100°C.). Days at 130°C. shown as abscissa and the neutralization number as ordinate.

# ITEM 62: Process for Removing Colcium from Cobolt Solutions

636

German Patent No. 683691 of November 13, 1939. This patent describes the removal of calcium as calcium fluoride by adding to the solution a water soluble fluoride.

#### ITEM 63: Catalyst Factory at Holton

640

These are 6 flow sheets showing the plant at Holten.

#### ITEM 64: Synthetic Scaps.

641

These are several reports on synthetic scaps especially from the fatty acids formed in CO hydrogenation and includes the separation of the higher fatty acids. The OXO synthesis is mentioned. This item also includes a report concerning the separation of alcohol from the waste water of the activated carbon plant which contains an average of O.4 per cent alcohols, aldehydes, and ketones.

#### ITEM 65: Aromatization

659

This is a report on the aromatization of straight chain aliphatic hydrocarbons from the Fischer Tropsch synthesis using chromium oxide and alumina-chromium oxide. There is a description of the work done and plans for large scale operations.

#### ITEM 66: Preparation of Isopetrol

673

A report on the preparation of iso- $\mathbb{C}_4$  hydrocarbons and aviation gasoline from synthesis products. Polymerization of  $\mathbb{C}_3$  and  $\mathbb{C}_4$  hydrocarbons with phosphoric acid catalysts and the cracking of Fischer Tropsch products for aviation gasolines is described (Houdry)

# ITEM 67: Preparation of Isopropyl Alcohol and Methyl-Ethyl Carbinol.

695

Description of a process for producing alcohols from olefins using sulphuric acid, describing the difficulties of the process and giving data with regards to acid concentration, ratio of acid to olefin and the temperature during olefin absorption.

# ITEM 68: Inhibitor for Resin Formation.

704

This is a description of the inhibitor "Stadisol" which inhibits gum formation in cracked gasolines. The practical use of Stadisol is described but nothing is mentioned concerning its composition. It appears to be hydroquinone or a similar compound, it is stated that alkalinity should be avoided.

These are tables showing yields in the Fischer-Tropsch synthesis and data on  $CO/H_2$  ratio, end gas composition, contraction,  $CO + H_2$  conversion, new formation of  $CH_4$  and  $CO_2$ , synthesis gas m<sup>3</sup>/hr. and age of oven.

# ITEM 70: Production Summeries, 1943-1944.

726

This is a collection of 10 day interval summaries for the synthesis which gives figures for oil and paraffin, synthesis gas, water gas, ratio synthesis gas to water gas, etc. (This is partly illegible).

# ITEM 71: Miscellaneous Analytical Methods

806

853

- 1. A determination of the tar and coke number of fresh oil 807 according to Ehlers.
- 2. Directions for testing aviation gasolines for use in diesel 809 motors. (Changes and explanations).
- 3. Determination of the distillation curve for product 812 boiling in the gasoline range region, for condensate oil and diesel cil, for solid products and for the total product.
- 4. Determination of content of elefins in aromatics.
- 5. Determination of the specific weight. 817
- 6. Determination of the iodine number. 821
- 7. Determination of the analine point. 823
- 8. Determination of the vapor pressure. 824
- 9. Determination of neutralization and saponification 825 numbers.
- 10. Determination of octane number. 827
- 11. Determination of the softening and the drop point. 828
- 12. Determination of moisture in gasolines. 830
- 13. Determination of the flash point and ignition point. 832
- 14. Determination of the solidifying point and melting point. 837
- 15. Determination of the evaporation tests and the bomb test, 842 with figures and illustrations.
- 16. Determination of the absorptive power of wax for oil. 852
- 17. Octane number according to the motor method, including determination of the content of aromatics in gasolines. This is hardly legible.

18. Rapid determination of cobalt.	873
19. Improved colorimetric determination of the heating value of liquid fuels according to Angewendte Chemie, p. 146 (1938).	875
20. Separation of propylene and ethylene in the presence of CO and ethane.	878
21. Testing of diesel oils for miscibility.	895
22. Investigation of alkaline mercury cyanide solution for absorptive power for ethylene and CO.	896
23. Determination of gaseous paraffin hydrodarbons.	899
24. Determination of the diene number of hydrocarbons.	908
25. Testing of diesel fuels.	910
26. Behavior of diesel fuels to zinc.	912
27. Filterability of diosel gas oils.	914
28. Determination of molecular weight according to Rast.	926
29. Determination of chlorine in dechlorinated paraffins.	927
30. Determination of resin in gases Brennstoffchemie p. 24(1940).	928
31. Determination of the residue on evaporation (resin content).	929
32. Change of method of determining the sulphur content (with illustrations).	934
33. Testing of the products made from fats.	940
34. Determination of the fatty acid content of fatty acid liquors.	942
35. Detection of benzene in gasolines (Rung method).	943
36. Determination of the CO and Rhodan number (thiocyanate number).	944
37. Same as No. 35.	, 953
38. Determination of olefins with phosphorus pentoxide- sulphuric acid.	954

	39. Modified method to determine the neutralization number and saponification number of gasolines, diesel oils and lubricating oils.	957
	40. Behavior of light gasolines to copper in the corrosion bomb Heinze-Marder.	986
	41. Dimethyl sulphate number.	987
	42. Total impurities.	988
ITEM 72: De	ecade Conferences 1943-1944.	989
Tropsch Synt	hese are 10 day interval reports on operation of the Fischer- thesis plant. They deal mainly with the difficulties in especially with emptying the ovens, overhauling the generators water gas delivered, repairs made during shut downs, different ies, etc.	,
ITEM 73: 0:	xidation of Waxes	1071
used, vield:	his is a short report of January, 1942, on the raw materials s and difficulties due to power shutdown for an experimental low sheet for the semi-commercial plant is included.	
ITEM 74: M	iscellaneous Patents of General Interest	1074
ir cr ir n	Patent 746,767, 8-23-44, Herman Pemsel. Process to increase the activity of Fullers' Earth. This is a continuation of patent 711,454. Claimed is an improvement in drying by soaking the purified Fullers' Earth with a cutral liquid of a boiling point higher than water and rying by bubbling through warm air.	1075
M s	Patent 746,573, 8-12-44, Gebr Herrmann, (Inventor ax Stauber). Process for production of colloidal silica olutions. This is a continuation of Patent 739,751 of 2-4-42. It describes a 2 stage method of neutralizing odium silicate solutions with ion exchange resins.	107.7
3 £	• Patent 746,512 of 6-19-44, Christian Stiasni. Device or feeding liquid gas into internal combustion engines.	1079
Ţ	• Patent 746,303, 7-21-44. Siemens-Lurgi Electrofilter Co. nventor Friedrich Wilhelm Hoss. Device for supplying high ension current into gas filled chambers such as electrofilters	1081
I a	• Patent 746,074, 6-17-44. I.G. Farbenindustrie.  nventor Heinrich Medick. Continuation of Patent 738,521  rocess for preparation of ethylene derivatives. This is a  harmaceutical patent for producing estrogenic compounds.	1084
• с	. Patent 740,772, 4-25-44, Karl Schmid. Process for continuously reacting mixtures of liquids which form separate hases of different specific weight.	1086

Ten

7. Patent 744,686, 6-21-44. Auer Gesellschaft A. G., inventor Walter Stiller. Protective filter against inorganic hydrides of multivalent metalloids. The patent describes the use of silver salts or finely divided silver or silver exide on activated carbons for H2 S, etc.

1088

8. Patent 740,013, 7-21-44. Aug. Schnakenberg & Co. Device for evaporating liquids which flow in thin layers over inclined planes with heating canals.

1090

9. Patent 746,636, 8-16-44. Julius Pintsch, Inventor Hellmut Stock. Still for continuous heating of light oils from low temperature carbonization for vapor phase refining over solid catalysts.

1093

- 10. Patent 746,541, 8-12-44. Felix Engelhardt. Process for 1096 production of citric acid soluble phosphate fertilizers. Heating of all phosphates with sodium nitrate from 500 to 700°C. under exidizing conditions.
- 11. Patent 745,338, 5-27-44. W. Cuypers & Stalling, Inventor, 1098 Paul Cuypers. Lubricating oils for watches and similar instruments. It is claimed that the addition of small amounts (approximately 1 to 2 per cent) of metal scaps especially aluminum stearate gave improvement in oiliness, retention of the oil in the bearing and makes unnecessary the addition of larger amounts of fatty oils as currently practiced.
- 12. Patent 746,787, 8-25-44. I. G. Farbenindustrie Inventor 1100 Fritz Fried. Cast waxes especially for dentistry. The patent claims an improvement by using synthetic resins instead of the natural resins currently used in compounding the waxes. The waxes show better properties. More synthetic resin can be added than natural resin. As an example cyclohexanone resins are used according to German patents 337,993; 357,091; 511,092; and 711,888.
- 13. Patent 745,684, 5-15-44. I. G. Farbenindustrie Process for making highly condensed polyamides from diamines and carbonic acid. These are super-polyamides similar to nylon using carbonic acid instead of long chain dibasic acid as in nylen.

1102

1105 14. Patent 744,318, 6-17-44. I. G. Farbenindustrie Process for making concentrated stable emulsions. The patent describes polymerization in emulsion for synthetic plastics. By polymerizing the mixture with small amounts of vinyl sulphonic acid or salts and an emulsifying agent of soapy character.

15. Patent 746,670, 8-18-44. Aktiengesellschaft der Kohlenwertstoff-Verbände Gruppe Benzin-Benzol-Verband. Inventors Rudolf Weller and Josef Gohm.

1107

Process for improving diesel fuels. The invention relates to an improvement of the filterability of diesel fuels containing amorphous paraffin to produce diesel oils of low cloud point by adding 0.05 to about 0.5 per cent of crystalline art paraffin.

16. Patent 746,861, 8-28-44. I. G. Farbenindustrie, inventor 1109 Rudolf Halder and George Weber. Device for determination of the lubricating property of cils. The device measures the wearing of a test piece which is pressed against a moving steel band. Two drawings are included.

17. Patent 746,304, 7-21-44. I. G. Farbenindustrie, inventor 1111 Hens Hauber and Josef Hirschbeck. Process for obtaining hydrocarbon oils suitable as insulating oils. The invention describes the production of synthetic lubricants made by polymerization of higher olefins such as propylene and butylene with Friedl-Craft catalysts such as aluminum chloride, zinc chloride, ferric chloride and especially boron fluoride. In contrast to synthetic lubricants made from ethylene the lubricants described are, with regard to viscosity, etc., especially suitable as insulating cils. The clefins may be obtained from Fischer-Tropsch synthesis or by cracking of petroleum. The gas used for polymerization should contain at least 30 per cent, preferably 40 per cent olefins. The following gas composition is given as an example:  $CO_2 - 12.00$  per cent,  $CO_2 - 1.25$  per cent,  $H_2 - 1.00$ per cent, ethylene - 1.70 per cent, propylene - 20.80 per cent, butylene - 19.60 per cent, amylene higher unsaturated hydrocarbons - 16.10 per cent, methane and ethane - 1.75 per cent, propene - 11.30 per cent, butene and higher saturated hydrocarbons - 14.50 per cent. This gas is moistened with water and polymerized with 3.3 volume per cent boron fluoride. After the condensation had proceeded far enough the catalyst is destroyed and the resulting oil is hydrogenated with hydrogen over a catalyst (for instance nickel catalyst) and then treated with superheated steam in the presence of a little caustic. The treatment of the superheated steam may precede the hydrogenation. An insulating oil.obtained by this method had the following properties: Flash point - about 170°C. specific weight at 20°C. .810, acid number - 0, saponification number - 0, indine number - 0, viscosity at 20°C. - 4.91° angler, viscosity at 38°C. - 2.09° angler. In considering this patent application the patent office cited the following referenced: German patents Nos. 402,990; 505,265; 507,919; 524,891; I. & E.C. 23 606 (1931). French patents Nos. 677,973; 800,956; 44,501 (addition). Dutch patent 36,210. British patents 323,805; 421,118. U.S. petent 1,756,154, J.S.C.I. (Japan) 39 21B, J.I.P.T. 21 952, Chemisches Zentralblatt (1936) I page 1760.

18. Patent 746,572, 8-16-44. Aktivkohle-Union Verwaltungs-G. 1113 s.m.b.H. Process for drying and cooling of absorbants. A special-arrangement for drying and cooling absorbers which are connected in series.

19. Patent 746,888, 6-15-44. N. V. DeBataafsche Petroleum 1116
Meatschapij in Den Haag, Netherland - inventors William E.
Vaughan and Frederick Farlow Rust. Process for chlorinating
hydrocarbons. This is a priority application from U.S.A. of
Sept. 2 and 30, 1939, and the corresponding U.S. patent will be
available. The patent claims chlorination in the presence of very
small amounts (about 0.005 mol per cent) of metal organic or azo
compounds which under conditions of the process give 3 radicals
such as tetraethyl lead. German patent office gave the following
reference: Chemisches Zentralblatt (1939) vol. II page 3047.

1122

This item consists of the following patents:

- 1. Patent No. 744,224, 7-21-44. Julius Pintsch, inventor Wermer Lohrisch. Process for production of synthesis gas. The invention describes the production of synthesis gas by reacting methane containing gases with CO<sub>2</sub> and steam which are obtained from the combustion gases on heating the methane reactor. Part of the initial gas is used to heat the reactor, the exit gases are scrubbed, the CO<sub>2</sub> liberated from the wash water and in mixture with steam is added to the main flow of the gas entering the reactor. A sketch is shown.
- 2. Patent 746,818, 8-25-44. Wintershall A. G. and Hans Schmalfeldt inventor Hans Schmalfeldt. Process for gasification of pulverized fuels according to patent 686,761. This is a process of gasification using fuels which are comparatively dry and rich in bitumen, to produce a gas poor in methane by taking off the gas between heater and gasifier. A sketch is included.
- 3. Patent 746,747, 8-21-44. Heinrich Koppers inventor 1124 Adolph Weissendorn. Water gas generator. The grate and distributor of the water gas generator are shown in a drawing.
- 4. Patent 247,426, 8-28-44. F. J. Collin A. G. Inventor 1127 Paul Stoller. Process for selectively washing out H<sub>2</sub>S from CO<sub>2</sub> containing gases. The gases are washed with ammonia water and the selectivity for H<sub>2</sub>S is claimed to be obtained by adding small amounts, approximately 1 per cent of phenol or homologues. A variation is to add, with the phenol, an equal amount of alkali carbonate. A sketch is shown.
- 5. Patent 746,886, 8-28-44. Karl Weiss. Dust Filter for Air 1129 and Gases. The filter employs several layers of perforated oil soaked paper. The papers can be easily removed for changing. A drawing is included.

# ITEM 76: A Metalgesellschaft Synthesis Patent

1131

Patent 746,887, 8-30-44. Metalgesellschaft, Inventors Helmut Weittenhiller and Wilhelm Herbert.

Process for production of hydrocarbons from CO and hydrogen. This is a patent in which the only change in procedure seems to be that fresh about catalysts are used in the 2nd or 3rd stage with high space velocities for about 1 to 2 months until methane formation increases and then the catalysts are used in the first stage at a lower space velocity whereby they operate more satisfactorily. It is claimed that this process gives an increase of 10 g liquid products per normal m<sup>3</sup> of gas.

#### ITEM 77: Filtration of Crude Paraffin

1134

Difficulties in the plant were encountered when using Fullers Earth. Laboratory experiments were carried out and satisfactory filtration

was obtained by the addition of 10 per cent and in another case 15 to 20 per cent "tonsil". The original wax was dark brown but after filtration at 150° C. with tonsil the color was good, one sample being pure white.

#### ITEM 78: Cracking of Residues from Wax Oxidation

1136

This is a report of Oct. 17, 1939, on the cracking of residues of synthetic fatty acids. It was found that cracking is possible but not suitable for the cracking plant because the residue has corrosive properties due to its high acid number and the coke residue of 4-1/2 per cent is too high. The distillate yield was 93.4 weight per cent.

#### ITEM 79: Synthesis of Paraffin and Benzine at Medium Pressure

1139

This is a report of 9-19-39 regarding paraffin and gasoline from the pressure experimental plant. A considerable number of tables of briling ranges and yields are given. The boiling range of paraffin samples and their suitability for exidation to fatty acids was investigated.

### ITEM 80: Determination of Fraction of Wax Boiling Below 400°C.

1163

This is an addition to the previous item and describes the distillation of the hard paraffin boiling below 450°C. under a vacuum of 1 mm pressure.

#### ITEM 81: Catalyst Wax

1165

This is a report on the analyses of oven paraffin from stages 1 and 2, hard paraffin in flakes and distillation analyses and investigation of catalyst paraffin.

#### ITEM 82: Refining hard wax.

1173

This is a laboratory report on the refining of paraffin wax with "tonsil" and a report on the production of an almost odorless cake paraffin obtained by passing steam which had passed first a tube filled with coarse "granosil" into refined cake paraffin at 120°C. A report is also included on the extraction of used tonsil from the paraffin plant with different solvents.

#### ITEM 83: Removing ash from wax, March 16, 1940.

1189

A report of several experiments on filtering paraffin to remove the ash, (removing of colloidal cobalt particles). Good results were obtained by acid treatment for 2 hours at 120 to 130°C. with 8.4 per cent of 75 per cent sulphuric acid, with 11 per cent of tartaric acid and with 9.2 per cent of oxalic acid. The process using the sulphuric acid is preferable.

# ITEM 84: Washing paraffin wax with alkali October 1938.

1197

Laboratory experiments on mixing paraffin with 10 per cent caustic solution to determine whether an emulsion would form. It was found that no emulsion was formed.

This is a report of experiments on cracking paraffin from the pressure synthesis. Liquid phase conditions (440°C., 100 atm.) for 20 minutes gave a conversion of 17% per pass to 8% gas, 12% gasoline and 80% Diesel cil (200-320°C.). Lower pressure and higher temperature or conversion gave more gas and gasoline, less Diesel cil. The cracking experiments are described and many tables and graphs are shown.

#### ITEM 86: Use of Synthetic Ward Wax in the Candle Industry.

1216

This is a report on discussions with the candle manufacturers concerning usability of synthetic hard wax. The candle industry did not like this hard wax because the ail content made it unsuitable and the price was too high. The report mentions that 2 to 5% of the wax could be added with good results in the production of drawn candles. The wax in this case being used for hardening and opacity.

# ITEM 87: Reactions of industry to the result of Kech and Billig, Oct. 1940

1221

This is a memo on the reaction of Ruhrbenzin personnel to an article of Koch and Billig in Brennstoffchemie 1940 No. 14 on investigations on solid paraffin hydrocarbons by determination of crystal form and reaction with chlorosulphonic acid to determine straight and branch chain molecules.

#### ITEM 88: Mixtures of synthetic Wexes. July 1940.

1224

This is a report on experiments determining the solidifying points of wax mixtures. Tables and graphs are given.

#### ITEM 89: Emulsions of Paraffin Wax, July 1940.

1232

This is a draft for a patent application on the production of wax emulsions suitable for polish and similar uses. The process consists of mixing exidized wax (from paraffin of C<sub>18</sub> or higher) with hard paraffin and emulsifying the mixture in water. As emulsifying agents may be used primary fatty acids from CO hydrogenation which are neutralized in the mixture with alkali hydroxide or alkali carbonate.

#### ITEM 90: Reducing the Lower Melting Fraction in Paraffin Wax.

1234

an investigation to determine whether specification hard wax could be obtained without forming a cake paraffin. Distillation was carried out up to 440°C. and the residue was hard wax which contained 10 per cent boiling between 440 and 450°C. (10% cake paraffin). Calculations are given as to how much cake paraffin may be added to soft paraffin.

Hydrogenation experiments were carried out on crude to determine whether the amount of cake paraffin could be changed. No change was observed.

A report on determination of cake paraffin in hydrogenated crude paraffin indicates that the alcohols and esters in the water gas recycle paraffin do not influence appreciably the crystallization of the cake paraffin.

A short report describes how cake paraffin determinations are carried out.

#### ITEM 91: Sample Analysis of Paraffin Wax From Ruhrchemie and Licensees.

Analysis of paraffin samples are given (distillation range and solidifying point). Numerous graphs are included, many of which are illegible.

#### ITEM 92: Distillation of Cold-Press Oil

1275

1239

Distillation analysis of cold press oil is given.

#### ITEM 93: Suitability of Synthetic Wax for Oxidation to Fatty Acids.

1278

This is a draft and final paper by Professor Martin concerning large scale production of fatty acids by the splitting of natural fats and ox idation of paraffins. It is an historical outline of the subject which describes generally which fatty acids are suitable for soaps and gives distillation analyses of synthetic paraffins to show how much of the paraffin is suitable for oxidation.

#### ITEM 94: Solubility of Paraffin Wax in Various Solvents

1318

The solubility of wax was determined in naphthas from the activated carbon plant (100-200°C.), from fractional distillation (150-200°C.), from diesel oil (200-300°C.) and from cracked gasoline (40-200°C.). Tables and graphs are given. It was found that the solubility does not depend on the kind of solvent as much as on the melting point of the paraffin and the temperature. A drawing of the apparatus used to determine solubility of solid materials in various solvents is shown.

# ITEM 95: The Benzene-alcohol Method of Extracting Natural Wax

1327

This is a report of an inspection of a candle factory which obtains paraffin by distillation of low temperature tar. The paraffin contained approximately 10 to 15% of phenolic and asphaltic oil which was washed out at 0° C. by 5 to 7 volumes of a benzene alcohol mixture (4 parts of 90° benzene and 6 parts absolute alcohol). The temperature during operation should never exceed 5°C. in order to avoid paraffin losses. The flow sheet of the plant is included.

#### ITEM 96: Oven 15 Filling 8.

1330

These are similar tables to those in Reel 66 Item 9 for Oven 11. Oven 15 is filled with a low temperature iron catalyst.

# ITEM 97: Oven 11 Filling 14 (Incomplete)

1337

These are numerous tables of yields and distillation analyses from operation with the low temperature iron catalyst at 9 atm. Much of the data is shown graphically.

END OF REEL