

FILM STUDY GROUP

REPORT ON MICROFILM REEL NO. 31

Prepared by

THE ATLANTIC REFINING COMPANY

SCANNING OF REEL #31

U. S. Government Technical Oil Mission

(Note: The film itself bears the designation #30, but the material corresponds to the index for film #31. Section 16 in the index is a continuation of subject of the same number in the previous reel #30.)

Manufacture of BZ Catalyst

Frames 1052 - 1062. April 1939.

Magnesium oxide, kaolin and nickel carbonate paste are treated with nickel nitrate solution and thoroughly mixed. The mixture is dried and calcined. The resulting product is ground, mixed with cement and water and is pressed into pellet or ring shape for catalyst mass. Directions are given for testing the activity of this catalyst, from which it is observed that the BZ catalyst is employed for the conversion of hydrocarbon to CO, CO₂ and hydrogen in the presence of steam.

BP Catalyst

Frame 1063 is devoted to a statement of the composition and the analytical procedure used in examining this catalyst. Neither the manufacturing method nor the application of the catalyst is given, although the high content of charcoal and P₂O₅ suggest use as a polymerization agent.

BS Catalyst

Several frames beginning with 1064 are devoted to manufacture and analysis of this catalyst whose application is not stated.

It appears farther on in this section that BS contact mass was developed by the I.G. at Oppau for the synthesis of methane from water gas, and Oppau's report dated July 19, 1940 will be found unusually complete (extends to frame 1096). The catalyst consisted of approximately 35% nickel oxide, 14% magnesium oxide, 28% (combined) carbon dioxide and minor amounts of other substances. The reaction rate is favorable at about 350° C.

17. CHLORINE PRODUCTION BY HCL OXIDATION (Oppau Process)

Frames 1097 - 1106.

The Oppau process employs a fluid catalyst mass (70% FeCl₃ and 30% KCl) which melts at 236°C. The reaction product, which is about a third chlorine, is dried with sulphuric acid and scrubbed with sulphur monochloride (S₂Cl₂). The sulphur monochloride dissolves up to about half its weight of chlorine. This solution can be stored at atmospheric pressure and consequently presents less danger than the storage of liquid chlorine. The solution when distilled gives up its chlorine. The operating pressure is about 100 lb./sq. in. The chlorine-free bottoms are returned through a cooler to the absorption tower.

Materials resistant to the corrosive effect of sulphur chloride were found to be steel with at least 15% chromium and V₂A. The overhead chlorine is cooled and is liquefied under the operating pressure of the distillation column.

18. CORRESPONDENCE ON POLYMERIZATION AT RAUXEL

Frames 1107 - 1149.

The initial discussions deal with a proposed polymerization plant for the Victor-Gewerkschaft at Castrop-Rauxel. The heat of reaction was assumed to be 300 kilogram calories per kilogram of polymer gasoline. A contract and bill of material for a plant with a capacity of 25 to 30 tons per day was drawn up in October, 1939.

In March, 1940 (frame 1139) a memorandum from I.G. to Victor discloses that a conversion of 65% of the C₃-C₄ olefinic charge was expected and that the catalyst would have a life of about 260 kilograms of raw polymer per liter of catalyst. The polymer distillation was as follows:

To 100°C. 7%
To 150°C. 68%
To 200°C. 93%
To 220°C. 95%

End Point 250°C.; recovery 97.5%. The octane number of the fraction up to 200°C. was 82 to 83 CFR motor method. It is presumed that this octane number is on unhydrogenated material.

A communication of June 25, 1941 discloses that the original filling of the catalyst case would require 420 kilograms of catalyst mass, although 12,000 kilograms were ordered sufficient for about 10 months' operation. Each liter was equivalent to 150 grams of activated carbon plus 180 grams H₃PO₄ (89%). The equivalent catalyst life in U.S. units is about 90 gal. polymer/lb. catalyst.

19. EXPERIMENTAL CROTONALDEHYDE PLANT

Frames 1150 - 1170.

A memorandum dated April, 1942 contains detailed design data calculations for a unit to produce 20,400 tons crotonaldehyde per year from about 60,000 tons of raw aldehyde oil. Despite the fact that there is no description of the properties of the charge stock, the material embodied in this section is considered excellent from the standpoint of description of the equipment. The intended location of this plant is not given, but the caption on diagrams indicate that the design data were prepared at Ludwigshafen.

Frame 1167 reveals that the Chemische Werke Huls at Marl-Recklinghausen is

interested in a crotonaldehyde experimental unit, and the I.G. office has transmitted to this firm a list of the equipment used in the croton plant at Oppau.

Reviewers should be alert to note the characteristics of the charge stock used for the plant described in this section.

20. IDENTIFICATION OF CONTAMINATING HYDROCARBONS
IN TTH (Brabag-Zeitz) Paraffin

Frames 1171 - 1187.

This report from the I.G. Laboratories at Oppau, February, 1941 describes procedure and results in the analysis of unpurified TTH paraffin with particular reference to the identification of non-normal-paraffinic hydrocarbons in material apparently derived by an oxidation process at Brabag Werk Zeitz. No identification is made of the TTH process and the intended use of the product.

21. RIKOL

Frame 1188. August 17, 1944.

A summary on a process to produce 12 tons Rikol per day is given. The location of the plant is probably at Merseberg, but the intended use of the product is unknown.

Four steps in the process are indicated: (a) - the splitting out of water from raw amylalcohol to make trimethyl ethylene; (b) - conversion of trimethyl ethylene with formalin solution in the presence of acid to trimethyl-1, 3-dioxan; (c) - splitting of the dioxan with 20% hydrochloric acid to Rikol; (d) - purification of the Rikol over phenylhydrazon.

For the production of 12 tons Rikol daily there was required the following quantities of material stated as tons per day:

Amylalcohol	-	30
Formalin solution (30%)	-	62
10% Hydrochloric acid	-	4.6
10% Caustic	-	5

22. RENAL - PROCESS FOR PRODUCING HYDROGEN-PEROXIDE

Frames 1189 - 1210.

The title may be a misnomer, in that a break in the file indicates that the subject matter is both a means of producing alcohol, and a process for concentrating hydrogen-peroxide. A memorandum of June, 1943 suggests that the Renal process was to be used for conversion of light fatty acids (C₄-C₆) to aliphatic alcohols used as special fuel. The process was to be installed in Heydsbreck.

A memorandum, November 16, 1943, states that the process, developed by Dr. Pfleiderer, through a ketonization of the fatty acids derived by oxidation and hydrogenation of ketones to secondary alcohols, yielded a product which they have called Paralk. There is no description of the process.

The next significant entry in August, 1944, describes bomb damage to the Renal plant at Heydebreck, from which it is inferred that the process had been placed in operation.

A memorandum dated October 20, 1944 at Oppau, discusses the Renal distillation for concentrating solutions of hydrogen-peroxide. There does not seem to be any connection between the manufacture of this product and the previously discussed use of the plant for making alcohols. It is inferred that the H_2O_2 can be concentrated to 85%. The microfilm inadequately reveals the working and products of the Renal process.

23. EDIBLE FATS FROM PARAFFIN

Frames 1211 - 1224.

A memorandum in July, 1943 outlines a project for the production of fatty acid material, involving several organizations. The extent of the project may be judged by the estimated expenditure of 244 million marks, steel requirements of 160,000 tons and the consumption of one million tons of coal annually. The initial plant was for the production of 100,000 tons of fluid primary products, which were expected to yield 34,000 tons of final products. The fatty acids were to be made through paraffin synthesis, the hydrogenation of the split-out hard BP 290-450°C. paraffin, and the conversion to fatty acids by the Oxo process. The film at this point is almost illegible so that the description is very brief.

(a) A Koppers type plant used oxygen to gasify hard coal to favorable ratio of carbon monoxide to hydrogen. Water washing was employed to remove CO_2 .

(b) The synthesis prescribed that 84,000 cubic meters of gas per hour be charged to 160 ovens in the first step and 35 ovens in the second step (each oven contained 10 cubic meters of catalyst).

(c) The paraffinic mixture (boiling from 290 to 450°C.) is given a hydrogenation step prior to the oxidation process. This hydrogenation employed an iron catalyst at 150 to 180°C. and a pressure of 20 to 100 (atmospheres?). The hydro-cracking of the high molecular weight paraffin yields 77% product at 200°C., and up to 400 atmospheres, the pressure apparently has little effect on yield. The original content of 44% hard paraffin is reduced to 34%. The 10% difference apparently is recovered as Kogasin II with a boiling range 200 to 290°C.

(d) In order to obtain the highest yield of fatty acids, the olefins of diesel oils or Kogasin II fractions are oxidized. A flow diagram at frame 1224 outlines the combined processes but lacks operating details and quantities involved in the several steps.

24. HYDROGEN CRACKING PROCESSES AS OF KOPPEL AND I.G.

Frames 1225 - 1233.

These processes were competitive. The analysis of charge gas shows 39% hydrogen, 30% methane, 15% ethane, 11% propane and minor quantities of other gases. Product gas contained approximately 16% CO, 6% CO₂, 75% H₂ and minor quantities of other gases. There were no technical details other than the inference that catalytic reforming in the presence of steam is employed.

25. STUDIES ON ULTRAVIOLET ABSORPTION, RAMAN SPECTRA, ETC., AT OPFAU PHYSICAL LABORATORIES

Frames 1234 - 1247.

A. Raman Spectra may be used for the analysis of mixtures which are not fluorescent and do not absorb the incident radiation excessively. The mixture must not contain more than four or five components (preliminary fractional distillation if necessary) in concentrations not less than 1-5% depending on components. The accuracy is about ± 5 to $\pm 10\%$ absolute. Components with very similar structure, such as the various methyl octanes, cannot be determined separately.

By a combination of fractional distillation and Raman spectra, paraffins may be determined up to and including the octanes; olefins up to and including hexenes; aromatics up to 200°C.; and naphthenes to 125°C. Beyond these limitations, the Raman analysis may be used to determine the general type of higher molecular hydrocarbons present in a mixture such as highly branched paraffins, monoalkylbenzenes, alkylbenzenes and cyclohexane derivatives.

Mixtures containing C₁-O₂ alcohols can be analyzed by Raman spectra, and for mixtures containing higher alcohols a type analysis for total alcohols may be made. In some cases the spectra may be used to differentiate between alpha and beta olefins, and between the cis and trans forms. It may be used to show whether a branch chain is connected to a carbon atom in a double bond, whether or not the double bond in the side chain of a substituted benzene is conjugated with the ring, or whether or not an ortho, meta or para compound is present.

Raman spectra have been used extensively for the determination of the constitution of terpenes. Raman spectra cannot be used generally for the analysis of inorganic compounds since heteropolar compounds do not show the Raman effect.

B. Ultraviolet Absorption permits a simple qualitative and quantitative type analysis of organic compounds, and is restricted to condensed ring systems and compounds containing the C=O group. Thus the total content of aldehydes and ketones in mixtures with alcohols may be determined with 10% accuracy from the absorption band at 1700 AU. The spectra of benzol, toluol, and xylol are sufficiently different to permit separate determination; however, the spectra of higher benzene homologues are practically identical, so that their total concentration may be determined, provided that olefins with conjugated double bonds,

if present, are removed with 80% H_2SO_4 at $0^\circ C$. Very small concentrations may be determined in this manner.

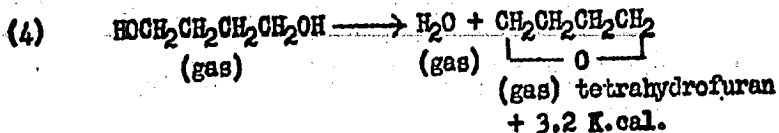
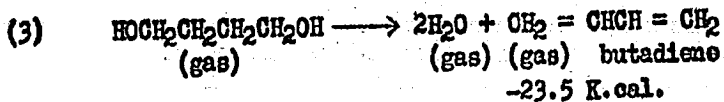
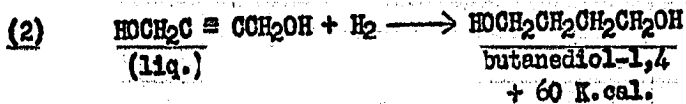
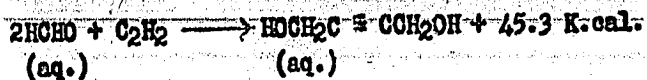
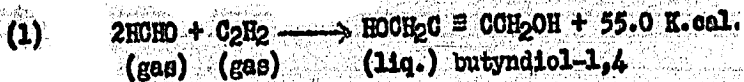
The spectra of benzene, naphthalene, anthracene, phenanthrene and higher condensed ring systems are widely different and are practically unchanged by substitution. This fact may be used for the determination of the constitution of organic compounds in hydrogenation fractions consisting almost exclusively of condensed aromatics such as coronenes ($C_{24}H_{12}$), benzocoronenes, naphthacoronenes, etc.

C. Researches have been undertaken by Dr. Hochheim on the Reflectivity of Metals, particularly of metal mirrors deposited on glass by vaporization in high vacuum.

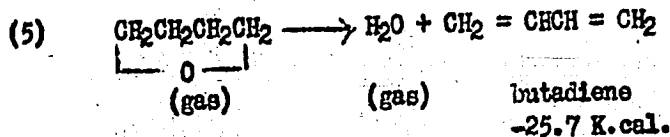
D. Work was done on the Vaporization of Salt in high vacuum. One piece of work led to making of very thin salt plates which showed remarkable light conductivity, and were used by I.G. Wolfen and Camerawerk München in the construction of optical instruments.

E. Iron Powder made from Iron Carbonyls has been used in electrical coils. The powder when formed into grains is used in telephones and radio equipment where low losses are required.

F. The study of Ammonia Synthesis with extremely pure gases, and the effect of poisons such as H_2S , PH_3 , O_2 and CH_4 led to the development of more resistant catalysts.



with value of 8.2 K.cal. if liquid butanediol and tetrahydrofuran are assumed.



PRODUCTION & ENGINEERING DATA ON THE MANUFACTURE
OF BUTANEDIOL AND BUTYNEDIOL

Reference: Index Page 27

(Items 1 - 43, Frames 0001 - 0245)

The first 14 frames of this section give the subject titles of 242 items, of which the first 92 are a part of the present reel, and presumably continues on to reel 32. The titles are in English, and correspond to the index.

Item 1. Summary of the Heats of Reaction for Compounds Entering into the Butadiene Synthesis

Frames 16 to 22 give the fundamental reactions involved in the synthesis of butadiene and the heats of formation involved. The reactions are given on facing page. Several pages showing calculations of raw material requirements and product yields are given. No date or location appears on these frames.

Item 2. Monthly Report of Diol Separation and Butanediol Distillation Plant - December 1944 and Jan. 1945

Frames 23 to 28 give plant production figures for the period December 1944 - January 1945. The location presumably is Ludwigshafen. These figures relate to operation and shed little light on the subject.

Item 3. Influence of Diffusion on the Reaction Rate in Butynediol Hydrogenation

Four frames - December 1944. This memorandum is speculation on the process and does not contain useful data.

Item 4. Report on the Removal of Silicic Acid from the Raw Butynediol Solution

Frames 31 - 40. The removal of small amounts of silicic acid from the aqueous butynediol solution can be effected by specially prepared magnesium hydroxide, as well as by calcium hydroxide suspension.

Item 5. Measurement of the Concentration of Butanediol Solutions

Frames 41 - 43. Give laboratory results in which the distillations of butanediol solution at several temperatures and pressures were made, and the change in water content recorded.

Item 6. Solubility of Silicic Acid in Water, Butanediol and Butynediol

One page, giving tabular results. These data were obtained in conjunction with work upon hydro catalyst 13340.

Item 7. Average Retention Time of a Carbon Particle in the Butadiene Plant

Frames 45 - 51. The calculations show residence time in the various portions of the plant. Of the total residence time of 104 hours, 60 hours were represented by storage in tankage.

Item 8. Heat of Formation of Butynediol

Frames 52 - 74. Memorandum undated, but appears to have been written in the summer of 1942. It discusses experimental work and calculations relating to the design of a butynediol plant, in which the product is synthesized from one mol acetylene and two mols formaldehyde. The discussion is in several parts:

- (a) Heat of reaction in butynediol synthesis.
- (b) Operating pressure (4.5 atmospheres was considered optimum for Plant B-III).
- (c) Removal of the heat of reaction.
- (d) Calculation of pressure drop in the recycle gas system.
- (e) Distribution of liquid.
- (f) Influence of pH values.

This section, because of the detail of calculations, will be of interest to persons studying synthesis of butynediol.

Item 9. First, Second, Third and Ninth Reports on Butynediol Manufacture

Frames 75 - 113. These appear to be monthly operating reports on the butynediol plant at Ludwigshafen and begin April 1943. Material, chemicals and utility requirements are given, with comment.

Item 10. Monthly Report on Butynediol Manufacture

Items 9 and 10 refer to similar but apparently separate plants and comment upon operating features.

Item 11. Monthly Utility Requirements for Butenediol, Butanediol and Butadiene Production

A single sheet probably of little significance.

Item 12. Distillation Equipment in Lu 392

A two-page memorandum August, 1943 discussing distillation equipment for separation of propylene-butadiene mixtures and the recovery of these hydrocarbons. The processing appears conventional.

Item 13. Visit to Dr. Steinhofer and Dr. Adam in Niles on May 20, 1943

A five-page memorandum discusses the suitability of certain equipment for the hydrogenation of butynediol and aldols. The description is lucid and despite the lack of a flow diagram, a reasonably good picture of the operation is given. The catalysts are described by number or by general terms. This section is probably of interest when correlated with similar information.

Item 14. Monthly Reports for October 1942 on Butadiene, Butenediol, Aldol Hydrogenation, Aldol Manufacture, Butenediol Hydrogenation, Butol Distillation, Aldehyde Manufacture and Catalyst Manufacture

Frames 125 - 147. These are operating reports for units at Schkopau, describing technical operation and production for current operations. There are no flow diagrams to explain the process involved but the following inferences are made:

- (a) From butanol and allyl carbinol (proportions 98:2) is made butadiene and minor quantities of propylene, butyraldehyde and butadienol.
- (b) The butynediol processing involves the manufacture of this material from acetylene and formaldehyde, giving a yield on these two components of 86 and 94% respectively.
- (c) Aldol hydrogenation. The text is not explicit, but it is believed that the aldol is in effect butyraldehyde which is hydrogenated in the process to produce a high quality, concentrated butanol.
- (d) Butynediol hydrogenation. This seems to be a conversion of the butynediol to butyraldehyde.
- (e) Butol distillation. This step seems to be the purification of butol by elimination of minor quantities of aldehydes and heavy residues.
- (f) Aldehyde manufacture in which mercury was used to catalyze production from acetylene. This latter material in turn seems to have been made from carbide.
- (g) Catalyst manufacture. This single page enumerates materials used in the catalyst production department, but does not disclose the application of this catalyst. Shown below is an analysis of the finished catalyst:

H_3PO_4	- 3.35%
$Na_2H_2P_2O_7$	- 7.29%
Trimeta (Phosphate ?)	- 1.78%
Water Soluble	- 11.53%
Maddrell	- 29.64%
Graphite	- 61.16%
$C_6H_{11}N$	- 0.81%
P_2O_5	- 24.23%

Item 15. Butadiene Distillation in Schkopau

Frames 149 - 154 describe what appears to be a conventional distillation plant for the purification of butadiene-propylene mixture in which there was minor quantities of aldehydes, alcohol, water, etc. The product propylene is 99.9% pure, while the butadiene is 99.4%.

of

Item 16. The Reaction of Butenediol and Propargyl Alcohol with Alkalies

Frames 156 - 161. It was shown that the reaction of alkalies on butynediol or propargyl alcohol produces a carboxylic acid, which in aqueous solution or as an alkaline salt solution has strong foam forming properties. The formation of the acid proceeds easily. The isolated carboxylic acid is presumably identical with the substance which produces foaming in the butynediol or butanediol solution.

Item 17. By-products of Butynediol Hydrogenation in the Reppe Process

Frames 162 - 195. An extensive report giving detailed figures on this subject. The nature of the by-products which were formed in the catalytic hydrogenation of butynediol-1,4 are described. The main by-products are butanol, gamma oxybutyraldehyde and high boiling residues which are formed from the intermediate butenediol.

Item 18. Use of Sucol S for Esterifying Colophonium

Illegible. A telegram or brief letter.

Item 19. Testing of Sucol DB as a Softening Agent for Paper

Frames 197 - 198. Sucol DB is the 4,4'-trioxydibutyl ether. Sucol DB is a good softener for technical paper, but results in some lessening of tensile strength. The product is non-corrosive to light metal.

Item 20. Conference on the Use of Butanediol Bottoms as Core Oil

A three-page memorandum discussing the use of butanediol residues as binder for granular material.

Item 21. Excerpts from the Record of the Conference on Sucols of February 25, 1944

Frame 202. Discusses the Sucols (which are the residues of butanediol manufacture) and their uses in lacquer industry, as binder for granular material, and for the paper industry.

Item 22. Utilization of 1,4-Butanediol Bottoms

These residues can be used as a substitute for glycerine and in other applications where a binder is required.

Item 23. Conference on Sucols February 25, 1944

Sucol G is a residue from glycerine. It appears to have been available in several grades as binders. Sucol T is a residue from trimethylolpropane and is used as a flux and in the lacquer industry.

Item 24. Further Discussion on Sucols Conference - February 25, 1944

Item 25. Results of Experiments to Determine the Constitution of the Lighter Fractions of Butanediol Bottoms

The distillation was conducted at pressures of 4 to 12 millimeters, and the resulting fractions were found to have molecular weights from 109 to 192. The calculated formulae led to the conclusion that the material was a mixture of oxygenated compounds, among which were $C_8H_{14}O_2$, $C_8H_{10}O_2$ and $C_8H_{16}O_2$.

Item 26. Letter Giving Properties and Designations of the Various Sucols

Item 27. Memorandum on Core Oils

Frame 220. A patent memo relating the possible uses of binder material derived from distillation-residues-of-polyalcohols.—This would include the so-called Sucols discussed in previous items.

Item 28. The Constitution of Asfluidols and Karboresins

Frame 222. The Asfluidols are complex mixtures apparently compounded of by-products from several I.C. plants.

Item 29. Toxicity of Diols

Item 30. Purity of Technical Butanediol - 1,4 for Special Purposes

Item 31. Tests of a Glycerine Substitute

Frame 223. Examination of Sucol DB as a glycerine substitute.

Item 32. Glycerine Substitute 5641

Use of glycerine substitutes in the printing ink industry.

Item 33. By-product from the Butanediol - 1,4 Distillation

Item 34. Cutting Oil

Item 35. Plasticiser Solution

The Sucol DB is proposed as a plasticiser. Its probable composition is 4,4'-dioxylbutyl ether. Frame 234.

Item 36. Plasticiser for Polyamide

The same material is proposed. The ester of ortho-oxybenzoic acid with butanediol-1,4 and the ester of ortho-oxybenzoic acid with butane triol-1,2,4 is proposed as plasticiser.

Item 37. Method for Obtaining 4,4'-Dioxylbutyl Ether

A patent application covering the manufacture of Sucol DB.

Item 38. Polyurethane

Polyurethane was made from 4,4'-dioxydibutyl ether and hexamethylene diisocyanate. For further examples of the preparation of polyurethane, see Item 39.

Item 39. Production of Polyurethanes from the Residue of 1,4 Butanediol Distillation

Item 40. Polyurethane from Butanediol Residues. Testing of Products

Physical properties of some polyurethanes.

Item 41. Utilization of 1,4 Butanediol Bottoms

One page brief properties - no interest.

Item 42. Testing of 4,4'-Dioxydibutyl Ether as a Glycerine Substitute

Sucol DB was found unsuitable for printing-rolls and had limited interest for manufacture of stamp-pad inks.

Item 43. Ammonium Chloride for Alkali (Core Oil)

The addition of ammonium chloride to the bottoms of butanediol distillation gave a foundry core material with excellent properties.

PLANT DESIGN FOR BUTANEDIOL AND BUTENEDIOL MANUFACTURE

Note: Many of the items in this section and the following "Flow Sheets and Drawings for Butadiene Manufacture" are simply drawings without descriptive text. Those which seem to have had their origin in an engineering department and which may be presumed to be applicable to actual or intended plants are marked with an asterisk.

Item 44. Plant Data on Butanediol, etc.

A note book sheet showing plant production.

Item 45.* Butanediol Plant Storage and Pumping Diagram

Item 46. Flowsheet of Butanediol Hydrogenation

Items 45 and 46 are flow diagrams showing butynediol hydrogenation, storage and piping.

Item 47. Impurities in Acetylene in Month of May, 1943

Item 48.* Layout and construction details of Tetrahydrofuran Plant

Item 49.* Flowsheet of Tetrahydrofuran Plant

Item 50. Chemical Requirements for Buna Manufacture

Frames 275 - 276. Give material requirements, including intermediates for Buna manufacture. The page is not dated, nor identified as to location.

Item 51. Cost Estimate for the (Butene ?) diol Plant

Item 52. Time Schedule for Starting up Ludwigshafen Buna Plant

Item 53. Flowsheet for 1,3 Butol Manufacture

Item 54.* Instrument Flowsheet for Tetrahydrofuran

Item 55. Flowsheet for Hydrogenation of Butanediol

Item 56.* Building Layout for Tetrahydrofuran Plant

Item 57. Production and Chemical Consumption for Butanediol Plant 1943

Item 58.* Flowsheet for Hydrogenation of Butanediol

Item 59.* Flowsheet for Hydrogen Purification

Item 60.* Hydrogenation Flowsheet

Item 61.* General Plan of Hydrogen Works

Item 62.* Instrument Flowsheet. (Frame 291. Bears the notation "missing".)

Item 63.* Flowsheet of Butadiene Reactor Section

Item 64.* Recovery of Residues from Butanediol Manufacture

Item 65. Material Balance on Butanediol Hydrogenation

Item 66. Memorandum on Thermal Cracking of 1,3 Butanediol Diacetate

Frames 296 - 301. A memorandum which discloses that the cracking of 1,3-butoldiacetate at 550 to 600°C. gave yields of 84% pure butadiene at a charge rate of 2-1/2 kilograms diacetate per liter per hour. Whether further increases in yield could be obtained by recycling the half-ester, remains to be proven.

Item 67. Butadiene from Mixtures of 1,3 and 1,4 Butylene Glycol

Frames 302 - 305. From a mixture of 1,3 and 1,4 butylene glycols, 62 tons of butadiene were made in 24 days, with 6.7 tons of catalyst. The average yield during this time was 89.4% pure butadiene.

Item 68. Experiments on Splitting Water from 2-Methyl Butanediol 1,3

Frames 306 - 314. Dehydration of 2-methyl butanediol-1,3 yields 70 to 80% of raw isoprene. The undesirable by-products of this reaction have been identified.

FLWSHEETS AND DRAWINGS OF BUTADIENE MANUFACTURE

Items 69 - 84, Frames 315 - 335, corresponding with Index, Page 29, are engineering drawings of good legibility and usually sufficient detail to permit reconstruction of the process employed.

ISOBUTYLENE MANUFACTURE

Frames 336 - 341. (Item 85 - Frame 336) Plan for a 4,000 ton per year iso-octane plant. Material dated October, 1937 and presumably located in Oppan. The charge is isobutyl alcohol. The process is conducted in four steps. The first is dehydration of isobutyl alcohol to isobutylene at 300°C. and 5 atmospheres. The second step is catalytic polymerization of liquid isobutylene at 15 to 20 atmospheres and 150 to 200°C., whereby a mixture of 80% di-isobutylene and 20% tri-isobutylene is obtained. The third step is the hydrogenation of the di-isobutylene at 200 atmospheres and about 180°C. The product is iso-octane with a boiling point 99 to 100°C. The fourth step is the cracking of the tri-isobutylene obtained in the second step to make isobutylene and di-isobutylene. There is no data on the type and weight of catalyst used.

Item 85. Operating Description of the ET 100 Plant (Iso-octane)

Item 86. Operating Description of Isobutyl Oil Distillation

Item 87. Isobutyl Oil Distillation Flowsheet

Item 88. Separation of Methyl Alcohol and Isobutyraldehyde

OXO PROCESS, ETC. (Items 89 - 92, Frames 342 - 603)

Item 89. Production of Aldehydes in the Oxo Synthesis

One page; no significance.

Item 90. Synthesis of Adipic Acid Through the Reaction of Carbon Monoxide on Cyclic Ethers, Lactones, and Diols

Frame 344+. A survey, dated February 1942, of research carried out at Ludwigshafen and is a comprehensive report of about 100 pages, giving great detail of chemical reactions and might be broken down into the following main headings:

Investigation of the Catalyst
Variation of Operating Conditions
Explanation of Side Reactions
Continuous Reactions - Their Catalysis
and Conditions
Regeneration of the Iodine

Frame 352+ discusses the synthesis of adipic acid from tetrahydrofuran, carbon monoxide and water. The reaction was brought about with a nickel iodide catalyst at 200 atmospheres and a temperature of 240 to 270°C.

Frames 414 - 416. Investigation of the corrosion resistance of different alloys towards adipic and valeric acids in the presence of catalyst containing iodine and carbon monoxide at 200 atmospheres and 260 to 280°C

Frames 417 - 428. Reaction between carbon monoxide and tetrahydrofuran.

Frames 429 - 449, February 1942. Continuation of work on adipic acid.

Item 91. Synthesis of Acetic Acid Through the Reaction of Carbon Monoxide and Methanol

Frames 450+, June 30, 1942. This is a comprehensive report of about 70 pages in which detailed laboratory work on the catalysis of acetic acid is described. This section will probably be a valuable approach to the subject by those interested in the synthesis.

Item 92. Reaction of Carbon Monoxide with Olefins and Alcohols

Frames 519 - 603. Another comprehensive report. This section is actually a series of reports denoting a continuing project in the I.G. Laboratories at Ludwigshafen.

(This ends reel No. 31)