

FILM STUDY GROUP

REPORT

T.O.M. REEL NO. 32 Part II

Prepared by

SOCONY-VACUUM OIL COMPANY, INC.

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REVIEW OF MICROFILM REEL #32 - PART II
U. S. GOVERNMENT TECHNICAL OIL MISSION

I.G. Farbenindustrie - Ludwigshafen
W.I.F.O. - Achim-Baden

Compiled by J. J. Somers
(Socony-Vacuum Oil Co.)

4-4-46

INTRODUCTION

This review of Microfilm Reel 32 - Part II (Microfilm Reel 32 is actually two separate reels) is presented in the form of brief descriptions of the individual items, arranged in the order of their location in the reel. For each item, the date and approximate length in pages are also indicated.

It will be noted that only that part of the reel which is included in BAG 2078 (Ludwigshafen) has been covered in this review; this comprises items 174 to 242 of BAG 2078. The remainder of Reel 32 (i.e., BAG 4043B, Items 1-46, and BAG 4043C, Items 1-2) contains little data on chemical processes, but is largely concerned with organizational details, petroleum specifications, analytical test methods, etc. Moreover, such material is very difficult to summarize. For these reasons, no abstracts of this material have been made, although the contents are indicated in the index.

In the index, many of the titles of the individual items of BAG 2078 have been revised to be more indicative of the contents. Also, the sub-headings listed in the original TOC index have not been used, since this break-down was found to be somewhat inadequate and misleading. Hence it will be necessary for anyone interested in this review to scan the entire list of titles (items 174 to 242).

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BAG 4043B 30/Opp. - WIFO Achim-Baden

Item

A. Organization and Code Abbreviations Used

- 1 Personnel Organization
- 2 Distribution System - France and Atlantic Coast Parts
- 3 Code System for Products
- 4 Statistics on Loadings - 1941 & 1942

B. Specifications and Tests on Products

- 5 Gasoline (including alcohol blends)
- 6 Diesel Fuel
- 7 General Naval Lube Oil Specifications - 1939
- 8 " " " " " - proposed, 1940
- 9 Contract with German High Command for Lube Oils, 1939
- 10 Miscellaneous Lube Oil Data by Various Manufacturers
- 11 Steam Cylinder Oil
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- 13 Corrosion-Protection Oil (Sea Water in Cooling Systems)
- 14 Transformer Oil
- 15 Weapon Oils
- 16 Castor Oil for Magnetic Compasses
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- 18 Glycerin (for Recoil Oil)
- 19 Fuel Oils
- 20 Greases

C. Miscellaneous Research Information

- 21 Emulsified Oils
- 22 ~~Mixing - Compatibility of Fuel Oil Components~~
- 23 Small LPG Apparatus (Duevag)
- 24 Bearing-Testing Machine
- 25 Summary Chart - Types of Crude Oils
- 26 Chart Showing Relations of Synthetic Products by Polymerization
- 27 Chart Showing Condensation Products from Phenol, Cresol, and Formaldehyde

D. Analytical Test Methods

- 28 Test for Corrosive Sulfur in Gasoline
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- 30 Alcohols in Oil
- 31 Tolerances in Lube Oil Testing
- 32 ~~Open Cup Flash (DVM)~~
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- 34 Viscosimeter (Dr. R. V. Dallwitz-Wegner)
- 35 Determination of Emulsibility

Item D. Analytical Test Methods (Cont'd)

- 36 Saponifiable Material in Mineral Oils
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- 38 Chart for Viscosity-Temperature Relationship
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- 40 Consistency of Greases
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- 45 Boiler Water Test Methods
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BAG 4043C Target 30/WIFO

- 1 Typical Tests on Lube Oils and Special Lubricants
(1939-1941)
- 2 Typical Tests on Components and Blends of Motor Gasoline,
Diesel Oil, Fuel Oils, and some Specialties

DESCRIPTIONS OF INDIVIDUAL ITEMS (#174-242)

174

2p

3-18-44

Engine Test Data for Several Motor Fuels and Additives

The following motor octane numbers (injection method) were determined for the indicated fuels:

	<u>M.O.N.</u>
S ₃ + 1 Vol. % n-butyl borate	66.5
S ₃ + 1 Vol. % tin tetrabutyl	66.0
S ₃ (no additive)	65.0
S ₁ + 25 Vol. % acetone	78.0

175

2p

2-9-44

Engine Tests of Several Diesel Fuels and Additives

The experimental data in the form of curves showing cetane number as a function of fuel composition, are missing from this short memo. The base fuels R300, V, and D688 ("flying Diesel") were blended with S₂ and S₃, and with the additive HoSR, which is quite effective. "The following maximum increases in cetane numbers were obtained:"

	<u>Cetane Number Unblended</u>	<u>Maximum Increase</u>	<u>Maximum Cetane No.</u>
Fuel V	22	25	47
" D688	52	53	105
" R300	174	100	274

176

10p

2-4-44

Use of Peroxide for Improvement of Diesel Fuels

This is a collection of 7 different short memos and letters from which it is difficult to extract much useful information. The memo of most recent date (2-4-44) states that research in preparing a peroxide for Diesel-fuel improvement has led to a technically applicable process; advice is asked on further development. The cetane value is increased about 10 units per percent addition of the peroxide (unspecified).

An earlier memo reports not very favorable tests on peroxides of cyclohexane and cyclohexanone.

177

1p

4-14-44

Micro-burning Chamber Experiments

A one-page letter discussing proposed work. The author says, in part: "We intend to study the use of catalysts (e.g. iron carbonyl) dissolved in the fuel or oxygen-carrier, as a means of increasing the rate of combustion."

178

1lp

3-9-44

"Höchst SR" (Tetranitromethane) : Diesel Oil Additive

This item contains several letters and memos in which problems related to the application of "HöSR" are discussed. The additive raises the cetane number of Diesel oils approximately as follows: 0.5% = 10 C.N., 1% = 15 C.N. The high freezing point of tetranitromethane, 13°C., introduces the danger of sludges of explosive compositions separating from the fuel. For this reason, solubilizing agents were investigated. The additive was also tested in gasolines.

179

2p

2-17-44

Explosion Experiments with Mixtures of Tetranitromethane and Fuels S₂ and S₃

The tabulated data show that up to 20 wt.% of tetranitromethane can be added to S₂ or S₃, without danger of explosions. On the other hand, TNM-rich mixtures containing only small amounts (5-10%) of S₂ or S₃ are quite explosive.

180

3p

12-29-43

Explosibility of Tetranitromethane-Fuel Mixtures

The explosive limits of mixtures of tetranitromethane with R300 and VT702 were determined; with both benzines, mixtures containing from 20 to 99% of tetranitromethane were found to be explosive. A sketch and description of the experimental apparatus are included.

181

16p

5-19-44

Acetone Purification at Oppau 64^a in 1943

Contents: I. Acetone Production, II. Purification (a) acetone rectification, (b) aldehyde rectification, (c) caustic wash (d) final purification, III. Material Balance.

A flow-sheet, numerous curves, and a diagram of the equipment are included.

The acetone is produced by the catalytic reaction of steam with an acetylene-containing gas resulting from thermal conversion of methane. The by-products, which must be removed, include acetaldehyde, acetic acid, and higher ketones and aldehydes.

182

2p

5-11-43

Acetone Purification at Knappsack (Memo of Visit)

Differences in processing, particularly regarding the caustic wash, were discussed by men from Oppau and Knappsack. In caustic-washing, some of the acetone is lost, along with impurities. To minimize this, Knappsack uses a relatively low alkali concentration, low temperature, and long contact time.

183

5p

8-24-44

Catalysts for the Production of Acetone by Reaction of Acetylene with Steam. (Pat. App.)

Three claims: (1) The zinc oxide catalyst is prepared by roasting basic zinc carbonate, (2) The basic zinc carbonate is obtained by precipitation of solutions of zinc salts with carbonic acid, in presence of ammonium ion, (3) the precipitation is carried out in the absence of alkali.

These catalysts effect nearly complete conversion of dilute acetylene (e.g. 8% $\text{CH} \equiv \text{CH}$ - 92% methane) to acetone, at temperatures in the range, 350-450°C.

184

5p

8-24-44

Aqueous Butynediol ("Golpanol B") as Inhibitor

This item includes 4 short memos, 3 of which are photostats of limited legibility. They relate to problems arising in the production and application of 37% aqueous butynediol as an inhibitor ("sparbeize"). The removal of objectionable impurities, such as formaldehyde, is discussed.

185

lp

7-8-44

Sucol B : Paper-softener and Impregnating Medium

This short memo indicates that Sucol B is an aqueous solution of 4,4-dihydroxydibutyl ether.

186

4p

5-X-44

Oxidation Products of Propargyl Alcohol
($\text{CH} \equiv \text{C}-\text{CH}_2\text{OH}$)

Aqueous propargyl alcohol (e.g., 20%) undergoes catalytic oxidation when heated to 80-90°C. in the presence of CuSO_4 . Working-up of the reaction mixture leads to the following products: (1) acrolein, (2) methyl glyoxal, (3) propionic acid, (4) a resinous product reminiscent of caramel (20% yield), (5) a dark-brown glassy resin (30% yield).

187

2p

5-25-44

Fractionation of Propargyl Alcohol Solution

A 200-ton sample of dilute (3.3%) propargyl alcohol was concentrated by distillation. In addition to water, formaldehyde and methanol were present in important amounts, and were difficult to separate by distillation. The concentrated product was an azeotrope containing 37.5% of propargyl alcohol but also 3.6% of formaldehyde.

188

4p

8-22-44

Application for Trade-mark "Golpanol"

Typing is blurred, no technical data.

189-191

9p

4-4-44

Propargyl Alcohol (Product PL) and Butynediol (Product B) as Inhibitors and Rust-removing Agents

These 3 items consist of a series of very short memos containing little technical information, but relating usually to the supply situation, physiological aspects, etc. Both chemicals are marketed as aqueous solutions.

2p

192 Distillation of Propargyl Alcohol Liqour 5-31-43

Distillation of 103,390 Kg of an aqueous liquor containing methanol and formaldehyde in addition to propargyl alcohol gave the following results:

<u>Fraction</u>	<u>Weight (Kg)</u>	<u>Formaldehyde Content</u>
1. Methanol	7,270	0.9%
2. Propargyl alcohol azeotrope	13,140	4.5%
3. " " "Hochproz"	1,680	0.8%
Total Distillate	22,090	

193A

4p (1532-1535)

11-13-42

Production of Propargyl Alcohol from Acetylene and Formaldehyde

The Reppe process ($\text{CH} \equiv \text{CH} + \text{HCHO} \longrightarrow \text{CH} \equiv \text{C}-\text{CH}_2\text{OH}$) is reviewed; thanks to recent advances, its commercial application is contemplated. A brief description follows:

A solution contg. 10% formaldehyde, 65% tetrahydrofuran, and 0.01-0.10% sulfuric acid, is passed, together with acetylene, thru a tube filled with catalyst, at 90-100°C. and 10-12 atm. The reactor effluent has the following approximate composition:

Acetylene	-	0.2	Wt. %
Formaldehyde	-	0.8	"
Propargyl alcohol	-	6.7	"
Butyne diol	-	6.0	"
Tetrahydrofuran	-	60.0	"
Water	-	26.25	"
Sulfuric Acid	-	.05	"
		<u>100.00</u>	"

Processing of this mixture is described, and utilization of the propargyl alcohol is discussed.

193B

2p (1536-1537)

6-13-42

Preparation of Hexaminediol

A hand-written memo.

194

2p

5-30-41

Memo on Acetylene Purification

Comments on methods used at Schkopau and Ludwigshafen; difficult to summarize.

195

1p

5-11-42

Absorption of Acetylene by a Tetrahydrofuran-
Formaldehyde-Water Mixture

Data are tabulated for the solubility of acetylene, at atmospheric pressure and various temperatures, in a solvent composed of 60% tetrahydrofuran, 10% formaldehyde, and 30% water. At 20°C., 4.5cc. of acetylene are absorbed per cc. of the solvent.

These determinations were evidently made in connection with the Reppe propargyl alcohol process.

196

3p

5-13-41

Memo: Acetylene Purification Plant

This is a badly blurred photostat. The processing details are described, and the required equipment is itemized, for the treatment of 2100 m³/hr. of impure acetylene. The acetylene is to be freed of sulfur compounds (mostly organic) PH₃, and NF₃.

197

2p

5-7-41

Conference on Acetylene Purification

Discussion of equipment and processing technique

198

19p

11-28-40

Purity and Hydrogenation of Dry-Process
Acetylene

The quality of dry-process (Piesteritz) acetylene was tested by studying its hydrogenation to ethylene over a palladium catalyst. The effect of chemical purification was investigated.

Summary: Piesteritz acetylene has about the same phosphorus content but from 5 to 10 times the sulfur content (mostly organic) of wet-process acetylene. Hydrogenation of the crude acetylene showed a surprisingly high initial rate, with excessive ethane formation, followed by rapid decline of the catalyst activity. Washing of the crude gas with $\text{Ca}(\text{OH})_2$, and especially with $\text{Ca}(\text{OH})_2$, chlorine water, and NaOH , minimized the rate of catalyst deterioration.

199

2p

5-22-40

Dry-Process Acetylene (Memo)

This memo is very blurred in places.

200

3p

5-6-40

Dry-Process Acetylene (Conference Memo)

A largely-illegible photostat.

201

4p

12-19-44

Method of Preparing Unsaturated Carboxy
Acid Amides (Patent)

~~Two claims are granted. In the first, ketone-~~
cyanohydrins of a specified general structure are treated with concentrated sulfuric acid, oleum, or an alkyl sulfuric acid, at temperatures up to 110°C ., the reaction mixture is further heated to a maximum of 140°C ., and the unsaturated carboxy acid amide is obtained by suitable processing of the product. In the second claim, the reaction between the cyanohydrin and the acid is conducted in the presence of a polymerization inhibitor.

202

2p

12-18-44

Method of Preparing Pentachlorobutadiene (Patent)

One claim. Trichloroethylene is heated in the presence of small amounts of iron chlorides which cause the HCl-splitting to proceed at a practical rate but do not retard too strongly the polymerization of trichloroethylene, the reaction being conducted under pressure and at temperatures in the range 150-300°C.

203

4p

11-25-44

Process and Apparatus for Continuous Sulfonation and/or Neutralization (Patent)

Two rather complicated process claims. Sketch of reaction vessel is included.

205

3p

10-6-44

Method of Preparing Fatty Acids of High Molecular Weight (Pat. App.)

One claim. High M.W. fatty acids are prepared from high M.W. alcohols or waxes by subjecting these substances to the action of aqueous alkalis at elevated pressure and temperature.

206

12p

6-5-44

Reaction of Water Gas with Tetrahydrofuran

Water gas reacts with tetrahydrofuran at 200 atm. and 140-180°C., to give 3 chief products: (1) C₅ and C₆ diols, (2) hydroxymethyltetrahydropyran,



CH₂OH,

and (3) δ-valerolactone. The catalyst must contain about 5% cobalt, nickel is no good. This report contains extensive experimental data regarding the effect of reaction variables, catalyst composition, apparatus, etc.

207

25p

5-30-44

Reaction of Carbon Monoxide with Tetrahydrofuran

"It was found that in the presence of cobalt catalysts, CO reacts with tetrahydrofuran to yield δ -valerolactone; this is in contrast to the reaction in the presence of nickel catalysts and halogens, known to give rise to adipic acid. Yields of δ -valerolactone of 60-80% are obtained, and the process can be conducted continuously with space-time yields of 1:0.2."

208

16p

5-25-44

Production of Adipic Acid from Tetrahydrofuran and Carbon Monoxide

This item is the experimental section of a general report, and consists largely of tables and curves. Contents:

- (A) Experiments with stoichiometric amounts of iodine or nickel carbonyl, effect of reaction velocity, reaction temp., ratio of valeric acid to adipic acid.
- (B) Corrosion and experiments to reduce this by addition of propargyl alcohol.
- (C) Experiments in platinum apparatus
- (D) Halogen-free and nickel-free experiments
- (E) Various activators
- (F) Bromine as halogen source

209

1p

4-22-44

Synthesis and Reactions of Nitroalkenes

The addition of aldehydes to nitroparaffins yields nitroalcohols which are dehydrated to nitroalkenes, e.g., nitromethane + formaldehyde \longrightarrow nitroethanol $\xrightarrow{-H_2O}$ nitroethylene. Also synthesized were 1-nitro-2-propene and 2-nitro-1-propene. The polymerization, copolymerization, reduction, and addition reactions of these compounds are briefly discussed.

210

4p

4-14-44

Production of Dichloroacetylene

This is a brief description of a continuous process for dichloroacetylene, based on DRP 495,787. Acetylene is contacted countercurrently with strongly alkaline hypochlorite solution, and the product is stripped from the effluent solution with air-free nitrogen. Yields up to 90% of theory, and conversions of 60-70%, are attained. A table lists known reactions of dichloroacetylene.

211

2p

4-12-44

Condensation Products of Lactames and Organic Acids.

"Up to now, we have investigated the condensation of ϵ -aminocaprolactame, α -pyrrolidone, and α -piperidine, with acetic anhydride, succinic acid, maleic anhydride, and phthalic anhydride." The esters of these new condensation products have plasticizing qualities, and are quite compatible with cellulose triacetate. Their medicinal action is to be investigated.

212

lp

1-26-44

Suberic Acid from 4,4'-Dichlorodibutylether

4,4-dichlorodibutylether is brought to reaction with metallic sodium. The reaction product, a "higher-molecular octyl ether," is then oxidized with nitric acid to give suberic acid ($\text{HOOC}-(\text{CH}_2)_6\text{COOH}$).

213

2p

2-23-44

Conversion of Butenediol or Butynediol to Hydroxybutyraldehyde

This memo requests revision of a recent patent application which had described the conversion of butynediol to hydroxybutyraldehyde in the presence of hydrogen and catalysts such as nickel. An example is adduced to show that butenediol also can be used as starting material, the conversion occurring on heating for 28 hours at 100°C. with water, in the presence of a finely-divided nickel catalyst.

214

lp

9-20-43

Dipropylcarbinol

Dipropylcarbinol prepared from 92% butyric acid differs from the product prepared from the pure acid, only in having a yellowish color. This can be removed by simple distillation.

215

5p

5-16-44

Symposium on Distillation (at Schkopau)

This item contains brief abstracts of some 17 different papers on distillation processes.

216

4p

7-5-44

Recovery of Metals in Catalytic Processes
(Pat. App.)

One claim is made: A process for the recovery of carbonyl-forming metals from the products of catalytic conversions characterized by heating these products in the presence of carbon monoxide, to temperatures that lie above the decomposition temperature (corresponding to the existing pressure) of the carbonyl of the catalyst metal in question. 4 examples.

217

2p

6-22-44

Method of Preparing Cyclic Ethers (Pat. App.)

Cyclic ethers of the type like tetra- and pentamethylene oxide are prepared by hydrogenating hydroxymethyl derivatives of these compounds or of their homologues at temperatures above 200 °C., under elevated pressure. Three examples are presented the first of which gives details for the conversion of tetrahydrofurfuryl alcohol to tetrahydrofuran and methane, by hydrogenation in presence of Ni catalyst at 290° and 200 atm.

218

2p

6-22-44

Method of Preparing Tetrahydrofuran
(Pat. App.)

A single claim specifies the catalytic hydrogenation of furfural at temperatures above 200°C. under elevated pressure, in neutral or acid medium. The aldehyde group is split off as methane.

219

6p

10-30-44

Method of Preparing Carboxy Acids (Pat. App.)

Two claims: (1) Method of preparing aliphatic or aliphatic-cyclic carboxy acids by heating cycloparaffinic carboxy acids having univalent functional substituents (particularly hydroxyl groups), or derivatives of these acids or the corresponding alcohols, aldehydes, and ketones, with alkalis or alkaline earths at temperatures above 100°C. (2) The reaction is performed in aqueous solution or in dilution with an organic solvent.

220

11p

4-25-44

Analysis of Products of the Glycerin Synthesis

Contents:

- I. Testing purity of n-propyl alcohol
- II. Water in Allyl chloride (a. with acetyl chloride
b. iodine-SO₂ solution)
- III. Allyl alcohol determination (a. bromometric
b. refractometer)
- IV. Analysis of monochlorohydrin-dichlorohydrin mixtures
- V. Analysis of crude and pure glycerin
 - (1) Water content (a. with I₂-SO₂ solution
b. with acetyl chloride)
 - (2) Residue by vacuum-drying
 - (3) Bichromate method without use of KI
 - (4) Glycerin content by density and refractive index.

221

10p

10-16-42

Method of Preparing Conversion Products of Allyl Compounds (Pat. App.)

One claim: The process is characterized by treating allyl alcohols (or compounds that go to allyl alcohols in the presence of alkali) at temperatures between 130° and 140°C., preferably 180-240°, with alkali or alkaline earth hydroxides.

222

3p

8-4-43

Recovery of Cobalt from Nickel Ores

This exchange of two letters discusses possible solutions of a problem arising at the Krupp works. A process of stepwise oxidation of the 3 metals involved (iron, cobalt, nickel) is recommended as suitable for immediate large-scale testing. The reverse process of fractionative reduction of the mixed oxides with reducing agents, is also discussed.

223

2p

8-30-43

Activated Charcoal Plant for Benzene Recovery

This letter discusses particulars of the design of a plant to be shortly constructed.

224

10p

10-11-44

Vapor Pressure, Density, and Viscosity of Aliphatic Amines

The importance of amines as rocket fuels gave rise to this compilation and correlation of physical data for amines.

225

7p

1-14-37

Explosion Experiments with Vinylacetylene

Contents:

1. Experiments with pure, gaseous "VA"
2. " " mixtures (i.e., with acetylene and vinylmethyl ketone)
3. Experiments with liquid "VA"
4. " " a mixture of "VA" and "di-VA"
5. Explosion prevention.

226

1p

5-16-44

Method of Preparing w,w'-dihydroxypolybutyl Ethers (Pat. App.)

"By distilling the residues obtained in the distillation of butanediol-1,4 (prepared by hydrogenating butynediol)."

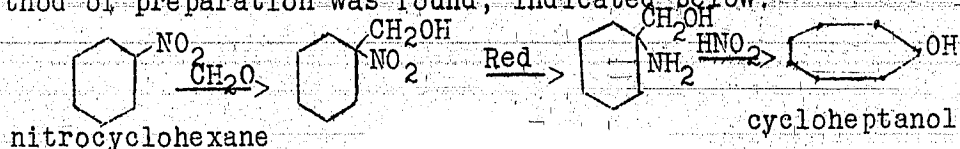
227

4p

4-22-44

Synthesis of Cycloheptanol

The processes known in the literature for the synthesis of cycloheptanol were improved and simplified. Finally, a new method of preparation was found, indicated below:



228

2p

4-3-44

Method of Preparing Polycondensation
Products (Pat. App.)

"by reacting polyisocyanates (e.g. 1,6-hexamethylene diisocyanate) with the distillable portion of the residue from the distillation of 1,4-butanediol (prepared from acetylene and formaldehyde, with butynediol as intermediate)."

229

5p

4-18-40

Batch Production of Hexamethylene Diamine

This short report describes the hydrogenation of adiponitrile to hexamethylene diamine in 120 Kg batches. With the cobalt catalyst, yields of 85-95% are obtained. A flow-sheet of the apparatus is included. Heat-transfer difficulties will be reduced by continuous processing on a larger scale.

230

4p

4-22-40

Hydrogenation of Adiponitrile to Hexamethylene Diamine

A summary of the technical development of the process to be shortly put into continuous use on a 70 liter (catalyst) scale. The product, hexamethylene diamine, is required for "the superpolyamide plant." A diamine yield of 80-90 is expected.

231

9p

5-7-40

Status of the Process for the Hydrogenation
of Adiponitrile to Hexamethylene Diamine

The chemistry of the reaction and process details are reviewed, and typical material balance data are presented. The formation of by-products, especially hexamethylene imine ($C_6H_{13}N$), is discussed.

The hydrogenation (with a cobalt catalyst) is conducted under 200 atm. pressure.

232

6p

2-19-40

Colorless, Water-soluble Sizing Based on
Polyvinyl Compounds

"By employing pure colorless acrylic acid one can obtain high-viscosity, clear, water-soluble polymerization products of acrylic acid and its salts. By copolymerizing with water-soluble or water-insoluble vinyl or acrylic compounds, such properties as solubility, viscosity, and film characteristics can be widely varied. By 'in-polymerization' of vinyl compounds with hydroxy or acid amide groups, one obtains polymers that give resins with aldehydes, especially formaldehyde. Products resinifiable with formaldehyde are also obtained by ammonolysis of polyacrylnitrile polymers." (from German summary).

233

1p

4-27-44

Removal of Silicic Acid from the Butynediol Solution

Experimental data are presented which show that $Mg(OH)_2$ and $[Mg(OH)_2 + Ca(OH)_2]$ are fairly effective for the precipitation of silicic acid. Neither of these reagents, nor $Al(OH)_3$, has any significant decolorizing effect on the butynediol solution; hence, the treatment with charcoal cannot be replaced.

234

5p

7-14-44

Separators for the Final Purification of
the Filtered Butynediol Solution

This memo discusses the effectiveness of the separators, contemplated revisions of equipment, analysis of separator influent, effluent, and sludge, etc.

235

6p

2-16-44

Silic Acid Content and pH of the Crude
Butynediol Solution

Contents:

1. Decrease in pH on standing
2. Silicic acid content

Several pages of tabulated data are included.

236

8p

1-26-44

Memo: Visit to the Hüls Chemical Works

Contents:

1. Hydrogenation experiments with butynediol at 700 atm.
2. Aldol hydrogenation
3. Shutting down in emergencies (air-raids)
4. "Butadiene oil" (Contains C₁ to C₄ aldehydes, ally-carbinol, acetic acid, etc.)
5. Butanol - final hydrogenation

Text is very technical and specific, not very informative to a stranger.

237

2p

3-27-43

Memo: Visit to Schkopau Experimental Plant

Contents:

1. Experimental plant for butynediol production and hydrogenation
2. Calculations
3. Catalysts
4. General

Text is not very coherent to a stranger.

238

15p

1-10-40

Butadiene by Cracking Butylene Glycol Diacetate

This report describes experimental work done on the thermal decomposition of 1,3- and 1,4-butylene glycol diacetate, to produce butadiene and acetic acid. The reaction goes best at 620-625°C., with yields of butadiene as high as 95% of theory. The effects of variables (temp., pressure, charge rate) and the production of the charge stock (by esterifying butylene glycol) are discussed in some detail.

239

4p

1-9-40

Butadiene by Cracking Butylene Glycol Diformate

A descriptive summary of the knowledge of the process as of Jan., 1940. Butylene glycol diformate is prepared by the addition of carbon monoxide to 1,3- or 1,4-butylene glycol, in the presence of sodium alcoholate. The diformate is then cracked thermally into butadiene and formic acid; it is important to minimize loss of the latter by undesirable side-reactions.

240

6p

12-15-39

Process for Producing Butadiene from
Formates of Butylene Glycols (Pat. App.)

Five claims: (1) Butylene glycols having hydroxyl groups on 2 different carbon atoms are converted to the diformate, which is cracked thermally or catalytically into butadiene and formic acid. (2) The formate is prepared by addition of CO to the glycol. (3) The esterification with CO is conducted in presence of less than 1% sodium in the form of sodium alcoholate, and the end-temperature is under 40°C. (4) Formic acid is added to complete the esterification. (5) The walls of the cracking reactor are constructed of materials that do not favor the decomposition of formic acid.

241

7p

4-27-39

Butadiene by Dehydration of 1,4-Butylene Glycol

1,4-Butylene glycol is dehydrated to tetrahydrofuran at temperatures in the range, 250°-400°, and over suitable catalysts the dehydration proceeds further to give butadiene. This report describes the development of a continuous process which features recycling of tetrahydrofuran and the use of phosphate-activated catalysts (P = 1 atm., T = 280°C.). Over a 4-week period, the yield of nearly pure butadiene was about 94% of theory.

242

18p

1-X-40

Principles of Heat Transfer and Design of
Heat-Exchangers

A summary of two lectures reviewing methods of calculation.