

FILM STUDY GROUP

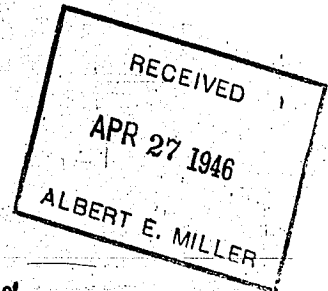
REPORT

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

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Review of Microfilm Reel #32 - Part I
U. S. Government Technical Oil Mission
I. G. Farbenindustrie - Ludwigshafen

Compiled by J. J. Somers
~~(Socony-Vacuum Oil Co.)~~
3-12-46

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Descriptions of Individual Items (#93-#173)..... Page 5-24

Introduction

This report presents a review of Microfilm Reel 32 - Part I (Reel 32 is actually two separate reels). The report consists of two parts: (1) An index of the items in the reel, arranged in the same order as their location in the reel, and (2) brief descriptions of the individual items.

The index is an improvement over that previously provided by the T.O.M. The original date and approximate length in pages are listed for each item, and the titles have been often revised to be more indicative of the nature of the contents.

Most of the descriptions of the individual items are essentially abstracts or brief summaries. For some items, however, the descriptions are limited, because of poor legibility or lack of German summaries or other difficulties, to brief comments designed to indicate the nature and scope of the subject matter.

The bulk of the items in Reel 32A relates to three general projects: (1) evaluation of rocket fuels, (2) research on the explosive decomposition of acetylene in the interests of safety, and (3) production of concentrated hydrogen peroxide (T-stuff).

The various reports on rocket fuels include an enormous amount of test data on "hypergol systems," i.e., fuel: oxygen-carrier combinations for use in engines which do not consume atmospheric oxygen, in which the two components react directly on mixing. Prominent among the oxygen-carriers mentioned are nitric acid (Ignol) and 85% hydrogen peroxide (T-stuff). As fuel components, blends rich in amines (particularly cyclohexylamine and aniline) or vinyl derivatives (various ethers) appear to have been important.

Judging from the collection of isolated research and development reports on this subject, T-stuff is produced in two steps. In the first, a 20 wt. % H_2O_2 solution is produced by the auto-oxidation of organic compounds which may be regenerated by hydrogenation, such as ethylanthrohydroquinone, and subsequent water-extraction. The second step is concentration of this solution by fractionation, a process made difficult by the ease of decomposition of H_2O_2 by traces of impurities and by the corrosive action of H_2O_2 on metallic equipment. Most of the items concerning T-stuff refer to these two steps, or to storage or testing of the product.

The section on acetylene decomposition indicates the importance of safety research in connection with various syntheses involving compressed acetylene, e.g., butynediol synthesis.

In addition to these three main topics, other subjects treated less extensively are indicated in the index. It should be mentioned here that the classification of material in the reel is not rigorous, and that anyone searching for specialized material should examine the entire index.

Index to Microfilm Reel 32 - Part I

Item No.	BAG 2078 Date	Approximate Length (Pages)	Descriptive Title
93	Aug. 23, '40	15	Research on the "Oxo" reaction (addition of CO + H ₂ to olefins and other unsats.)
94	Jan. 8, '44	5	(f) Evaluation of Rocket Fuels
95	Oct. 1, '44	13	Progress report on ergol fuels.
96	Sept. 30, '44	12	Effect of the oxygen-carrier: fuel ratio on ignition delay, in hypergol systems.
97	Aug. 28, '44	7	An apparatus for measuring ignition delay.
98	Aug. 28, '44	20	Ignition delay in a ternary hypergol system: Furfuryl alcohol + aniline + 2-methyl-tetrahydroprrole.
99	Aug. 20, '44	11	Physical and chemical studies of T-stuff (hydrogen peroxide solutions; catalytic decomposition).
100	Aug. 20, '44	5	Corrosion of metals by various oxygen-carriers.
101	Aug. 28, '44	12	Calculations for the combustion of organic compounds with nitric acid and hydrogen peroxide.
102	May 31, '44	16	Experiments with the hypergol ignition tester.
103	Jan. 5, '44	11	Hypergol reactivity of various organic compounds (ignition with HNO ₃ and H ₂ O ₂).
104	Feb. 10, '42	15	Effect of additives on the ignition of Methanol by air.
105	Apr. 15, '42	32	Hypergols having nitric acid as the oxygen-carrier, and various amines as the fuel-component.
106	Dec. 5, '42	15	Mixtures of cyclohexylamine and aniline as hypergol fuel-components.
107	Jan. 6, '43	15	Hypergol fuels based on mixtures of vinyl-n-butyl ether and butanedio-divinyl ether.
108	Jan. 6, '43	23	Summary of results obtained up to end of 1942 with fuels based on amines and vinyl derivatives.
109	Aug. 1, '39	20	Tables to be added to Item 107.
110	July 1, '38	95	Investigations of explosives based on hydrogen peroxide - Part II.
111	Undated	1	Investigations of explosives based on hydrogen peroxide - Part I.
112	'42-'43	3	Vapor-liquid equilibrium diagram: water-hydrogen peroxide. Graphs representing the decomposition of hydrogen peroxide concentrates.
113-114	Undated	2	(g) Explosive Decomposition of Acetylene Graphs representing the decomposition of acetylene-methane and acetylene-nitrogen mixtures.

Item No.	Date	Approximate Length (Pages)	Descriptive Title
115	Nov. 25, '43	5	Conference regarding safety in acetylene lines, particularly during air-raids.
116	Sept. 2, '43	3	Memo on the acetylene fire in the butynediol plant on Sept. 1, 1943.
117	June 11, '43	9	Acetylene content of air.
118	Sept. 24, '42	31	Summary report: Research on acetylene decomposition.
119	Nov. 11, '42	3	Drying acetylene (with potassium hydroxide).
120	Aug. 22, '42	13	Conference report discussing an acetylene explosion (decomposition) in the butynediol reactor at Schkopau.
121	Jan. '42	5	Commercial production and purification of acetylene.
122	Dec. 29, '41	2	Chlorine and sulfur content of Ludwigshafen (606) acetylene.
123	Nov. 25, '41	5	Conference on safety in handling acetylene (butynediol production).
124	July 15, '41	4	Preventing decomposition of acetylene flowing under pressure (patent appl.).
125	Dec. 10, '40	7	Conference on acetylene decomposition.
126	Aug. 28, '40	4	Acetylene explosion-arresters.
127	- '40	5	Preventing acetylene-air explosions by addition of CO ₂ or N ₂ .
128	June 15, '40	1	Explosion-arrester for acetylene decomposition.
129	June 6, '40	1	Ignition experiments with acetylene-CO ₂ mixtures.
130	Feb. 5, '40	6	Experiments on acetylene decomposition.
131	May 8, '30	38	The decomposition of concentrated and dilute acetylene, by employment of initial ignition.
132	Feb. 3, '36	17	Explosion experiments with vinylacetylene. (h) Manufacture and use of High-Concentration Hydrogen Peroxide (T-stuff)
133	Feb. 28, '45	1	Standardized testing of catalysts for T-stuff decomposition.
134	Feb. 23, '45	1	Discussion of further work on the decomposition of T-stuff.
135	Dec. 8, '44	60	Development of distillation process for concentrating hydrogen peroxide.
136	July 28, '44	2	Letter concerning aluminum tanks for storage of H ₂ O ₂ concentrate.
137	June 24, '44	2	Catalytic decomposition of hydrogen peroxide (T-stuff).
138	Aug. 17, '44	3	Instructions for storing hydrogen peroxide concentrate.
139	July 1, '44	24	Instructions for handling and storing hydrogen peroxide concentrate.
140	July 3, '44	12	Directions for various analytical tests on D1R & D2R (H ₂ O ₂ concentrates).
141	Mar. 7, '44	16	Safety in storage of hydrogen peroxide concentrate, particularly against explosive and incendiary bombs.
142	Feb. 21, '44	1	Drawing of aluminum tank for T-stuff storage.

Item No.	Date	Approximate Length (Pages)	Descriptive Title
143	Undated	1	Flow sheet of plant for manufacturing T-stuff (H ₂ O ₂ concentrate)
144	June 24, '44	10	Detail drawings of flanges and tubes for T-stuff storage.
145	July 18, '44	1	Letter concerning aluminum tanks for T-stuff storage.
146	Aug. 7, '44	2	Transport of T-stuff (letter).
147	Aug. 16, '44	13	Hydrogen peroxide distillation process: 2-stage vaporization in one column with two bottoms.
148	Aug. 28, '44	18	Physical and chemical data on T-stuff.
149	Dec. 20, '43	3	Stabilizers for T-stuff (to inhibit H ₂ O ₂ decomposition)
150	Dec. 1, '43	3	Preparation 77 (T-stuff stabilizer).
151	Nov. 16, '43	2	Solvents for use in hydrogen peroxide production.
152	Oct. 16, '43	7	Proposed process for concentrating the 20% H ₂ O ₂ solution.
153-5	June 4, '44	3	Drawings of aluminum tanks for storing H ₂ O ₂ concentrate.
156	Mar. 30, '45	2	Flow sheet of process for obtaining 85% hydrogen peroxide from ethylanthraquinone.
157	Apr. 29, '43	4	Requirements of alloy steels, aluminum, etc., for the hydrogen peroxide plant.
158	Jan. 5, '42	3	Hydrogen peroxide from alkylanthraquinones.
159	Apr. 8, '37	2	Process for producing hydrogen peroxide (patent).
160	June 1, '40	5	Process for the catalytic reduction of free fatty acids to the corresponding alcohols.
161	June 20, '38	7	Process for producing peroxides, especially H ₂ O ₂ .
162	Nov. 9, '37	8	Process for producing peroxides (especially H ₂ O ₂).
163	Apr. 3, '36(?)	9	Production of hydrogen peroxide and alkali peroxides by auto-oxidation of organic compounds.
164	Oct. 9, 1935	5	Process for producing hydrogen peroxide.
165	Feb. 8, '43	7	(i) Hydrogen Supply of Ludwigshafen
166	Nov. 22, '40	10	Memorandum: Hydrogen supply at Ludwigshafen and Oppau. Electrolytic hydrogen balance.
167	Apr. 20, '44	3	(j) Physical data on Butadiene and Related compounds
168	Undated	26	Thermodynamic constants of butadiene.
169	Undated	7	Physical properties of butadiene and related compounds.
170	May 23, '41	16	(k) Physical and Chemical Properties of Acetylene and Derivatives
171	Nov. 22, '44	9	Properties of acetylene and some derivatives thereof.
172	July 12, '44	3	Determination of chlorine, sulfur, and phosphorus in acetylene
173	June 1, '44	28	(l) Ignition characteristics of motor fuels; Improvement of Same Current problems under consideration at the Oppau Engine Labs. Engine ratings of various fuels and additives. The preparation, properties and engine behavior of some tertiary butyl ethers.

DESCRIPTIONS OF INDIVIDUAL ITEMS (#93-#173)

93

15p

8/23/40

Research on the "OXO"-Reaction

This report contains extensive experimental data for the addition of CO + H₂ to numerous olefins and other unsaturated compounds, and for the subsequent conversion of the aldehydes thus formed to alcohols (hydrogenation) or acids (oxidation). Among the charge stocks used were octadecene, cetene, cracking-olefins, acrylic acid, vinyl chloride, etc. Several different catalysts were employed. With the Fischer-Tropsch catalyst (Cobalt-Thoria on Kieselguhr) approximate conditions for the aldehyde step are 100-150°C. and 120 atm.

94

5p

1/8/44

Progress Report on Ergol Fuels

The composition of Ergol 51 is:

- 21.3% amine mixture
- 20.0% Optol I
- 20.0% Visol 6, crude (cyclohexylamine)
- 18.7% Xylol
- 20.0% heavy gasoline

In the work reported here, the effect of replacing several of these components with other materials was investigated; ignition tests were made at room temperature and at -60°C., by dropping nitric acid on the test mixture contained in a crucible.

95

13p

10/1/44

Effect of the Oxygen-Carrier:Fuel Ratio on
Ignition Delay, in Hypergol Systems

Six different combinations of O₂-carrier and fuel component were investigated. Extensive tabulated and graphical data.

96

12p

9/30/44

An Apparatus for Measuring Ignition Delay

An apparatus is described which measures (by photocells) the time elapsed between contact and ignition, in hypergol systems. The oxygen-carrier (e.g., nitric acid) is dropped into a crucible containing the fuel mixture. Amply illustrated.

97

7p

8/28/44

Ignition Delay in a Ternary Hypergol System:
Furfuryl Alcohol + Aniline + 2-Methyltetrahydropyrrole

Ignition delay was determined as a function of composition for a number of mixtures. It is possible to construct a three-component diagram from the data for binary mixtures.

98

20p

8/28/44

Physical and Chemical Studies of T-Stuff
(H_2O_2) Solutions; Catalytic Decomposition

The decomposition of H_2O_2 solutions was studied with and without catalysts. The decomposition was found to be greatly influenced by the material of the containing vessel, and by temperature, catalysts, H_2O_2 concentration; also by the chemical composition, amount, and surface character of the catalysts.

The catalytic effect of aluminum was especially investigated, since this metal is used to store T-stuff.

The gradual decomposition of T-stuff (H_2O_2) at room temperature is minimized by the addition of stabilizers, such as phosphoric acid.

99

11p

8/20/44

Corrosion of Metals by Various Oxygen-Carriers

Some 16 different metals and alloys were tested for their resistance, at 20° and 40°C., against 80% T-stuff (H_2O_2), "Mixed acid MS10," and "Salbei K4." The tests were of 24 to 72 hours duration.

Against all three O_2 -carriers, pure aluminum was most resistant. Of the iron alloys, V2A steel was best. Welded aluminum was attacked much more strongly than unwelded aluminum.

Iron alloys are fairly stable toward 80% H_2O_2 .

100

5p

8/20/44

Calculations for the Combustion of Organic
Compounds with HNO_3 and H_2O_2

Stoichiometric calculations, no data.

101

12p

8/28/46

Experiments with the Hypergol Ignition Tester

Ignition delay is measured electronically in a set-up where the O₂-carrier is dropped through a nozzle into a crucible containing the fuel-component. The operation of the tester was studied as a function of (a) crucible size, (b) temperature, (c) height of dropping, (d) chemical nature of O₂-carriers and fuel-components.

102

16p

5/31/44

Hypergol Reactivity of Various Organic Compounds (Ignition with HNO₃ and H₂O₂)

Extensive tabular and graphical data are presented to compare the hypergol fuel reactivity of numerous types of compounds, e.g. (1) organic amines, (2) organic compounds with high reduction potentials, especially aldehydes, polyhydroxyphenols, and unsaturates, (3) metal-organic compound.

Amines (e.g., aniline and cyclohexylamine) react very strongly with HNO₃ and H₂O₂. Organic amines were found to be the best fuels for HNO₃, and inorganic amines (hydrazine hydrate) were found best for H₂O₂.

The effect of catalysts on the ease of ignition of various systems was also investigated.

This is "Hypergol Report 8, Part I."

103

11p

1/5/44

Effect of Additives on the Ignition of Methanol by Air

No additive could be found which would increase the ignition temperature of methanol and prevent its "glow-ignition." In a series of experiments conducted in an apparatus swept through with fuel:air mixtures, the general rule was found that O₂-rich compounds have lower ignition temperatures than compounds containing little or not oxygen. The nitroparaffins particularly have very low ignition points.

104

15p

2/10/42

Hypergols Having Ignols as the Oxygen-Carrier
and Various Amines as the Fuel-Component

Pure amines in general react very well with nitric acid. However, only cyclohexylamine is available cheaply and in large amounts, and none of the amines have satisfactory properties in the cold (F.P., viscosity, D_{4}^{20} , stability, etc.)

In this investigation it was found that amine mixtures rich in cyclohexylamine ("Gola 6R") give improved results on the addition of solvents such as ethanol. Table I lists 6 fuel-mixtures which are recommended on the basis of high reactivity, low viscosity at low temperatures, availability, and cheapness.

This is Hypergol Report 2, Part I.

105

32p

4/15/42

Mixtures of Cyclohexylamine and Aniline as
Hypergol Fuel-Components

Mixtures of cyclohexylamine and aniline are very reactive with HNO_3 , but not all satisfy the required properties in the cold (must be liquids of viscosity ≤ 40 c.st. @ -40°C .). Moreover, because of the great affinity of cyclohexylamine for CO_2 , these mixtures are liable to be contaminated with solid salts (carbamates) which settle out and hinder the reaction.

It was found that the addition of diluents (benzene, gasoline, tetralin, vinylisobutyl ether) to cyclohexylamine-aniline mixtures not only improved their cold behavior, but greatly repressed carbamate formation. Table IV is a compilation of the best mixtures.

Data for a great number of mixtures.

This is Hypergol Report 2, Part II.

106

15p

12/5/42

Hypergol Fuels Based on Mixtures of Vinyl-n-Butyl
Ether and Butanediol-divinyl Ether

As the result of a great number of experiments, fuel mixtures of the composition indicated in the title, with the addition of important amounts of amines (aniline, pyrrolidine) are deemed quite satisfactory for ignition with nitric acid, from the stand-points of reactivity, properties @ -40° , availability, and power. The ignition tests were conducted at -40° , with and without iron catalysts. Two compositions are particularly recommended.

This is Hypergol Report 4, Part II.

107

15p

1/6/43

Summary of Results Obtained Up to End of 1942
with Fuels Based on Amines and Vinyl Derivatives

The twenty best fuel compositions are listed together with their properties and test results. The fuels based on amines ("Golas") are generally good, but their supply-situation is unsatisfactory. The fuels based on "Visol 41A" (mixtures of Vinyl-n-Butyl ether and butanediol-divinyl ether) have such good properties including availability that they are henceforth recommended as the substances to be used for ignition by nitric acid.

This is Hypergol Report 6, Part II.

108

21p

1/6/43

Tables to be Added to Item 107

Very extensive experimental data to supplement the foregoing report.

109

20p

8/1/39

Investigations of Explosives Based on
Hydrogen Peroxide - Part II

An experimental study of the detonation of mixtures of H_2O_2 with various organic compounds, especially alcohols and carboxy acids. A short summary at the start says: "All the mixtures investigated show multiple detonation. The chemical structure of the admixed compound affects the lowering of the maximum velocity. Multiple ignitions were released by percussion waves in the tube wall. Pure H_2O_2 up to now has been found to detonate with only one velocity."

Many tables and photos.

Investigations of Explosives Based on
Hydrogen Peroxide - Part I

Note the unusual length of this very complete and detailed report. A short summary at the start states: "Existence of several characteristic forms of detonation. Practical application possible."

The explosive character of H_2O_2 -fuel mixture was established, and it was shown that even pure H_2O_2 can be made to detonate.

Of the 3 alcohol mixtures investigated the one with ethanol proved most stable, decomposition being even less than with pure H_2O_2 . The maximum explosive action was obtained with the stoichiometric ratio for complete combustion.

Replete with tables, photos, and graphs; the graphs however are photostats, and not very legible.

111

1p

(Undated)

Vapor-Liquid Equilibrium Diagram for Water-Hydrogen Peroxide System

Concentrations in "Wt. %". Data for 40 mm. Hg pressure (?)
Two different curves plotted - separate investigations.

Equilibrium diagrams for this system are also given in several other items of this reel, i.e., those referring to T-stuff (H_2O_2) concentration. (See items 135, 147, 152)

112

3p

'42-'43

Graphs Representing the Decomposition of
Hydrogen Peroxide Concentrates

This item is mistakenly stamped 121 instead of 112.

Fig. 1: Decomposition End-Temperatures of T-Stuff at
Various Pressures and Initial Temperatures

Fig. 2: Same as Curvel, different range of variables.

Fig. 3: Decomposition End-Temperature and Moisture
Content at Various Pressures, as a Function
of the Heat of Formation and H_2O_2 Concentration
(Initial temperature = 0°C.)

No accompanying text.

113-114

2p

(Undated)

Graphs Representing the Decomposition of
Acetylene-Methane (113) and Acetylene-Nitrogen (114)
Mixtures

Three-dimensional plots:

X = acetylene concentration

Y = pressure of mixture

Z = temperature of mixture

115

5p

11/25/43

Conference Regarding Safety in Acetylene Lines,
Particularly During Air-Raids

Excerpts from this memo:

"To begin, Dr. Frost reported on the causes" of two
explosions "which occurred during the air-raid of Nov. 17, 1943"
(acetylene in lines detonated by bombs falling near-by).

"It is proposed to blow off the acetylene with air
through turbine-nozzles, carefully regulating the dilution with
air to prevent the occurrence of explosive mixtures."

116

3p

9/2/43

Memo on the Acetylene Fire in the Butynediol
Plant on Sept. 1, 1943

Damage not very great; fire caused by acetylene leak-
ing from defective welded joint.

117

9p

6/11/43

Acetylene Content of Air

At both Gendorf and Ludwigshafen, troubles have been
experienced in keeping the acetylene content of the air below a
safe minimum (at least one explosion has occurred). Causes and
corrective measures are discussed.

118

31p

9/24/42

Summary Report: Research on Acetylene
Decomposition

Experiments had been initiated in 1940-41, to determine the pressures attained in acetylene decomposition, and to discover means of preventing the change from explosive to detonative decomposition.

There is no summary to this rather lengthy report, but it appears that the safety-measure adopted consists of inserting tube-bundles or Roschig rings into acetylene lines. The higher the operating pressure, the smaller the diameter of the bundled tubes must be to prevent detonation. Tests have been conducted at pressures up to 15 atm. Hollow spaces must be avoided as they render useless the most elaborate tube-bundle protection.

Many graphs, tables, and photos.

119

3p

11/11/42

Drying Acetylene (With Potassium Hydroxide)

An exchange of letters discussing safety in drying $\text{CH} \equiv \text{CH}$ with KOH. Among matters covered: effect of phosphorus compounds, effect of chloroacetylenes, solid VS aqueous KOH, open VS closed tank for receiving spent alkali. Several explosions reported.

120

13p

8/22/42

Report Discussing an Acetylene Explosion (Decomposition)
in the Butynediol Reactor at Schkopau

No summary, many photos.

121

5p

1-42

Commercial Production and Purification of Acetylene

This is an article taken out of a technical journal for reference: *Kunststoff-Technik*, Jan., 1942, pp. 14-18.
Author: W. Scheruhn. Both the wet and dry processes are reviewed.

122

2p

12/29/41

Chlorine and Sulfur Content of Ludwigshafen
(606) Acetylene

Analytical data are listed for a number of samples.
No text.

123

5p

11/25/41

Conference on Safety in Handling Acetylene
(Butynediol Production)

Research on safety reviewed.

"To summarize, it may be said that the favorable outcome of the experiments requested by the CTR now makes safe the handling of acetylene compressed to 5-6 atmospheres, and that no more doubts can be raised against the Reppe butynediol process.... For newer syntheses employing pressures up to 15 atm., the series of experiments described herein constitute the necessary preliminary safety-research."

A more detailed report is promised.

124

4p

6/15/41

Preventing Decomposition of Acetylene Flowing
Under Pressure (Patent App.)

Liquids (gas oil or water) injected in finely-divided form into the tube wherein the compressed acetylene is flowing, control or completely prevent its decomposition. One example (18 atm.)

125

7p

12/10/40

Conference on Acetylene Decomposition
Questions up for discussion:

1. To how high a pressure may pure or diluted acetylene be safely compressed, i.e., without danger of a detonation, for transporting through long-distance pipe lines?

2. What tube diameter is necessary to supply 3000 c.b.m. acetylene at an initial pressure of 0.3 atmosphere to a point 83 km. distant; how great is the pressure loss?

No summary.

126

4p

8/28/40

Acetylene Explosion-Arresters

Test data show that the insertion of grooved sheet steel of specified dimensions insures the safe transport of acetylene under commercial conditions (20°C., 6 atm., 92% $\text{CH} \equiv \text{CH}$:8% N_2).

127

5p

'40

Preventing Acetylene-Air Explosions by
Addition of CO_2 and N_2

This is an article removed for reference from a technical journal: Autogene Metallbearbeitung, Vol. I. 1940, p. 2-6. Author: W. Gliwitzky. No. summary.

128

1p

4/15/40

Explosion-Arrester for Acetylene Decomposition

A "viscose sponge" was tested as an explosion-arrester for acetylene at 6 atm. and 25°C., and found to be ineffective.

129

1p

6/6/40

Ignition Experiments with Acetylene-Carbon
Dioxide Mixtures

A partly illegible photostat.

130

6p

2/5/40

Experiments on Acetylene Decomposition

~~The work reported here was conducted under conditions~~ similar to those employed in the "Reppe" (butynediol) process, in the interests of safety. The effects of temperature, pressure, and contact (copper acetylide) were studied. The variation of pressure during explosions was studied by means of an oscillograph. Several explosion-arresters were tested.

131

38p

5/8/30

The Decomposition of Concentrated and Dilute Acetylene, by Employment of Initial Ignition

This old photostat (1930) is illegible in places; this is particularly true of the graphs included in the report.

132

17p

2/3/36

Explosion Experiments with Vinyl Acetylene

The work described in this report was instigated by an explosion which occurred in the vinyl acetylene hydrogenator on Aug. 27, 1935.

The typing is blotted and rather difficult to read in some places.

133

1p

2/28/45

Standardized Testing of Catalysts for T-Stuff
(Conc. H_2O_2) Decomposition

This memo describes two different evaluation tests.

134

1p

2/23/45

Discussion of Further Work on the Decomposition
of T-Stuff

This one-page memo concerns planned cooperation, particularly in catalyst testing.

"Since deliveries of T-stuff can no longer be depended on to possess the original purity, because of the events in Lud. and Oppau, Dr. Fischbeck will, for the present, himself distill small amounts for his scientific work."

135

60p

12/8/44

Development of Distillation Process for Concentrating
Hydrogen Peroxide

The concentration of H_2O_2 by distillation, theoretically easy to perform, is made difficult by several important technical problems, e.g., the tendency of impurities to catalyze the decomposition of H_2O_2 , and the corrosive attack of metals by H_2O_2 . This report is concerned chiefly with ascertaining the best system of distillation (to minimize loss by decomposition), and with deciding on materials of construction. The experimental data are very extensive: tables, flow-sheets, curves. The final concentrate cannot be taken off as a bottoms product, but must be a vapor steam, to remove traces of non-volatile impurities; this necessitates 2 or more columns.

136

2p

7/28/44

Letter Concerning Aluminum Tanks for Storage
of H_2O_2 Concentrate

In part, the author says: ".....for this substance, only aluminum of 99.5% purity or non-rusting steels like V2A, Ramanite, etc., are stable."

137

2p

6/24/44

Catalytic Decomposition of Hydrogen Peroxide (T-Stuff)

An outline of experiments planned to be made. No particular catalysts are mentioned.

138

3p

8/17/44

Instructions for Storing Hydrogen
Peroxide Concentrate

An almost illegible photostat.

139

24p

7/1/44

Instructions for Handling and Storing
Hydrogen Peroxide Concentrate

Contents: A. Properties of T-Stuff, B. Storage of
T-Stuff, C. Rules for Conduct in Event of Danger, D. Accident
Protection, E. Storage Book-keeping.

140

12p

7/3/44

Directions for Various Analytical Tests on
D1R and D2R (H₂O₂ Concentrates)

Instructions are detailed for taking samples and for
carrying out various determinations, e.g., density by hydrometer,
density by Mohr-Westphal balance, purity by KMnO₄ titration,
acid content, evaporation residue, stability test, etc.

141

16p

3/7/44

Safety in Storage of Hydrogen Peroxide Concentrates,
Particularly Against Explosive and Incendiary Bombs

Among subjects discussed: Catalytic decomposition by
inorganic substances, such as FeCl₃, thermal decomposition,
burning with organic compounds, detonation, supplementary experi-
ments on safety against enemy-action, large-scale experiments
(photos)

142

1p

2/21/44

Drawing of Aluminum Tank for T-Stuff Storage

143

1p

(Undated)

Flow Sheet of Plant for Manufacturing T-Stuff

144

10p

6/24/44

Detail Drawings of Flanges and Tubes for
T-Stuff Storage

145

1p

7/18/44

Letter Concerning Aluminum Tanks for
T-Stuff Storage

"We need for experiments

3 tanks of 10 cubic meters capacity
3 " " 2.1 " " " "

3 paragraphs of discussion

146

2p

8/7/44

Letter Concerning Transport of T-Stuff

147

13p

8/16/44

Hydrogen Peroxide Distillation Process: 2-Stage
Vaporization in One Column With Two Bottoms

In this report a process for concentrating the raw 20 wt. % H_2O_2 solution to 85 wt. % H_2O_2 (75 mol %), is proposed and described. Two considerations are decisive in determining the distillation technique:

1. The separation of the H_2O_2 from high-boiling contaminants should be done at as low a concentration as possible.
2. The finished product must not come into contact with metal.

To meet these requirements, the purification of the feed and of the concentrated product are carried out as simple vaporizations, with intermediate fractionation to attain the desired concentration.

This report contains calculations and flow-sheets, no experimental data. See Items 135 and 152.

Physical and Chemical Data on T-Stuff

Collection of technical data taken from several reports issued in fall of 1944. No descriptive text, mostly graphs and tables. Included among contents: purity of T-stuff (H_2O_2) vs specific gravity, thermal decomposition of T-stuff, decomposition in presence of various catalysts, effect of material of containing vessel on decomposition, fuel test data, effect of ratio of O_2 -carrier to fuel on ignition delay, diagram of ignition-delay apparatus.

Probably all of this data is taken from reports appearing elsewhere in this reel (e.g., items 95, 96, 98, 101, 102).

Stabilizers for T-Stuff (to Inhibit H_2O_2 Decomposition)

In an exchange of letters, the supply of several inhibitors is discussed. One of these has the following composition:

hydroxyquinoline	41%
citric acid	59%

Another stabilizer mentioned is a mixture of quinoline and phthallic acid.

Preparation 77 (T-Stuff Stabilizer)

Two photostated memos, scarcely legible.

Solvents for Use in Hydrogen Peroxide Production

In the Pfeleiderer process a special solvent is used which consists of a mixture of secondary alcohols prepared by ketonizing and hydrogenating C_4 - C_6 fatty acids. In this memo, the supply situation and methods of producing substitute solvent are discussed, e.g., from butyric acid and from aldehydes obtained in the "OXO" process.

152

7p

10/16/43

Proposed Process for Concentrating the 20% H₂O₂ Solution

The 20% wt. % H₂O₂ solution is distilled in 3 steps. In the first step the solution is vaporized without reflux; the overhead is then rid of water by fractionation to the point where the bottoms product on re-vaporization gives an overhead of the desired concentration, i.e., 85 wt.% H₂O₂. 5% of the product is removed as sump from the bottoms of each of the two vaporizers.

Calculation, operating diagrams, flow-sheet.

See items 135 and 147.

153-5

3p

6/4/44

Drawings of Aluminum Tanks for Storing
H₂O₂ Concentrate

156

2p

3/30/45

Flow Sheet of Process for Obtaining 85% Hydrogen
Peroxide from Ethylanthraquinone

A pencil sketch. The ethylanthraquinone, dissolved in a solvent mixture, is hydrogenated in the presence of Raney nickel. The product is then oxidized with O₂ in 4 steps, and the H₂O₂ formed is extracted with iron-free water to yield a 20% H₂O₂ solution. This solution is concentrated by a special distillation technique to give 80-85 wt. % H₂O₂ (T-stuff). The quinone solution from the top of the extraction column is treated with K₂CO₃ to remove dissolved water and rid of impurities before recycling.

The solvent employed in the process is a mixture of benzene and cyclohexanol (or better, higher aliphatic alcohols (C₇-C₁₁)).

157

4p

4/29/43

Requirements of Alloy Steels, Aluminum, Etc., for
the Hydrogen Peroxide Plant

A conference report listing the individual items of equipment needed in the various stages of the process, and specifying the dimensions and the appropriate materials of construction.

158

3p

-1/5/42

Hydrogen Peroxide from Alkylanthraquinones

This is a photostat of a memorandum, illegible in places, describing the Pfleiderer process. The chemistry of the process is illustrated below:

1. Ethylanthraquinone + H₂ ———> Ethylanthrahydroquinone
2. Ethylanthrahydroquinone + O₂ ———> Ethylanthraquinone + H₂O₂

159

2p

4/8/37

Process for Producing Hydrogen Peroxide (Patent)

This is a copy of a patent issued to Riedl and Pfleiderer on April 8, 1937, for the production of H₂O₂ from easily auto-oxidizable organic compounds (such as hydrazobenzene and anthrahydroquinone). The distinguishing feature of the process is the use of a mixed solvent containing a component which dissolves well the starting material plus a component which dissolves well the oxidation product, and from which solvent the H₂O₂ formed is easily separated by settling or extraction.

In an example, the solvent employed for the hydrogenation-oxidation of 2-ethylanthraquinone is a mixture of 40 vols. Anisol and 60 vols. i-Heptanol.

160

5p

6/1/40

Process for the Catalytic Reduction of Free Fatty Acids to the Corresponding Alcohols

"On behalf of Director Dr. Reppe, work was begun in Sept. 1939, on the direct catalytic reduction of free fatty acids. The charge stocks first used were mixtures of C₅-C₁₁ fatty acids formed by paraffin oxidation, since these acids were available in larger amounts than were the acids from soap-fractions."

This short report includes data obtained on 25 kg. and 100 kg. scales, and a description and sketch of the hydrogenation unit. The two different fatty acid mixtures hydrogenated had mean molecular weights of 132 and 223.

161

7p

6/20/38

Process for Producing Peroxides, Especially H₂O₂

This patent application adds 3 claims to an application already on file for producing H₂O₂ from auto-oxidizable organic substances. In the cited process, oxygen dissolved in the liquid is removed before reduction by evacuating or by stripping with an inert gas. Now it is proposed to absorb the O₂ thus evolved, preferably by the reduced solution of the auto-oxidizable substance used in the same process.

162

8p

11/9/37

Process for Producing Peroxides (Especially H₂O₂)

This patent application discloses an improvement in the general process of producing peroxides, especially H₂O₂, from auto-oxidizable organic substances. The deterioration of the hydrogenation catalyst used to regenerate the starting compound is prevented, by completely removing (before hydrogenation) dissolved oxygen and organic peroxides still remaining after oxidation and separation of the bulk of the peroxides. Chemical methods (e.g., addition of ferrous compounds) are used to remove the O₂ and peroxides. 8 claims.

163

9p

4/3/36(?)

Production of Hydrogen Peroxide and Alkali Peroxides by Auto-Oxidation of Organic Compounds

This photostat is illegible in places. It is entirely descriptive in nature, and cannot be readily summarized. The chemical equations for the formation of H₂O₂ from anthraquinone (by hydrogenation-oxidation) are given on p. 1. The article is a review of the process developed by Pfleiderer.

164

5p

10/9/35

Process for Producing Hydrogen Peroxide

There are 5 claims to this patent application, the first of which follows:

"A process for the production of hydrogen peroxide consisting of the oxidation of cyclic compounds (especially polynuclear and substituted cyclics) which yield quinoid or indigoid compounds, the oxidation being conducted in neutral or weakly acid or alkaline medium with gases containing oxygen, and separation of the H₂O₂ formed."

165

10p

2/8/43

Memorandum: Hydrogen Supply at Ludwigshafen
and Oppau

"Repeated disturbances in recent months of the H₂ supply of the Ludwig. and Oppau plants, and the intended starting-up of the first butynediol hydrogenation unit in March, make necessary a new inspection of the present and future H₂-supply situation."

Descriptive matter and several tables and graphs in which the H₂-requirements are broken down.

166

3p

11/22/40

Electrolytic Hydrogen Balance

This photostat is almost entirely illegible.

167

26p

11/20/44

Thermodynamic Constants of Butadiene

Contains a lengthy descriptive summary including calculation methods, and eight tables of various thermodynamic constants listed as functions of temperature.

168

7p

(Undated)

Physical Properties of Butadiene and Related Compounds

Physical properties (including ignition points) tabulated for butadiene, butynediol, tetrahydrofuran, etc.

169

16p

(Undated)

Properties of Acetylene and Some Derivatives Thereof

Contents:

- A. Physical and chemical properties of acetylene
- B. Technical data for acetylene, butadiene, tetrahydrofuran, 1,3- and 1,4-butanediol, etc.
- C. Vapor-liquid equilibrium curves for water-tetrahydrofuran, water-butanediol, water-butynediol.

170

9p

5/23/41

Determination of Chlorine, Sulfur, and Phosphorus, in Acetylene

A largely-illegible photostat.

171

3p

11/22/44

Current Problems Under Consideration at the Oppau Engine Laboratories

It is revealed in this letter that the technical test-station had been largely destroyed by air-raids. However, plans are discussed for resuming work on a limited scale.

172

2p

7/12/44

Engine Ratings of Various Fuelds and Additives

A. Octane Nos. by the Injection Method:

3-nitrobutyl-methylketone	59.
3-nitropropyl-methylketone	56.5

B. Diesel Tests:

	<u>Cetane No.</u>
D717	54.0
Diesel Oil Op. 381	60.0
Light Oil Op. 381	46.0
Allyl-tetrasulfide Prep'n A (2 Vol % in D717)	76.0
Allyl-tetrasulfide Prep'n B (2 " " ")	82.0
Nitropropylmethylketone (5 " " ")	55.0
3-Nitrobutyl-methylketone (5 " " ")	54.5

173

28p

6/1/44

The Preparation, Properties, and Engine Behavior of Some Tertiary Butyl Ethers

Eight different ethers (e.g., i-propyl-t-butyl ether, glycol-mono-t-butyl ether) were synthesized. For most of these, motor octane numbers were determined, and for some their supercharge behavior was also studied.

Methyl-t-butyl ether, i-propyl-t-butyl ether, and perhaps too ethyl-t-butyl ether, give higher ratings than Diisopropyl ether.

The following methods of preparation were compared:

- (1) Norris & Rigby: Azeotropic distillation of the two alcohols in the presence of dilute acids
- (2) Addition of isobutylene to the alcohol, under pressure
 - (a) Rosinsky: FeCl₃ or ZnCl₂ catalysts
 - (b) A new process using slightly concentrated H₂SO₄.