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QUARTERLY PERIODIC STATUS REPORT  
OF THE  
HYDROGEN PEROXIDE LABORATORIES

**FC**

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## I. Stability of Hydrogen Peroxide (C. C. Lane and H. L. Kimball)

### A. pH of Diluted Hydrogen Peroxide Samples of Different Origins

Some question had arisen as to a possible variation in the pH on dilution of different samples of unstabilized concentrated hydrogen peroxide, as produced by the electrolytic, or the autoxidation processes, or from the oxidation of propyl alcohol. Accordingly, specimens of unstabilized hydrogen peroxide, supplied by different manufacturers were diluted with conductivity water and the pH measured over the range 90-10%  $H_2O_2$  for the samples of electrolytic and propyl alcohol origin, and 50-10% for the sample from an autoxidation process as a further check, the pH of each sample was also measured after tenfold dilution. The results are shown in the accompanying Figure 1. It is apparent that the behavior of all three samples is similar on dilution, although at any given concentration of hydrogen peroxide the pH of the autoxidation-process sample ran somewhat lower than the average of the other two.

It is concluded that the differences in pH reported must have been due to a variation in the quantity and/or kind of stabilizer employed in the particular samples involved.

### B. The Effect of Aluminum Upon Stability with Tin

A further study has been made of the mechanism whereby the stabilization of concentrated solutions of hydrogen peroxide afforded by the addition of sodium stannate declines on storage in aluminum containers. Further tests have been carried out which establish the fact that when metallic aluminum is in contact with aqueous solutions (in the absence of  $H_2O_2$ ) containing small concentrations of sodium stannate, a deposit is formed fairly promptly on the aluminum surface, but if concentrated hydrogen peroxide, containing the same proportion of stannate, is used, no change in the aluminum surface is noticed.

The white, even deposit obtained in about 5 min. on an aluminum strip, when aqueous stannate solutions were employed (best at  $85^\circ C$ ), was shown to contain tin when carefully scraped off, dissolved in hydrochloric acid, and treated with hydrogen sulfide. Considerable corrosion of the aluminum was visible and the solution gave a positive test with aluminum reagent.

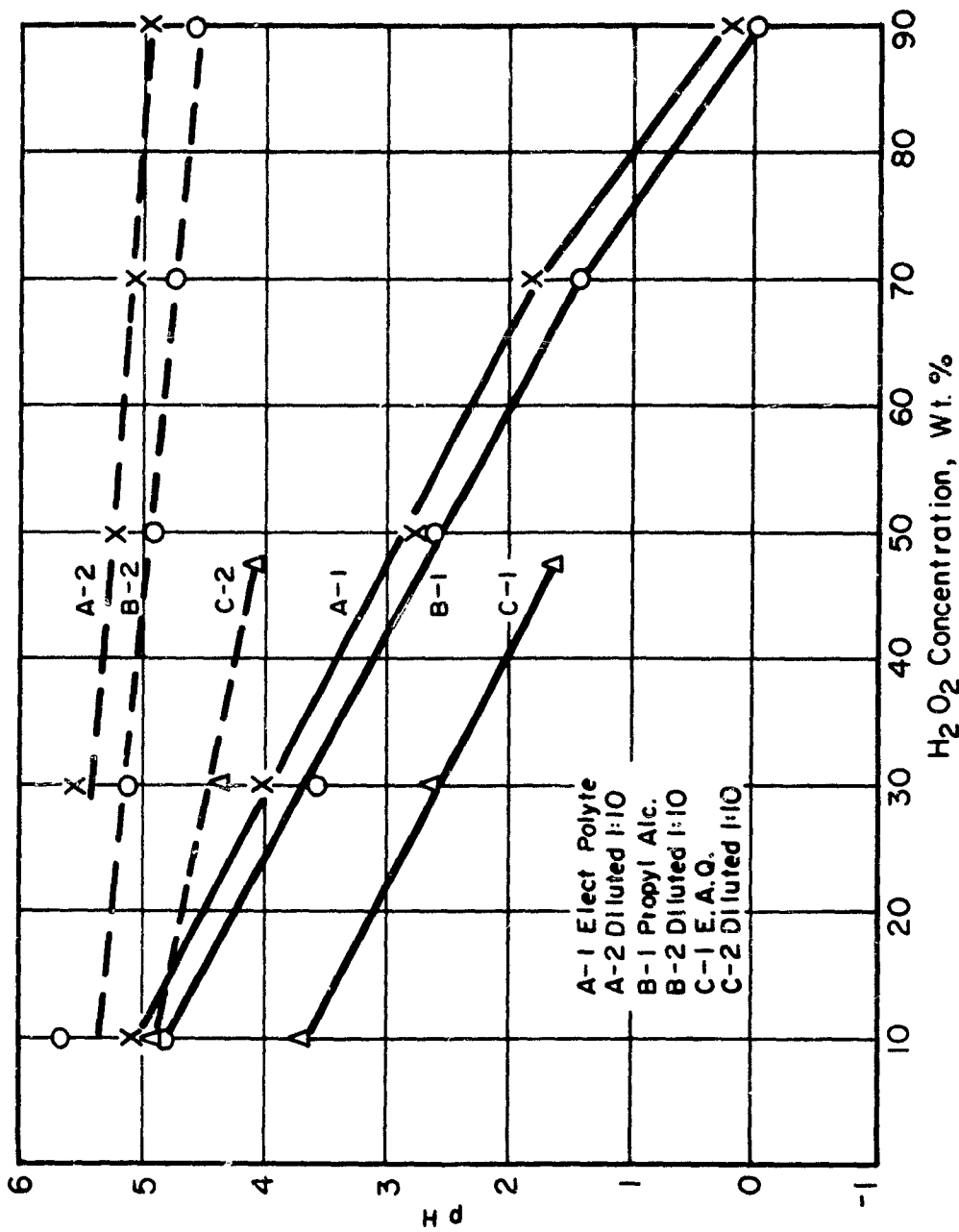


FIG. 1 - pH ON DILUTION OF H<sub>2</sub>O<sub>2</sub> MANUFACTURED BY VARIOUS PROCESSES

Comparative tests with aluminum strips, immersed at room temperature in aqueous and 90% hydrogen peroxide solutions, respectively, and containing 10 to 50 p.p.m. Sn (in steps of 10 p.p.m.) added as sodium stannate, were made over a period of two months. The strips immersed in the aqueous stannate solutions all became coated in a few days with a film, initially brownish in color but later turning to a grey-white. The aluminum strip in the most dilute stannate solution (10 p.p.m. Sn) did not change in appearance till near the end of the period of observation. In the case of the stannate solutions in hydrogen peroxide, no change in appearance was visible in any of the tests throughout the period.

In view of the negative results with the  $H_2O_2$  solutions, other tests have been started with 90% hydrogen peroxide solutions containing from 50-400 p.p.m. Sn (as stannate), in the pH kept definitely above that of the untreated hydrogen peroxide solution (approximately 1.5). In another test, the possibility of corrosion of aluminum by  $H_2O_2$  vapor, using aluminum, which has become coated by means of immersion in aqueous stannate solution, is being investigated. Other tests are planned in which contact of aluminum with solutions of more dilute hydrogen peroxide--30, 50, 70%  $H_2O_2$ --containing stannate is to be studied, to determine, if possible, at what concentration of hydrogen peroxide the deposition of tin ceases.

In connection with the work described above, decomposition rate measurements were carried out at 50°C on 90% hydrogen peroxide solutions containing 30 p.p.m. of tin (added as sodium stannate), some of which contained strips of aluminum. After a two week period at 50°C, 0.1 p.p.m. of  $Fe^{+++}$  was added (as the sulfate) to each sample. It was the expectation that in the case of the solutions containing the aluminum strips, conceivably enough tin may have been deposited from the solution so that addition of the ferric ion would cause a definitely greater rate of decomposition than that shown by the solution not in contact with aluminum. However, the amount of stannate used proved sufficient to hold back the catalytic effect of the 0.1 p.p.m.  $Fe^{+++}$ , as no appreciable difference in the rates was observed.

(It may be remarked in passing that we had demonstrated earlier--see Dec. 31, 1956 report-- that when a soluble aluminum salt is added to such a solution, precipitation of the tin does occur in a degree sufficient to show a resulting rise in the decomposition rates.)

Further similar trials in which less stannate was used (10 p.p.m. Sn) have shown a greater initial rise in the decomposition rate when 0.1 p.p.m.  $\text{Fe}^{+++}$  was added to the peroxide solution containing the metallic aluminum strips than occurred without aluminum in contact with the solution. This experiment is still in progress, however, and some convergence of the decomposition rates is noted.

In another series of decomposition rate studies, two different samples of 90% hydrogen peroxide were made up with the following additives:

Sample (1) 30 p.p.m. Sn (as stannate); 50 p.p.m. pyrophosphate ion (added as  $\text{Na}_2\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$ ); 30 p.p.m.  $\text{Al}^{+++}$  (added as sulfate).

Sample (2) 30 p.p.m. Sn; 50 p.p.m.  $\text{P}_2\text{O}_7^{4-}$ ; metallic strips of aluminum.

When these solutions were left at  $50^\circ\text{C}$  for 13 days, the decomposition rates remained essentially constant at about 0.0018% per hr. When 0.2 p.p.m.  $\text{Fe}^{+++}$  was then added to each sample, the rate for Sample 1 rose to about 0.08%/hr., while that for Sample 2 rose only to 0.013%/hr. The pH of Sample 1 was found to be 3.5 at the end of the run. This sample contained considerable flocculent precipitate, no doubt following hydrolysis of the aluminum and pyrophosphate ions, causing removal of considerable stabilizer from the solution. In the case of the sample containing the metallic aluminum strips, there was no visible precipitate in the flask bottom, indicating that the extent of hydrolysis in this sample was not appreciable. The pH at the end of the run was 5.3. The addition of 0.2 p.p.m.  $\text{Fe}^{+++}$  to this solution caused an increase in the rate from about 0.0018 to about 0.013%/hr--a much lower rate, by a factor of 6--than was obtained with Sample 1.

The conclusion to be drawn from the experiments conducted during the period of this Report is that in the presence of 90%  $\text{H}_2\text{O}_2$  aluminum metal does not appear to react with stannate, whereas with aqueous stannate solutions, rapid corrosion with deposition of a film containing tin, takes place. The mechanism whereby stannate disappears from concentrated hydrogen peroxide when stored for some time in aluminum containers remains uncertain. Further studies will be required with hydrogen peroxide solutions of intermediate concentration, containing stannate, in contact with aluminum, in the hope of discovering the cause of the observed phenomena.

## II. Flame Velocities in Hydrogen Peroxide Vapor (M.A.T. Mendes)

During the period covered by this report more data were obtained on the decomposition flame velocities above liquid hydrogen peroxide.

The results of flame velocities measurement by this technique are summarized in Fig 2 and are compared with velocities measured with the Bunsen burner technique as outlined in the quarterly report of June 30, 1956. In making this comparison only those velocities based on calculations made for point conditions for the "Bunsen" flame velocities were included since these appeared to be of higher accuracy. The data presented in the graph for a flame above the liquid phase are also summarized in Table 1.

The flame velocities listed in Table 1 and shown on Fig. 2 were calculated on the assumption that the liquid which disappeared was vaporized and then underwent decomposition in the flame. This velocity was calculated by considering a unit cross section (1 sq.cm.) of the liquid and determining the rate of vapor generation from the liquid at the measured liquid temperature assuming that the vapor acted as a perfect gas. The decomposition flame was assumed to be planar above the liquid.

With these assumptions, the flame velocities were calculated from the following relationship:

$$v = \rho_{T_L} \left( \frac{x}{34} + \frac{1+x}{18} \right) 22400 \left( \frac{cc}{mole} \right) \frac{T_L + 273}{273} (0.0328)$$

where

$v$  = flame velocity, ft/sec.

$\rho_{T_L}$  = density at  $T_L$ , g/cc

$x$  = weight fraction of hydrogen peroxide in one mole of the liquid, g

$T_L$  = liquid temperature, °C

$\ell$  = drop in liquid level per second, cm/sec.

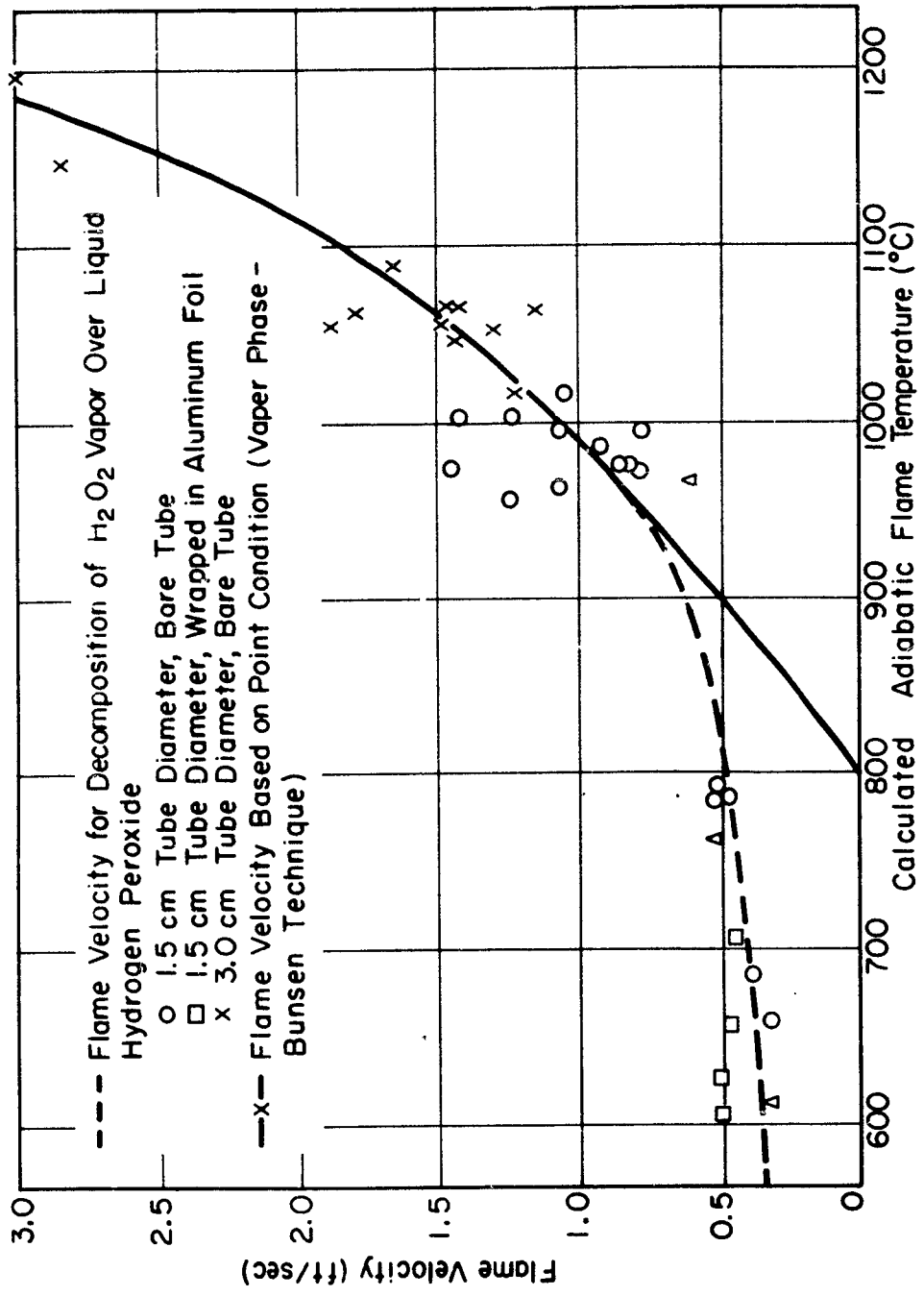


FIG. 2 - VARIATION OF VELOCITY OF HYDROGEN PEROXIDE DECOMPOSITION FLAME VELOCITY WITH ADIABATIC FLAME TEMPERATURE

TABLE I

Run	Liquid Concentration mole %	Flame Velocity ft/sec.	Liquid Temperature °C	Calculated Adiabatic Flame Temp. °C.
7	95	0.84	117	978
11	95	0.92	120	987
12	94.5	0.79	117	977
13	96	0.82	110-114	978
14	98	0.77	110-114	995
15	87	0.51	110	792
17	85.5	0.53	112	785
18	83	0.47	114	787
19	73	0.32	129	660
20	74	0.38	129	687
21	93.8	1.06	122	980
23a	94.6	1.43	136	1002
23b	92.8	1.46	136	975
24a	95	1.07	122	997
24b	96.8	1.05	122	1017
25a	95	1.24	130	1002
25b	92.7	1.24	130	957
26a	70.8	0.49	127	625
26b	69.8	0.49	127	607
27a	75.6	0.45	127	707
27b	72.8	0.46	127	657
28	97	0.59	114	967
29a	81	0.51	120	762
29b	71.2	0.3	129	612

The liquid temperature was measured with an iron-constantan thermocouple inserted into a well in the bottom of the test tube.

The adiabatic flame temperature was calculated by the relation:

$$\begin{aligned} & \chi \Delta H_0 - [\Delta H_v + mC_p \Delta T] = \\ & = \int_{T_B}^{T_P} \left[ (8.22 + 0.00015T + 0.00000134T^2) + \frac{\chi}{2} (8.27 + \right. \\ & \quad \left. 0.000258T - \frac{187700}{T_s}) \right] dT \end{aligned}$$

where

$\chi$  = mole fraction of hydrogen peroxide in the liquid

$\Delta H_0$  = heat of combustion of one mole of solution, cal.

$\Delta H_v$  = heat of vaporization of one mole of solution at its boiling point, cal.

$mC_p$  = heat capacity of one mole of solution, cal/°C

$\Delta T = T_B - T_2 = (T_2)$  difference between ( $T_B$ ), liquid boiling temperature and actual liquid temperature, °C.

$T_P$  = adiabatic flame temperature °K

The data now cover the entire range of both concentrations and liquid temperatures for which it was possible to obtain decomposition flames over the liquid. Taking into consideration the differences between the two methods by which the velocities were determined, it seems that a good correlation of data was obtained within the common region of adiabatic flame temperature. This region corresponds to the lowest values that could be obtained in the vapor phase Bunsen experiments and the highest that were obtainable in measurement of a flame over the liquid phase.

According to Fig. 2 the flame velocity increases with the adiabatic flame temperature. However in the region of the higher liquid concentrations corresponding to adiabatic flame temperatures of the order of 1000°C considerable scatter of the data was apparent.

In analyzing the data the liquid temperature appeared to influence the flame velocity beyond the effect of liquid temperature on the adiabatic flame temperature. It should be noted that for the decomposition above the liquid, flames were obtainable over the liquid of lower concentrations only at temperatures close to the boiling point of the liquid. At the higher concentration, flames could be obtained over a considerable range of liquid temperatures. Hence only at these higher concentration is it possible to search for any effect of liquid temperature on the flame velocity. The possible significance of liquid temperature remained uncertain at the time of the last quarter report. It now appears that there is some effect of liquid temperature on the flame velocity in this region. This effect is shown by plotting in Fig. 3 some of the data of Fig. 2 characterized by circles and identifying the individual liquid composition and temperature for each point.

The most important results obtained during the present work are:

1. Flames can be obtained above at liquid concentration over the range of 71.2 to 100 mole percent of hydrogen peroxide.
2. If the tube in which the liquid is burned is wrapped with aluminum foil.
  - a) flames may be obtained at liquid to concentrations as low as 69.8 mole percent,
  - b) at the same liquid concentration, flame velocities are slightly higher, and
  - c) the actual flame temperature is closer to the calculated adiabatic temperature.
3. If the tube diameter is increased no apparent influence is observed on the flame velocity except when working at high concentrations and at liquid temperatures as low as the temperature at which a flame is obtainable.

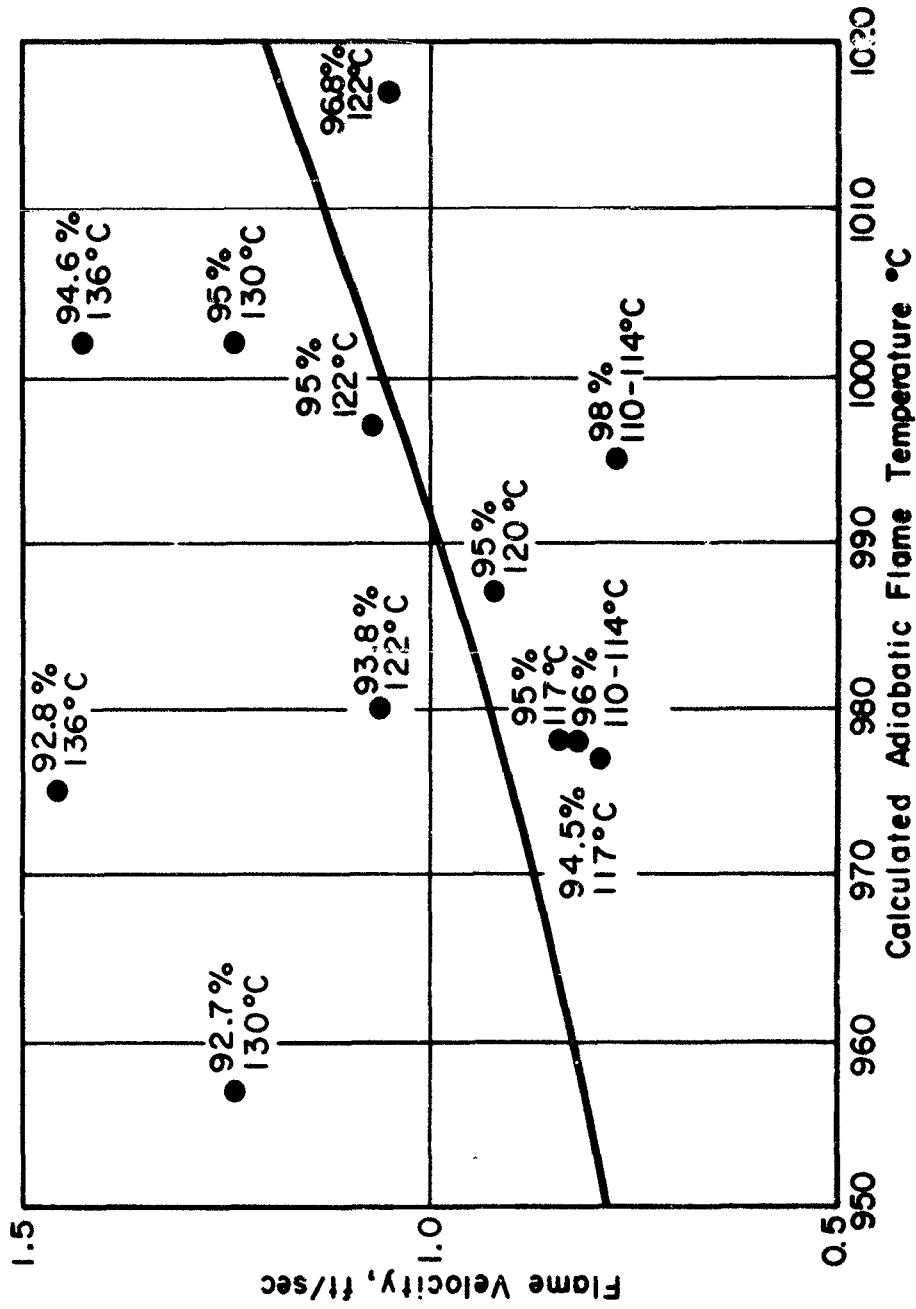


FIG 3 - EFFECT OF LIQUID TEMPERATURE ON VELOCITY OF DECOMPOSITION FLAME ABOVE LIQUID HYDROGEN PEROXIDE. (POINTS ARE LABELLED WITH LIQUID TEMPERATURE & CONCENTRATIONS)

In continuing with this study, future work will include determinations of flame velocities above liquid hydrogen peroxide at sub-atmospheric pressures. It is hoped that experiments at low pressure will provide information on the nature of the rate controlling step in the decomposition flame.

### III Supercooling of Liquid Hydrogen Peroxide (J. Roberts)

Studies have been made of the addition of 37-43 micron sized particles of both Pyrex and alumina to 98-99% hydrogen peroxide to determine their effect on the tendency of the hydrogen peroxide to supercool. The seeding materials were added to the hydrogen peroxide both above and below the equilibrium freezing point for  $H_2O_2$  of  $-0.43^\circ C$ . The results of the tests may be summarized as follows:

1. The Pyrex and alumina particles have no significant effect on the tendency of hydrogen peroxide to supercool.
2. The hydrogen peroxide did not freeze homogeneously down to the lowest temperature recorded in the tests ( $-68^\circ C$ ).
3. The freezing process tends to kill the active centers for crystal formation.
4. The freezing temperature of a peroxide sample is not raised by allowing the open test tube to accumulate dust from the air. (Six samples were left open to the air for periods of from 24 to 72 hours).
5. Some seven samples of hydrogen peroxide at temperatures below  $-54^\circ C$  froze following repeated finger tapping of the test tube. However, four samples thus agitated at temperatures from  $-48^\circ C$  to  $-54^\circ C$  showed no change.
6. Scraping the test tube wall with the glass stirrer caused supercooled hydrogen peroxide to freeze (six samples <sup>tried</sup> over the temperature range of  $-45^\circ C$  to  $-62^\circ C$  and each froze immediately on scraping.
7. Heating the sample before cooling does not have a noticeable effect on the freezing temperature of the sample.

The next phase of the study will be to determine the effect of adding a tetragonal seeding crystal, cassiterite, to hydrogen peroxide (hydrogen peroxide crystals belong to the tetragonal system). This will complete the study of the effect of seeding crystal shape on the freezing temperature of the peroxide. Then 5-9 $\mu$  crystals of the three substances, Pyrex, alumina, and cassiterite, will be added to the peroxide to determine the effect of crystal size on freezing point.

#### IV High Pressure Ignition Limits of Hydrogen Peroxide Vapor (F. Feskes)

During the past period, the construction and assembly of the equipment described in the previous Quarterly Report was completed. However, limited tests with the apparatus have indicated that the rate of decomposition of the hydrogen peroxide in the equipment is so high that several modifications will be required. First steps must be taken to decrease the area of aluminum exposed to the peroxide vapor. This will be accomplished by replacing parts of the aluminum inlet and outlet lines of the apparatus with Pyrex tubing. In addition, it is planned to line the compression cylinder with Teflon and to replace the head of the piston with the same material.

Those sections of the present apparatus which have been made of Teflon have been found to be satisfactory. According to Stein\* the rate of decomposition of hydrogen peroxide vapor on Teflon is some 5 to 7 times lower than on pickled aluminum at the temperatures to be used in the present study.

By decreasing the area of aluminum exposed to the vapor and increasing the vapor rate it is hoped that useful results will be obtainable.

While making the above alterations, construction will be initiated on a separate apparatus in which only Pyrex glass will come in contact with the hydrogen peroxide vapor. The apparatus will be similar in principle to that

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\* Stein, T. W., "Decomposition of Hydrogen Peroxide Vapor" So.D. Thesis M.I.T. (1955)

used by Satterfield, Kavanagh, and Resnick\*\* in the studies of the ignition limits of hydrogen peroxide vapor at atmospheric and sub-atmospheric pressures. Pressures in excess of one atmosphere will be obtained by enclosing the whole apparatus in a vessel pressurized with oxygen or helium.

#### V. Heavy Metal Catalysis of Hydrogen Peroxide Decomposition (J. W. Rice)

The purpose of this study is to determine quantitatively the rate of decomposition of hydrogen peroxide when catalyzed by the ions of lead, silver, and iron in aqueous solutions, and the factors affecting this rate.

The following experimental procedure has been used. A sample, (250ml.) of a 20% weight solution of hydrogen peroxide of known pH was placed in a 1000 ml. flask. A known amount of the metal nitrate in dilute aqueous solution was then added to the hydrogen peroxide and the rate of oxygen evolution measured with a wet test meter. The temperature was kept constant throughout the reaction by regulating the temperature of the surrounding water bath. The reaction mixture was agitated by means of a magnetic stirrer. Up to the present time, data have been obtained for lead nitrate as the catalyst in concentrations up to 0.5 g.  $Pb(NO_3)_2$  per liter of solution. The results indicate that the rate of decomposition of hydrogen peroxide is zero order with respect to hydrogen peroxide concentration for hydrogen peroxide concentrations up to 20%. The catalyzed reaction rate increased approximately three-fold as reaction temperature was raised from 25°C to 30°C. The reaction rate increased with pH from practically zero at a pH of 5.5 to a moderate value at pH 7.0. At a pH of 8.5 it became extremely vigorous. It is now planned to repeat the above type of tests using both silver nitrate and ferric nitrate as the catalysts.

#### VI. Visitors

Mr. L. Powell, Office of Naval Research, Boston, Mass.  
Mr. R. Levine, Rocketdyne

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\*\* Satterfield, C. N., Kavanagh, G. M. and Resnick, H.,  
Ind. Eng. Chem. 43, 2507 (1951)

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Experiment Incorporated Richmond, Virginia Attn: Dr. J. V. Miller, II	Naval Inspector of Ordnance Applied Physics Laboratory Johns Hopkins University 6411 Georgia Avenue Silver Spring, Maryland	1	Commander New York Naval Shipyard (Attn: Code 2444) Naval Base Brooklyn 1, New York	1	
Detroit Controls Company Research Division Redford City, California	Asst. Inspector of Naval Material 377 Middlefield Rd. Redford City, California	1	Shell Development Company Emeryville, California	1	
Fairchild Engine and Airplane Corp. Fairchild Engine Division Farmingdale, Long Island, New York Attn: Mr. S. M. Lester	Bureau of Aeronautics Rep. Fairchild Engine & Airplane Corp. Farmingdale, Long Island, New York	1	Office of the Chief of Ordnance Department of the Army Washington 25, D. C. Attn: GMSB	1	
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