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UNCLASSIFIED
QUARTERLY PERIODIC STATUS REPORT
OF THE
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DIC 6552

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I. Stability of Hydrogen Peroxide (E. C. Lincoln)

A. Conductivity of Hydrogen Peroxide Solutions

It was considered to be of interest to compare the conductivity of the purest hydrogen peroxide available at this time with that reported in the measurements carried out several years ago; especially as the results of similar measurements reported by the Leporte Laboratory in England showed a "flat maximum" in the curve of conductivity vs concentration extending over an appreciable range of concentration.

Accordingly, samples of "Becco" 90% peroxide were evaporatively redistilled at low pressure, avoiding actual ebullition and resulting contamination from spray. The concentrated solutions were diluted with triply distilled "conductivity water" to the desired concentrations. Measurements of the specific conductivity were made at 15°C in the same conductivity cell with cast tin electrodes as had been employed in earlier measurements.

The results of these measurements are indicated in the accompanying figure, which does not indicate any pronounced "flat maximum", although the change in specific conductivity for the range 33-60 wt.% H₂O₂ is only from 4.0 to 4.5 x 10⁻⁶ ohm⁻¹ cm⁻¹. These results indicate that the form of curve originally presented (Ind. Eng. Chem., 41, 992 (1949)) is correct, although the maximum values in the vicinity of 50% H₂O₂ may be revised downward slightly in line with the latest determinations.

B. Effect of pH upon Decomposition Rate

It has been observed previously that when 90% unstabilized H₂O₂ is diluted with water the rate of decomposition, expressed as percent decomposed in unit time, will not increase, provided that the apparent pH of the solution is adjusted so as to be held constant at 0.0. However, when acid is not added to maintain a constant pH the change in apparent pH brought about by mere dilution with pure water is from approximately 0.0 for 90% H₂O₂ to nearly 5.0 for 10% H₂O₂ -- the pH being measured directly with the glass electrode, without tenfold dilution. Figure 2 shows the variation in pH as dilution of the H₂O₂ is carried out, and also the increase in the decomposition rate at 50°C attending the dilution.

Further work is planned in which the pH of the solution will be held at other values than 0.0, such as 0.5 or 1.0, during the process of dilution of the 90% H₂O₂.

C. The effect of Surface-to-Volume Ratio on Decomposition Rate

Experiments carried out at the Leporte laboratories, Luton, England, in which ground glass particles were mixed with hydrogen peroxide in small glass vessels in order to offer a large area of glass in contact with the hydrogen peroxide solution under test, indicated that the surface-to-volume ratio was without significant effect upon the observed decomposition rate. In an attempt to explore this matter somewhat more completely, two sets of experiments were carried out. In one, the rate of

decomposition of 90% H_2O_2 was measured in the usual way, with a Pyrex gas evolution flask filled with different volumes of the solution. The flasks used for this purpose are 250 ml Florence flasks, having flattened bottoms but essentially round and with a radius, R , near 4 cm. The surface area exposed was calculated from measurements of the height of the liquid in the flask, h ; thus, $Area = 2\pi Rh$. The volume of liquid was determined by weighing the amount of hydrogen peroxide of known concentration and density added to the flask. The variation of S/V ratio possible under these conditions is shown in the curve "no rods" in Figure 3. The rates of decomposition measured are shown in Figure 4, which resembles earlier results from this laboratory (Prog. Rept., Mar., 1952), as well as the results quoted by Roth and Shanley (Ind. Eng. Chem. 45, 2343 (1953).) It was also found that the rate was the same when the flasks were half and totally full. Since the flasks used are spherical the surface-to-volume ratio is the same in both cases, and the duplication of results indicates a negligible contribution from vapor phase decomposition, either homogeneous or heterogeneous, under the conditions of these experiments.

To further increase the surface-to-volume ratio, in a second experiment, small pieces of Pyrex rod 2 mm in diameter and 1 cm in length were fire-polished to smooth the ends and conditioned in the same manner as the flask into which they were introduced. These rods had approximately 0.69 cm^2 area each, and the contribution to the S/V ratio calculated on this basis and including the flask surface is shown in the curves of Figure 3 for various fixed numbers of added rods. Figure 5 shows the results of decomposition rate studies on 90% H_2O_2 in which the surface-to-volume ratio was altered in this manner. Curve A is similar to that shown on a larger scale in Figure 1; the other curves were obtained by the addition of from 40 to 400 pieces of Pyrex glass rod to the flask with the proper amount of 90% H_2O_2 to yield the indicated surface-to-volume ratio. Although for a fixed number of rods immersed in varying volumes of liquid the rate of decomposition is proportional to S/V ratio, it is of note that for a given S/V ratio--say, 9--the decomposition rate observed declined from about 0.021% per hr. at 50°C when 100 rods were present, to about 0.011% when 400 rods were used. This result implies that if the procedure of adding more and more surface were carried far enough, the slope of the decomposition rate curve would decline to the horizontal, or that the rate of decomposition would be independent of the surface-to-volume ratio. This conclusion is in agreement with the observations of the Laporte laboratories, but its interpretation is not immediately apparent. It is conceivable that in a heap of glass particles immersed in the solution the full effect of the surface is not exerted, due to the many direct contacts between the particles, which prevent access of the solution.

D. The Effect of Radiation on Stability

It is clear that ultraviolet light can have a significant effect on the stability of hydrogen peroxide. This has been appreciated in the past, as evidenced by the fact that hydrogen peroxide solutions have been frequently sold in colored glass bottles or otherwise packaged to avoid illumination. However, the degree to which sunlight or other radiation causes decomposition under common conditions of handling and use is not well delineated. It is apparent that exposure to sunlight may result in heating, with concomitant rise in decomposition rate, as well as photochemical decomposition. At the same time there exists the obvious inconsistency of using a colored glass, which will ordinarily be of inferior quality with regard to stability because of its metal or alkali content, to overcome another source of decomposition. Coupled with this is the well-known fact that glass, hard or soft, is generally considered opaque to most of the ultraviolet portion of the spectrum.

The possible contribution of heating and relative activity of surfaces to supposed photochemical deterioration of hydrogen peroxide cannot be evaluated in a general way, but the facts concerning the likelihood of encountering photochemical decomposition can be estimated qualitatively by reference to Fig. 6. This representation shows that artificial light sources are not likely to cause appreciable difficulty; their radiation is diminished nearly to zero at wavelengths where absorption by hydrogen peroxide is only beginning and where quantum yields are presumably very low. In the exposure to direct sunlight, on the other hand, there appears to exist considerable risk of appreciable decomposition, particularly at the higher concentrations. Perhaps 4 percent of the sun's radiation occurs at wavelengths under the "toe" of the intensity curve shown which may be judged effective for decomposition. Furthermore, experience indicates that when samples of 90% unstabilized hydrogen peroxide, contained in glass vessels, are illuminated by direct sunlight, they may undergo decomposition to the extent of about 1% per day at room temperature. The curves of Fig. 6 also imply that clear glass cannot be counted upon to retard photochemical decomposition induced by the usual sources of illumination; this is particularly true for the Pyrex common in the laboratory. Glass (but not quartz or special glasses) is effective in screening the ultraviolet radiation shorter than 2800Å. This accounts for the fact that hydrogen peroxide contained in Pyrex has been found to be unaffected by illumination with a special ultraviolet lamp; such lamps, the so-called germicidal lamps, are operated so as to concentrate the radiation at the 2537Å mercury line, which is completely blocked by Pyrex.

These conclusions are of course only general; in any instance of importance it would be necessary to evaluate the absolute intensity and wavelength distribution of the incident radiation as well as the quantum yield and absorption in the particular concentration and the container geometry. It will be noted that the circumstances favor rapid changes in decomposition rate as

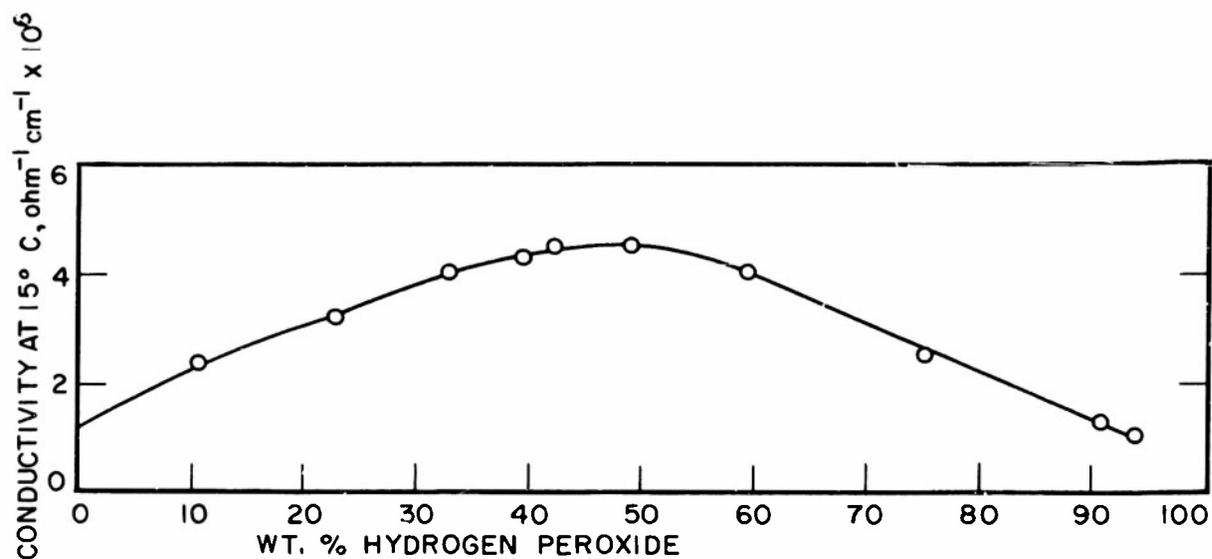


FIG. 1 - CONDUCTIVITY OF REDISTILLED AQUEOUS HYDROGEN PEROXIDE.

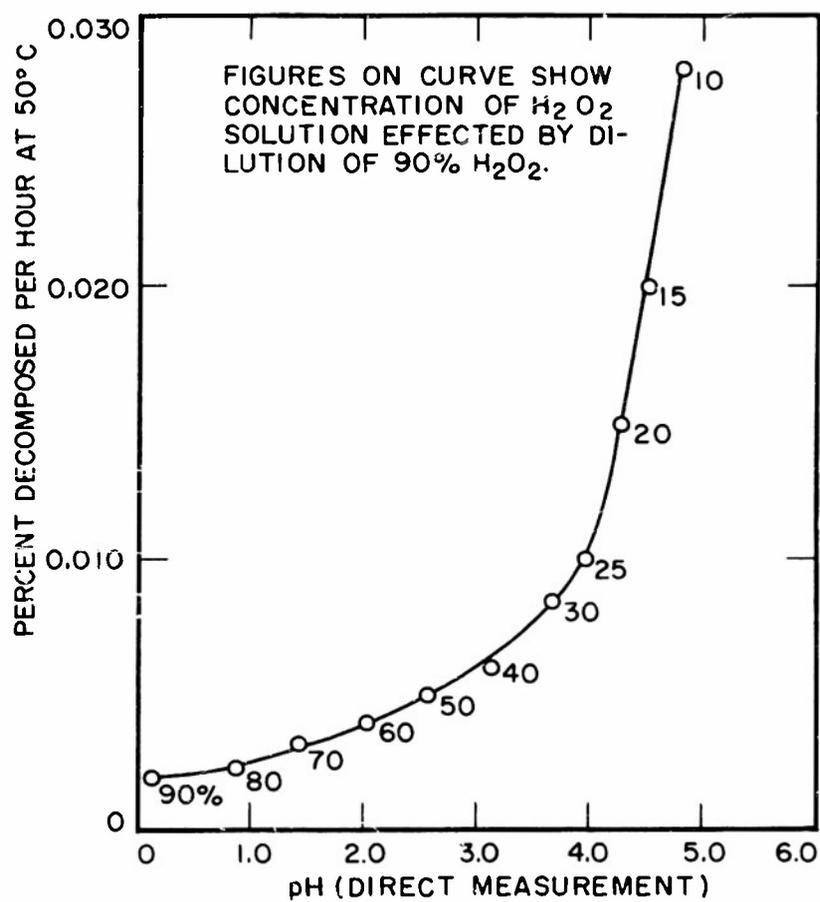


FIG. 2 - DECOMPOSITION vs. pH (pH CHANGE BY DILUTION ALONE)

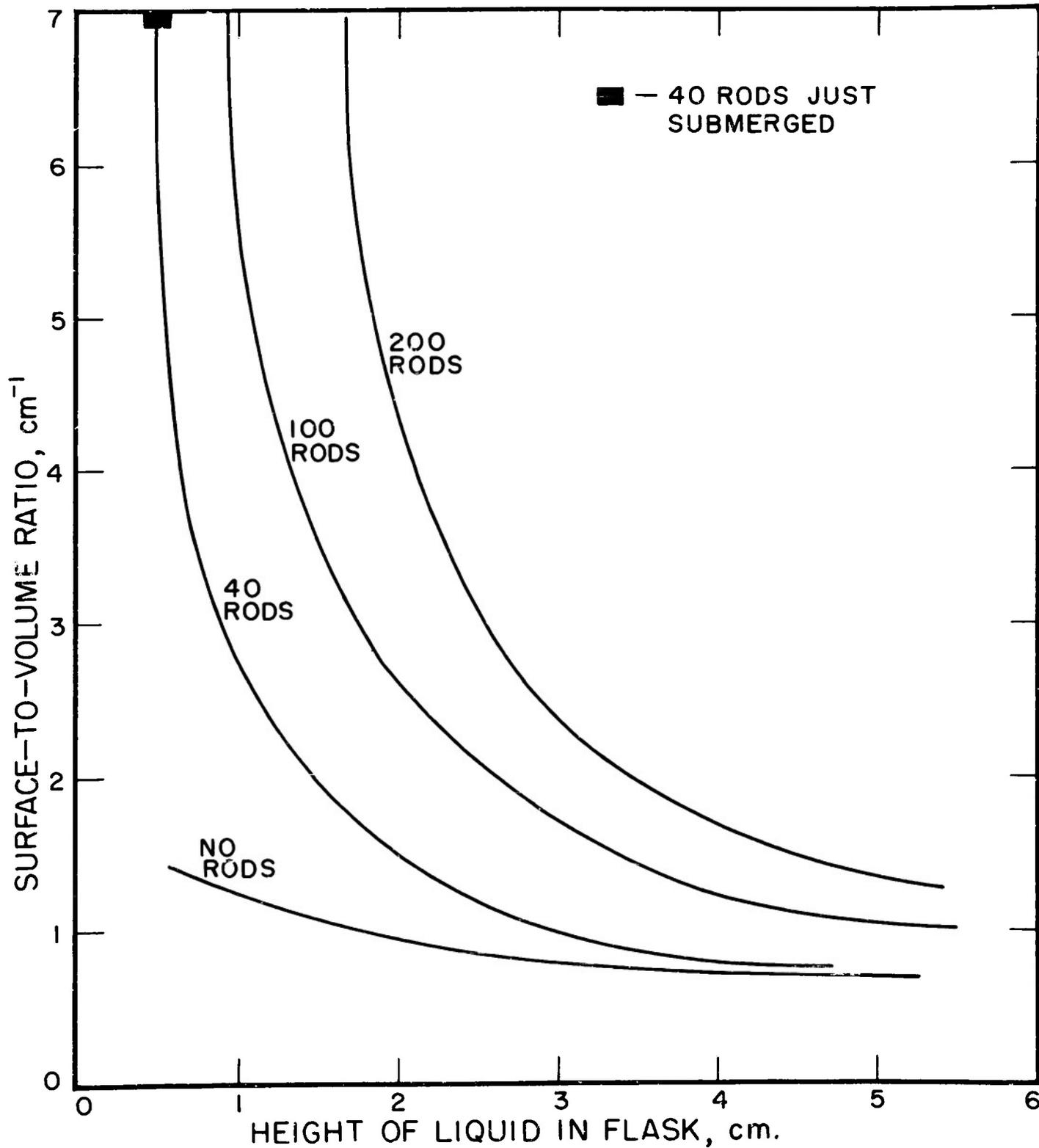


FIG. 3 - VARIATION OF SURFACE-TO-VOLUME RATIO WITH FILLING OF 8 cm dia. SPHERICAL FLASK.

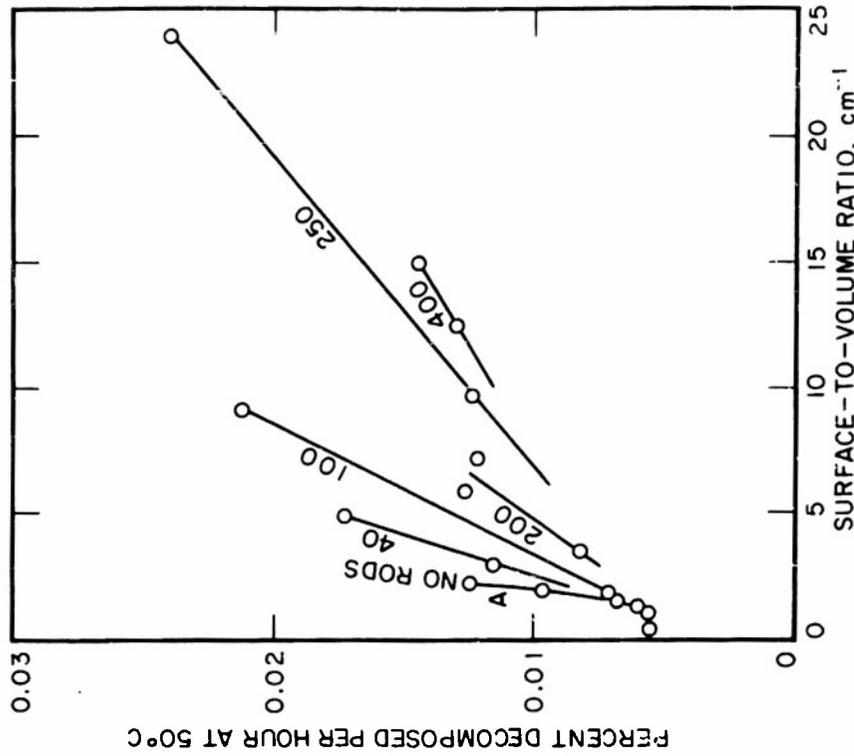


FIG 5 - DECOMPOSITION OF 90% H₂O₂ IN AN 8 cm dia. SPHERICAL FLASK CONTAINING GLASS RODS (0.69 cm²)

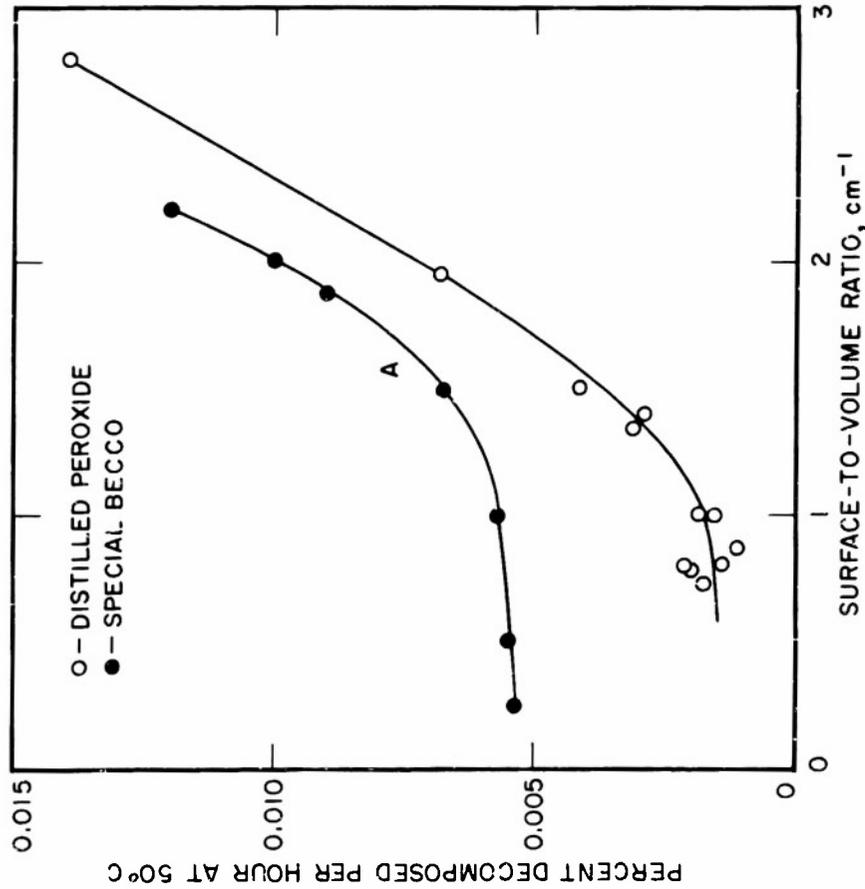


FIG 4 - DECOMPOSITION OF 90% H₂O₂ IN AN 8 cm dia. SPHERICAL FLASK.

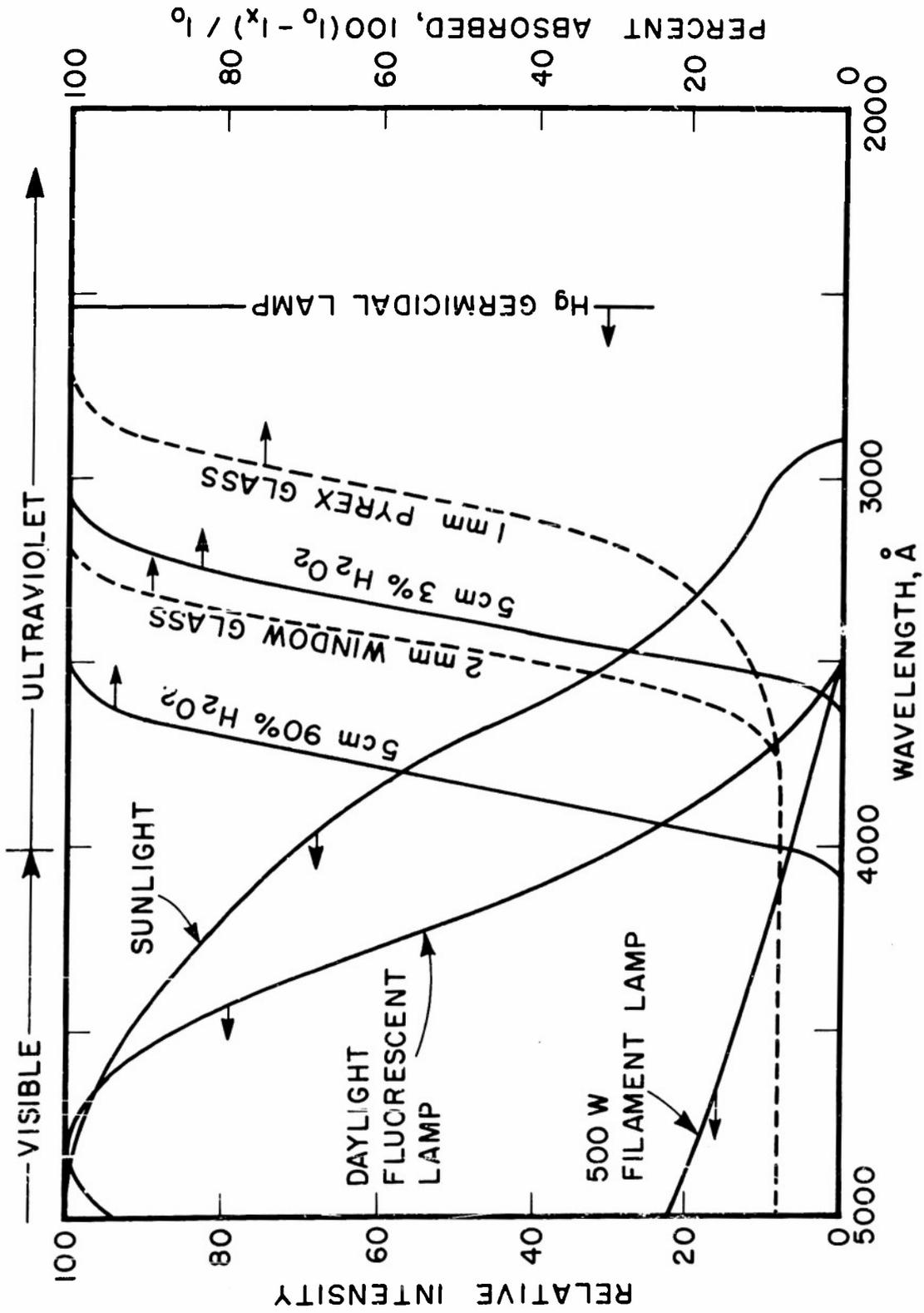


FIG. 6 - FACTORS AFFECTING STABILITY OF HYDROGEN PEROXIDE UNDER ILLUMINATION

wavelength and concentration are altered. Since diffuse reflection effectively reduces both the intensity and proportion of ultraviolet in sunlight it is concluded that as carried out under usual laboratory conditions the effect of diffuse daylight upon the decomposition rate of hydrogen peroxide solutions is small enough to introduce no significant error into the results obtained. If comparative measurements are to be carried out under conditions of illumination which are variable in intensity or frequency, this situation may introduce a source of error.

II. Partial Oxidation of Propane (C. E. Bodington)

It has been shown in previous work that the yields of hydrogen peroxide and acetaldehyde formed in the partial oxidation of propane are adversely affected by the presence of an active surface. In order to determine the extent of heterogeneous reactions occurring in the Pyrex flow reactor used earlier it was attempted to carry out the reaction under such conditions that heterogeneous reactions would be minimized.

The apparatus designed for this purpose consisted of a porous walled Alundum tube sealed into a Pyrex glass jacket. Glass tubes, for the inlet and outlet gases, supported this tube inside the jacket. The propane and oxygen, both 99+ % pure, were preheated, separately mixed, and injected into the top of the porous tube. Pure nitrogen gas was blown into the surrounding jacket. The gas went from this jacket through the walls of the porous tube at right angles to the reacting gases inside. This positive flow of inert gas toward the reacting propane-oxygen mixture inside the tube was intended to prevent the diffusion of these gases to the walls of the tube where heterogeneous reaction takes place. In order to compare the results with those of previous workers the experimental conditions were made as similar as possible. The propane to oxygen mole ratio used was 5.5:1 and the temperature was fixed at 475°C. These conditions were used by Reid in determining the effect of propylene on the mechanism, by Wei in determining the effect of nitrogen and helium on the mechanism and by Wilson in his work leading to the formation of a mechanism for the reaction. Wei found that nitrogen had little effect on the rate of the reaction, the slope of a curve of percent of inlet oxygen reacted versus the residence time, but that it increased the length of the induction period of this free radical reaction. Due to this effect on the induction period of the reaction, the nitrogen to oxygen ratio was fixed at 3.5:1, 5.5:1 and 6.5:1 for three series of runs. In order to obtain data at varying amounts of reaction, different residence times were used, from 1.5 to 6 seconds. Since the gas inside the reactor accelerates as more and more nitrogen enters through the walls, the residence time, defined as the length of time it takes a small volume of the inlet propane, oxygen mixture to traverse the length of the tube, is based on a length average flow rate. Assuming constant temperature and uniform gas flow along the length of the tube, it can be shown that the average flow is

equal to the arithmetic mean of the propane and oxygen input entering the tube and the flow rate of the gases leaving the bottom of the tube.

The results of these experiments show that the yield of hydrogen peroxide is lower in this reactor than in the Pyrex reactor used by Reid and Wei. The yield was found to be a function of the ratio of the wall gas rate (nitrogen) to the residence time. Since the accessibility of the walls to the reacting gases should also be a function of this ratio, the data indicate that the formation of hydrogen peroxide is a homogeneous reaction and that the decomposition is heterogeneous. Since the effect of the wall will be a function of this ratio in any reactor of this type, it would be desirable to make this ratio as high as possible in order to minimize the wall effects. The yield, compared to that obtained by Reid, of the other products of this reaction was also affected by this reactor, but in most cases it is nitrogen dilution rather than the surface that can best account for the change. All the products stem from either the splitting or oxidation of a propyl radical. The products formed through the oxidation of a propyl radical such as acetaldehyde, methanol and propylene, are depressed in yield, while those formed by the splitting of the propyl radical such as ethylene and formaldehyde, were obtained in amounts equal or greater than those obtained by Reid in the Pyrex reactor. These results are probably due to the fact that the rate of oxidation of the propyl radicals must be a function of the oxygen concentration. The rate is therefore reduced when the gases are diluted with nitrogen.

The particular design used in these experiments did not completely inhibit the diffusion of hydrogen peroxide or any other species to the walls of the reactor. Leakage of the wall gas around the seals at the top and bottom of the porous tube may be partly responsible, since such leakage would reduce the net flow through the walls of the tube itself. The extent of the heterogeneous reactions is in part due to the large surface to volume used (48:1 l/ft) and to the extremely active surface used. It is intended at a later date to report some of these studies in a porous tube made of sintered Pyrex, which should be much more inert than the Alundum used here.

III. Heterogeneous Vapor Phase Decomposition (T. W. Stein)

As was stated in the last report, work during the second quarter of 1954 was to be directed towards determining whether there is some homogeneous decomposition in addition to the heterogeneous decomposition of hydrogen peroxide vapor in the various tubes being studied. Studies at high concentrations of hydrogen peroxide had indicated that the precision of the data became poor as the concentrations of hydrogen peroxide vapor approached the explosion limit. It was proposed to vary the surface to volume ratio in several tubes and to compare the rate of decomposition per unit area. To make such a comparison, it must be certain that the surface activities towards hydrogen peroxide

are the same in the tubes to be compared. As was pointed out in the last report, investigations have shown that a coating of boric acid on a surface gives a fairly uniform surface.

Before undertaking this study, several changes were made in the construction of the equipment in an attempt to improve the precision of the data. The element for supplying heat to the boiler was changed. Previously the heating coils were wrapped around the boiler in such a manner that they were quite close to the boiler surface. Consequently there was the possibility of "hot spots" on the boiler walls. To improve this situation, the heating wires were embedded in a ceramic tube which is placed coaxially around the pyrex boiler. This leaves an air space of about a quarter of an inch between the heating coils and the boiler which results in a more uniform distribution of heat over the boiler walls. In addition to the improvements of the boiler, improvements were made on the "collectors" which collect the liquid condensate from the condensers. With these improved collectors the liquid samples can be removed without affecting the pressure in the system; therefore, there is no inducement for the rate of vapor flow in the system to change. Additional minor changes were made in the construction and operation of the equipment. The sum result of these changes was an increase in the precision of the data.

The first tube which was investigated was a Pyrex tube which was coated with boric acid and had an area of 635 cm^2 and a volume of 360 cm^3 . The rate of decomposition at a total pressure of 1 atm. was measured at three temperatures-- 180°C , 215°C , and 250°C --- and at various averaged hydrogen peroxide partial pressures from 0 up to about 0.22 atm. This upper limit of concentration in the tube being studied corresponds to an explosive concentration (26 mole per cent hydrogen peroxide) of vapor leaving the boiler.

The second tube had a surface area of 1486 cm^2 and a volume of 265 cm^3 and was prepared by adding to the first tube a bundle of twenty-three boron coated pyrex rods which were about 26 cm long and whose diameters varied from about 4 to 5 mm. The temperatures investigated in this tube were the same as in the other and the averaged partial pressures of hydrogen peroxide varied from 0 to 0.14 atm. This highest averaged concentration is lower in the case of the second tube. It was again necessary to have an explosive mixture leaving the boiler to produce these concentrations, but because of the increased surface area more hydrogen peroxide is decomposed, and consequently the average is lower. Nevertheless, the same range of concentrations of hydrogen peroxide vapor leaving the boiler was investigated in both tubes.

The results showed that in the two tubes, one with a surface to volume ratio of 1.75 and the other with a ratio of 5.6, the rates of decomposition per unit area of surface were very nearly the same. In fact, the rate per unit area in the tube with the high surface area was slightly larger which would tend to indicate that no detectable homogeneous decomposition is occurring in the range of conditions studied. In these tests it was found that the precision of the data was good even at high concentrations. It is believed that the erratic behavior in earlier work was a result of inadequacies of the equipment which were corrected as was mentioned above. There definitely appears to be no indication of homogeneous decomposition.

The next phase of the work was to correlate the data and to represent them by an equation. The approach which was used was to propose several mechanisms and then to attempt to fit the data to the equations which the Langmuir monomolecular adsorption theory would predict in accordance with the proposed mechanisms.

Fourteen different mechanisms were proposed but of these proposals only three were mathematically acceptable. These three mechanisms are:

- 1 and 2 Reaction between an adsorbed OH radical and molecularly adsorbed hydrogen peroxide
 - 1) Desorption of water controlling
 - 2) Reaction rate controlling

- 3 Reaction between an adsorbed OH radical and a molecule of hydrogen peroxide in the gas phase, where the reaction rate is controlling.

It is realized that the Langmuir theory is an over simplification of what is actually occurring on the catalyst surface. However, on the assumption of the validity of the Langmuir theory there can be proposed the following picture of the decomposition of hydrogen peroxide. A hydrogen peroxide molecule is chemisorbed on the surface and splits homolytically into two OH radicals. This initial step can be stated with some certainty. Uncertainty does exist, however, as to whether one of these free radicals reacts with a physically adsorbed hydrogen peroxide molecule or whether it reacts with a hydrogen peroxide molecule impinging from the gas phase.

This proposal for the initial step of the decomposition is in accordance with the hypothesis of Giguère (1), who imagined the initial step to be the breaking of the O-O bond of hydrogen peroxide as it is adsorbed. Also this heterogeneous mechanism parallels the chain mechanism for the homogeneous decomposition

of hydrogen peroxide which was proposed by Satterfield, Kavanagh, and Resnick (2). They propose that an hydrogen peroxide molecule thermally decomposes to give two OH radicals which then propagate the chain by reacting with an hydrogen peroxide molecule. Thus the mechanism discussed here is consistent with those proposed by other investigators.

As was mentioned above, there are three forms of equations which are acceptable as a means of correlating the data. The third proposal mentioned above was arbitrarily chosen and the data for the decomposition of hydrogen peroxide in a boron coated pyrex tube were applied to this equation. The final equation is:

$$-\frac{dn}{A d\theta} = \frac{\alpha K H_2 O_2 P H_2 O_2^2}{1 + K H_2 O_2 P H_2 O_2 + K H_2 O P H_2 O}^2$$

where

$$K H_2 O_2 = 7.34 \times 10^7 \quad - \frac{11.3 \times 10^3}{RT}$$

$$K H_2 O = 3.82 \times 10^{-3} \quad \frac{7.76 \times 10^3}{RT}$$

$$\alpha K H_2 O_2 = 1$$

and where

- n = moles of hydrogen peroxide decomposed
- $P H_2 O_2$ = partial pressure of hydrogen peroxide (atm)
- $P H_2 O$ = partial pressure of water (atm)
- A = surface area (cm²)
- θ = time (min)
- T = temperature (°K)

The average deviation at 180°C is 7%, at 215°C, 16%, and at 250°, 9%.

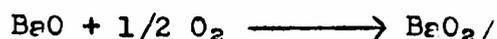
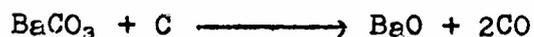
Future work is to be directed to the examination of a glass surface in the attempt to find what factors contribute to the activity of a surface as a catalyst for the decomposition of hydrogen peroxide as a means for finding ways to passivate surfaces. Secondly work will be begun on the examination of the activities of metallic surfaces.

1. Giguère, P.A., Can. Journal of Research 25B, 135-50, 1947
2. Satterfield, C. N., Kavanagh, G. M., and Resnick, H., Ind. Eng. Chem., 43, 2507, (1951)

IV. Cyclical Barium Peroxide Processes (F. Feakes)

An investigation has been undertaken of (1) the mechanism of the calcination of barium carbonate, and (2) the mechanism of the formation of barium peroxide from barium oxide obtained by the calcination of barium carbonate.

During the period April 1 - Jun 30, 1954, detailed investigations of the following reactions were initiated:



This work aims at establishing the optimum conditions for the production of barium peroxide, which may then be converted into hydrogen peroxide by treatment with acid.

The rate and degree of conversion of barium oxide to barium peroxide in an oxygen atmosphere is largely determined by the conditions used for the calcination reaction. The activity (peroxide forming capacity) of the barium oxide is considered to be a function of the following variables.

a) Calcination Reaction

1. Temperature
2. Carbon monoxide concentration in the gas phase
3. Time
4. Amount and surface activity of admixed carbon
5. Porosity of calcined material
6. Crystal size and purity of barium carbonate used,

b) Peroxidation Reaction

1. Temperature
2. Oxygen concentration
3. Time
4. Porosity of BaO & BaO₂
5. BaO crystal size

It has been proposed that close control of the above variables should assist in the determination of the optimum conditions for peroxide formation. For this purpose the construction of a small electrically heated tube furnace has been commenced. It is proposed to calcine spheres of barium carbonate admixed with carbon in an atmosphere of known composition within the tube furnace. The rate and degree of the reaction will be determined by measuring the gas evolution during the reaction. Provision is being made for automatic temperature control of the furnace. Temperatures within the sphere undergoing reaction will be measured with a platinum-rhodium thermocouple. A knowledge of the temperature distribution through the sphere during reaction should make it possible to determine whether heat transfer or some other variable controls the reaction rate.

Following the calcination reaction, an inert gas will be substituted for the gas in the furnace, the temperature of which will then be readjusted to that of the peroxidation reaction. After evacuation of the inert gas, oxygen will be admitted and the rate and degree of peroxidation measured.

In this manner it is hoped that it will be possible to measure both the rate and degree of the calcination and peroxidation reactions.

V. Visitors:

Mr. Chard - British Joint Services Mission
Mr. Ratten - Bur Aer, Washington
Mr. N. Davis - Buffalo Electro-Chemical Co.
Mr. L. Powell - ONR, Boston
Mr. Horstein - ONR, Washington
Mr. A. G. Whittaker - Naval Ordnance Test Station

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