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## The Production of Hydrogen Peroxide in Water by Alpha-Rays\*

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(Received July 27, 1936)

The ultraviolet absorption spectrum of air-free water irradiated with alpha-rays has been photographed between 2200 and 2800Å. The spectrum appears to belong to a dilute aqueous solution of hydrogen peroxide. The gases evolved from the irradiated water contain hydrogen in excess of an electrolytic mixture. The parallel variation of excess  $H_2$  pressure and  $H_2O$  absorption is consistent with Duane and Scheuer's hypothesis. Apparatus has been developed in which excess  $H_2$  and  $H_2O$  absorption can be measured simultaneously at different times without disrupting the normal course of an experiment. The method is applicable to other radio and photochemical reactions.

### INTRODUCTION

MANY radio and photochemical reactions in aqueous solutions are ascribed to the primary decomposition of the water molecules and to the secondary formation of hydrogen peroxide. In 1912 Rosanoff<sup>1</sup> suggested that the absorption of ultraviolet rays by radioactive substances is conditioned by the emanations emitted, and by the hydrogen peroxide formed under the influence of this emanation. Other investigators at that time regarded the part played by hydrogen peroxide as insignificant.

Kernbaum<sup>2</sup> found that distilled water free from air when exposed in a quartz vessel to the sun's rays shows evolution of gas after a few hours. Using Shonbein's reagent, hydrogen peroxide was detected. The quantity was larger when the experiments were carried out in the presence of air. He concludes that water under-

goes decomposition and that hydrogen peroxide is an intermediate compound in the reaction. Hydrogen peroxide production by radiant energy is of biological interest, for there are those who believe that ultraviolet rays exert an influence on microorganisms by virtue of the  $H_2O_2$  they produce, and that the intense action of the direct rays is due to a production of peroxide within the organisms themselves.<sup>3</sup>

Clark, in his review of the biological effects of x-radiation,<sup>4</sup> lists the formation of hydrogen peroxide when water containing dissolved oxygen is irradiated among the biologically significant effects of x-rays. Later, in the same paper, he mentions the important conclusion of Fricke that the activated water molecule rather than the direct effect of the rays or the formation of hydrogen peroxide may be responsible for the action on living cells. The role of hydrogen peroxide in the complex organic molecular systems is extremely difficult to ascertain and its formation in the more simple systems has not been proved experimentally in many cases.

\* The experimental work was done in the Laboratory of Biophysics and the Cancer Institute, Minnesota General Hospitals, Minneapolis.

<sup>1</sup> Rosanoff, J. Russ. Phys. Chem. Soc. **44**, 1146 (1912); J. Chem. Soc. **102**, 875 (1912).

<sup>2</sup> Kernbaum, Farm. Polsk. **71**, 16 (1913).

<sup>3</sup> T. H. B. Bedford, Brit. J. Exp. Path. **8**, 437 (1927).

<sup>4</sup> George L. Clark, Radiology **26**, 295 (1936).

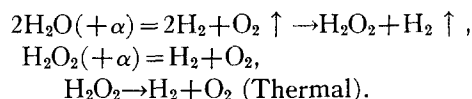
Eight-tenths normal sulphuric acid in water added to ferrous sulphate after x-ray irradiation oxidizes the ferrous ion to the ferric ion, although in an irregular manner.<sup>5</sup> The reaction is ascribed to the formation of  $\text{H}_2\text{O}_2$  in the acid. The reduction of chromate by x-rays has been found to be related also to the production of  $\text{H}_2\text{O}_2$ .<sup>6</sup> Fricke found that oxygen dissolved in water is reduced to  $\text{H}_2\text{O}_2$  when irradiated with x-rays.<sup>7</sup> If the water is air-free then x-rays do not decompose it and  $\text{H}_2\text{O}_2$  is not formed.<sup>8, 9</sup> On the other hand, air-free water is decomposed by alpha-rays as has been shown by the writer in some work not yet published. The inability of x-rays to decompose air-free water in contrast to the ability of alpha-rays to do so suggests two distinct mechanisms which hitherto have been considered similar.

The notion of  $\text{H}_2\text{O}_2$  synthesis in aqueous solutions probably originated from the well-known investigations of Duane and Scheuer<sup>10</sup> in which water was irradiated with alpha-rays. Hydrogen and oxygen were liberated. The gases, however, were not present in electrolytic proportions but always contained an excess of hydrogen. The excess was less than 50 percent, and gradually decreased as the experiment proceeded. The writer has found that if the experiments continued long enough then the excess hydrogen disappeared almost entirely.<sup>11</sup> In a more recent work<sup>12</sup> the excess hydrogen from air-free water has been found to be 100 percent early in the experiments.

Duane and Scheuer concluded that hydrogen peroxide was formed by the action of nascent oxygen on water and thereby giving an excess of hydrogen in the liberated gases. They explained the decrease of excess hydrogen as occurring when the quantity of  $\text{H}_2\text{O}_2$  reached an amount such that its rate of decomposition, both spontaneous and that caused by the rays, is equal to its rate of formation. Their hypothesis

seems logical in part but no conclusive experimental evidence has been produced either to prove or disprove it. The various reported steps in the reaction are mostly speculative. Whether nascent  $\text{O}_2$  acts on the  $\text{H}_2\text{O}$  molecules or  $\text{H}_2$  and  $\text{O}_2$  combine to form  $\text{H}_2\text{O}_2$  has not been confirmed one way or the other; however, the results on air-free water indicate that if nascent oxygen is present it has originated from decomposed water and not from dissolved oxygen.

For the sake of clearness the reactions may be described by the following expressions:



From these reactions there result two experimentally measurable end products, namely  $\text{H}_2\text{O}_2$  and the gases  $\text{O}_2$  and  $\text{H}_2$  which have certain reasonable quantitative relationships. For example, each  $\text{H}_2\text{O}_2$  molecule formed or decomposed should give rise to one more or one less excess  $\text{H}_2$  molecule (excess of  $\text{H}_2$  over electrolytic proportions of  $\text{H}_2$  and  $\text{O}_2$ ). Therefore,  $\text{H}_2\text{O}_2$  concentrations calculated from measured excess  $\text{H}_2$  gas should agree with measured concentrations. The rise and fall of  $\text{H}_2$  pressure should also run parallel to  $\text{H}_2\text{O}_2$  concentrations. In order to test these suggestions an experimental method has been developed in which  $\text{H}_2\text{O}_2$  in the irradiated water and excess  $\text{H}_2$  in the liberated gases can be measured simultaneously at many different times without disrupting the normal course of the experiments. The method gives promise of being applicable to the study of many other radiochemical reactions. Another desirable feature is that the irradiated water is made gas-free. Therefore it is possible to determine if alpha-rays and x-rays are alike in the latter's inability to decompose air-free water and to cause the formation of  $\text{H}_2\text{O}_2$ . A description of the method and of some of the first results are described. Because of the author's transfer to a limited stay in China further experiments have ceased temporarily.

## EXPERIMENTAL METHOD

The water was purified in an all Pyrex vacuum still (Fig. 1) and was subjected to the rays in a

<sup>5</sup> Hugo Fricke and Samuel Morse, *Phil. Mag.* **7**, 129 (1929).

<sup>6</sup> Hugo Fricke and E. R. Brownscombe, *J. Am. Chem. Soc.* **55**, 2358 (1933).

<sup>7</sup> Hugo Fricke, *J. Chem. Phys.* **2**, 349 (1934).

<sup>8</sup> O. Risse, *Zeits. f. physik. Chemie* **A140**, 133 (1929).

<sup>9</sup> Hugo Fricke and E. R. Brownscombe, *Phys. Rev.* **44**, 240 (1933).

<sup>10</sup> Wm. Duane and O. Scheuer, *Le radium* **10**, 33 (1913).

<sup>11</sup> Carl E. Nurnberger, *J. Phys. Chem.* **38**, 47 (1934).

<sup>12</sup> Not yet published

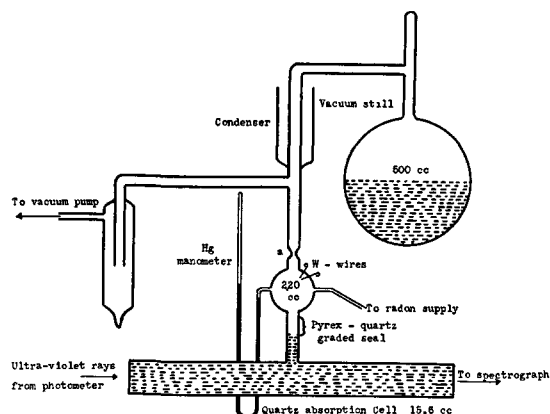


FIG. 1. Vacuum still, reaction chamber and absorption cell for the study of alpha-ray irradiation of air-free water.

quartz chamber. The usual precautionary measures common to the preparation of conductivity water were carefully observed. The reaction chamber was sealed directly to the still in order to eliminate contamination of the pure water by the air in the laboratory and by contact with the apparatus which otherwise would have been used to transfer the water from the still to the chamber.

The reaction chamber consists of two parts. At the bottom a 10 cm quartz cell serves as a reservoir for the irradiated water and as absorption cell in the spectrophotometric measure of hydrogen peroxide. Above the cell, but connected to it by means of a small tubular Pyrex to quartz graded seal is a 2.20 cc Pyrex bulb. The evolved gases collected and were analyzed in this bulb. The quantity of gas was measured at constant temperature and volume. Pressures were read on the 2 mm bore mercury manometer. The excess hydrogen was separated from the gaseous mixture by means of explosions set up by repeated sparks between tungsten wires sealed in the small bulb. The residual gas was all hydrogen.

Some difficulty was experienced by cracking of the quartz-Pyrex graded seal. Pyrex glass is known to become cracked by penetrating irradiation. Many of the Pyrex reaction chambers which the author used in earlier work had to be discarded after prolonged alpha-ray bombardment because of deep cracking, particularly around sealed in metal electrodes. Similar phenomenon has not been observed in quartz by

the author, but in the quartz-Pyrex seal deep cracks appeared after much less irradiation than required to crack Pyrex alone. Most of the deeper cracks came in the Pyrex end of the seal; however, some shallow ones extended into the quartz. The cracked tube was readily fixed by heating to the softening point. Although quartz is colored by alpha-rays, the absorption characteristics of the cell itself were not altered because the water protected the surfaces from intense exposure.

After water had been distilled into the quartz cell and the tube had been sealed at *a*, radon was admitted through the capillary radon supply tube. The latter was subsequently sealed a few millimeters from the chamber. The method of releasing the radioactive gas has been described in detail elsewhere.<sup>9</sup> At this stage, the reaction chamber and the manometer were completely separated from the still and the radon supply. The small unit was portable and could be moved readily from the constant temperature water bath to the optical analyzing system.

A portion of the radioactive gas diffused into the water in the quartz cell. The amount of dissolved radon, and therefore that which determines the principal effective alpha-ray action, depends on the dimensions of the reaction chamber. If quantitative gas yields per unit of irradiation are desired, then these physical factors cannot be neglected without introducing considerable errors. In the experiments described here gas yields per unit of exposure are not important and so no attempt is made to calculate the effective radon. It will suffice to say that the results of previous experiments point to the conclusion that the diffusion of radon into the water was nearly complete after two days.

The concentration of hydrogen peroxide in the irradiated water was determined by measuring its absorption coefficients for ultraviolet rays. A Judd Lewis sector photometer and an Adam Hilger quartz spectrograph were used. The experimental procedure with these instruments is described elsewhere.<sup>13</sup> A low pressure hydrogen discharge tube was used for light source. The tube was built in the Minnesota laboratory. It operated continuously at 2100 volts a.c., 0.21

<sup>13</sup> Carl E. Nurnberger and L. Earle Arnow, *J. Phys. Chem.* **38**, 71 (1934).

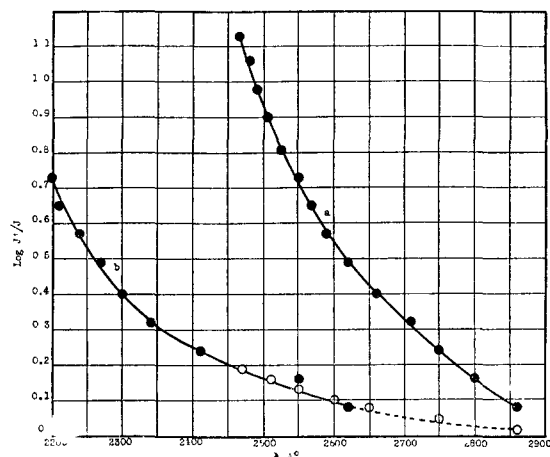


FIG. 2. Ultraviolet absorption curves of hydrogen peroxide and of irradiated water. (a) 0.018 percent  $\text{H}_2\text{O}_2$  in  $\text{H}_2\text{O}$ . 10 cm cell. (b) Irradiated  $\text{H}_2\text{O}$  in 10 cm cell.

ampere, and hydrogen pressure of approximately 3 mm of mercury. The easy and rapid alignment with the other optical instruments and the steady, intense, and continuous ultraviolet spectral emission of the tube are some of the advantages over the older under-water spark sources. The absorption of the cell and its contents was always measured relative to the absorption of pure water in a cell with quartz end plates.

The concentration of  $\text{H}_2\text{O}_2$  in the water was determined by Beer's absorption law as expressed in the following equation,

$$c = (1/\epsilon d) \log (J'/J) \quad (1)$$

where  $c$  is the number of  $\text{H}_2\text{O}_2$  molecules per cubic centimeter,  $d$  is the length of the absorption cell,  $\epsilon$  is the molecular absorption coefficient, and  $\log J'/J$  is called the absorption factor. The latter was read directly from the photometer scale. Slight corrections were applied according to directions furnished by Hilger. The  $J$  and  $J'$  refer to the intensities of the incident and emergent light respectively.

### RESULTS

Three photographic plates of the absorption of a known solution of  $\text{H}_2\text{O}_2$  in the quartz cell (0.018 percent in 10 cm cell) were made for comparative purposes. Their average values are plotted in curve (a) in Fig. 2. The maximum

deviation of  $\log J'/J$  among the three plates was not greater than 0.05. The solid portion of curve (b) is the average of two plates exposed one immediately after the other of irradiated  $\text{H}_2\text{O}$ . Curve (b) is similar to the ultraviolet absorption curve of  $\text{H}_2\text{O}_2$ . If points on the known  $\text{H}_2\text{O}_2$  curve are reduced by the factor 0.18, then the reduced points, indicated by the circles, all fall very close to curve (b). Since the concentration of  $\text{H}_2\text{O}_2$  in curve (a) is 0.018 percent, then the concentration of absorbing substance in curve (b), presumably  $\text{H}_2\text{O}_2$ , is 0.018 percent  $\times 0.18$ , or 0.00327 percent. Both curves were obtained with the same absorption cell. Henri and Wurmer<sup>14</sup> found the absorption of  $\text{H}_2\text{O}_2$  to be continuous. More recently Urey, Dawsey and Rice measured the absorption of  $\text{H}_2\text{O}_2$  both for the liquid and gaseous states.<sup>15</sup> They also found the absorption to be continuous for wave-lengths of ultraviolet light from 3000 to 2200 Å. They determined the molecular absorption coefficients of carefully purified  $\text{H}_2\text{O}_2$  for these wave-lengths. In Table I the molecular absorption coefficients of  $\text{H}_2\text{O}_2$  and of the irradiated water may be compared. The latter were calculated by means of Eq. (1) and by assuming the concentration of  $\text{H}_2\text{O}_2$  to be 0.00327 percent or  $0.00149 \times 10^{20}$  molecules per cc. Values for the longer wave-lengths were obtained by the extension of the experimental curve (b) through the reduced points of curve (a).

TABLE I. The molecular absorption coefficients of  $\text{H}_2\text{O}_2$  and of irradiated  $\text{H}_2\text{O}$ .

$\lambda$ , Å	Molecular Absorption Coefficients		
	$\text{H}_2\text{O}_2$ Urey, Dawsey, Rice (a)	Author's $\text{H}_2\text{O}_2$ (c)	Irradiated $\text{H}_2\text{O}$ (e)
2800	$2.4 \times 10^{-20}$	$2.0 \times 10^{-20}$	$2.0 \times 10^{-20}$
2700	4.1	4.0	3.8
2600	6.9	6.7	6.8
2500	10.2	11.3	10.8
2400	14.7	17.6	16.8
2350	17.9		20.8
2300	22.0		26.1
2250	25.6		35.6
2200	28.9		48.3

<sup>14</sup> Henri and Wurmer, *Comptes rendus* **156**, 1012 (1913); **157**, 126 (1913).

<sup>15</sup> H. C. Urey, L. H. Dawsey and F. O. Rice, *J. Am. Chem. Soc.* **51**, 1371 (1929).

The agreement between Urey's and the author's coefficients for  $\text{H}_2\text{O}_2$  is fairly good. The coefficients for the irradiated  $\text{H}_2\text{O}$  are very close to  $\text{H}_2\text{O}_2$  for wave-lengths longer than about 2400Å, but there is a decided increase for the shorter wave-lengths. The reason for this discrepancy is unknown. A portion of the difference may be due to the experimental methods, for there is a slight tendency in the author's coefficients for pure  $\text{H}_2\text{O}_2$  to rise above Urey's for the shorter wave-lengths. Another possible explanation may be that impurities are liberated from the quartz surfaces under alpha-ray bombardment which absorb or scatter ultraviolet rays shorter than 2400Å more than for the longer rays. Impurities are thought to be released from Pyrex and the increased conductivity of irradiated water is ascribed to them. Some sodium and calcium was found in earlier experiments.

The question might arise regarding the absorption of ultraviolet rays by radon and its decay products with the supposition that a part of the absorption of the irradiated  $\text{H}_2\text{O}$  is due to these materials. The absorption cannot be ascribed to radon. In 3.85 days 50 percent of the radon is destroyed, while the absorption factor as shown in the last column of Table II has maximum value in 4.74 days. Moreover, the rate of decrease is much slower than the disintegration rate of radon. It is doubtful if a part of the molecular absorption coefficient for wave-lengths shorter than 2400Å can be attributed to the active deposits of radon. For of the most likely of these, the long life ones, only radium *E* might show absorption in this region. Radium *E*

and bismuth might be expected to have similar absorption characteristics. Bismuth has not been measured for wave-lengths shorter than 2570Å.

Table II contains the results of an experiment in which residual  $\text{H}_2$  pressure and the absorption factor,  $\log J'/J$ , of the irradiated water were measured. Both increase, reach a maximum, and then decrease simultaneously in the course of the experiment. This sort of variation is consistent with Duane and Scheuer's hypothesis. However, no simple quantitative relationship exists between the excess  $\text{H}_2$  and the absorption factor as was originally expected. On the contrary, the  $\text{H}_2$  pressures at the beginning and end of the experiment were 10 mm and 42 mm respectively, while the absorption factor at these times had the same value, 0.32. Furthermore, in a period of 3.5 days the excess  $\text{H}_2$  pressure increased 7.7 fold while the absorption factor for any specific wave-length increased only 1.7 fold. These figures indicate that either the excess  $\text{H}_2$  cannot be due to the production of  $\text{H}_2\text{O}_2$  alone or the method of measuring  $\text{H}_2\text{O}_2$  does not give the true number of peroxide molecules. In order to carry the analysis further, the total maximum quantity of  $\text{H}_2\text{O}_2$  molecules was calculated from the measured excess  $\text{H}_2$  and from the measured absorption factor at 2250Å. Assuming one  $\text{H}_2\text{O}_2$  molecule for each excess  $\text{H}_2$  molecule, the total maximum number of  $\text{H}_2\text{O}_2$  molecules is given by

$$\begin{aligned} & [(273 \times 77 \times 2.20) / (760 \times 301)] \times 2.70 \times 10^{19} \\ & = 54 \times 10^{17}, \end{aligned}$$

where the part in brackets is the volume of  $\text{H}_2$  reduced to standard pressure and temperature and  $2.70 \times 10^{19}$  is Loschmidt's number. From absorption measurements the total maximum number of  $\text{H}_2\text{O}_2$  molecules is given by

$$(0.53 / 35.6 \times 10^{-20} \times 10) \times 15.6 = 23 \times 10^{17},$$

where 0.53 and  $35.6 \times 10^{-20}$  are the absorption factor and molecular absorption coefficient respectively for 2250Å, 10 cm is the length of the absorption cell, and 15.6 cc is the volume of irradiated water. The lack of quantitative agreement between the two results may be due to the formation of clusters  $(\text{H}_2\text{O}_2)_n$ . This supposes that the absorption of ultraviolet rays by the cluster is less than the sum of the individual absorptions of its constituents and that,

TABLE II. *A comparison of excess  $\text{H}_2$  pressure and the absorption factor of the irradiated  $\text{H}_2\text{O}$ .*

Radon = 69.8 mc       $\text{H}_2\text{O}$  volume = 15.6 cc

Time days	$\text{H}_2\text{O}$ Temp °C	Gas Volume cc	Gas Pressures (mm)		Abs. factor $\log J'/J$ $\lambda 2250$
			$P_{\text{O}_2 + \text{H}_2}$	$P_{\text{H}_2}$	
0	28.0	2.20	0	0	0
1.21	28.2	2.20	24	10	0.32
1.93	28.2	2.20	79	36	.40
2.72	28.0	2.20	153	68	.49
4.74	28.2	2.20		76	.53
5.64	28.2	2.20		77	.47
6.89	28.2	2.20		66	.41
9.68	28.2	2.20		62	.45
12.9	28.2	2.20		44	
13.7	28.2	2.20		42	.32

TABLE III. *Spontaneous decrease of the absorption factor of a dilute aqueous solution of H<sub>2</sub>O<sub>2</sub>.*

Days	0	0.8	2.8	4.8	8.0
Log $J'/J$ , $\lambda 2500$	0.98	0.90	0.81	0.81	0.73

therefore, the second calculated value of total H<sub>2</sub>O<sub>2</sub> is too small. The ratio  $54 \times 10^{17} / 23 \times 10^{17}$ , which is approximately equal to 2, might be supposed to mean that  $n$  for the cluster is equal to 2, although there is no experimental evidence to support the supposition.

The decrease of H<sub>2</sub> has been assumed to occur as the result of the spontaneous decomposition of H<sub>2</sub>O<sub>2</sub> and of alpha-ray decomposition. This assumption can be carried over now to explain the decrease in the absorption factor for a given wave-length. Since ultraviolet rays decompose H<sub>2</sub>O<sub>2</sub>,<sup>14, 16</sup> some of the reduction in (log  $J'/J$ ) might be due to ultraviolet exposures during the photographing of the spectrum. The exposure times were short, so not much decomposition could be expected. However, some experiments were performed to determine how much change might occur in the absorption of a dilute solution of H<sub>2</sub>O<sub>2</sub> on standing for several days at constant temperature without alpha-ray irradiation, but subjected to the usual ultraviolet ray exposures involved in the photographing of the spectrum. The results of a typical experiment are given in Table III. In 8 days the initial absorption had reduced approximately 25 percent. Most of this is most probably pure temperature or spontaneous decomposition. Under alpha-ray irradiation the maximum absorption decreased 40 percent in 9 days. Therefore, approximately one-fourth of the decomposition can be ascribed to alpha-rays and three-fourths to spontaneous decomposition.

Some oxygen is produced also with the hydrogen, for in each instance sparking of the gas reduced the pressure. After about the third day

<sup>16</sup> Tian, J. Chem. Soc. **108**, 828 (1915); J. Soc. Chem. Ind. **35**, 114 (1916).

of the experiments, the combined O<sub>2</sub> and H<sub>2</sub> pressures were too large to be recorded by the manometer. However, explosions reduced the pressure to readable values. It is therefore concluded that alpha-rays do decompose air-free water and that hydrogen peroxide is formed. The mechanism of the reaction of alpha-rays and of x-rays on water appear to be unlike.

### SUMMARY

1. The absorption spectrum of air-free water irradiated with alpha-rays has been found to be continuous beginning at approximately 2850Å and extending to 2200Å.

2. Similarities of the absorption curves of hydrogen peroxide and of irradiated water are noted. Points on the hydrogen peroxide curve when reduced by a common factor fall remarkably close to the irradiated water curve.

3. Molecular absorption coefficients of the irradiated water agree closely with the coefficients for H<sub>2</sub>O<sub>2</sub> in the spectral region 2800–2400Å; the agreement is not so good for shorter wave-lengths, which may possibly be due to absorption and scattering by impurities released from the quartz cell under alpha-ray bombardment.

4. Excess H<sub>2</sub> in the evolved gases and the H<sub>2</sub>O<sub>2</sub> concentration in the irradiated water rise, reach a maximum, and decrease simultaneously.

5. Excess H<sub>2</sub> is not entirely due to formation of hydrogen peroxide as shown by quantitative determination of the total H<sub>2</sub>O<sub>2</sub> from absorption measurements.

6. The major portion of the decomposition of H<sub>2</sub>O<sub>2</sub> is spontaneous.

7. It is finally concluded that air-free water is decomposed by alpha-rays and that hydrogen peroxide is formed.

I wish to express my appreciation of the cooperation of Dr. W. K. Stenstrom of the Division of Biophysics and Cancer Institute, University of Minnesota, Minneapolis.