THE EQUILIBRIUM CONSTANT OF THE HYDROGEN-OXYGEN REACTION

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INTRODUCTION

The purpose of this research problem is the determination of the equilibrium constant of the Hydrogen-Oxygen reaction on the basis of Hydrogen Peroxide formed as the product of the reaction. This constant may be expressed mathematically as

$$K = \frac{C_{H_2O_2}}{C_{H_2} \times C_{O_2}}$$

in which C is the concentration of the reacting substances in mols per liter at the point of equilibrium.

A thorough search of the literature revealed no published value of the constant, and, furthermore, no information was available regarding any attempts having been made toward its determination. On the other hand, a relatively large amount of research has been done on the decomposition of Hydrogen Peroxide in the presence of various catalytic substances. Kargin[1] studied the decomposition of Hydrogen Peroxide with a Platinum catalyst, and by employing a glass electrode to obtain the titration

1. Kargin,* Z. anorg. allgem. Chem., 183, 77-80 (1929); abstract in Chem. Abstracts, 24, 280(1930)
* Only the abstract was read.

curve with Potassium Hydroxide, calculated the dissociation constant from an ionic standpoint to be 1.55×10^{-12} at twenty degrees. Unfortunately all of this experimental work was directed toward the determination of the velocity of decomposition, and no figures are available for conditions at equilibrium.

It has been only in the last few years that any intensive investigation has been made upon the formation of Hydrogen Peroxide by the direct union of Hydrogen and Oxygen. Lately much work has been done to determine the mechanism of the reaction, and to measure the energy of activation of the Hydrogen atom. One very interesting phase of this research is the reaction of gaseous Hydrogen and Oxygen in the presence of resonance activated Mercury vapor, excited by ultra violet radiation from a quartz Mercury vapor lamp. The production by this method of measurable quantities of Hydrogen Peroxide is recorded, but the reaction was not permitted to attain equilibrium.[2] A maximum yield of 0.06% of the gas

2. Taylor, Marshall, and Bates,* Proc. Am. Phil. Soc., 65, 90-8(1926); abstract in Chem. Abstracts, 20, 2792(1926)
* Only the abstract was read.

passed through the reaction chamber was obtained as Hydrogen Peroxide, but this was considered of sufficient interest to protect.[3]

The reaction has been activated also by the X-ray. Marshall describes a procedure in which mixtures of gaseous Hydrogen and Oxygen are allowed to flow across the Nickel window of a cathode ray tube.[4] Small amounts of Hydrogen Peroxide and Ozone were produced. When the flow rate was decreased in attempts to secure equilibrium, explosions always occurred accompanied by the formation of water vapor.

Hydrogen Peroxide has been formed by the direct burning of a jet of Hydrogen in an atmosphere of Oxygen at elevated pressures. This method of manufacture has been covered by a patent specifying 300 atmospheres pressure and catalyst of the noble metals deposited on asbestos.[5]

Bates and Salley passed mixtures of Hydrogen and Oxygen saturated with Mercury vapor through a quartz tube exposed to ultra violet radiation.[6] The

3. Taylor, U. S. Pat. 1,659,382. (August 18, 1927)
4. Marshall, J. Am. Chem. Soc., 50, 3197-3204(1928)
5. Society L'Air Liquide, Br. Pat. 309, 602 (April 14, 1928)
6. Bates and Salley, J. Am. Chem. Soc., 55, 110(1933)

best result recorded was eleven milligrams of total product from thirty liters of a two to one Hydrogen-Oxygen mixture, of which eighty-five per cent was Hydrogen Peroxide. The reaction was considered entirely from a mechanism and velocity standpoint, and no attempt was made to reach equilibrium.

It is at once obvious from the foregoing that the difficulties attendant upon the carrying out of this problem were greatly enhanced by the absence of previous research dealing with the subject at hand, namely, the determination of the equilibrium constant.

PREPARATION OF THE CATALYST

The Palladium catalyst was prepared by a method given by Thorpe.[1]

Twenty grams of washed, acid treated, asbestos was finely ground, boiled again with dilute hydrochloric, filtered by suction, and washed on the filter. This was suspended in 500 ml. of distilled water, and sixty-eight mg. of Palladious Chloride dissolved in one ml. of concentrated Hydrochloric Acid and diluted to 250 ml. was added. The mixture was heated to eighty degrees Centigrade, then twenty-five grams of Sodium Formate and ten grams of Sodium Hydroxide, dissolved in 250 ml. of water, was added quickly and vigorously stirred. The temperature was maintained at eighty degrees with constant stirring for a period of fifteen minutes, at the expiration of which time the mass had assumed a dark gray color. This was filtered by suction, washed with hot water, then with hot dilute Hydrochloric Acid, and finally with boiling distilled water until the wash water gave no test for chlorides. The catalyst

1. Jacobsen and Landesan,* Ber., 3117, 1907; abstract in Thorpe's Dictionary of Applied Chemistry.
* Only the abstract was read.

was finally transferred to a two liter flask after being accurately adjusted to a volume of 2000 ml. The Palladium coated asbestos settled slowly after vigorous agitation, and the assumption that fifty ml. of this mixture contained one mg. of Palladium should be well within the limit of experimental error.

The Nickel catalyst was prepared by the method of Wimmer and Higgins,[2] which was used commercially at an oil refinery at which the senior author of this paper was formerly employed. According to this method, 3.1400 grams of Nickel Formate was dissolved in the least possible quantity of water, intimately mixed with twenty grams of washed, acid treated asbestos, which had been powdered, and the mixture evaporated to dryness on the water bath. The mass was finely ground and introduced into a 500 ml. distillation flask. After replacing the contained air with Hydrogen, the flask and contents were heated for one hour in an air bath at 180° C., while a slow stream of Hydrogen was maintained through the flask. The flask was cooled, and, without discontinuing the gas stream, about 100 ml. of distilled water was introduced. It was necessary

2. Wimmer and Higgins, U. S. Pat. 1,081,182. (December 9, 1913)

to protect the catalyst particles with a layer of water to prevent the rapid adsorption of of atmospheric Oxygen, which would be accompanied by the liberation of so much heat that the finely divided metallic Nickel would become incadescent and converted to the oxide. The flask was carefully washed, and the catalyst suspension diluted to 1000 ml. Fifty ml. of this suspension was equivalent to 0.0500 grams of metallic Nickel. In order to be certain that no unchanged Nickel Formate remained, one fifty ml. portion was filtered, the filtrate acidified and titrated with standard Potassium Permanganate solution. No permanganate was reduced, hence the catalyst was considered free from unchanged formate.

APPARATUS

The construction of the apparatus used is shown diagrammatically by Figure 1. The rubber sleeve A is for the admission of gas to the apparatus and for the exhaustion of the catalyst chamber. Two wide mouthed reagent bottles B_1 and B_2, each of 500 ml. capacity, are used for gas vessels. A bent glass tube extends from the bottom of B_1 to the lower face of the stopper of B_2. The tube C is used as the inlet for water under pressure and as the exhaust when filling the gas vessels. The Mercury reservoir D is movable and may be adjusted to any desired height. It is connected to B_2 by means of tubing which extends to the bottom of the gas chamber. The rubber sleeves E_1 and E_2 are used as stop cocks by compressing with screw clamps. The sleeve E_3 allows movement of the catalyst vessel F by means of connecting rod G which transmits motion from a motor driven crank.

All rubber connections between the vessels were closely wrapped with cotton cord to reinforce them. To prevent the blowing out of the glass connections

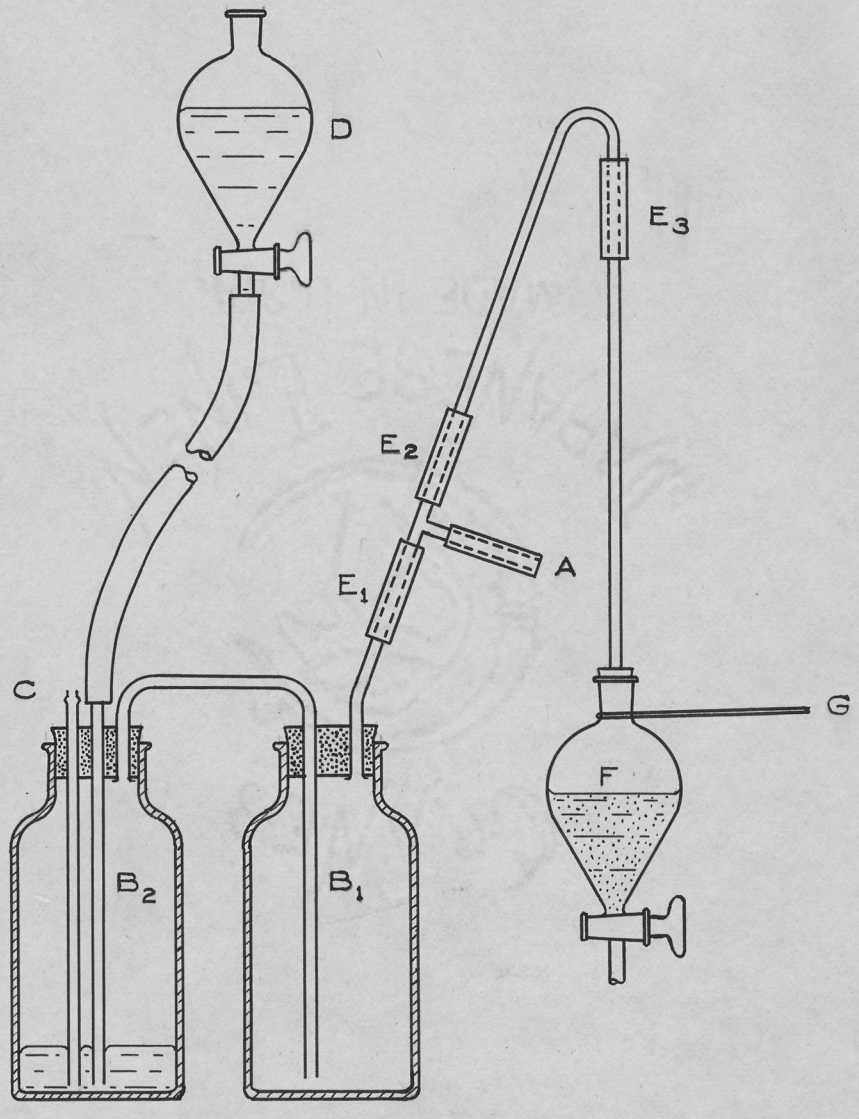

Figure 1.

A,	gas inlet.
B_1, B_2	gas vessels.
C,	pressure water inlet.
D,	mercury rerservoir.
E_1, E_2, E_3,	rubber sleeves.
F,	catalyst chamber.
G,	connecting rod.

from the rubber stoppers of the chambers, the glass tubing was flanged at the ends. The connections of the rubber tubing to the glass tubes were made secure by blowing small bubbles in the tubes of a diameter slightly larger than that of the tube. These were fitted tightly into the rubber connecting lengths and wrapped closely with cotton cord. After the apparatus was assembled, the rubber stoppers of the gas bottles and the reaction chamber were fixed rigidly in place by tying them to the necks of the vessels.

The completed apparatus was subjected to a hydrostatic pressure equivalent to a column of Mercury 200 cm. in height.

TECHNIQUE

The gas vessels, after the connections to the reaction chamber had been closed by means of a screw clamp, were filled to capacity with water from the tap. Hydrogen was then introduced into the chambers from a cylinder of the compressed gas. The volume of the gas displaced an equivalent volume of water which was collected in a graduated cylinder and measured. The desired volume of Oxygen was then introduced from an Oxygen tank and measured in a like manner. Water from the tap was admitted into the first gas chamber until the gases were compressed into the second chamber. This procedure was necessary to insure a complete mixing of the two gases. The pressure was then adjusted to a value slightly higher than that desired for the run by means of pressure from the water tap.

After vigorous agitation of the catalyst stock flask in order to insure a homogeneous suspension, a fifty ml. portion was quickly measured and introduced into the bottom of the reaction chamber, through the stopcock, by the aid of gentle suction. After closing the stopcock, the chamber was evacuated to the full capacity of the pump. This was

done for two purposes. First, the occluded gases were removed from the catalyst, and, second, the air in the reaction chamber was withdrawn. The water pump connection was now closed, and communication between gas containers and reaction chamber established. The pressure was adjusted to the value desired by the judicious admission of pressure water. Agitation was now started, and, as gas absorption proceded, pressure was maintained constant by the slow admission of water into the gas vessels. When the reaction had proceded for the desired time, the total volume of aqueous mixture was blown out of the chamber by gas pressure.

For the first few runs the reaction mass was filtered by suction through an asbestos mat on a Büchner funnel as rapidly as possible, and immediately titrated with standard Potassium Permanganate solution in the presence of dilute Sulphuric Acid.. This method gave results which did not agree satisfactorily for identical experimental runs. Possibly the reason for this variation was that the equilibrium conditions were altered by the change in the partial gas pressures during the suction filtration. Then

the following method was adopted. The reaction mass was blown directly into an excess of acidified standard Potassium Permanganate solution, filtered by suction through an asbestos mat, and the excess permanganate titrated with standard Ferrous Sulphate solution. This latter method gave results which were concordant.

Several runs were made with Palladium catalyst from previous runs. It was washed thoroughly, suspended in water, diluted to fifty ml. and the foregoing procedure followed.

EXPERIMENTAL DATA

TABLE No. I

Conditions of experiment using 50 ml. of palladinized asbestos suspension, equivalent to 1 mg. of Pd.

Run Number	Time of run	Pressure cm. of Hg.	Temp. C°	Gas Ratio H_2:O_2
1a	24 hrs	73.8	23	1 1
2a	24	74.7	22	1 1
3a	1	74.4	22	1 1
1b	24	74.6	24	1 1
2b	1	74.1	26	1 1
3b	30 min.	73.9	22	1 1
4b	30	74.7	24	1 1
5b	4 hrs.	74.5	21	2 1
6b	24	74.5	23	2 1
7b	5 min.	74.4	22	2 1
8b	5	74.4	22	2 1
9b	10	74.4	23	2 1
10b	10	74.4	23	2 1
11b	20	74.3	25	2 1
12b	30	74.3	25	2 1
13b	15	74.3	25	2 1
14b	15	74.3	22	2 1
15b	2 hrs.	74.6	24	2 1
16b	17	73.8	21	2 1
17b	5	75.1	23	2 1

TABLE No. I
(Continued)

Run Number	Time of run	Pressure cm. of Hg.	Temp. C°	Gas Ratio H_2:O_2
18b	15 min.	75.1	23	2 1
19b	15	137.0	24	2 1
20b	15	137.0	24	2 1
21b	15	137.0	24	2 1
22b	15	137.0	24	2 1
23b	30	137.0	24	2 1
24b	30	137.0	24	2 1
25b	15	165.0	23	2 1
26b	15	165.0	22	2 1
27b	15	165.0	22	2 1
28b	30	165.0	23	2 1
29b	30	165.0	22	2 1
30b	15	195.0	20	2 1
31b	15	195.0	23	2 1
32b	15	195.0	23	2 1
33b	15	195.0	24	2 1
34b	30	195.0	22	2 1
35b	30	195.0	22	2 1
36b	15	225.0	22	2 1
37b	15	225.0	22	2 1
38b	15	225.0	22	2 1
39b	15	225.0	22	2 1
40b	15	225.0	22	2 1

TABLE II

Reaction mass filtered, filtrate acidified and titrated with 0.0302 N $KMnO_4$ solution.

Run Number	$KMnO_4$ ml.
1a	0.7
2a	1.2
3a	0.4

TABLE III

Reaction mass blown into excess of acidified $KMnO_4$, filtered, and the filtrate titrated with 0.0245 N $FeSO_4$ solution.

Run Number	$KMnO_4$ ml.	$FeSO_4$ ml.	Remarks
1b	25.0	28.4	
2b	10.0	9.6	
3b	5.0	3.6	
4b	5.0	3.7	
5b	10.0	9.3	
6b	25.0	28.7	
7b	5.0	4.4	
8b	5.0	4.7	Uncertain result; slow discharge
9b	5.0	4.1	
10b	5.0	3.9	
11b	5.0		No result; discharge obstructed

TABLE III
(Continued)

Run Number	$KMnO_4$ ml.	$FeSO_4$ ml.	Remarks
12b	5.0	3.4	
13b	5.0	5.9	Uncertain result; slow discharge
14b	5.0	3.9	
15b	5.0	3.7	
16b	5.0	3.6	
17b	5.0	3.7	
18b	5.0	3.5	
19b	5.0	2.1	
20b	5.0	1.8	
21b	5.0		No result; discharge obstructed
22b	5.0	2.2	Catalyst recovered and reused
23b	5.0	2.0	
24b	5.0	1.9	
25b	5.0	1.4	
26b	5.0	1.6	
27b	5.0	1.4	Catalyst recovered and reused
28b	5.0	1.5	
29b	5.0	1.7	
30b	7.0	3.7	
31b	7.0	3.2	
32b	7.0		No result; discharge obstructed

TABLE III
(Continued)

Run Number	$KMnO_4$ ml.	$FeSO_4$ ml.	Remarks
33b	7.0	3.6	Catalyst recovered and reused
34b	7.0	3.2	
35b	7.0	3.4	
36b	7.0	2.9	
37b	7.0	2.6	Catalyst recovered and reused
38b	7.0	2.2	Uncertain result; slow discharge
39b	7.0	2.8	
40b	7.0	2.6	Catalyst recovered and reused
Blank	5.0	6.0	Catalyst alone
Blank	5.0	6.1	Recovered catalyst alone

TABLE IV

Experimental runs using 50 ml. of nickelized asbestos suspension, equivalent to 50 mg. of Ni, blowing reaction mass into acidified $KMnO_4$ and titrating with standard $FeSO_4$ solution.

Run Number	Time of run	Pressure cm. of Hg.	Temp. C°	Gas Ratio $H_2:O_2$	$KMnO_4$ ml.	$FeSO_4$ ml.
1c	15 min.	74.6	23	2 1	5.0	0.0
2c	15	74.6	23	2 1	25.0	0.0
Blank, catalyst alone					50.0	0.0

TABLE V

Experimental runs using 50 ml. of nickelized asbestos, filtering, and titrating filtrate with 0.0302 N $KMnO_4$ solution.

Run Number	Time of run	Pressure cm. of Hg.	Temp. C°	Gas Ratio H_2	Gas Ratio O_2	$KMnO_4$ ml.
1d	15 min.	74.4	24	2	1	0.4
2d	15	74.4	21	2	1	0.7
3d	15	74.4	24	2	1	0.3
4d	30	74.1	22	2	1	0.2
5d	30	74.1	22	2	1	0.5
6d	30	74.1	23	2	1	0.6

CALCULATIONS

Gas concentrations were calculated by means of a solubility table given by Hodgman.[1] It was assumed that Henry's law holds true at the concentrations used.

The following expression was deduced and used in the calculations:

$$C_{H_2} = \frac{(P - P_w) \times R_h \times S_h}{(760 - P_w)(R_h + R_o) \times M_h}$$

P is the observed pressure; P_w is the water vapor tension; R_h and R_o are the volume ratios respectively of Hydrogen and Oxygen; S_h is the solubility of Hydrogen at 760 mm. pressure and the observed temperature; M_h is the molecular weight of Hydrogen. A similar expression was employed in the calculating of C_{O_2}.

1. Hodgman, "Handbook of Chemistry and Physics" p. 674, 1932

TABLE VI

Results of calculations of equilibrium constant.

Run Number	$(C)H_2 \times 10^4$	$(C)O_2 \times 10^4$	$(C)H_2O_2 \times 10^4$	$K \times 10^{-5}$
	(Concentrations in mols per liter)			
1b	3.8	6.1	6.1	2.7
2b	3.7	6.0	6.7	3.0
3b	3.8	6.3	6.3	2.6
4b	3.8	6.1	6.1	2.7
5b	5.2	4.2	7.4	3.3
6b	5.1	4.1	5.3	2.5
7b	5.2	4.2	4.4	2.0
8b	5.2	4.2	3.6	1.6
9b	5.1	4.1	5.1	2.4
10b	5.1	4.1	5.5	2.6
11b	No result			
12b	5.0	4.0	6.8	3.4
13b	5.0	4.0	0.7	0.3
14b	5.1	4.0	5.5	2.7
15b	5.1	4.3	6.1	2.9
16b	5.1	4.3	6.3	2.9
17b	5.1	4.2	6.0	2.8
18b	5.1	4.2	6.5	3.0
19b	9.2	7.5	10.0	1.45
20b	9.2	7.5	10.7	1.55

TABLE VI
(Continued)

Run Number	$(C)H_2 \times 10^4$	$(C)O_2 \times 10^4$	$(C)H_2O_2 \times 10^4$	$K \times 10^{-5}$
	(Concentrations in mols per liter)			
21b	No result			
22b	9.2	7.5	9.6	1.39
23b	9.2	7.5	10.2	1.47
24b	9.2	7.5	10.2	1.52
25b	11.5	9.3	11.7	1.09
26b	11.6	9.5	11.2	1.02
27b	11.6	9.6	11.7	1.06
28b	11.5	9.3	11.4	1.07
29b	11.6	9.5	10.9	0.99
30b	13.9	11.8	12.2	0.75
31b	13.6	11.1	13.3	0.88
32b	No result			
33b	13.5	10.9	12.3	0.82
34b	13.7	11.4	13.3	0.86
35b	13.7	11.4	12.8	0.82
36b	15.8	13.1	14.1	0.68
37b	15.8	13.1	14.8	0.71
38b	15.8	13.1	15.7	0.76
39b	15.8	13.1	14.3	0.69
40b	15.8	13.1	14.7	0.71

DISCUSSION OF RESULTS

Concordant results were not secured by filtering off the catalyst and titrating the filtrate with Potassium Permanganate. This may be explained by the fact that during suction filtration the pressure, and thus the concentration of the gas in solution, was greatly changed. This resulted in completely upsetting the equilibrium conditions, and, therefore, any conclusions drawn from the experimental runs of Series A are likely to be invalid.

Results obtained by running the Palladium catalyst suspension into an excess of Potassium Permanganate, filtering, and titrating with Ferrous Sulphate are somewhat erratic at atmospheric pressure, but are more nearly concordant at the higher pressures.

This may be accounted for by the experimental difficulty of removing the mixture from the reaction chamber at atmospheric pressure. At the higher pressures, the reaction mass was rapidly discharged, and reaction with the acidified permanganate solution secured before and appreciable amount of Hydrogen Peroxide had decomposed.

As the gas pressures were increased, an increase

in the concentration of Hydrogen Peroxide was noted. Consideration of the reaction

$$H_2 + O_2 \rightleftharpoons H_2O_2 \rightleftharpoons H_2O + \tfrac{1}{2}O_2$$

from the standpoint of Le Chatelier's principle may point out an explanation. Pressure increase upon the system would tend to shift the reaction toward an increase in the concentration of Hydrogen Peroxide.

Values of this equilibrium constant show a continual decrease with increasing pressure. This seems unusual, and the following explanation is suggested. In addition to the above reaction, it is possible that the secondary reaction

$$H_2O_2 + H_2 \rightleftharpoons 2H_2O$$

occurs. Pressure increase upon the system would tend to shift this equilibrium toward a decrease in Hydrogen Peroxide concentration. Thus, the equilibrium concentration would be the resultant of the two reactions.

For the first reaction $K = \dfrac{C_{H_2O_2}}{C_{H_2} \times C_{O_2}}$, and for the second $K_1 = \dfrac{C^2_{H_2O}}{C_{H_2O_2} \times C_{H_2}}$. Upon dividing K by K_1, and eliminating the constant $C^2_{H_2O}$, the

expression now obtained is $K_2 = \frac{C^2_{H_2O_2}}{C_{O_2}}$.

The calculated values of this new equilibrium constant at various pressures are shown in Table VII. Temperature variations are neglected, since they are relatively small, and the values of C_{O_2} and $C_{H_2O_2}$ are the mean of results at the pressures indicated. This data is taken from the runs of Series B.

TABLE VII

Pressure cm. of Hg.	$(C)O_2 \times 10^4$ (Concentrations in mols per liter)	$(C)H_2O_2 \times 10^4$	$K_2 \times 10^4$
74.5	4.16	6.24	9.4
137.0	7.50	10.10	13.6
165.0	9.42	11.24	13.4
195.0	11.32	12.78	14.4
225.0	13.10	14.72	16.6

The above values of K_2 are reasonably concordant, if one keeps in mind the fact that the experimental data at atmospheric pressure was subject to considerable error and probably gave low results for the concentrations of Hydrogen Peroxide.

Experimental runs of Series C gave no useful data whatever. It was impossible to secure an end point during the titrations, because of the reduction of the Potassium Permanganate by the catalytic agent itself.

Series D, also, gives such incongruent results that no conclusions may be derived from it.

SUMMARY

1. Measurements have been made to determine the equilibrium constant of the catalyzed Hydrogen-Oxygen, Hydrogen Peroxide reaction.

2. Palladium and Nickel were used as the catalytic agents.

3. Increasing pressure caused an increase in Hydrogen Peroxide concentration, and, at the same time, a decrease was noted in the value of the equilibrium constant.

4. An attempt has been made to reconcile these results with Le Chatelier's principle.

5. The results secured indicate that $K = \frac{C^2_{H_2O_2}}{C_{O_2}}$.

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APPROVAL

The foregoing thesis is hereby approved as a creditable study of an engineering subject, carried out and presented in a manner sufficiently satisfactory to warrant its acceptance as a prerequisite to the degree for which it has been submitted. It is to be understood that by this approval the undersigned does not necessarily endorse or approve any statements made, opinions expressed, or conclusions drawn therein, but approves the thesis only for the purpose for which it was submitted.

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