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THE PHOTOCHEMICAL
DECOMPOSITION OF HYDROGEN
PEROXIDE

BY

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Introduction.

IN 1900 KISTIAKOWSKY¹ found that potassiumferro- and ferri cyanide were able to act as an optical sensitiser on the decomposition of hydrogen peroxide. This process exhibits several interesting features. It shows, f. inst., a very marked after-action. KISTIAKOWSKY's measurements showed that a quite evanescent insolation (1 minute) was sufficient to give the same velocity as a continued insolation, a fact which suggests the formation by the insolation of a catalyst of very great longevity. The catalyst formation was directly shown by WEIGERT², by first exposing a solution of potassiumferro-cyanide to light and then adding it to hydrogen peroxide in the darkness, whereafter a vivid decomposition followed. J. AMANN³, too, was able to show ultra-microscopically that by exposure of potassiumferro-cyanide, colloid particles are formed, by which the oxygen-formation of the hydrogen peroxide mainly takes place. The mechanism of the process, hence, seems fairly well elucidated, if we disregard the fact that no determinations of colour-sensitiveness are present.

With regard to the order of reaction, KISTIAKOWSKY found that the process in special cases, i. e. at certain mixtures of ferro- and ferri-cyanide, was mono-molecular, but that the constant in all other cases gradually increased somewhat. This

¹ Zeitschr. f. physikal. Chemie **35**, 431 (1900).

² Ann. d. Physik (4) **24**, 261 (1907).

³ Kolloid-Zeitschr. **8**, 13 (1911).

vagueness cannot give rise to any astonishment, because of the exclusive employment of mixed light (arc) for the exposure.

By trying to prepare this process for lecture purposes it proved, however, to my great astonishment, that the reaction velocity was constant everywhere, the process, thence, was zero-molecular. At my desire Mr. S. WEIBEL therefore examined a series of different concentrations of hydrogen peroxide, potassiumferro- and potassiumferri-cyanide, amongst which also those for which KISTIAKOWSKY had found the first order. But the velocity was found to be constant everywhere both in monochromatic and mixed light, with arc and with quartz-mercury-lamp, and under extremely different conditions with regard to the exposure of the single portions and the taking out of samples, and we have not succeeded in finding the cause of the discrepancies between KISTIAKOWSKY's and our results.

The final measurements which were carried out by myself, were for the special purpose of 1) determining the efficiency of the absorbed radiation, 2) to find by which law the light-catalyst is formed and disappears and 3) to calculate the rate of reaction in the mixture of hydrogen peroxide and potassiumferro-cyanide from the knowledge of the formation and effect of the catalyst comprised by the two previous points.

The employed arrangement was quite the same as by the quantitative investigation of the oxidation of the hydriotic acid¹, only differing by the omission of the air-supply which was compensated by mechanical stirring by means of a very rapidly rotating spiral glass stirrer. All these exposures were executed in a couvette of 1 cm. thickness of layer and furnished with glass walls. The quartz-mercury-lamp, with in-

¹ Kgl. Danske Vid. Selsk, Math.-fys. Medd. II, 2.

serted ray filters, served as a light-source. In order to obtain constant results it proved necessary to cleanse all the glass apparatus with strong hydrochloric acid.

Formation of the Catalyst.

For this 0,075 gm. potassiumferro-cyanide was dissolved in 50 cm.³ water (0,0035-m). After having exposed this solution for t minutes, 5 cm.³ was taken out and added to 5 cm.³ diluted hydrogen peroxide (abt. 0,08-m and 0,04-m). The mixture was left in darkness for 30 minutes and was then acidified with sulphuric acid and titrated with 0,02-m potassium permanganate.

Table 1.

405 $\mu\mu$ (Fig. 1).

Experiment 1.			Experiment 2.		
360 Watt, $E = 4,7$ Erg.			340 Watt, $E = 4,4$ Erg.		
t	$KMnO_4$	$\Delta H_2O_2 \cdot 10^4$	t	$KMnO_4$	$\Delta H_2O_2 \cdot 10^4$
0	20,10	—	0	20,25	—
3	20,00	0,02	3	20,15	0,02
6	19,80	0,06	6	20,05	0,04
9	19,60	0,10	9	19,85	0,08
12	19,60	0,10	12	19,85	0,08
15	19,10	0,20	15	19,35	0,18
18	19,35	0,15	18	19,65	0,12

366 $\mu\mu$ (Fig. 2 and 3).

Experiment 3.			Experiment 4.			Experiment 5.		
330 Watt, $E = 6,4$ Erg.			410 Watt, $E = 6,6$ Erg.			440 Watt, $E = 7,0$ Erg.		
t	$KMnO_4$	$\Delta H_2O_2 \cdot 10^4$	t	$KMnO_4$	$\Delta H_2O_2 \cdot 10^4$	t	$KMnO_4$	$\Delta H_2O_2 \cdot 10^4$
0	20,20	—	0	20,22	—	0	20,30	—
3	19,00	0,24	3	18,55	0,33	3	18,10	0,44
6	18,60	0,32	6	18,25	0,39	6	17,80	0,50
9	18,60	0,32	9	18,45	0,35	9	17,90	0,48
12	18,35	0,37	12	18,35	0,37	12	18,00	0,46
15	18,45	0,35	15	18,45	0,35	15	18,18	0,42
18	18,40	0,36	18	18,30	0,38	18	18,02	0,46

Experiment 6.				Experiment 7.				Experiment 8.			
415 Watt, $E = 13,9$ Erg.				390 Watt, $E = 11,2$ Erg.				430 Watt, $E = 12,9$ Erg.			
t	$KMnO_4$	$\Delta H_2O_2 \cdot 10^4$	k	t	$KMnO_4$	$\Delta H_2O_2 \cdot 10^4$	k	t	$KMnO_4$	$\Delta H_2O_2 \cdot 10^4$	k
0	19,75	—	—	0	10,10	—	—	0	10,45	—	—
1	18,60	0,23	0,18	1	9,45	0,13	—	1	9,77	0,14	—
2	17,70	0,41	0,21	2	8,80	0,26	—	2	8,90	0,31	—
3	17,05	0,54	0,25	3	8,55	0,31	—	3	8,60	0,37	—
4	16,70	0,61	0,25	4	8,20	0,38	—	4	8,50	0,39	—
6	16,63	0,62	0,22	6	8,30	0,36	—	6	8,35	0,43	—
8	16,60	0,63	0,19	8	8,15	0,39	—	8	8,45	0,40	—
			$c_\infty = 0,65$				$0,22$				

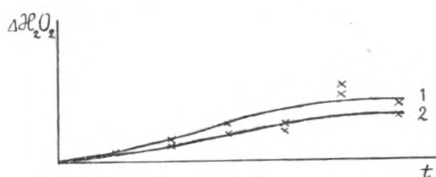


Fig. 1.

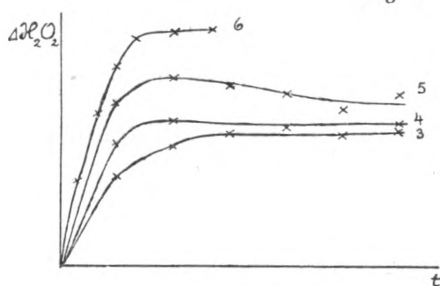


Fig. 2.

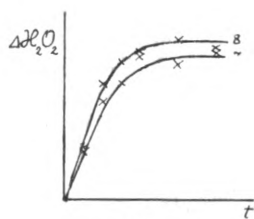


Fig. 3.

The experiments show that the catalyst formation increases with the time of exposure until a certain maximum concentration, which rises with the applied energy of light, and — to a far slighter degree — with the hydrogen peroxide-concentration. This maximum concentration for the hydrogen peroxide-concentration 0,08-m (Exp. 1—6) can with good approximation be put equal to:

$$c_\infty = 0,071 \cdot 10^{-4} \cdot E_{\text{abs}} + 0,14 \cdot 10^{-4}$$

where E_{abs} is the energy absorbed per mm^2 and second in erg. The agreement is seen in table 2.

Table 2.

Experiment	λ	E_{abs}	C_{∞}		
			found	calcul.	diff.
1	405	0,31	0,15	0,16	-0,01
2	>	0,29	0,12	0,16	-0,04
3	366	3,3	0,36	0,37	-0,01
4	>	3,4	0,37	0,38	-0,01
5	>	3,6	0,46	0,40	+0,06
6	>	7,1	0,65	0,64	+0,01

Also the velocity with which the catalyst is formed increases, as shown by the figures, with the intensity, and the initial velocity seems to be proportional to the absorbed energy of light.

The fraction of the penetrating light, absorbed in 1 cm. layer was found to be 0,065 for λ 405 (with spectral photometer) and for λ 366 (with fluorimeter) 0,51. In the experiments 1—6, the amount of substance transformed in the beginning per absorbed energy-unit (erg.) will in mol. 10^{10} hereafter be:

Table 3.

Experiment	λ	t	$\Delta H_2O_2 \cdot 10^4$	E_{abs}	$(\Delta H_2O_2 : E_{\text{abs}}) \cdot 10^{10}$
1	405	3	0,02	$2,1 \cdot 10^4$	1,0
2	>	3	0,02	$2,1 \cdot 10^4$	1,0
3	366	3	0,24	$2,9 \cdot 10^5$	0,8
4	>	3	0,33	$3,0 \cdot 10^5$	1,1
5	>	3	0,44	$3,2 \cdot 10^5$	1,4
6	>	1	0,23	$2,1 \cdot 10^5$	1,1
					<hr/> 1,1

The experiments 7 and 6 (half hydrogen peroxide-concentration) give the following values:

7	366	1	0,13	$1,7 \cdot 10^5$	0,8
8	>	1	0,14	$1,9 \cdot 10^5$	0,7
					<hr/> 0,75

The efficiency is here, perhaps, a little lower than for the double concentration. For the most complete and accurate of the velocity curves (experiment 6) a fairly good reaction

constant of the first order may be calculated (as seen in the table). The velocity formula may therefore be written:

$$\frac{dc}{dt} = k_1 E_{\text{abs}} - k_2 c$$

where E_{abs} is the absorbed amount of energy, c the concentration of the catalyst to the time t , k_1 and k_2 the velocity constants by the formation and transformation of the catalyst. The found constant—typified in the table—is then = 0,4343 k_2 and refers to the retrogressive transformation of the catalyst.

The Transformation of the Catalyst.

The magnitude 0,4343 k_2 in mean being equal to 0,22, it is easy to calculate that the retrogressive transformation in darkness ought to occur so rapidly, that after the course of 1 minute 60 per cent., after 3 minutes 22 per cent., and after 10 minutes 0,6 per cent. should be left of the maximum amount of catalyst. These predictions prove very far from the mark here.

The 0,0035-molar potassiumferro-cyanide-solution was insolated for 8 minutes with the wave length 366 μm (hence, to maximum catalyst-concentration). After t minutes, 5 cm^3 were taken out in darkness and added to 5 cm^3 diluted hydrogen peroxide (abt. 0,08-m). After having been left for 30 minutes, the mixture was acidified and titrated.

Table 4.

Experiment 9. 420 Watt.		Experiment 10. 430 Watt.		Experiment 11. 430 Watt.	
t	$\Delta H_2O_2 \cdot 10^4$	t	$\Delta H_2O_2 \cdot 10^4$	t	$\Delta H_2O_2 \cdot 10^4$
0	0,36	0	0,56	0	0,30
1	0,47	10	0,34	3	0,25
2	0,49	20	0,30	6	0,31
3	0,47	30	0,38	9	0,33
4	0,45	40	0,28	12	0,26
6	0,47	60	0,27	15	0,28

It is only in the beginning of experiment 10 that a decrease in the amount of catalyst seems to take place, but thereafter this quantity keeps fairly constant instead of falling to zero. In the two other experiments the amount of catalyst oscillates irregularly around a mean value. The first value in experiment 10 must then, presumably, be due to an experimental error, inasmuch as the subsequent section shows that the catalyst does not decrease perceptibly in the course of several hours.

The apparent disagreement between this extraordinary stability and the existence of a stationary state might, perhaps, be explained in the following way. KISTIAKOWSKY has found that a potassiumferro-cyanide-solution becomes alkaline by insolation. But according to HENRI and WURMSER¹ the decomposition of hydrogen peroxide in light is inhibited by bases, whereas the decomposition of the same substance in darkness is accelerated by bases. Insolated potassiumferro-cyanide-solution must with its contents of light catalyst be considered as light in regard to hydrogen peroxide, and there is consequently the possibility that the alkali formed by the insolation can act as a negative catalyst to such a degree, that a compromise occurs between the effect of the catalyst and the hydroxylion, the concentrations of which both increase by continued exposure.

It is difficult to know what quantities can come into question. Taking for granted what is certainly exaggerated, that each molecule of potassium ferro-cyanide transformed to light catalyst thereby yields a molecule of potassium hydroxide, we can by means of the law of critical efficiency mentioned later on², calculate that, f. inst. in experiment 6 0,00035 mol. potassium hydroxide per liter will be formed in the

¹ Compt. rend. 157,284 (1913).

² Kgl. Danske Vid. Selsk. Math.-fys. Medd. II, 3.

course of 4 minutes. According to the results of HENRI and WURMSER such an alkali concentration will lower the decomposition of the hydrogen peroxide to abt. 30 per cent. of the value in neutral solution, it would, then, act as a powerful negative catalyst.

This explanation cannot after all be correct, for as the inhibiting effects of the potassium hydroxide increase far more than its concentration, while the light catalyst must be supposed to be formed with constant velocity, the absorption remaining practically constant, the effects on the hydrogen peroxide should necessarily first pass a maximum and then fall in a marked degree by further exposure, while the experiments point decisively in the direction of a stationary state.

Another explanation might be that an inhibiting substance was formed simultaneously with and proportionally to the catalyst itself. If the inhibiting effect of this substance be proportional to its concentration, we obtain a velocity formula of similar form as the one discussed above, with the difference only that the constant k_2 now — one constant factor excepted — corresponds to the formation of the catalyst.

Above was found:

$$c_{\infty} = 0,071 \cdot 10^{-4} E_{\text{abs}} + 0,14 \cdot 10^{-4}$$

The velocity formula which in the stationary state should give this result, must be:

$$\frac{dc}{dt} = K (0,071 \cdot 10^{-4} E_{\text{abs}} + 0,14 \cdot 10^{-4} - c).$$

From experiment 6 we have:

$$K = \frac{0,23 \cdot 10^{-4}}{0,071 \cdot 10^{-4} \cdot 7,1 + 0,14 \cdot 10^{-4}} = 0,35.$$

By application of this formula we obtain the following values for the quantity transformed in the first minute:

Experiment:	1	2	3	4	5
	0,05	0,05	0,13	0,13	$0,14 \cdot 10^{-4}$,

while in 3 minutes was found:

0,02 0,02 0,24 0,33 $0,44 \cdot 10^{-4}$.

Table 5 shows the accordance between the found and the calculated values for experiment 6.

Table 5.
 $H_2O_2 \cdot 10^4$

found	cal.
0,23	(0,23)
0,41	0,38
0,54	0,48
0,61	0,54
0,62	0,61
0,63	0,63

The formula is evidently fairly well adapted for the experiments with rather large decompositions, whereas it quite fails us for the small alterations in experiments 1 and 2, where they are found as very small differences between two titrations having fairly large quantities of liquid, and they cannot be very correct, which is also shown by the two said experimental tables. Any importance can, therefore, hardly be attached to this disagreement.

Considering the formula to be correct, we obtain for the initial velocity no simple proportionality with the absorbed energy, as the experiments seemed to show, but a lineary relation expressed by:

$$\frac{dc}{dt} = 0,35 (0,71 \cdot 10^{-4} E_{\text{abs}} + 0,14 \cdot 10^{-4})$$

Fig. 4 contains this line together with the found values calculated for the first minute, which no doubt gives too small values.

What significance the constant magnitude $0,14 \cdot 10^{-4}$ possesses is an unsolved question. The mixture of hydrogen peroxide and potassiumferro-cyanide showing, also in darkness, a faint decomposition, under the given circumstances being

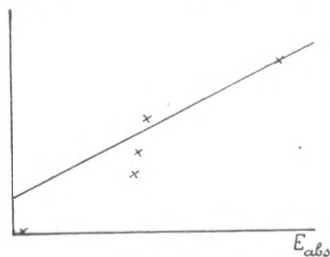


Fig. 4.

abt. $0,13 \cdot 10^{-4}$ in 30 minutes, it is an obvious conclusion, that the potassiumferro-cyanide-solution already previously to the exposure contains a corresponding amount of the colloidal catalyst. But this darkness-process is beforehand subtracted from all the given numbers, and it is not easy to see how this originally present catalyst could act twice as powerfully by exposure as in darkness, all the experiments showing that the catalyst formed by exposure acts with exactly the same power in darkness as in light, as soon as it has been formed. The assumption that the nuclei of the darkness catalyst should be able to serve as specially fruitful starting points for the formation of light catalyst, only introduces the absorbed energy also in this term of the formula, deprives it, then, of its constancy, and is consequently incapable of removing the difficulty.

The Action of the Catalyst.

In all of the previously adduced experiments the decomposition of the hydrogen peroxide was measured after the mixture with potassiumferro-cyanide had been left in darkness for 30 minutes. This period was chosen for purely practical reasons and is, as will be shown by the following experiments, as applicable as any other period within reasonable limits.

Potassiumferro-cyanide solution (0,0035-m) was in 1 cm. layer exposed to light for 3 minutes with $\lambda 366$, and thereafter added to equal volumes of hydrogen peroxide-solution (0,08-m). The mixture was left in darkness for t minutes, after which 10 cm³ were taken out and titrated.

Table 6.
Experiment 12.
400 Watt, $E = 13,2$ Erg.

t	5	10	20	40	60	
$\Delta H_2O_2 \cdot 10^4$	0,12	0,20	0,37	0,61	0,90	
$\Delta H_2O_2 \cdot 10^4$	0,024	0,020	0,019	0,015	0,015	Cal. 0,021
$\frac{\Delta H_2O_2 \cdot 10^4}{t}$						

In another experiment with 2 minutes primary exposure was found:

Experiment 13.					
416 Watt, $E = 14,2$ Erg.					
t	30	60	120	150	
$\Delta H_2O_2 \cdot 10^4$	0,40	1,23	2,67	3,04	
$\frac{\Delta H_2O_2 \cdot 10^4}{t}$	0,013	0,021	0,022	0,020	Cal. 0,022

A portion, being quite uninsolated, in 150 minutes lost $0,13 \cdot 10^{-4}$ mol H_2O_2 .

A third experiment with 8 minutes' primary exposure gave:

Experiment 14.					
445 Watt, $E = 19,5$ Erg.					
t	30	60	120	180	
$\Delta H_2O_2 \cdot 10^4$	0,44	0,73	1,83	3,04	
$\frac{\Delta H_2O_2 \cdot 10^4}{t}$	0,015	0,012	0,015	0,017	Cal. 0,029

These results are exhibited in fig. 5 from which it appears that the catalyst acts with almost constant velocity until the greater portion of the hydrogen peroxide is decomposed. The decrease of velocity which then

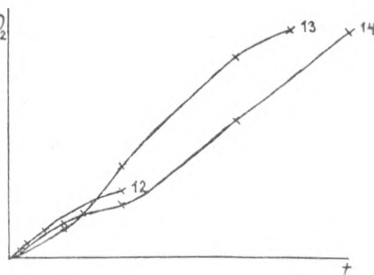


Fig. 5.

appears may very well be due to the mass action, so that the catalyst, then, does not presumably lose any of its activity by the decomposition itself, but keeps this activity unaltered through several hours and perhaps through a much longer period. The values for the decomposition per minute which are given under »cal.» are calculated from the formula for c_∞ (v. p. 6), which implies that the formation of catalyst has been complete.

The Total Process.

When the velocity with which the catalyst is formed by exposure as well as the constant velocity with which the catalyst exerts its action are both known, the velocity of the total process should be computable from this, if new complications do not appear by exposure of the mixture of the two substances. The provisional experiments indeed pointed in the direction of the presence of such complications, the mixture rather quickly becoming much darker (greenish) coloured by exposure, whereas the pure potassiumferro-cyanide-solution keeps its colour unaltered. An experiment to determine the extinction-coefficient of the mixture for $\lambda 405$ for this reason remained without result. During the measurement itself with the spectral photometer the magnitude αc increased from 0,55 to 0,79 in the course of abt. 10 minutes, whereby the quartz-mercury lamp, screened with dull glass, served as a light-source. For the corresponding pure solution of potassiumferro-cyanide is $\alpha c = 0,029$, and it has been impossible for me to decide how much of the increase up to 0,55 is due to the mixing process itself and how much to the exposure.

In spite of this considerable increase in the absorption, the mixture is only very little sensitive to $\lambda 405$. The found losses of hydrogen peroxide are so small and vary so irregularly that there is no reason to state the numbers.

With regard to $\lambda 366$ it is quite another matter. The extinction was measured behind the spectograph by means of the fluorimeter. There was measured partly a fresh unisolated mixture, and partly a mixture having served for one of the experimental exposures given below, whereby abt. half of the hydrogen peroxide was decomposed. In the first case (fresh

solution) $\alpha c = 0,31$, in the last case (exposed solution) 0,43 while the pure solution of potassiumferro-cyanide has $\alpha c = 0,31$. In this case there is thus only a comparatively slight increase of the absorption by exposure, and the great increase of $\lambda 405$ must therefore be due to quite a new absorption band in this part of the spectre, a band which has evidently nothing to do with the formation of the light catalyst. We might therefore hope to avoid complications by only exposing the mixture to $\lambda 366$. Two such experiments of exposure were carried out. In both cases 50 cm.³ potassiumferro-cyanide-solution (0,0035-m) was mixed with 50 cm.³ hydrogen peroxide (0,08-m) and exposed for t minutes in 2 cm. layer. Thereafter 10 cm.³ were taken out and titrated immediately.

Table 7.

Experiment 15.					Experiment 16.				
440 Watt, $E = 15,9$ Erg.					405 Watt, $E = 16,6$ Erg.				
t	$\Delta H_2O_2 \cdot 10^4$				t	$\Delta H_2O_2 \cdot 10^4$			
found	calculated				found	calculated			
	I	II	III		I	II	III		
10	0,15	0,19	0,25	0,20	5	0,04	0,08	0,10	0,08
20	0,53	0,44	0,58	0,60	10	0,18	0,19	0,25	0,20
43	1,32	1,02	1,34	1,28	15	0,36	0,32	0,42	0,33
68	2,09	1,65	2,17	2,10	25	0,74	0,57	0,75	0,74
					35	1,19	0,82	1,08	1,04

As shown particularly by experiment 16 and fig. 6, there is also in this process — as in the oxidation of iodine hydrogen¹ — a period of induction arising here as there, from the fact that the catalyst is formed slowly by the exposure itself. There is, however, the difference between the two processes, that in the case before us, the substance concerned — potassium-

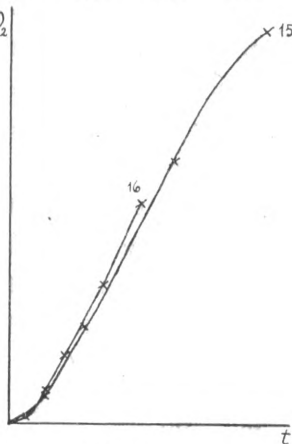


Fig. 6.

¹) Kgl. Danske Vid. Selsk., Math.-fys. Medd. II, 2.

ferrocyanide — is light-sensitive, the catalyst thus being able to be formed even if none of it is found beforehand, whereas it is an absolute condition of the sensitivity of the hydriotic acid that some of the catalyst is found beforehand, the hydrogen iodide not being sensitive in itself.

The magnitudes given in the columns »calculated, I, II and III« are calculated in the following manner.

The velocity of reaction by the formation of the catalyst was found above (p. 10) to be:

$$\frac{dc}{dt} = 0,35 (0,071 \cdot 10^{-4} E_{\text{abs}} + 0,14 \cdot 10^{-4} - c)$$

from which:

$$c = (0,071 \cdot 10^{-4} E_{\text{abs}} + 0,14 \cdot 10^{-4}) (1 - 10^{-0,4343 \cdot 0,35 t}).$$

In the way this formula is derived the velocity is expressed by the amount of hydrogen peroxide which is decomposed by being left for 30 minutes with the newly formed catalyst. This velocity of decomposition for the hydrogen peroxide is — as shown above — constant. In 1 minute, consequently, $\frac{1}{30}$ of the amount which is denoted by the above formula is decomposed. If no unforeseen complications arise by exposure of the mixed solution, the hydrogen peroxide must by this exposure be decomposed with the same velocity as by primary exposure of potassiumferro-cyanide, and subsequent mixing with hydrogen peroxide in darkness, the activity of the catalyst being apparently of unlimited duration. The velocity of decomposition of the hydrogen peroxide by exposure of the mixed solution is consequently:

$$\div \frac{dc'}{dt} = \frac{1}{30} c = (0,0024 \cdot 10^{-4} E_{\text{abs}} + 0,0047) (1 - 10^{-0,4343 \cdot 0,35 t}).$$

Column I in the above table is calculated on the presupposition that the extinction of the mixture has kept unaltered ($\alpha c = 0,31$) during the exposure; the calculated figures being so much more behind the found the longer the exposure lasts. It is taken for granted in the calculation of column II that the extinction has all the time had the slightly greater value which was found for the exposed mixture ($\alpha c = 0,43$). In this place the calculated figures are too large to start with, but agree so much the better with the found ones, the longer the exposure lasts. Assuming, finally, the alteration from 0,31 to 0,43 to have taken place proportionally to the time in the 68 minutes the mixture has been exposed before the extinction was measured last time, we obtain column III, which agrees as well as can be expected with the found values.

The velocity of reaction by exposure of the mixture of potassiumferro-cyanide and hydrogen peroxide can consequently be calculated when the velocity by which the light-catalyst is formed, and the velocity with which this light-catalyst decomposes hydrogen peroxide in darkness are known.

Results of This Work.

By exposure of a mixture of potassium ferrocyanide and hydrogen peroxide in diluted solution with ultra-violet the hydrogen peroxide is decomposed at a rate, which at first is very small, afterwards increases, and finally becomes constant.

This is explained by the slow formation of a constant amount of a substance, which catalyses the decomposition of hydrogen peroxide, and the stability of which is very great.

The rate of decomposition of hydrogen peroxide in the

mixed solution can be calculated, when the rate of formation of the catalyst and the rate of its effect on the hydrogen peroxide is known.

*Royal Technical College of Copenhagen,
May 1919.*