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# The decomposition of potassium chlorate: II, The spontaneous decomposition temperatures using various proportions of manganese dioxide, and of ferric oxide, and a mixture of manganese dioxide and ferric oxide as catalysts

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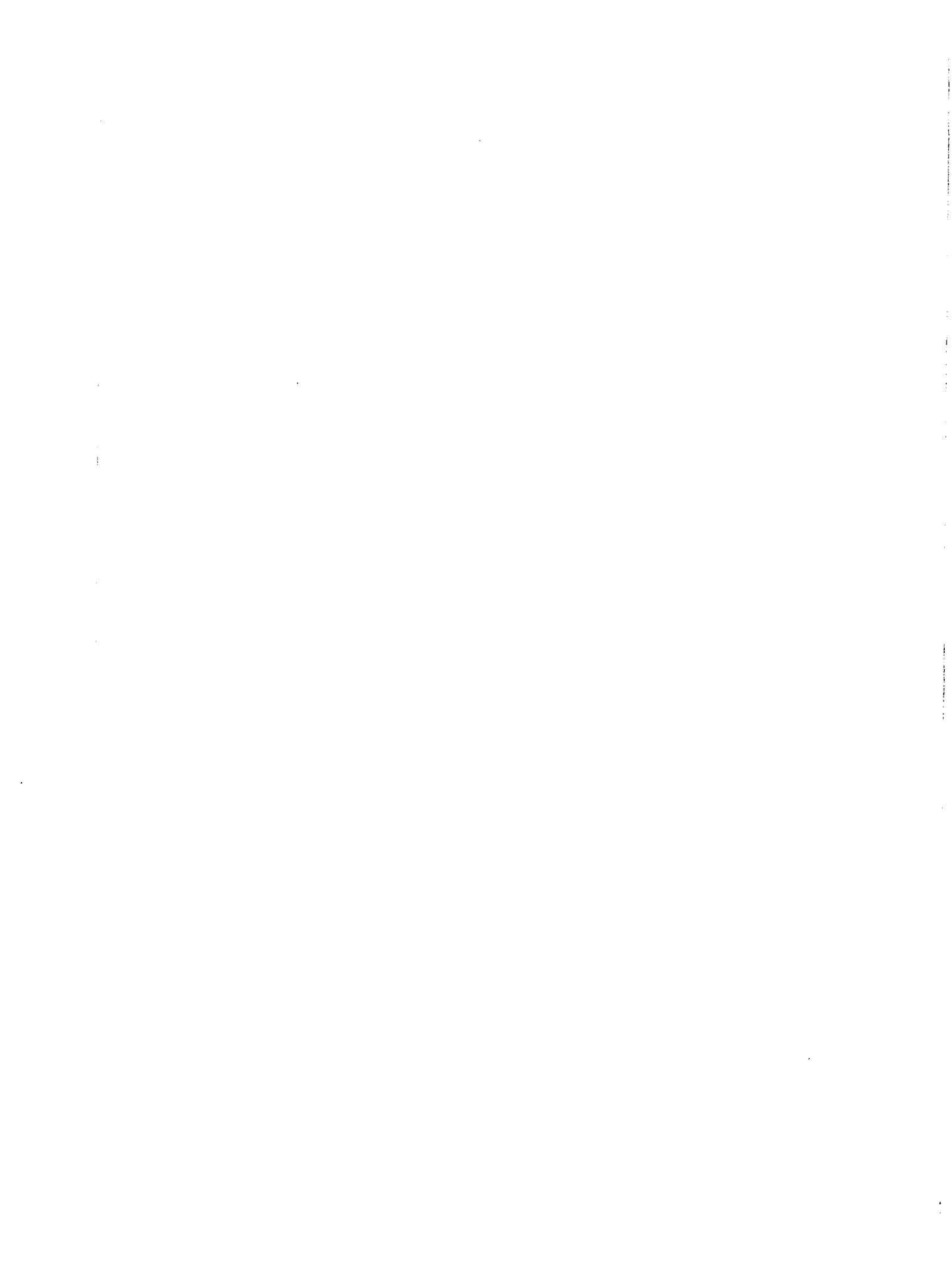
THE DECOMPOSITION OF POTASSIUM CHLORATE

II. The Spontaneous Decomposition Temperatures Using

Various Proportions of Manganese Dioxide, and of

Ferric Oxide, and of a Mixture of Manganese

Dioxide and Ferric Oxide as Catalysts

By

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Ames, Iowa                                    James Austin Burrows.

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# INTRODUCTION

In a previous paper (1) was reported the spontan-
eous decomposition temperature of potassium chlorate in the
presence of manganese dioxide - the "spontaneous decompo-
sition temperature" being the temperature at which the
mixtures began to evolve oxygen violently, and produce
enough heat to carry on its own reaction and produce a rise
in temperature. The object of the present work is to rede-
termine this temperature for the various mixtures of potas-
sium chlorate and manganese dioxide, as well as to determine
the temperatures for mixtures of potassium chlorate with
ferric oxide, and to check the promoter action of ferric
oxide on the potassium chlorate-manganese dioxide mixtures
as reported by Neville (2). The redetermining of the
temperatures for the potassium chlorate-manganese dioxide
mixtures was made because the manganese dioxide used in the
previous work was commercial manganese dioxide and Neville
stated that commercial dioxide contains 8.8 percent of ferric
oxide and that the ferric oxide had a promoter action upon
the manganese dioxide. The manganese dioxide used in this
later work was especially prepared, purified and tested.

The determining of the temperatures for the potassium chlorate-ferric oxide mixtures was deemed necessary to clear up some wquestions of the purity of the ferric oxide used in the previous work done on these mixtures by F. E. Brown and W. C. O. White (3). The ferric oxide used by them was prepared by the use of ammonium hydroxide and ferric chloride. These authors and others (4) have shown that the presence of salts of ammonium give an apparent lower decomp osition temperature than the pure catalyst. This is believed to be due to the oxidation of the ammonium salts at a low temperature into nitrogen, water and possibly certain oxides. Furthermore Brown and White heated some of their ferric oxide to a high temperature and it is believed this heat treatment has some effect upon its catalytic effect.

Neville (2) has shown that ferric oxide exerts a promoter action on the catalytic effect of manganese dioxide and it was decided to determine the relationship between the promotion produced and the spontaneous decomposition temperatures of a mixture containing both these oxides.

## PREPARATION OF MATERIALS

The potassium chlorate used was the C.P. grade of Baker and Adamson which had previously been analyzed by H. M. McLaughlin in this laboratory and found to be free from chlorides, heavy metals, calcium, bromates, nitrates, and sulfates, and except for a small amount of absorbed moisture was 100 percent pure potassium chlorate. This was then pulverized and passed through a 200 mesh sieve.

The manganese dioxide was prepared from Merck's highest purity "C.P." manganese carbonate according to the method of Forchhammer (5). The method consisted in heating the manganese carbonate in air at a temperature ranging from 255° to 260° on a sand bath and stirring at frequent intervals. This was continued until samples of it evolved no carbon dioxide when treated with dilute HCl. The material was then washed with diluted HCl for same time and finally washed free of any chlorides by means of distilled water. It was then tested and found to be free of iron, silica, nickel, and ammonium compounds. Its oxidizing power was determined by the ordinary reaction of first dissolving it in sulfuric acid to which a known amount of oxalic acid was added. It was then titrated back with standard permanganate solution. Two-tenths of a grain of this mang-

anese dioxide required 15.8 cc of $\frac{N}{5}$ oxalic acid. (The theoretical amount of $\frac{N}{5}$ oxalic acid for 0.2 g of manganese dioxide is 2.3 c c.) The total manganese content was then determined by the bismuthate method by first dissolving the sample in a sulfuric-nitric acid mixture and then, after diluting to a known volume an aliquot part is analyzed as usual. The net amount of 0.0661 N $FeSO_4$ oxidized by the permanganate produced in samples containing 0.01 g of the manganese dioxide for each of six trials was: 8.84 cc, 8.87 cc, 8.56 cc, 9.13 cc, 8.84 cc, 8.99 cc. (The theoretical amount of the same strength $FeSO_4$ required for 0.01 g of $MnO_2$ is 8.696 cc.) It is believed that the discrepancy between the results obtained from the oxidizing power and these last results can be accounted for by the fact that the manganese dioxide contains some lower oxides of manganese. It is believed that the catalysis of $KClO_3$ is always associated with those substances which can be alternately oxidized and reduced. Therefore the total manganese content is the imp ortant factor and the presence of lower oxides would not constitute an impurity in this reaction. It might be stated further that the catalytic effect of manganese carbonate was tried and was found to be almost as efficient a catalyst as the equivalent moleculer prop ortion of mang-

anese dioxide. The spontaneous decomposition came just a few degrees higher for the carbonate.

The ferric oxide was prepared from Merck's Reagent Ferric chloride by dissolving it in water, filtering and precipitating the iron with sodium hydroxide in an iron container. The material was allowed to settle and decanted many times. This was continued until the decanted material was free from chlorides as shown by silver nitrate. Toward the end of this procedure it required weeks for the ferric hydroxide to settle due to its tendency to stay colloidal. Care was taken that the best sodium hydroxide was used by taken Merck's pure sticks, making a saturated solution and filtering off whatever sodium carbonate was present since it is insoluble in this concentration. This filtering was accomplished through clean iron filings and powder on a Gooch crucible which was paraffined to prevent the introduction of silica from the porcelain. The ferric hydroxide obtained was then dried for about two weeks in an oven at 110° and finally powdered and sifted through a 200 mesh sieve. The oxide so formed was then analyzed by the Zimmermann-Reinhardt (6) method. A hydrochloric acid solution was treated with stannous chloride and then oxidized with standard permanganate solution in the presence of manganese sulfate solution. The

oxide thus analyzed tested 98.5 percent. The remaining 1.5 percent was thought to be due to moisture more or less closely adhering. This oxide was found to be free from manganese, silica, nickel and chlorides.

## APPARATUS

The different mixtures were each heated in a hand glass test tube 32-35 cm. long to which a side arm had been sealed about 10 cm. from the open end. This side arm was connected to an invèrted two and a half liter bottle by means of a glass tube which extended through a two-hole rubber stopper in the bottle to near the bottom of the bottle. A second inverted bottle was connected to the first by means of an inverted U tube, the end of one arm of which was bent up and passed through the second hole in the first bottle and the other end of which bent up in same way, extended through one of the holes in the stopper in the second bottle. The second bottle was necessary in order to keep the water from scattering widely when the spurt of oxygen came and also gave a means of collecting and measuring the water displaced as it drained from the second hole in the stopper. The first bottle was filled with water up to near the bottom of it.

Through the one-hole stopper in the test tube was inserted a 360° or 550° mercury thermometer. The test btube was then insebted in a furnace which was electrically heated

and controlled. In the bottom of the furnace was placed
a vessel containing metals of low melting points which when
the furnace was operating became molten and helped keep the
temperature from fluctuating.

# EXPERIMENTAL

## With Manganese Dioxide as a Catalyst

The potassium chlorate was mixed with the manganese dioxide (both having been dried, pulverized, and sifted through a 200 mesh sieve) in the following molecular proportions: $KClO_3$: $Mn O_2$ :: 1 : 4, 1:2, 1:1, 2:1, 4:1, 6:1, 8:1, 16:1, 32:1, and 64:1. The mixing was accomplished by sifting the weighed amounts of the two together through a 200 mesh sieve, and then twice through a 150 mesh sieve. The resulting mixture appeared to be very uniform and homogeneous. Portions of these mixtures were then placed in the hand glass text tube, such that each portion contained 2 g. of potassium chlorate and its proportionate amount of catalyst. The test btube was inserted into the furnace which had previously been heated a few degrees hotter than the temperature at which it was thought the mixture would spontaneously decompose. Wathhing the rate of flow of water out of the second bottle and the rise of the mercury in the thermomete r inserted in the mixture, the temperature was recorded when the mercury suddenly started to rise very much faster than before. This happened either coincident with or just after the water spurted up in the second bottle. The rate of

evolution of oxygen from the potassium chlorate could also
be followed by measuring the amount of  water flowing out
of the second bottle at definite intervals.  The experiment
was repeated with each mixture at least three times and the
average of the temperatures obtained was recorded  as the
spontaneous decomposition temperature of that mixture.
The following is a  representative  table of data:

### TABLE I.

| Mixture taken; $2.KClO_3$: 1 $MnO_2$ (2.0793 g of mixture) | | | |
|---|---|---|---|
| Time<br>June 30 | Temperature<br>Mixture(Therm.#5) | Furnace (Therm.#3) | $H_2O$ displaced<br>and remarks |
| 3:06 P.M. | 96 | 320 | |
| 3:10 | 205 | 310 | 47 cc    white |
| 3:12 | 252 | 308 | 48  cc  fumes |
| 3:14 | 283 | 306 | 70 cc occurred |
| 3:15½ | 297 Temp, rose<br>rapidly | 308 | 475 cc in bottle |

The temperatures thus obtained with the percent  of
$KClO_3$ for each of the mixtures in  the various experiments are
as  follows:
1 $KClO_3$ to 4 $MnO_2$ or 20%;325°, 325°,  327° with average of 326°.
1 $KClO_3$ to 2 $MnO_2$ or 33-1/3%;  314°, 300°,  300°, 308°, 308°,

  314° with average of 307°.

1 $KClO_3$ to 1 $MnO_2$ or 50%: 295°, 295°, 293° -average 294°.

2 $KClO_3$ to 1 $MnO_2$ or 66 ½%: 277 9  292 9  293 9 298 9 313 9 301 9

  302 9 293 9 297 9 321 9 289 9 294 9 average 297 °

4 $KClO_3$ to 1 $MnO_2$ or 80%: $304^O$, $302^O$, $303^O$, average $303^O$.

6 $KClO_3$ to 1 $MnO_2$ or 85.7%: $318^O$, $315^O$, $316^O$, $317^O$, average

$317^O$.

8 $KClO_3$ to 1 $MnO_2$ or 88.8%: $330^O$, $333^O$, $336^O$, average $333^O$.

16 $KClO_3$ to 1 $MnO_2$ or 94.1%: $350^O$, $350^O$ $352^O$, average $351^O$.

32 $KClO_3$ to 1 $MnO_2$ or 96.9%: $383^O$, $383^O$, $383^O$, $378^O$, $378^O$,
average $381^O$.

64 $KClO_3$ to $MnO_2$ or 98.4%: no sudden rise in mercury nor

sudden spurt of water when heated above $420^O$ in two

different trials.

The average of all these results are tabulated

in Table II.

| Mols $KClO_3$ | Mols $MnO_2$ | Molal % $KClO_3$ | Temperatures |
|---|---|---|---|
| 1 | 4 | 20 | 326 |
| 1 | 2 | 33 | 307 |
| 1 | 1 | 50 | 294 |
| 2 | 1 | 67 | 297 |
| 4 | 1 | 80 | 303 |
| 6 | 1 | 86 | 317 |
| 8 | 1 | 89 | 333 |
| 16 | 1 | 94 | 351 |
| 32 | 1 | 97 | 381 |
| 64 | 1 | 98 | |

The temperatures were plotted against the molal

percentage compositions as shown in Figure 1.

Figure I.

Temperature of Decomposition

A — — $KClO_3 - MnO_2$ (alone)  Curve
B — — $KClO_3 - Fe_2O_3$ (alone)  Curve
C — — $KClO_3 - MnO_2 + Fe_2O_3$ (Combined weight equal to $MnO_2$ in A) Curve

Molar Percent Potassium Chlorate
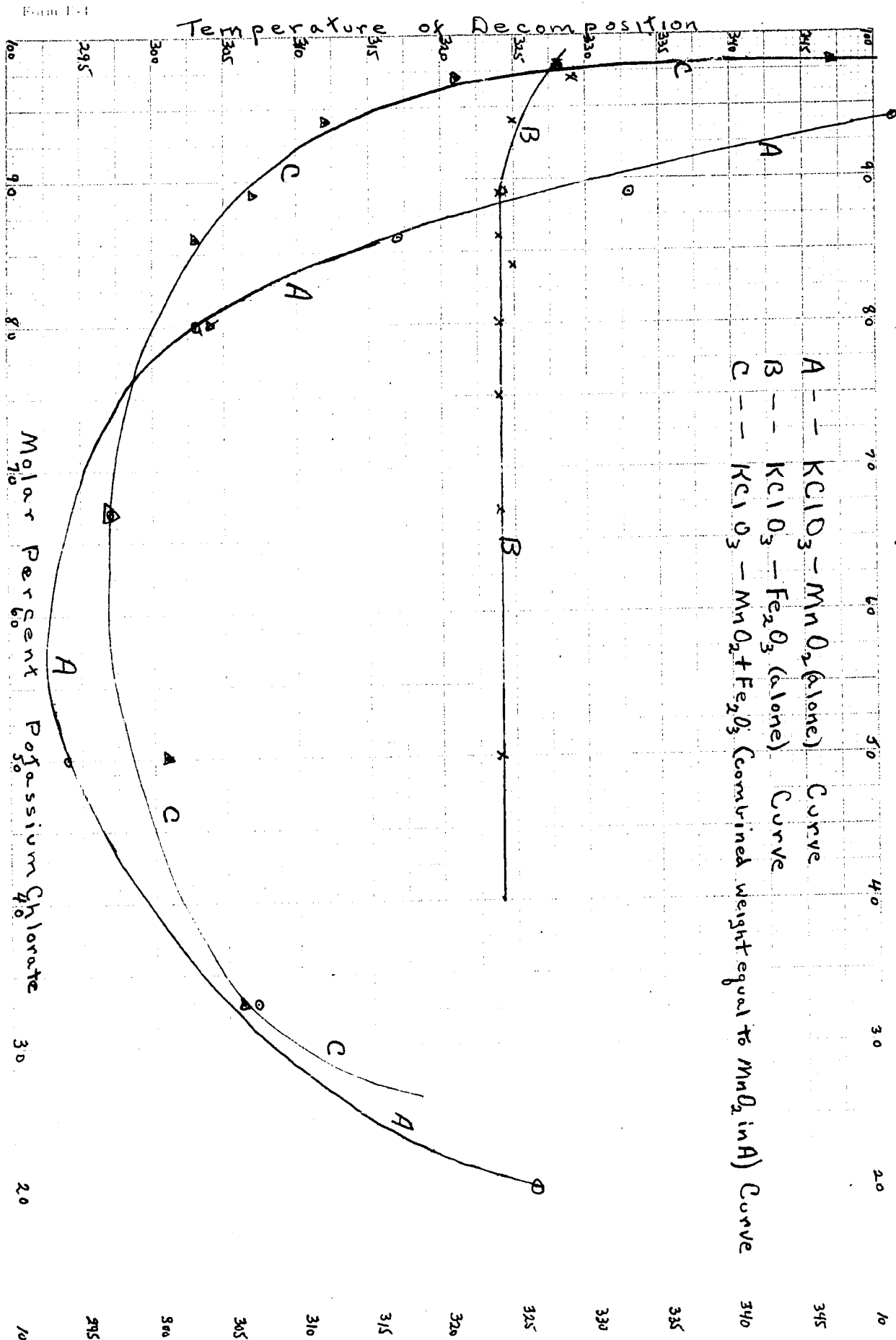
## With Ferric Oxide as a Catalyst

Ferric oxide was used with potassium chlorate in the same way as the manganese dioxide, the same care being taken in getting intimate mixtures. The following molal proportions were used: $KClO_3$ :$FE_2O_3$ :: 1:8, 1:6, 1:5, 1:4, 1:2, 1:1, 2:1, 4:1, 5:1, 6:1, 8:1, 16:1, 32:1, 64:1. Table III shows the data for a representative experiment.

TABLE III

| Mixture taken :$8KClO_3$:$1FE_2O_3$ | | (2.3257 g of mixture) | |
|---|---|---|---|
| Time | Temperature | | $H_2O$ displaced; |
| June 20 | Mixture (Therm.#11) | Furnace (Therm.#3) | remarks |
| 4:28 | 100 | 335 | |
| 4:30 | 175 | 300 | 15 cc |
| 4:32 | 239 | 311 | 9 cc |
| 4:34 | 284 | 323 | 24 cc |
| 4:36 | 308 | 330 | 30 cc |
| 4:38 | 318 | 332 | 40 cc |
| 4:38¼ | 323 temp. rose rapidly | | 520 cc big spurt of water |

The following are the results obtained for each of the mixtures in the different trials:

In the mixtures containing 1 Mol $KClO_3$ with 8,
6, 5, 4, 3, or 2 Mols of $Fe_2O_3$ no sudden rise in the temperature nor sudden spurting of the water was obtained up to temperatures above $350^{\circ}$.

1 $KClO_3$: 1 $Fe_2O_3$ - 50%: $323^{\circ}$, $325^{\circ}$, average $324^{\circ}$.

2 $KClO_3$: 1 $Fe_2O_3$ - 67%: $327^{\circ}$, $323^{\circ}$, $322^{\circ}$, $322^{\circ}$, average $324^{\circ}$.

3 $KClO_3$: 1 $Fe_2O_3$ - 75%: $322^{\circ}$, $323^{\circ}$, $326^{\circ}$, $322^{\circ}$, average $324\frac{1}{2}^{\circ}$.

4 $KClO_3$: 1 $Fe_2O_3$ - 80%: $323^{\circ}$, $324^{\circ}$, $326^{\circ}$, average $324^{\circ}$.

5 $KClO_3$: 1 $Fe_2O_3$ - 84%: $324^{\circ}$, $326^{\circ}$, $324^{\circ}$, average $324\frac{1}{2}^{\circ}$.

6 $KClO_3$: 1 $Fe_2O_3$ - 86%: $323^{\circ}$, $323^{\circ}$, $321^{\circ}$, $325^{\circ}$, $322^{\circ}$, average $323^{\circ}$.

8 $KClO_3$:1 $Fe_2O_3$ - 89%: $324^{\circ}$, $324^{\circ}$, $325^{\circ}$, $323^{\circ}$ $326^{\circ}$ $323^{\circ}$ $324^{\circ}$, average $324^{\circ}$.

16 $KClO_3$:1 $Fe_2O_3$ - 94%: $324^{\circ}$, $327^{\circ}$, $323^{\circ}$, average $325^{\circ}$.

32 $KClO_3$:1 $Fe_2O_3$ - 97%: $332^{\circ}$, $326^{\circ}$ $326^{\circ}$, average $329^{\circ}$.

64 $KClO_3$:1 $Fe_2O_3$ - 98%: $328^{\circ}$, $328^{\circ}$ $327\frac{1}{2}^{\circ}$, average $328^{\circ}$.

128 $KClO_3$:1 $Fe_2O_3$ no sudden rise in temperature up to $420^{\circ}$.

These temperature averages were plotted against their corresponding molal percentages as shown in Figure 1.

## With a Mixture of Manganese Dioxide
## and Ferric Oxide as a Catalyst.

The finely powdered ferric oxide was intimately mixed with an equal weight of manganese dioxide in the manner described before, obtaining a very homogeneous appearing mixture. Separate portions of this mixture were then mixed with potassium chlorate in exactly the same way as had been done in the case of the manganese dioxide alone. Thus in making up the 1:1 combination 12 g of $KClO_3$ were mixed with 8.5114 g of this $Fe_2O_3$ - $MnO_2$ mixture, just as for the 1:1 combination before 12 g of $KClO_3$ had been mixed with 8.5114 g of $MnO_2$ alone. Experiments were run with the following combinations in exactly the same way as in the previous cases: $KClO_3$ : catalyst (assuming all the weight of the mixture to be $MnO_2$) ::1:4, 1:2, 1:1, 2:1, 4:1, 6:1, 8:1, 16:1, 32:1, 64:1.

The results obtained in the various treats for these mixtures are as given below:

1 $KClO_3$ : 4 catalyst - 20%:no spontaneous decomposition up to 358°.

1 $KClO_3$:2 catalyst - 33%: 290°, 319°, 319°, 297°, average 306°.

1 $KClO_3$:1 catalyst - 50%: 290°, 292°, 314°, 322°, 297°,

average 301°.

2 $KClO_3$:1 catalyst - 67%: 306°, 308°, 297°, 296°, 290°,

Average 297°.

4 $KClO_3$:1 catalyst - 80%:304°, 305°, 294°, 310°, 308°,

average 304°.

6 $KClO_3$:1 catalyst - 86%:298°, 310°, 300°, average 303°.

8 $KClO_3$:1 catalyst - 89%: 305°, 309°, 308°, average 307°.

16 $KClO_3$:1 " - 94%: 318°, 297° , 315°, 317°, average

312°.

32 $KClO_3$:1 " - 97%: 321°, 318°, 324°, average 321°.

64 $KClO_3$:1 " - 98%: 345°, 348°, 350°, average 348°.

These averages were likewise plotted against the
percent composition, which is also shown in Figure 1. Table
IV shows the data for a typical experiment which gave no
spontaneous decomposition temperature.

TABLE IV

| Mixture 1 KClO$_3$:4(50:50% each of MnO$_2$ and Fe$_2$O$_3$)(7.6743 taken) | | | | |
|---|---|---|---|---|
| Time | Temperature | | H$_2$O displaced | |
| July 8 | Mixture (Therm.#5) | Furnace (Therm.#3) | | Remarks |
| 4:58 PM | 80 | 350 | | |
| 5:02 | 175 | 347 | 64 | |
| 5:06 | 277 | 342 | 150 | |
| 5:08 | 301 | 339 | 86 | |
| 5:10 | 312 | 336 more current | 76 | no sudden |
| 5:12 | 318 | 335 | 50 | rise in temp. or |
| 5:14 | 325 | 337 | 115 | big spurt of |
| 5:16 | above 342 | 352 | 150 | water |

Table V permits a comparison of the results of the 3 sets of experiments one with the others.

TABLE V - Spontaneous Decomposition Temperatures

| % KClO$_3$ | MnO$_2$ alone | Fe$_2$O$_3$ alone | MnO$_2$ Fe$_2$O$_3$ |
|---|---|---|---|
| 20 | 326 | --- | --- |
| 33 | 307 | --- | 306 |
| 50 | 294 | 324 | 301 |
| 67 | 297 | 324 | 297 |
| 80 | 303 | 324 | 304 |
| 86 | 317 | 324 | 303 |
| 89 | 333 | 324 | 307 |
| 94 | 351 | 325 | 312 |
| 97 | 381 | 329 | 321 |
| 98 | --- | 328 | 347 |

The results in the final set of experiments where the $MnO_2$ - $Fe_2O_3$ mixture was used, were then tabulated, on the basis of the $MnO_2$ content and compared with the results of the experiments using $MnO_2$ alone. Table VI brings out this comparison.

TABLE VI

| % $KClO_3$ | Temperatures with $MnO_2$ alone | Temperatures with $Fe_2O_3$ and same % $MnO_2$ |
|---|---|---|
| 20 | 326 | |
| 33 | 307 | |
| 50 | 294 | 306 |
| 67 | 297 | 301 |
| 80 | 303 | 297 |
| 86 | 317 | |
| 89 | 333 | 304 |
| 94 | 351 | 307 |
| 97 | 381 | 312 |
| 98 | --- | 321 |
| 99 | --- | 347 |

Table VII brings out the same comparison between the use of $Fe_2O_3$ alone and the use of an equal amount of $Fe_2O_3$ accompanied by $MnO_2$.

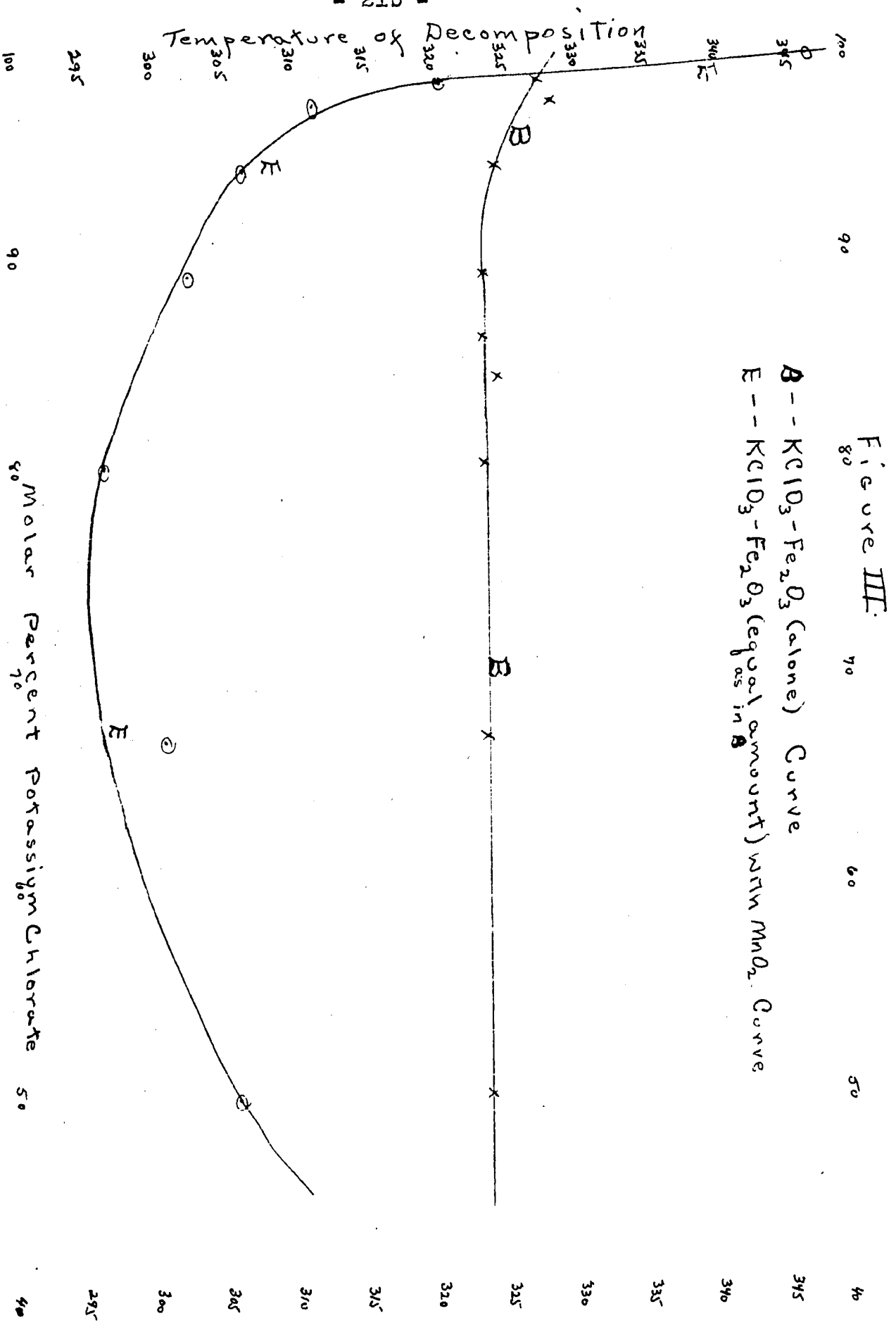| % $KClO_3$ | Temperatures with $Fe_2O_3$ alone | Temperatures with $MnO_2$ and some % $Fe_2O_3$ |
|---|---|---|
| 20 | --- | --- |
| 33 | --- | --- |
| 50 | 324 | 306 |
| 67 | 324 | 301 |
| 80 | 324 | 297 |
| 89 | 324 | 304 |
| 94 | 325 | 307 |
| 97 | 329 | 312 |
| 98 | 328 | 321 |
| 99 | --- | 347 |

The data in the last two tables is plotted in Figures 2 and 3 respectively.

Figure II.

Temperatures of Decomposition.
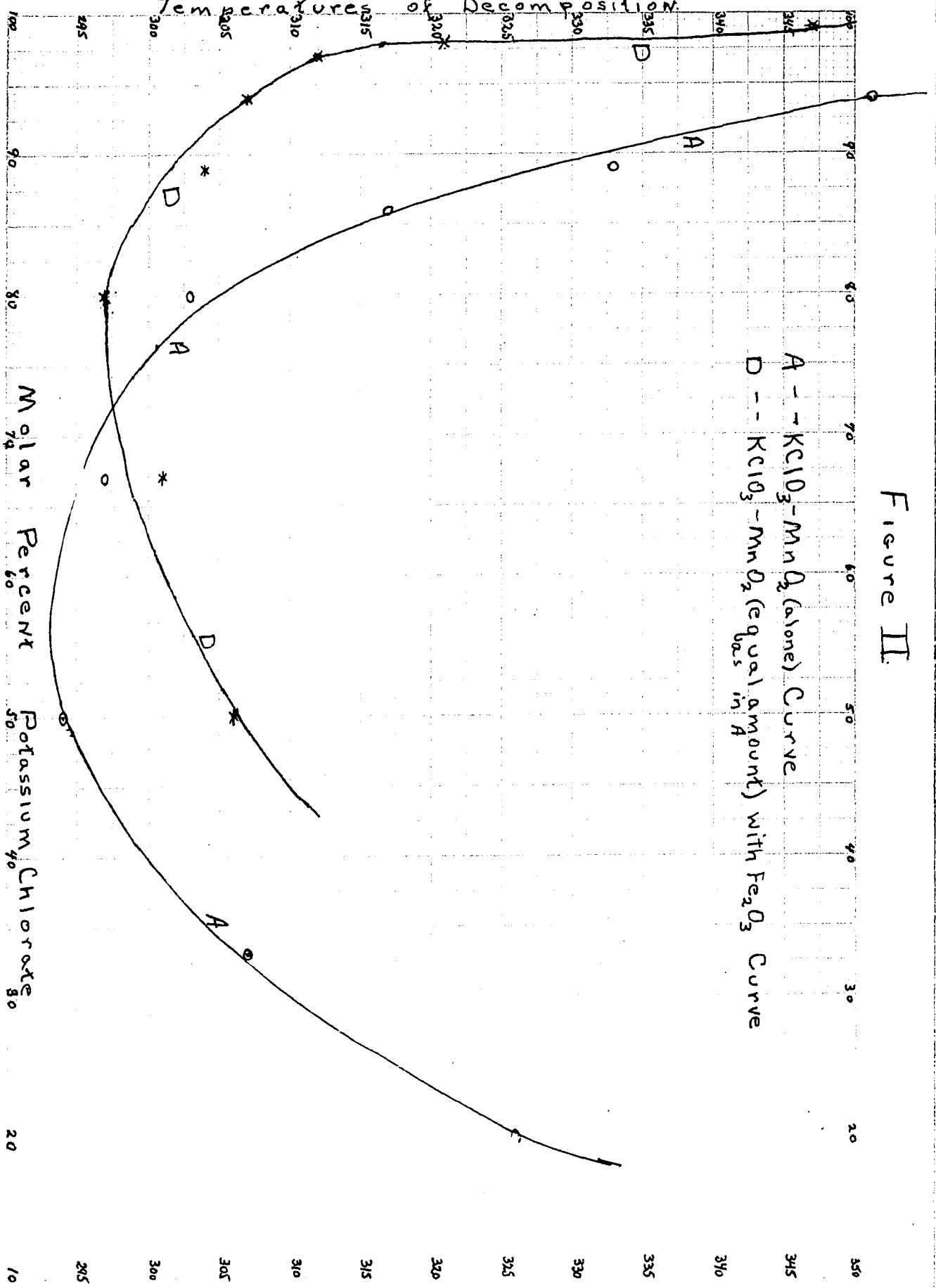
A -- KClO₃-MnO₂(alone) Curve
D -- KClO₃-MnO₂(equal amount) with Fe₂O₃ Curve

Molar Percent Potassium Chlorate

Figure III.

A -- $KClO_3$-$Fe_2O_3$ (alone) Curve

E -- $KClO_3$-$Fe_2O_3$ (equal amount) with $MnO_2$ Curve

Temperature of Decomposition

Molar Percent Potassium Chlorate

## DISCUSSION OF RESULTS

The data given strengthens the conclusions which the authors established in their previous paper (1) viz: that the spontaneous decomposition temperature of mixtures of potassium chlorate and manganese dioxide depends upon the percentage composition of the mixture and that those containing too much manganese dioxide as well as those containing too little manganese dioxide suffer no spontaneous decomposition. This is believed to be due in the former case to the fact that the heat given off by the potassium chlorate and which would otherwise be utilized in increasing the rate of its decomposition is not sufficient to do this as well as to increase the temperature of the large amount of inert material which is present. In the latter case no spontaneous decomposition is produced due to the fact that much of the potassium chlorate is not in contact with the catalyst since too little of it is present.

In general it is noted that ferric oxide is not as good a catalyst as manga nese dioxide although at some of the concentrations in which very little catalyst is present, the ferric oxide gives a spontaneous decomposition

temperature lower than manganese dioxide. This is very probably due to the fact that the ferric oxide was in a very much finer state of division than the manganese dioxide since much of it was practically colloidal as was stated in its method of preparation. This gave an enormous surface and therefore the small amount of it could be in contact with a large amount of potassium chlorate and thus catalyze it. This work as well as that of Brown and White (3) establishes the fact that for a wide variety of mixtures of potassium chlorate and ferric oxide there is a constant spontaneous decomposition temperature. The reason for this has not been thoroughly established.

The work of Neville has been confirmed in so far as establishing the fact that for some concentrations ferric oxide acts as a promoter with manganese dioxide. Nevertheless, it is evident from Figure 2 that for concentrations of manganese higher than about 25 percent this is not true. It is also evident that the promoter action is mutual since Figure 3 brings out the fact that manganese dioxide at certain concentrations of ferric oxide exerts a promoter action. In the light of the results which we have obtained it seems strange that Neville obtained no spontaneous decomposition at the temperature ($328^{o}$) at which

he worked whereas  we get very definite spontaneous decom-
position for a wide  range of mixtures, including the one he
used, at temperatures lower than his.  This may be accounted
for in  the  way in  which he mixed his ingredients.  The
method  used in the present  work is believe to give more
intimate and homogeneous mixtures  than by the other method.
It is  also believed  that our material contained less mois-
ture which may also account in part for Neville's results.

## SUMMARY

1. For a wide range of mixtures manganese dioxide produces a lowering of the decomposition temperature of potassium chlorate and the amount of heat produced by the decomposition of part of it is sufficient to cause spontaneous decomposition of the remainder, this spontaneous decomposition taking place at very definite temperatures for each mixture.

2. Ferric oxide produces spontaneous decomposition of potassium chlorate also over a wide range of mixtures.

3. The spontaneous decomposition temperature for ferric oxide mixtures is quite constant for quite a variety of mixtures.

4. Ferric oxide when added to some mixtures of potassium chlorate and manganese dioxide exerts a promoter affect but when added to other mixtures, high in manganese dioxide, has no such affect on the contrary it exerts a retarding effect upon the catalytic action of the manganese dioxide.

5. Manganese dioxide acts in a similar way as a promoter upon ferric oxide within a certain range of concentrations of ferric oxide.

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