LXII.—*Effect of Heat on Iodates and Bromates.*

I. *Potassic Iodate and Bromate.*

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IN order to compare the effects produced by heating bromates and iodates as well as to determine whether any other oxy-compounds are formed during the process, the experiments described in this paper were undertaken. It is intended to extend them to other salts.

The preliminary experiments soon showed that if trustworthy results were to be obtained the ordinary reagents sold as pure could not be relied upon. It, therefore, became necessary to specially purify each one before use; this was done in the usual way, but exceptional difficulties were experienced in the case of potassium iodide. Every commercial sample examined gave a separation of iodine on treating it with pure hydrochloric acid and allowing it to stand. Hitherto such a separation has been supposed to indicate the presence of iodate, but quite recently, since the commencement of these experiments, Robineau and Rollin have published (*J. Pharm.*, **26**, 485—490) a research on the "Detection of Iodates in Iodides," in which they state that "in presence of air and moisture all the acids, even carbonic anhydride, decompose potassium iodide, and that this salt is also decomposed when moist in presence of air and light," and "similar results are obtained with solutions of the salt, iodine being set free whilst the solution becomes alkaline." They, therefore, conclude that this test for the presence of iodates in the iodide is useless, and they propose the alternative one founded upon the immediate production of a colour with starch paste on the addition of hydrogen tartrate.

The immense importance of these statements to those who rely on potassium iodide as a reagent is obvious, and I have, therefore, made a series of experiments with the view of testing them.

Numerous attempts had been made to prepare a salt which would not give any separation of iodine when its solution was acidified with acid. The methods of purification adopted were:—

1. Repeated crystallisation from water of the better shaped crystals.
2. Fusion with charcoal powder and subsequent crystallisation from water.
3. Heating the solution of potassic iodide with the copper-zinc couple.
4. Crystallisation from absolute alcohol.

In no case, however, was a product obtained which failed to

become brown when its solution in water was acidified with hydrochloric or sulphuric acid.

These experiments extended over a considerable time, and it is quite improbable that a pure substance was not obtained in some of them; moreover, it is very difficult to believe that such a powerful reducing agent as the copper-zinc couple should not act on the small quantity of iodate present. It is, therefore, justifiable to say that the separation of iodine from a solution of potassium iodide on adding an acid and allowing the mixture to stand is no proof that the salt contains iodate.

One of the methods of analysis adopted depended on titrating the iodine with thiosulphate, and, therefore, it became important to overcome the difficulty caused by the separation of free iodine from the iodide itself. This was finally accomplished by setting a blank experiment going side by side with the original. In this way good results were obtained.

In some measure this peculiar behaviour of potassium iodide confirms the results of Robineau and Rollin, although my experiments were made before theirs. In order, however, to test the truth of their statements the following experiments were made.

Experiments with Potassium Iodide.

I. Some of the best defined crystals from a sample of potassium iodide, sold as pure, were recrystallised from water. A solution was then made of 1 part of the salt in 10 parts of water, care being taken to well boil the latter directly before use, so as to dispel any dissolved oxygen. This solution was then treated as follows:—

a. Starch paste and tartaric acid were added to the solution, but no *immediate* coloration was produced. Therefore, according to Robineau and Rollin, the sample was free from iodate.

b. Two test-tubes were partly filled with the solution, one was placed in the dark, and the other in the light. A gradually increasing colour due to separated iodine was noticed in the one exposed to light, whereas the one in the dark gave no colour after three days. Both tubes were freely open to the air.

c. Ten test-tubes were now partly filled with the iodide solution, and a small quantity of solutions of the following acids added, the water used having been, as before, well boiled just before use and allowed to cool; hydrochloric (diluted pure), nitric (diluted pure), sulphuric (diluted pure), acetic (diluted pure), phosphoric, oxalic, tartaric, citric, salicylic, and arsenious. The whole of the solutions were very weak and the mixing was done in the cold. In every case, except with tartaric, oxalic and arsenious acids an *immediate* separation of iodine was observed. In the case of tartaric and

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oxalic acids, a colour was produced after about 4 or 5 minutes, but no production of colour was noticed in the arsenious acid even after 24 hours standing.

d. Two tubes of the same size were about half filled with the iodide solution, and into one of them well-washed carbon dioxide was passed, so as to displace the air above the liquid by this gas. Both tubes were now corked and placed side by side in the dark for 24 hours; after this time, a distinct brown coloration was noticed in the carbon dioxide tube, but not in the other.

These results would, therefore, seem to confirm those of Robineau and Rollin, but remembering previous experience, it was decided to repeat them on specially purified material. A quantity of potassium iodide was, therefore, crystallised from absolute alcohol, and the tests repeated with the material thus obtained.

a. Tartaric acid did not liberate any iodine from the solution even after standing for 24 hours.

b. The solution of the salt in water freely exposed to the air, and a powerful light did not give up iodine.

c. The following acids produced a separation of iodine from the solution after a minute or two, namely, hydrochloric, sulphuric, acetic, oxalic. Solution of arsenious oxide in water produced no Other acids were not tried.

d. Carbon dioxide failed to separate any iodine even after standing in contact with the solution freely exposed to the light for several weeks.

These experiments, therefore, show :—

1. That exposure of a solution of ordinary purified potassium iodide to air and light produces a separation of iodine. But if the iodide has been purified by crystallisation from absolute alcohol no separation of iodine can be produced in such a way.

2. That weak acids cause a separation of iodine from solutions of potassic iodide. If the iodide has been prepared by crystallisation from absolute alcohol, tartaric acid will not do this.

3. That carbon dioxide will not separate iodine from a solution of potassium iodide which has been purified by crystallisation from absolute alcohol.

Assuming then that potassic iodide, prepared by crystallisation from its solutions in absolute alcohol is pure, the solution in water of such pure iodide is not coloured by exposure to light, or by carbon dioxide, or by tartaric acid solution.

Analysis of the Iodate and Bromate.

The determination of the halogen in the oxy-salt was done in one of three ways.

- (a.) By digestion with potassic iodide and hydrochloric acid.
- (b.) By reduction with sulphurous acid.
- (c.) By reduction with the copper-zinc couple.

The first two methods have been employed before, but, so far as I am aware, the last process has not, although Thorpe proposed it for use in the analysis of chlorates.

In the course of this work, a considerable number of determinations have been made by each method. The digestion with potassium iodide and hydrochloric acid was done in the usual way in stoppered bottles, but a blank experiment with potassium iodide and hydrochloric acid was made side by side with the main one, as otherwise good results could not be obtained. Care must be taken not to remove the stoppers until the bottles and their contents are quite cold. Results which agreed with each other within 0.1 per cent. were constantly obtained.

The reduction by sulphurous acid was effected by passing the washed gas through the solution until a colourless or (usually) slightly brown solution was produced, adding nitric acid and then silver nitrate.

In reference to the reduction by the copper-zinc couple, Thorpe has shown that it reduces potassium chlorate completely (*Chem. Soc. J.*, 1876, i, 856), and Gladstone and Tribe have investigated the action with a view to discover if any intermediate compounds are produced (*ibid.*, 1878, 147). So far as I am aware, however, the action of the couple on iodic and bromic acids has not been tried. Naturally it would be supposed that these compounds would be reduced in a similar manner to the chlorates. It was first necessary to ascertain this, and in order to do so a series of experiments were undertaken. I quote the first two of these as typical of the whole.

Impure Sample of Potassic Iodate.—The salt was dissolved in water, and poured on to a recently prepared couple. The mixture was gradually heated and kept boiling for 10 minutes, after which it was filtered, and the iodide precipitated with silver nitrate. Three determinations gave the following results:—

- (1.) 0.5000 gave 0.5646 AgI; I = 61.02.
- (2.) 1.0000 gave 1.1410 AgI; I = 61.66.
- (3.) 0.5000 gave 0.5720 AgI; I = 61.82.

The mean of the percentages is 61.50, whilst the mean found by the digestion process was 61.52.

Potassic Bromate.—The same process was adopted as in the case of the iodate.

- (1.) 0.5000 gave 0.5570 AgBr; Br = 47.40.
- (2.) 0.5000 gave 0.5560 AgBr; Br = 47.32.

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The mean of the percentages is 47·36, whilst the mean found by the digestion process was 47·40.

There can therefore be no doubt that the oxy-salts are completely reduced by the action of the couple.

The mode of employing the method is very simple. The couples are prepared according to the directions of Gladstone and Tribe (*ibid.*, i, 1877, p. 561), and the solution of the oxy-salt poured on. The mixture is then very gradually warmed for half an hour, and finally boiled for the same length of time, the solution filtered, the residue well washed, the washings added to the filtrate, and the whole precipitated with silver nitrate. Fresh couples must be prepared for each experiment.

All three methods give excellent results if proper precautions be taken. The digestion process with potassium iodide is untrustworthy unless a blank experiment, in which the same quantities of iodide and acid are used, is carried on side by side with the main determination. In fact, remembering the peculiar behaviour of potassium iodide, it is difficult to see how accurate results could have been obtained without doing this.

Effect of Heating Potassic Iodate.

This action has been studied by Gay-Lussac, Rammelsberg, and Schönbein, and the results they arrived at are thus summarised in Muir and Morley's edition of *Watts's Dictionary*. "Decomposes at higher temperatures than KClO_3 into KI and O without production of KIO_4 ."

In the latest edition of *Watts's Dictionary*, 1882, the action is more particularly described, thus:—"At a low red heat, the salt melts and froths up, and is converted, with loss of 22·59 per cent. of oxygen, into KI . If only part of the O is expelled, the residue consists of iodide and iodate, without any periodate."

Repeated crystallisation from water failed to give a salt of sufficient purity for experiment. The plan finally adopted was therefore to crystallise the commercial sample from water, then to redissolve the salt in a convenient amount of water, and add to the solution a little pure alcohol. An immediate precipitate is produced; this is collected, washed with a little water, and then dissolved in a larger quantity of the same liquid; on careful evaporation, crystals of pure potassium iodate separate. The salt thus prepared forms nodular masses of minute cubical crystals, the crystals showing a tendency to agglomerate in masses in the form of a cross.

As an indication of the purity of the salt thus obtained, and therefore of that used in the following experiments, these analytical numbers were given on analysis.

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	I.	II.	Theory for KIO_3 .
Potassium	18.42	18.30	18.27
Iodine	59.21	59.21	59.28
Oxygen (by diff.) . .	22.37	22.49	22.43

The salt did not alter in weight when kept in the water oven at 100° for several hours.

Apparatus Employed.—The apparatus finally employed for the heating experiments was of a very simple character. Into the open end of a piece of hard combustion tube, a second piece of just sufficient size to fit tightly was inserted. This second tube thus formed a hollow stopper for the first. The end of the stopper tube which was inside the other was shaped into the form of an inverted cone with the apex inwards. This apex was open, as was also the other end of the tube; thus any gas produced in the first tube could pass freely into the air through the second. The stopper tube was loosely filled with glass wool, and this was moistened with a solution of potassium iodide, the object being to arrest any iodine or bromine which

might be given off during the heating. The inversion of the bottom end of the tube prevented any liquid dropping into the first tube by allowing a space for it to collect in around the bottom of the cone. The heating tube was inclined, and a screen shut off the heat from its upper portion, so that it did not reach the second tube. The disposition of the apparatus is shown in the sketch.

Change in Appearance on Heating.—The effect of heat on potassium iodate is very well marked. No change whatever takes place in the

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appearance of the salt at 100° , neither does it lose weight even if kept at this temperature for several hours. At about 120° , the salt begins to assume a brown tint, and at 160° it has become quite brown, the colour increasing in depth up to its melting point (560° , Carnelly). Just as the substance begins to fuse, iodine vapour is observed to be given off in very small quantity, and the substance becomes white. During the fusion no iodine is given off. Fusion goes on quietly for some time, but at last the whole mass becomes solid. The temperature must now be raised very considerably to fuse the remainder. The formation of this solid mass shows the decomposition of the iodate to be complete, as no further loss of weight occurs even on prolonged heating.

Testing the Brown Compound.—The first thing necessary was to ascertain what was the cause of the production of the brown colour. Samples were therefore taken and carefully heated (1) up to 160° ; (2) up to 300° ; and (3) in boiling sulphur, that is up to 449° (Regnault). On cooling, and examining the product, it was noticed that the depth of colour increased with the temperature, but on weighing it was found that no loss of weight whatever had taken place, and no deposit of iodine could be observed on the sides of the tube. On dissolving the salt in water, a *colourless* and *neutral* solution was obtained, thereby showing that no free iodine had been produced. Silver nitrate gave a *white* precipitate, soluble in nitric acid, thus proving the absence of periodate, and also of iodide. The latter was also proved to be absent by the non-production of colour on the addition of dilute hydrochloric or sulphuric acid.

That the brown compound was formed without any alteration in composition of the potassium iodate taking place was proved by the following experiment. Two quantities of pure potassium iodate, of 0.3 gram each, were heated for five hours in a bath of boiling sulphur. The substance became quite brown, but no change in weight took place, and on estimating the iodine precisely the same percentage was found as in the original substance. It would thus appear that the brown compound is caused by some intra-molecular change of the ordinary iodate without any decomposition accompanied by loss or gain in weight taking place. It may be that the brown colour is caused by a separation of the iodine, which is afterwards given off on fusion of the salt, this iodine being held by the crystalline structure of the salt in such a way that it cannot escape, until fusion breaks up this structure and sets it free. When the heated salt is dissolved in water, the iodine recombines with the potassium and oxygen from which it was separated, reforming iodate.

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Complete Decomposition of Potassic Iodate.

Quantities of the pure salt in a finely divided state were weighed out in the combustion tubes, and carefully heated until decomposition had entirely ceased. This usually occupied about four hours, care being taken to regulate the heat so as to prevent any sudden increase in temperature. When decomposition was complete, the combustion tubes were allowed to cool and then weighed. The contents of the stopper tubes were washed out with a little potassium iodide solution, and the amount of the iodine estimated by standard thiosulphate. The following were the results obtained.

	KIO ₃ taken.	Residue.	Per cent. loss.	Iodine evolved.	Oxygen evolved.
	grams	grams		per cent.	per cent.
1	0·5000	0·3850	23·00	—	—
2	0·7500	0·5800	22·66	0·305	22·355
3	1·000	0·7710	22·90	0·308	22·592
4	1·0018	0·7708	23·05	0·310	22·740
5	1·000	0·7700	23·00	0·306	22·694
6	1·000	0·7720	22·80	0·310	22·490
7	0·500	0·3850	23·00	0·309	22·691
8	0·500	0·3860	22·80	—	—

	Total loss.	Iodine.	Oxygen.
	per cent.	per cent.	per cent.
Maximum differences.....	0·390	0·005	0·385
Mean results	22·901	0·308	22·593

Analysis of the Residue.—The substance remaining in the tubes after the heating was found to be easily soluble in water, forming a neutral solution. No separation of iodine took place on the addition of dilute acid, thereby proving the absence of iodate. The iodine was estimated as silver iodide.

Weight of residue.	Amount of iodine found.	Percentage.	Theory for KI.
0·7708	0·5876	76·24	76·50
0·7700	0·5880	76·32	76·50

The residue is therefore pure KI.

It has been proved, therefore, that when pure potassic iodate is heated until it is completely decomposed, it loses 22·59 per cent. of oxygen, and 0·308 per cent. of iodine, leaving a residue of potassic iodide.

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Partial Decomposition of Potassic Iodate by Heat.

The total decomposition having been studied, and no other oxy-compounds of iodine having been formed, it was thought that perhaps these might be produced if the process was stopped before all the oxygen was evolved. In order to test this, pure iodate was heated very carefully, the heating being discontinued after varying amounts of oxygen had been evolved. In the first case, a loss of 4.5 per cent. had taken place; in the second, of 6.7; and in third, of 15.6. These numbers were quite fortuitous, and in no sense indicate that any change whatever could be noticed at these particular stages of the decomposition. The analysis of the residue presented unexpected difficulties, as it was found that very great care indeed had to be taken in order that the silver iodate, which was precipitated on the addition of silver nitrate to the solution of the mixed iodate and iodide, should be dissolved in nitric acid. This only takes place with difficulty, especially when the proportion of silver iodate to silver iodide is large; in fact, several trials proving unsatisfactory, the method was abandoned, and the following substituted in its place. The residue, after heating, was dissolved in water, and the solution made up to a known volume. A certain proportion was then taken, hydrochloric acid and solution of potassium iodide added, and the mixture digested on the water-bath for three or four hours. The iodine thus set free was estimated by standard thiosulphate, and the amount of potassium iodate in the residue was then calculated, a blank experiment with potassium iodide and hydrochloric acid being carried on side by side. The iodide was estimated by adding an excess of acetic acid, and digesting, dissolving the liberated iodine in carbon bisulphide, washing, and titrating with thiosulphate. The reason why this process is possible is that 1 mol. of potassium iodate reacts with 5 mols. of iodide (roughly, 1 part by weight of KIO_3 to 4 of KI), and that therefore so long as the amount of iodide does not exceed four times that of the iodate, there will be a decomposition of the whole of the iodide, but not of the iodate. Five-sixths of the iodine liberated will be derived from the iodide, and hence the amount of the latter present may be calculated. The following results were thus obtained.

- I. 1 gram of KIO_3 , heated until it fused, lost 4.5 per cent. The residue contained an amount of KIO_3 equal to 82.87 per cent. of the original KIO_3 , and 11.78 per cent. of KI .
- II. 1 gram of KIO_3 heated until it lost 15.6 per cent. The residue contained KIO_3 , equal to 32.1 per cent. of the original, and 52.54 per cent. of KI .

	I.	II.
KIO ₃	82·87	32·16
KI	11·78	52·54
Loss on heating	4·50	15·60
	<hr/> 99·15	<hr/> 100·24

The close agreement of these numbers with 100 shows that no periodate is formed in the process, and this was still further confirmed by the production of a white precipitate on the addition of silver nitrate. The solution of the residue in water is quite colourless and neutral.

Action of Heat on Potassic Bromate.

Less difficulty was experienced in purifying this substance than was the case with the iodate. The pure salt was obtained after six crystallisations from water. It did not lose weight at 100°; as, however, it absorbs a little moisture (about 0·6 per cent.) very readily, it was dried before each heating. The heating was effected in the same apparatus as was used for the iodate. No change could be observed in the appearance of the salt until the temperature reached about 180°, when decrepitation began, notwithstanding that the substance had been finely powdered. These decrepitations continued until fusion, which took place at 434° (Carnelly). In order to see if any bromine was evolved, the glass wool was moistened with solution of potassic iodide; in this way it was shown that bromine is given off, and the amount was easily determined. Experiments made to ascertain at what stage of the process the bromine was evolved, showed that it *began when decrepitation began, and continued up to fusion, when it ceased*. On comparing this with what occurs in the case of the iodate, it will be observed that the latter commences to become *brown* at a somewhat lower temperature than that at which decrepitation commences with the bromate, but that *no iodine is evolved until the fusion begins*. This somewhat supports the idea that the brown colour of the iodate is due to liberated iodine, which is prevented from escaping by the crystalline structure of the salt until fusion breaks down the structure and allows it to escape. In the case of the bromate, the crystals break up before fusion, and allow the bromine to pass off.

The salt fuses to a colourless liquid, and oxygen is evolved in a gentle stream; after some time, however, the evolution gradually ceases and the mass becomes solid, indicating the complete decomposition of the bromate and formation of bromide. The following results were obtained.

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	KBrO ₃ taken.	Residue.	Per cent. loss.	Br evolved.	Oxygen evolved.
	grams	grams		per cent.	per cent.
1	0·7795	0·5500	29·44	—	—
2	1·0000	0·7050	29·50	—	—
3	1·0000	0·7050	29·50	0·0772	29·4228
4	1·0000	0·7039	29·61	0·0775	29·5325
5	1·0000	0·7020	29·80	0·0774	29·7226

	Total loss.	Bromine.	Oxygen.
	per cent.	per cent.	per cent.
Maximum differences	0·36	0·0003	0·2998
Mean results	29·57	0·07736	29·5593

Care is necessary in the heating to see that no spurting of the salt occurs during decrepitation, whereby some of it is projected up the tube and escapes decomposition. This was avoided by heating until fusion commenced, then allowing to cool, and again heating after brushing down any particles adhering to the sides.

Analysis of Residue.—The residue was white and easily soluble in water, forming a neutral solution which gave no coloration with acids (absence of oxy-compounds). Analysis gave the following numbers.

Weight of residue.	Amount of bromine found.	Percentage.	Theory for KBr.
0·7140	0·4784	67·003	67·23
0·7077	0·4736	66·921	67·23

The residue is therefore pure potassium bromide.

Thus when pure potassium bromate is heated until it is completely decomposed, it loses 29·56 per cent. of oxygen and 0·08 per cent. of bromine, and leaves a residue of potassic bromide.

Partial Decomposition of Potassic Bromate by Heat.

This was studied in the same way as in the case of the iodate, but the end of the stopper tube was connected with a delivery tube which was attached to a vessel standing in the pneumatic trough so that the rate of evolution of the oxygen could be watched. Heat was applied by a carefully-regulated burner or by immersing the tubes for a certain length of time in boiling sulphur. It was found, if the heat was *very* carefully applied so that the temperature rose *very* slowly, that after a certain amount of oxygen had been evolved the evolution slackened and eventually quite ceased. Attempts were now

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made to find if this break took place at any constant percentage of decomposition. The following results were obtained.

- I. 0.7795 gram heated until first evolution ceased; it lost 0.0203 gram = 2.60 per cent.
 II. 0.4921 gram lost 0.0131 gram = 2.66 per cent.
 III. 0.5082 „ „ 0.0142 „ = 2.79 „ „
 Mean, 2.68 per cent.

It would thus appear that a well-marked break takes place when about 2.7 per cent. of oxygen is evolved. Further experimenting, however, showed that this number depends on the amount of salt taken and the apparatus used. It is probably caused by the decomposition of the layer of bromate in contact with the heated surface of the glass, which, when decomposed, becomes bromide, through which the heat has to penetrate in order to reach the undecomposed salt inside. Thus varying numbers have been obtained with different tubes and quantities. The loss of the 2.7 per cent. therefore does not represent any definite reaction taking place throughout the mass; but in order to ascertain if any oxy-compounds were formed the residue was analysed. The bromate was estimated by digestion with potassic iodide and hydrochloric acid, and the bromide by precipitating with silver nitrate, adding nitric acid and filtering hot. In this case also, difficulty was experienced in dissolving the argentic bromate, and the method gives good results only with the greatest care. The silver bromate is, however, more soluble in hot nitric acid than the iodate, but on allowing the hot solution to stand, crystals of pure anhydrous silver bromate separate. The following results were obtained.

- I. 0.4921 gram of pure KBrO_3 was heated until it lost 0.0131 gram; the residue contained 91.59 per cent. of KBrO_3 and 5.60 per cent. of KBr .
 II. 0.5082 gram of KBrO_3 was heated until it lost 0.0142 gram; the residue contained 89.22 per cent. of KBrO_3 and 8.14 per cent. of KBr .

	I.	II.
KBrO_3	91.59	89.22
KBr	5.60	8.14
Loss	2.66	2.79
	<hr/> 99.85	<hr/> 100.15

There can therefore be no doubt that even when partially heated no other oxy-compounds are formed.

Heating under Reduced Pressure.

The non-formation of oxy-compounds in the previous experi-

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ments may, it was thought, be caused by the decomposition of the salts taking place under ordinary atmospheric pressure. Experiments were therefore undertaken in order to see if a reduction of pressure caused any change in the results previously recorded. For this purpose, the stopper tube was connected to an ordinary exhaust pump, which was kept rapidly working during the whole period of the heating. The pressure of course fluctuated as the decomposition of the salt varied, but by working the pump *rapidly* it was found possible to keep the changes within 20 mm. The lowest pressure obtained was about 30 mm. Under these conditions absolutely no difference could be detected from what took place when the pressure was normal. Analysis of the residues thus produced also gave the same results as before, and the decompositions took place at the same temperatures.

Decomposition of Potassic Chlorate.

It will be noticed that the iodate loses 0.3 per cent. of iodine and the bromate 0.08 per cent. of bromine on heating. It will also be seen that the iodine is given off as soon as the salt melts, and that the evolution lasts for a short time only. On the other hand, the bromine is evolved during the decrepitation of the salt. In order to test the behaviour of the chlorate under similar conditions, experiments were made with that substance; these showed very clearly, as did the others, the absolute necessity of having neither cork nor indiarubber in contact with the gas evolved; for in the first kinds of apparatus used no chlorine whatever could be obtained. As a final result of heating pure potassium chlorate until it ceased to lose weight it was found that 0.03 per cent. of chlorine was evolved. The whole of this was given off during the short time elapsing between the first melting of the salt and its complete melting. The salt did not decrepitate like the bromate. Thus the chlorate and iodate behave similarly in this respect whilst the bromate differs. This supports the explanation previously given of the cause of the change in colour produced in the iodate on heating, because it shows that the evolution takes place simultaneously with the breaking up of the physical structure of the solid, and the reason why bromine is not evolved at the same time is because, owing to the decrepitation of the salt, the element can pass off at an earlier stage than in the other cases.

The examination of other salts is in progress.

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