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# V.—*Lead Subiodide, and an Improved Method for Preparing Lead Suboxide. The Solubility of Lead Iodide.*

By Henry George Denham.

The existence in aqueous solutions of subvalent salts of lead has been demonstrated by the joint work of Denham and Allmand (T., 1908, **93**, 424), wherein it is shown that in the presence of platinised platinum hydrogen is capable of reducing bivalent salts of lead to a lower state of valency. Further support of this was obtained from the "circulation" experiments, in which it was shown that by maintaining a constant flow of an aqueous solution of lead acetate over a heated column of lead appreciable quantities of the metal could be dissolved and again precipitated on cooling the solution. These two results were summarised by the authors in the equations

$$Pb^{++} + H \rightleftharpoons Pb^{+} + H^{+}$$
$$Pb^{++} + Pb \rightleftharpoons 2Pb^{+}.$$

A further possible explanation has since been suggested by Pick and Ahrens (Abegg's "Handbuch," 4te Gruppe, Blei, 637), the mechanism of the second reaction being represented according to their view by the equation

$$Pb^{++} + Pb \rightleftharpoons Pb_2^{++}.$$

Further evidence as to the existence in aqueous solutions of the ions $Pb^{+}$ or $Pb_2^{++}$ has recently been furnished by the work of Bell (*Trans. Faraday Soc.*, 1916, **11**, 1, 79), who investigated the problem by comparing the weight of lead actually dissolved in various electrolytes during the passage of an electric current with that calculated from the electrochemical equivalent for bivalent lead. The discrepancy observed was found to substantiate the earlier work of Denham and Allmand.

The obvious method to obtain sub-salts of metals which give definite suboxides, for example, lead (Tanatar, *Zeitsch. anorg. Chem.*, 1901, **27**, 304; Brislee, T., 1908, **93**, 154), is to act on this suboxide with the necessary acid; but this method failed utterly owing to the following decomposition:

$$Pb_2O \longrightarrow Pb + PbO.$$

Other methods, such as the action of finely divided lead on the various solutions of lead salts, also gave negative results.

It has been shown that the vapour of methyl iodide acting on heated cupric oxide gives cuprous iodide without the liberation of iodine (Denham, *Zeitsch. anorg. Chem.*, 1911, **71**, 303). This sug-

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gested that the vapour of methyl iodide acting on lead suboxide might yield a lower iodide of lead, and although many unforeseen difficulties have been met with, this method has ultimately proved successful.

A preliminary experiment was carried out by distilling methyl iodide over lead suboxide at a temperature of 250—260°. After a distillation lasting about thirty minutes, the apparatus was cooled and the reaction tube examined. The substance in the tube was found to have changed from its original dark grey colour to a dark yellow with globules of lead scattered throughout the mass, but in the immediate neighbourhood of the glass walls a homogeneous, bright yellow band was clearly to be seen. The inner mass was extracted with hot water, and the filtrate gave, on cooling, a copious crop of lead iodide crystals, whilst the yellow band under similar treatment not only gave no such deposit, but the filtrate gave no trace of a precipitate with potassium chromate. This reaction, several times repeated, pointed to the desired reaction having proceeded with considerable evolution of heat—sufficient to raise all but the layer in contact with the relatively cool walls to a temperature at which the subiodide decomposed into a mixture of lead iodide and lead, the melting of the metal proving that the temperature had risen at least 70°. This supposition was at once tested by mixing the suboxide with four times its weight of silica, and it was found that the tendency to decomposition was by this means completely checked.

Before describing in detail the apparatus and method used in preparing the subiodide, the details of the method for obtaining pure samples of lead suboxide merit attention.

### *Methods of Preparation of Lead Suboxide.*

Two methods have been described for the preparation of this suboxide, namely, that of Glaser (*Zeitsch. anorg. Chem.*, 1903, **36**, 1) and that of Tanatar (*loc. cit.*), but the actual working details of their methods have been so sparingly given that it is wellnigh impossible to follow their work without repeating the whole of their experiments.

Glaser, for example, records that so long as the temperature does not exceed 235°, lead oxide is quantitatively reduced to the suboxide, and not to lead. The duration of this reduction is, however, not mentioned. On the other hand, the author has found that samples of lead oxide prepared by the decomposition of lead oxalate in a stream of air are reduced to lead at temperatures that do not exceed 220°. In an experiment at this temperature

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lasting 100 hours not the slightest sign of a halt in the neighbourhood of a composition approximating to that of lead suboxide was found. Glaser has pointed out in the case of copper oxide that the actual temperature of reduction depends on the previous history of the oxide, and it is highly probable that the samples of oxide used in this research possessed a finer grain than the specimens used by Glaser, hence the difference in the temperature of reduction. Obviously a method of preparation depending for its success on a knowledge of the particular temperature of reduction of each sample used is of little value as a method of preparation, and attention was then turned to that of Tanatar.

This author describes how he prepared the suboxide "by heating the lead oxalate in a combustion tube at the lowest possible temperature, carbon dioxide being led through the apparatus during the decomposition." It has required a considerable amount of work to rediscover the conditions that enabled Tanatar to obtain a product showing the properties of lead suboxide; under no conditions has the author been able to obtain a pure product when using a stream of carbon dioxide, for considerable traces of this gas were always tenaciously retained, possibly as subcarbonate. If nitrogen is used, according to Tanatar's suggestion, this objection is removed, but the inordinate length of time necessary for the decomposition—nearly a week at 300°—is a great disadvantage. Consequently, the author has been compelled to introduce various modifications in Tanatar's original method, and to lay down precisely those conditions that enable an independent worker to repeat his experiments.

In the first place, the dilution effect obtained by the passage of an inert gas was obtained by removing the products of decomposition (carbon monoxide and carbon dioxide) by an automatic three-fall Sprengel pump. Provided the total pressure does not exceed 5 cm., the carbon monoxide does not reduce the suboxide to lead. The main part of the decomposition was carried out at 270—275°, but when the pressure had fallen nearly to zero the temperature was steadily increased to a maximum of 335°. Under these conditions it is possible to convert the oxalate into suboxide in twenty-six hours. The product is a dark powder exhibiting all the properties ascribed to it by Tanatar. So far as the preparation of the suboxide itself is concerned, the author has been unable to find any reason why the temperature of decomposition should not rise as high as 375°. Even at this temperature no decomposition into lead and lead oxide occurs, but the product becomes distinctly paler in colour, and in this form is much less reactive, especially towards methyl iodide.

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Table I gives the composition of the last nine samples of lead suboxide prepared according to the method outlined above.

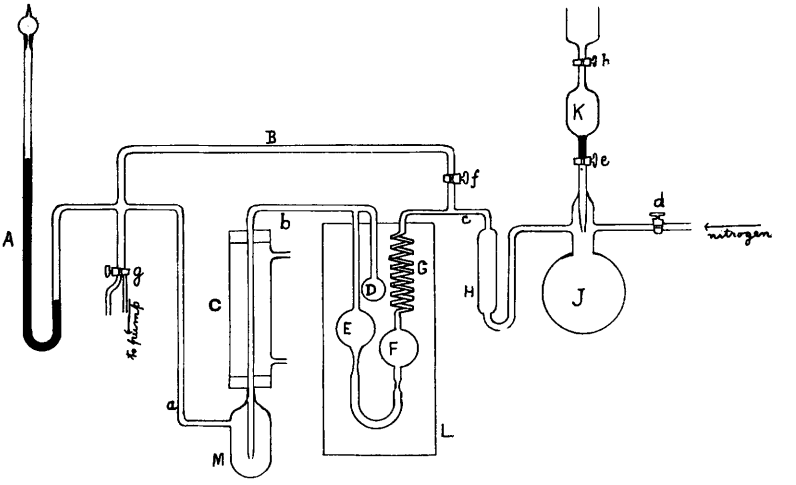
TABLE I.

| Experiment... | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|
| Lead per cent. (theory, 96·28) | 96·16 | 95·93 | 95·99 | 96·04 | 95·74 | 95·86 | 96·16 | 96·19 | 96·01 |

*Method of Preparation of the Subiodide.*

Although it is relatively easy to prepare samples of the subiodide approximating in composition to the theoretical, it has been found that the pure substance may be obtained only by the closest attention to the conditions stated below.

FIG. 1.




The apparatus found to be most satisfactory for the preparation of the subiodide is shown in Fig. 1.

*J* was a distillation flask into which methyl iodide could be introduced from *K* at the completion of the decomposition of the oxalate into suboxide; *H* was a tube containing phosphoric oxide; *G* a spiral of thin glass; *F*, *E* bulbs containing the reaction mixture of lead oxalate and silica; *D* a bulb containing lead oxalate capable of being sealed off at the conclusion of the decomposition of the oxalate into suboxide and separately analysed; *C* a water condenser; *M* a receiver into which the tube from the condenser projected to a considerable extent, so that after the first few c.c. of methyl iodide had been condensed the evolution of any gas

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could be noted; *A* a manometer; *B* a by-pass to enable the pump to remove evolved gases rapidly from both bulbs, thus ensuring that the manometer registered the pressure ruling throughout the whole apparatus—a very necessary precaution, for it may be noted that when more than two bulbs are used, owing to the difficulty of keeping down the pressure in the inner bulbs, reduction of the suboxide by the carbon monoxide ensues, and the inner bulbs give a variable product much richer in lead than the suboxide, and this occurs even although the gauge does not register a pressure above 5 cm. The taps *f*, *e*, *d* were protected with mercury seals, as the vapour of methyl iodide rapidly attacks the tap grease during the distillation; *g* was a three-way tap; *L* an electrically heated oven.

All temperatures were recorded on a platinum resistance thermometer, which was repeatedly checked against a standard.

In an experiment, about 0·5 gram of oxalate intimately mixed with four times its weight of silica was introduced into each of the bulbs *E* and *F*, a little glass-wool being first placed at the bottom of each bulb; and into the sample bulb *D* was brought about 0·4 gram of oxalate. The apparatus, after being sealed together as in the figure, was exhausted until the pump "hammered" and its tightness was tested. The electric oven was then steadily heated until a temperature of 270—275° was reached. At this stage a steady evolution of gas set in, and with three fall-tubes in use a pressure of 4—5 cm. was generally reached in an hour after the evolution of gas first set in. The decomposition then proceeded as already described.

When no further gas was evolved, the oven was cooled to 250°, and a slow stream of dry nitrogen freed from traces of oxygen by slow bubbling through alkaline pyrogallol and over red-hot copper was introduced until the pressure had risen to 75 cm. The bulb *D* containing the sample of suboxide was then sealed off; methyl iodide was introduced into *K*, oxygen thoroughly boiled out, and the tap *h* closed, a little mercury being also dropped into the cup. In this way the iodide could be easily introduced into the flask *J* without a trace of oxygen entering. By cutting out the by-pass *B*, the vapour of the iodide could be driven at any desired rate through the bulbs by suitably adjusting the temperature of the flask *J*. The most satisfactory rate of distillation proved to be such that 5 c.c. condensed in the receiver *M* in about twenty minutes. The total duration of the distillation was generally between forty-five and fifty-five minutes. In many cases the reaction, as shown by the evolution of gas in the receiver, was complete in fifteen minutes, but occasionally the suboxide appeared to

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react more slowly, and a longer distillation was necessary. After the distillation had begun the temperature of the oven was slowly raised to a maximum of 262°, and it was so arranged that this maximum temperature was maintained for the last twenty minutes of the distillation. After the completion of the distillation, the tap *f* was opened and the pump started, whilst the receiver *M* was cooled with a mixture of ether and carbon dioxide. When the pressure had fallen to 4 cm., the methyl iodide receiver was sealed off at *a* and *b*, likewise the apparatus to the right of the point *c*; at these points, it may be noted, constrictions had been made in setting up the apparatus. The object of reducing the pressure to 4 cm. before sealing off is that only when the pressure has fallen to this point is it possible to seal off any parts of the apparatus without the liberation of iodine. The pump was allowed to continue in action for twelve hours, the oven being kept at a temperature of 245°±5°. The bulbs were then sealed off and the contents were available for analysis.

The necessity for introducing nitrogen prior to the distillation arises from the fact that the methyl iodide reacts much more readily under atmospheric than under diminished pressure; for example, in two experiments a spiral condenser cooled by carbon dioxide and ether was used, and no nitrogen introduced into the apparatus. After one hour's distillation scarcely any reaction between the methyl iodide and the suboxide had occurred.

So far as the limits of the temperature are concerned, the reaction has been found to proceed too slowly below 250°, and at 264° a slight decomposition of the methyl iodide occurs with the liberation of iodine. The result is that at all temperatures above 263° there is a slow evolution of gas throughout the whole of the distillation, and an examination of the product shows that it always consists of a mixture of lead suboxide and considerable quantities of lead iodide, the actual amount of each depending on the temperature and the duration of the distillation. Thus at 280°, when the distillation lasted ninety-five minutes, the mixture contained 44 per cent. of iodine, whilst at 270°, with a duration of eighty-three minutes, the iodine content was 43 per cent. ($PbI_2$ contains 55·1, and PbI 38 per cent. of iodine). Moreover, it is to be noted that so long as the temperature does not exceed 262° it is possible to distil the vapour through the reaction bulbs for an hour after the evolution of gas has ceased, and the percentage of iodine does not rise above that of the suboxide (38), whilst even a few minutes' distillation at any temperature above 263° will give a product containing distinctly more than 38 per cent. of iodine.

The velocity of the distillation must be such that the vapour

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approximately reaches the temperature of the oven before coming into contact with the suboxide. This is ensured by the presence of the spiral; but if this precaution is neglected the reaction will rarely proceed to completion, and one obtains a mixture of the suboxide and subiodide. This can at once be seen, since such a mixture, even though containing but 2 per cent. of the suboxide, has no longer the characteristic yellow colour of the pure subiodide, but has a distinct green tint.

The object of the lengthy exhaustion of the apparatus at the completion of the experiment is that a slightly volatile, yellow product condenses in the reaction bulbs during the distillation, and this can be best removed by thorough exhaustion at as high a temperature as is consistent with the stability of the salt. This difficultly volatile product slowly distils off during exhaustion and condenses in a clearly defined yellow band just outside the oven. It contains neither lead nor iodine, and appears to be a condensation product of an aldehyde formed during the reaction. The following experiment illustrates the effect of insufficient exhaustion: The exhaustion was continued for two hours, and a bright yellow product was obtained which contains I = 36·6, Pb = 59·7 per cent. The ratio Pb : I is exactly that for lead subiodide (62 : 38), but the sample was evidently vitiated by the presence of the above-mentioned condensation product.

*Methods of Analysis.*

(*a*) In the case of lead oxalate, the lead was estimated by introducing a weighed quantity into a platinum crucible, adding dilute sulphuric acid, and weighing as sulphate after evaporation and ignition.

(*b*) The lead in the suboxide was estimated by introducing a weighed quantity from a bulb into a platinum crucible, dilute nitric acid was added, and the whole evaporated to dryness, the lead being finally weighed as sulphate according to the method outlined in (*a*).

(*c*) In the analysis of the lead in the subiodide, a weighed quantity of the mixture of subiodide and silica was introduced into a weighed platinum crucible; dilute nitric acid was added, and the whole evaporated to expel iodine and excess of nitric acid, and the total weight of lead sulphate and silica obtained as in (*a*). The weight of silica was subsequently obtained by washing out the lead sulphate with hot, concentrated ammonium acetate, and hence the weight of lead sulphate derived from the original subiodide estimated.

(*d*) In the estimation of the iodine, the weighed quantity of

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subiodide and silica was brought into a beaker and moistened with moderately concentrated acetic acid; after remaining about fifteen minutes on a water-bath, 250 c.c. of water were added, and the whole digested until complete solution of the subiodide had occurred. The silica was filtered off and estimated, the iodine in the filtrate being weighed as silver iodide. (A slight deposit of carbonaceous matter, never more than 0·1 per cent., was always left undissolved with the silica.)

*Analytical Results.*

(a) *Lead Oxalate.*—Samples of this salt were prepared by the action of oxalic acid on an acid solution of lead acetate.

| | Found, Pb per cent. |
|---|---|
| Sample 1 ... ..... | 70·11 |
| | 70·13 |
| ,, 2 ..... ..... | 70·11 |
| | 70·04 |
| ,, 3 ..... ..... ..... | 70·14 |
| ,, 4 ..... .. | 70·17 |
| Theoretical ..... | 70·18 |

(b) *Methyl iodide* was prepared by the usual method, and after washing repeatedly with sodium hydroxide and water was distilled from phosphoric oxide. The boiling point of the iodide did not vary by more than 0·25°.

(c) *Lead Subiodide.*

| Expt. | Found, Pb per cent. in suboxide. | Found, Pb per cent. in subiodide. | Found, I per cent. in subiodide. |
|---|---|---|---|
| 1 | — | 62·1 | 37·45 |
| 2 | 95·87 | 62·7 | 37·4 |
| 3 | 96·16 | 62·3 | 38·2 |
| 4 | 95·74 | 62·4 | (36·0) |
| 5 | 96·04 | 61·8 | 37·5 |
| 6 | 95·70 | 61·0 | 38·3 |
| 7 | 96·16 | 62·3 | 37·65 |
| 8 | — | 62·0 | 37·8 |
| Mean ..... | 95·94 | 62·07 | 37·76 |
| Theoretical | 96·28 | 62·01 | 37·99 |

(In experiments 1 and 8 no samples of the suboxide were taken; in taking the mean of the iodine percentages, experiment 4 has been omitted.)

*Properties of the Subiodide.*

The much vexed question as to whether the substance the formula of which is $Pb_2O$ is a true suboxide, and not an equimolecular mixture of lead and lead oxide, has been convincingly

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answered by Tanatar (*loc. cit.*) and by Brislee (*loc. cit.*). That the subiodide is also a true chemical compound, and in no sense a mixture, is clearly demonstrated in the first place by its colour. The bright yellow of the subiodide is only obtained when the product has a composition very close to the theoretical value. The presence of even 1 per cent. of the dark suboxide in the sample of subiodide is sufficient to change the colour from the clear yellow to a green or even a dark yellow. Thus in one experiment, wherein the suboxide had been heated to 360° during its formation, thereby forming the pale grey unreactive form, the subiodide obtained had a distinct green appearance, and analysis showed that the ratio of lead to iodine was 65 : 35 (theory is 62 : 38).

If the subiodide is heated to 300° in a vacuum, the colour slowly becomes much darker, and with a lens it is possible to distinguish the heterogeneity of the mixture. By the action of heat, the substance decomposes in accordance with the equation

$$2PbI \longrightarrow Pb + PbI_2.$$

The presence of the lead iodide in this dark mixture can readily be demonstrated by boiling it for a few minutes with water, filtering, and cooling; a copious crop of lead iodide crystals is at once obtained, whilst if the undecomposed yellow subiodide is boiled with oxygen-free water and rapidly filtered, not only is there an entire absence of any such separation of lead iodide crystals, but the filtrate gives no trace of a precipitate with potassium chromate or with hydrogen sulphide, the latter reagent producing a faint darkening only.

By the action of acids (hydrochloric, sulphuric, and acetic) a decomposition similar to that brought about by heat takes place.

The subiodide appears to undergo slow oxidation on exposure to the air. Thus, after removing the silica from the reaction mixture by sifting in the open air, the following analysis was obtained:

Lead = 60·9 per cent.
Iodine = 37·2 „
Oxygen = 1·9 „

the oxygen being determined by reduction in a stream of hydrogen.

As a final proof of the chemical individuality of the substance, its solubility, as well as that of the normal iodide, was determined by the conductivity method. A vessel of the type shown in Fig. 2 was constructed. Into the side-tube, before the capillary tap was sealed on, there was introduced a glass tube containing mercury, and also a small bulb containing the salt under investigation. After the tap had been sealed on, a stream of conductivity water

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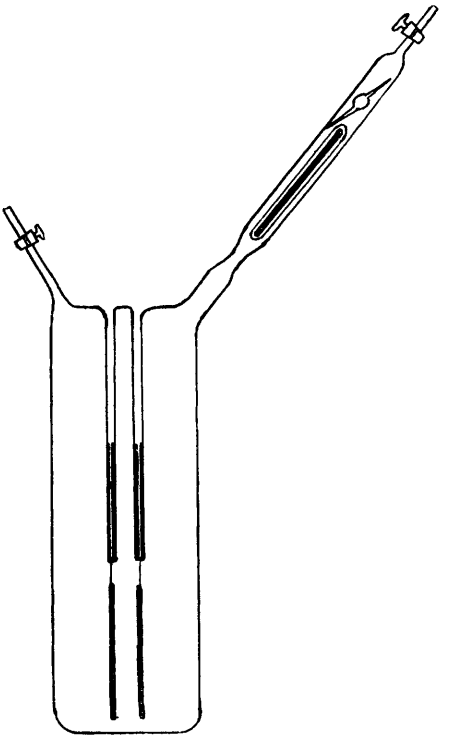
was drawn through the apparatus in order to remove all traces of impurity arising from acid products of combustion. When a constant resistance of 20,000 ohms had been registered, both side-tubes were connected to a water-pump, and the water boiled for ten to fifteen minutes under diminished pressure in order to remove traces of oxygen from the apparatus. After closing both taps and cooling the contents to 35°, the bulb was broken by means of the hammer, and after thorough shaking the vessel was

FIG. 2.



placed in a thermostat at 25°, and conductivity readings were taken until constant.

In the case of lead iodide, about 0·3 gram was brought into a small bulb, and after thorough exhaustion by a Töpler pump the bulb was sealed off, in order to make the conditions quite comparable with those under which the solubility of the subiodide was determined. In ten minutes after the bulb was broken in the conductivity vessel a resistance of 138 ohms was recorded, and this

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remained constant until the apparatus was opened to the atmosphere.

In order to obtain the solubility of the subiodide, a small bulb of this substance was prepared by the usual method, and a second and larger bulb retained for a control analysis. It is essential in this determination that oxygen be rigidly excluded both from the bulb and the conductivity vessel itself. In ten minutes after breaking the bulb a resistance of 1250 ohms was recorded, and during the next twenty-four hours this underwent no change.

It appears, therefore, that if the ionic mobilities of the iodine and the lead ions from the subiodide (whether $Pb^+$ or $Pb_2^{++}$) be assumed to be the same as in the case of the normal iodide, the solubility of the subiodide is 0·35 milli-equivalent per litre, that is, about one-ninth that of the normal salt. This relative insolubility of the lower iodide is interesting, inasmuch as it falls into line with the corresponding cases of copper, mercury, etc.

### *The Solubility of Lead Iodide.*

Numerous determinations of the solubility of lead iodide have been recorded, notably by Lichty (*Amer. Chem. J.*, 1903, **25**, 469), v. Ende (*Zeitsch. anorg. Chem.*, 1903, **26**, 162), and Böttger (*Zeitsch. physikal. Chem.*, 1903, **42**, 602). Lichty and v. Ende both used the direct method in their estimation of the solubility, the former obtaining 1·65 millimols and the latter 1·58 millimols per litre at 25°. On the other hand, Böttger arrived at a distinctly lower value. His measurements were made by the conductivity method, and on the assumption that 97 per cent. of the salt is dissociated in the saturated solutions, he finds that 1·31 millimols dissolve in a litre of water at 20·1°.

By using the apparatus just described, the following readings were obtained at 25°:

| | | | |
|---|---|---|---|
| in | 10 | min. | 140 ohms |
| „ | 40 | „ | 141 „ |
| „ | 100 | „ | 141 „ |

whence $L$, the specific conductivity, is 461·7 (corrected for the conductivity of water).

A second sample of lead iodide was prepared by precipitation, and after repeated washing it was recrystallised three times from water. A saturated solution in conductivity water was then prepared at 40°, and brought into the conductivity vessel without exposure to the air. In ten minutes a constant reading was obtained, whence $L=472·2$ (corrected for the conductivity of the water).

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Assuming complete dissociation, and that the ionic conductivities of lead and iodine are 71·8 and 76·4 respectively, the solubility is 1·58 millimols per litre at 25°, in exact agreement with the value obtained by v. Ende, but decidedly higher than that obtained by Böttger (1·31 at 20·1°).

On the other hand, it has been noticed that if in the preparation of the solution of lead iodide it is exposed appreciably to the atmosphere, the conductivity is abnormal. The resistance set up by a solution that had been thus exposed amounted to 139 ohms in ten minutes, and in sixteen hours it had risen to 148 ohms. This is attributed by the author to the following reactions:

$$PbI_2 + CO_2 + H_2O \longrightarrow PbCO_3 + 2HI,$$
$$4HI + O_2 \longrightarrow 2H_2O + 2I_2,$$

for it was possible by admitting gaseous impurities to induce an exactly similar change in a solution prepared by breaking a bulb of lead iodide in the conductivity vessel. When a perfectly constant reading for the solution, prepared, as already described, in the absence of air, had been obtained, a little carbon dioxide and oxygen were introduced into the apparatus and the whole shaken. In seventy hours the resistance rose steadily from 140 to 152 ohms.

It appears, therefore, that in using the conductivity method for the determination of the solubility of lead iodide, and possibly also of lead bromide (see Böttger, *loc. cit.*, p. 575, on the variation noted in the conductivity of this salt), extreme care must be exercised in excluding gaseous impurities, but with due precautions the method gives rapid and accurate results.

*Summary.*

(1) Lead suboxide may be best prepared by heating in a vacuum lead oxalate at a temperature as high as 375° provided the pressure of the evolved gases does not rise above 5 cm.

(2) Above 335° the suboxide becomes paler in colour, and at the same time much less reactive.

(3) Lead subiodide may be obtained by distilling the vapour of dry methyl iodide at a maximum temperature of 262° through the suboxide.

(4) The subiodide thus prepared is of a pure yellow colour, which darkens on heating above 300° owing to decomposition into lead and lead iodide.

(5) The subiodide has a solubility about one-ninth that of the normal iodide, is slowly oxidised by air, and is completely decomposed by acids.

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Further work on the sub-salts of lead, etc., is now in progress. The author desires to record his appreciation of the facilities placed at his disposal by the Walter and Eliza Hall Trust for the prosecution of this research.

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