

XLVII.—*On the Preparation of Salts of Nitrous Oxide.*

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IN the course of some analyses of cast-iron, an experiment was made to ascertain whether fusion with nitre and carbonate of soda would readily yield the phosphorus in the form of an alkaline phosphate.

If the operation be properly carried out, a bulky, yellow precipitate is obtained with nitrate of silver, which it is impossible to regard as phosphate. Professor Bloxam suggested the possibility of this body being the salt which Dr. Divers obtained by the action of sodium-amalgam on *nitrate of soda* (*Proc. Roy. Soc.*, xix, 425) and subsequent precipitation with silver nitrate. Several analyses of the silver salt were made.

Analysis of the Silver Salt.

The silver was estimated by dissolving in nitric acid and precipitating as chloride. Amount of substance taken = .6625 gram. On boiling with a little nitric acid, a little silver chloride, existing as impurity, remained undissolved: this was collected on a filter, fused, and weighed, that adhering to the filter-paper being burnt to metallic silver. The amount of silver chloride found, including the metallic silver calculated as chloride, = .092 gram, which when subtracted

from the amount of original substance taken, leaves $\cdot 5705$ of the pure silver salt.

The solution in nitric acid was then precipitated with hydrochloric acid. The silver chloride was collected on a filter, fused and weighed. The amount of silver chloride found was $\cdot 599$ gram. $\cdot 599 \text{ AgCl} = 78\cdot 17$ per cent. of silver.

A second analysis was made in a similar manner, the amount of substance taken being $\cdot 175$ gram. The silver chloride found as impurity was $\cdot 014$ gram, leaving $\cdot 161$ gram of the pure silver salt.

The amount of silver chloride obtained from the nitric acid solution was $\cdot 167$ gram. $\cdot 167$ of silver chloride = $78\cdot 01$ per cent. of silver.

| | Pure substance taken. | Silver chloride found. | Per cent. of silver. |
|---------|-------------------------------|---------------------------|-------------------------|
| I..... | $\cdot 5705$ | $\cdot 5990$ | $78\cdot 17$ |
| II..... | $\cdot 1610$ | $\cdot 1670$ | $78\cdot 01$ |
| | Mean = $78\cdot 09$ per cent. | | |

The amount of silver found, agrees, therefore, with the formula AgNO_3 , which requires $78\cdot 26$ per cent. of silver, and also with the analyses of Dr. Divers.

Sodium nitrate may be substituted for the potassium salt, and furthermore the admixture of carbonate of soda at first used may be left out, the salt being satisfactorily prepared by the simple fusion of iron filings with sodium nitrate. Regarding the best temperature for the operation, experiments were made with a Bunsen burner, a Gore's gas furnace, a coal furnace, and a charcoal furnace, the best results being obtained with a charcoal furnace. The silver precipitate is very bulky, but shrinks greatly when dried. The silver salt being reduced slightly at 100° , it is best to dry it in a vacuum over oil of vitriol, taking care to protect it from the light.

The silver salt is soluble in ammonia and in nitric acid, from solutions in either of which it can be again thrown down, as was shown by Dr. Divers, on the addition of acid in the one case, and of alkali in the other. When the silver salt is strongly heated it turns black owing to the reduction of the silver.

Preparation of the Sodium Salt.

To prepare the sodium salt, iron filings were fused with sodium nitrate in an iron crucible over a charcoal furnace, boiled with water, filtered from the oxide of iron, evaporated down to a small bulk, and left to crystallise. White needle-shaped crystals were deposited having the composition $\text{NaNO}_3 + 3\text{H}_2\text{O}$.

In order to obtain the best yield of the salt the crucible ought to be kept at a red-heat for about an hour after the deflagration has taken

place, otherwise the sodium nitrate will not be properly converted into hyponitrite, and crystals will be got with great difficulty.

Analysis of the Sodium Salt.

The sodium was estimated by conversion into sulphate.

| | Weight of substance taken. | Sodic sulphate found. | Per cent. of sodium. |
|----------|----------------------------|-----------------------|----------------------|
| I..... | ·478 | ·313 | 21·19 |
| II..... | ·3995 | ·2615 | 21·20 |
| III..... | ·3309 | ·2215 | 21·39 |
| IV..... | ·2205 | ·1455 | 21·35 |

Mean = 21·28.

The water of crystallisation was next determined by drying at 100° C. in the water-bath until a constant weight was obtained, which generally took about three or four hours. The following are some of the results :—

| | Weight of substance taken. | Loss. | Per cent. of water. |
|----------|----------------------------|-------|---------------------|
| I..... | ·202 | ·1025 | 50·74 |
| II..... | ·122 | ·0615 | 50·39 |
| III..... | ·442 | ·240 | 50·43 |
| IV..... | ·662 | ·350 | 50·62 |

Mean = 50·54.

The nitrogen was estimated by burning with soda-lime and passing the ammonia gas through standard sulphuric acid, also by precipitating as ammonio-platinic chloride. The following numbers are the results :

| | Amount of substance taken. | Ammonia obtained. | Nitrogen per cent. |
|----------|----------------------------|----------------------------|--------------------|
| I..... | ·466 | ·077129 | 12·95 |
| II..... | ·524 | ·0872175 | 13·70 |
| | Amount of substance taken. | Ammonio-platinic chloride. | Nitrogen per cent. |
| III..... | ·3625 | ·740 | 12·80 |

Mean = 13·15.

It will be seen that the mean numbers of the several analyses agree well with the formula $\text{NaNO}\cdot 3\text{H}_2\text{O}$:—

| | Found. | Calculated. |
|--------------------|-----------------|-----------------|
| Na = | 21·26 per cent. | 21·49 per cent. |
| N = | 13·15 " | 13·08 " |
| H ₂ O = | 50·54 " | 50·46 " |
| O = | | 14·95 " |
| | | 100·00 |

Experiments were then made, substituting zinc for iron in the reduction of the nitre, but the reaction was too violent, and there was not the slightest indication of the salt being formed.

The sodium salt is very soluble in water, to which it imparts a slightly alkaline reaction, but it is quite insoluble in ether and alcohol. It is also slightly efflorescent, and was found to lose water over sulphuric acid. I have not yet succeeded in obtaining crystals of the potassium salt.

An aqueous solution of the sodium salt was found to give the following reactions with some of the metals:—

With *copper sulphate*, a turquoise-blue precipitate soluble in acetic acid.

With *manganese sulphate*, a white precipitate soluble in acetic acid.

With *magnesium sulphate*, a white precipitate soluble in acetic acid.

With *bismuth nitrate*, a white precipitate insoluble in acetic acid.

With *zinc sulphate*, a white precipitate soluble in acetic acid.

With *aluminium sulphate*, a white precipitate insoluble in acetic acid.

With *baryta*, a white precipitate soluble in acetic acid.

With *calcium chloride*, a white precipitate soluble in acetic acid.

With *mercuric chloride*, a white precipitate, which afterwards became yellow and then brownish-red, which dissolved in acetic acid, leaving a white precipitate.

With *cobalt nitrate*, a mauve precipitate soluble in acetic acid.

With *mercurous nitrate*, a black precipitate, apparently metallic mercury at the bottom, and whitish-yellow suspended matter at the top, insoluble in acetic acid.

With *gold chloride* the gold was reduced to the metallic state as a purple powder.

With *platinic chloride*, a reddish-white precipitate, which on the addition of acetic acid was largely increased.

With *lead acetate*, a white precipitate, which was insoluble in acetic acid.

With *stannous chloride*, a white precipitate insoluble in acetic acid.

With *nickel sulphate*, a whitish-green precipitate soluble in acetic acid.

With *strontium nitrate*, a white precipitate soluble in acetic acid.

With a *cerous salt*, a white precipitate insoluble in acetic acid.

With *ferrous sulphate*, an olive-green precipitate resembling that obtained by caustic alkalis, and changing, on addition of acetic acid, to a yellowish precipitate.

With *ferric chloride*, a yellow precipitate soluble in acetic acid.

At the suggestion of Mr. Hartley, I acted upon the silver salt in a sealed tube with iodide of ethyl. As soon as the tube is sealed up,

ebullition commences. If heated in a water-bath for some hours the silver is converted into iodide. On fractionating the distillate, evidence of the formation of an ethereal salt of low boiling point is obtained. As the vapour-density of this would fix the molecular formula of the salt, I hope soon to be able to communicate success with further experiments in this direction.
