



# On the separation of antimony from arsenic

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quantity with bismuth; and the following very simple process for that purpose has occurred to me, founded on the known reduction and precipitation of salts of bismuth by metallic lead—a method which I have found to succeed perfectly with brittle alloys containing upwards of 85 per cent. of bismuth. A small fragment or little crystal of nitrate of bismuth is placed in a porcelain capsule, and moistened with a few drops of water, the greater part of which is afterwards poured off; and the metallic button of the mixed metals, as obtained by the blowpipe, having been slightly flattened on the anvil until it begins to crack at the sides, is then placed in the midst of the subsalt of bismuth formed by the action of the water, when in the course of a minute, or even less, according to the amount of lead present, minute arborescent crystals of metallic bismuth form and collect around the assay. Copper does not affect this reaction; but if either zinc or iron were present, the precipitation would ensue from that cause alone. Zinc, however, if originally contained in the assay-matter, would be volatilized by the action of the blowpipe, especially if a little carbonate of soda were added to it before subjecting the globule to the above test; and iron might be easily separated by treating the assay in the reducing-flame with a mixture of carbonate of soda and borax—the latter reagent serving to dissolve the iron, and to prevent its reduction to the metallic state. If a single operation do not effect this, the globule must be removed from the saturated dark green glass, and treated with a further supply of the mixture on another piece of charcoal until the resulting glass be no longer coloured.—*From the Chemical Gazette for September 15, 1848.*

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#### ON THE SEPARATION OF ANTIMONY FROM ARSENIC.

BY C. MEYER.

The following method is founded on the insolubility of the antimoniate of soda, and the conversion of arseniferous antimony into arseniate and antimoniate of soda. The author first convinced himself of the perfect insolubility of calcined anhydrous antimoniate of soda. When antimony is deflagrated with nitrate of soda and the mass exhausted with cold water, no antimony can be found in the liquid. The residuous antimoniate of soda is  $\text{NaO}$ ,  $\text{SbO}_3$ , and anhydrous. When a solution of antimoniate of potash is precipitated with sulphate of soda, and the liquid after some time filtered off clear, no antimony can be detected in it. The crystalline precipitate was proved by analysis to be also  $\text{NaO}$ ,  $\text{SbO}_3$ , but it contains 6 atoms or 21·23 per cent. of water; experiment gave 21·5. The antimony was estimated in some cases by precipitating it with sulphuretted hydrogen from a solution of the salt in a mixture of muriatic and tartaric acids; in others by the method recently proposed by Rose\*, igniting the salt with chloride of ammonium, which gave

\* Chem. Gaz., vol. vi. p. 166.

the most accurate result. This hydrated salt is not quite insoluble in boiling water.

To test the applicability of the insolubility of the calcined antimoniate of soda to its separation from arsenic, a known weight of pure antimony was mixed with arsenic, and the mixture deflagrated with three times its weight of a mixture of nitrate and carbonate of soda. After washing the mass with cold water and ignition, a quantity of antimoniate of soda was obtained, which almost exactly corresponded with the weight which should have been obtained according to theory. On examination before the blowpipe, the salt proved to be perfectly free from arsenic. The arsenic was precipitated from the solution with sulphuretted hydrogen, and determined in the usual manner; and its quantity likewise agreed perfectly well with the amount employed.

0.10 grm. tartar-emetic and as much arsenious acid were dissolved in a thick soup, the organic matters destroyed as much as possible by a long-continued current of chlorine, the heated liquid filtered, the mass upon the filter well-washed, the solution saturated with sulphuretted hydrogen and left for some time in contact with it; the precipitate filtered, washed, and then dissolved together with the filter in hot nitric acid, the solution saturated with carbonate of soda, some nitrate of soda added to it, the whole evaporated, and the residue heated in a porcelain crucible until the sulphurets and organic matter were completely oxidized, and the mass finally fused. On exhausting the mass with water, 0.057 grm. antimoniate of soda was obtained, while according to theory 0.058 should have been obtained. The solution which contained the arseniate of soda was evaporated to dryness; all the carbonic, nitric and nitrous acids expelled by concentrated sulphuric acid; the salt dissolved in water, mixed with sulphurous acid; and, after expelling the latter, the arsenic precipitated by sulphuretted hydrogen. To separate the free sulphur, the precipitate was dissolved in very dilute ammonia, and reprecipitated with sulphuric acid; it weighed 0.120 grm., which corresponds to 0.096 arsenious acid, which also is very near to the amount employed.

The author applied this insolubility of the antimoniate of soda likewise to the preparation of antimony free from arsenic acid with complete success. Commercial antimony was fused together with  $\frac{1}{4}$ th its weight of arsenic, the powdered regulus mixed with  $1\frac{1}{4}$ th part of crude nitrate of soda (cubic nitre), and  $\frac{1}{2}$  part carbonate of soda, heated to faint redness, and the mass exhausted with water. The residue of antimoniate of soda was, when dry, fused with half its weight of cream of tartar, when a very beautiful white regulus was obtained, which gave off not the slightest odour of arsenic before the blowpipe, and was characterized by the ease with which it continued to burn away. Moreover the regulus neither contained sodium nor potassium, which latter it generally contains when reduced from the antimoniate of potash.—Liebig's *Annalen*, May 1848.