IDENTIFICATION OF ARSENIC AND ANTIMONY.

BY JAMES T. ANDERSON.

The method of separating arsenic and antimony by passing H_2S and then dry HCl gas through the tubes in which the metals have been deposited as in Marsh's test, has been modified by the writer so as to be conveniently applied in cases where it is desired to identify as arsenic or antimony metallic deposits on porcelain.

Add a drop of ammonium sulphide to the deposit, which converts the metal into the sulphide. Allow the excess of ammonium sulphide to evaporate, and with an ordinary mouth blowpipe blow across the open mouth of a bottle containing concentrated HCl, directing the stream of gas into the porcelain dish upon the sulphide. If it be antimony sulphide, it will disappear entirely, while arsenic sulphide will remain unaffected in appearance.

AGR. & MECH. COLLEGE, Auburn, Ala., July 27, 1891.

ON METATITANIC ACID AND THE ESTIMATION OF TITANIUM BY HYDROGEN PEROXIDE.

BY PROF. F. P. DUNNINGTON.

The detection and estimation of small amounts of titanic acid which has long been very tedious and unsatisfactory, has been rendered exceedingly simple through the publication of A. Weller in the *Ber. d. Chem. Ges.*, 1882, of a method depending upon the production of a compound of intense yellow color by the addition of hydrogen peroxide to the solution of titanium.

Since its publication I have made considerable use of this method, but have recently noted a feature in the test which must be kept in view to avoid error. The solution of the melt obtained by fusion with acid sodium sulphate when made with dilute sulphuric acid of five or more per cent., gives constant results, which tally with those made gravimetrically; but when water only or very dilute acid is employed, one may obtain lower results. Upon one occasion a coloration was obtained which corresponded to only about one-third of the titanium, which was afterwards found to be present. Moreover, if we take a slightly acid solution of titanium sulphate, dilute it and heat until it is partially precipitated, cool, redissolve with sulphuric acid and then add hydrogen peroxide, the yellow coloration will correspond to only a portion of the titanium present. I find an explanation of these facts in the formation of some meta-titanic acid. It is uniformly stated that when titanic acid is precipitated by heating a not too acid solution, it separates in the form of meta-titanic acid.

In order to further examine the metatitanic acid which was in part described by Weber (*Jahr.*, 1863, p. 210), I have isolated it as follows: Heat a dilute solution of titanium sulphate to 100° C.; gradually add ammonia to excess, filter, treat the washed precipitate with hydrochloric acid and warm; there results a residue which is nearly insoluble in strong hydrochloric acid. Now wash this once or twice by decantation with cold water, and, when most of the hydrochloric acid is removed, there will be left a mass perfectly and readily soluble in cold water.

Again, if the above precipitate is treated with hydrochloric acid, and then evaporated upon the water bath to remove the latter acid, there is left a mass of scales of a gummy appearance, which dissolves in a moderate amount of water, giving an opaline solution. This substance is practically free from chlorine and contains one molecule of water to one of titanic acid—and so presents the composition of the air dried metatitanic acid of Merz. If either of these water solutions of meta-titanic acid be treated with hydrogen peroxide, it will not be colored yellow, but afford a white precipitate.

It therefore appears probable that after the fusion of titanic oxide with acid sodium sulphate, if the melt is digested in water only, the solution of the free acid may occasion sufficient heat to form some metatitanic acid, which when redissolved by the further admixture of acid would not be colored by the hydrogen dioxide.

And we conclude that in making the estimation of titanium by the method of A. Weller, the "melt," after cooling, is to be digested in dilute sulphuric acid of such strength as will prevent the formation of a precipitate even in warm solutions of titanic sulphate; for this purpose 5 per cent. acid is found to answer.

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