

arsenic. The author has reinvestigated De Haen's method, employing thiosulphate for the titration of the iodine. When free acids are absent, the results are only satisfactory if the solutions are sufficiently concentrated, but in presence of free sulphuric acid correct results are obtained, even when the dilution and amount of free acid are largely varied. Hydrochloric and nitric acids as acidifying agents are inadmissible, but acetic acid may be used. Although the action of potassium iodide on a cupric salt indubitably consists at first in the formation of cupric iodide, in the presence of free acid the decomposition into cuprous iodide and free iodine is so rapid and complete that titration may be commenced two minutes after making the mixture; a delay of ten minutes does not materially affect the results, but after an hour higher numbers are obtained. The following is the form in which the process is recommended: the copper solution (50 c.c. containing about 0.15 gram of copper as sulphate) is placed in a 300 c.c. stoppered bottle; 5 c.c. of 10*N* sulphuric acid and 2 grams of solid potassium iodide are added and the bottle is shaken for two minutes. The free iodine is then titrated with *N*/10 thiosulphate, adding starch when near the end.

To render the method applicable in presence of iron and arsenic, the solution of the metals (as sulphates) is treated with an excess of sodium pyrophosphate sufficient to redissolve the precipitate at first formed. If the original solution contained free sulphuric acid, some sodium acetate is added, then 4—5 grams of potassium iodide and 5—10 c.c. of 80 per cent. acetic acid. In these circumstances, the copper alone liberates iodine, but to obtain good results it is advisable to avoid dilution as far as possible, wherefore solid sodium pyrophosphate should be used for the precipitation, and if the amount of iron exceeds that of copper by more than one-third, the quantity of potassium iodide should be increased; a somewhat longer time (15 minutes) is allowed before titrating, and the thiosulphate run in very slowly.

M. J. S.

Detection and Estimation of Minute Quantities of Mercury.

CONSTANTIN ZENGELIS (*Zeit. anal. Chem.*, 1904, 43, 544—547).—For the detection of mercury in urine, the urine is feebly acidified with hydrochloric acid and left in contact for 12 hours with a spiral of copper and platinum wires. The spiral is then washed with dilute sodium hydroxide, water, alcohol, and ether, and dried completely over sulphuric acid. It is then thrust to the bottom of a narrow test-tube, the upper part of which is moistened with a solution of iodine in anhydrous ether. Holding the tube horizontally, the coil is heated, and the evolved mercury vapour is arrested by the ring of iodine solution and converted into mercuric iodide. Complete absence of water is indispensable: 0.2 milligram of mercury can be detected in 200—300 c.c. of urine. For quantitative estimation, the urine is boiled with an excess of sodium hydroxide and a little of a reducing sugar. The phosphate precipitate is dissolved in nitric acid and the solution electrolysed at 40—50° with a weighed piece of platinum foil as cathode.

M. J. S.