Iodometric Titration of Cu in Brass

Procedure

Standardize one liter of 0.1 M sodium thiosulfate solution using the procedure in Harris's experiments. Do not add HgI_2 to your starch solution. Make only sufficient starch solution for your needs.

Prepare 4 samples of brass simultaneously. For each, accurately weigh about 0.3 g of brass into a 250 mL conical flask. *In the hood*, add 5 mL of 6 M HNO_3 and warm until solution is dissolved. Still in the hood, add 10 mL of concentrated sulfuric acid and evaporate until copious white fumes of SO_3 are given off. Allow mixture to cool. Cautiously add 30 mL of DI water, boil for 1-2 minutes and again cool.

Add concentrated ammonia and with thorough mixing to produce the intensely blue $Cu(NH_3)_4^{2+}$; the solution will smell faintly of ammonia (but don't sniff it!). Make dropwise additions of 3 M sulfuric acid until the color of the complex just disappears (returns to the original blue color) and then add 2.0 mL of concentrated phosphoric acid.

Treat each sample individually from this point to minimize the air-oxidation of iodide ion!

Add 4.0 g of KI to the sample and titrate immediately with standard sodium thiosulfate until the solution becomes pale yellow. Add 5 mL of starch indicator (from standardization experiment) and continue the titration until the blue/purple color becomes faint. Add 2 g of KSCN; swirl vigorously for 30 s. Complete the titration, using the disappearance of the blue starch/I_2 color as the end point.

From: Fundamentals of Analytical Chemistry, 5th edition, by Skoog, West and Holler, Saunders College Publishing, 1988.