LABORATORY EXPERIMENT 7

The Iodometric Determination of Copper in Brass

Discussion

The method is relatively simple and applicable to brasses with less than 2% iron. A weighed sample is treated with nitric acid, which causes the tin to precipitate as a hydrated oxide of uncertain composition.

$$Cu(\text{metal}) + 4HNO_3 \rightarrow Cu(NO_3)_2 + 2NO_2 + 2\,H_2O$$

$$3Cu(\text{metal}) + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + 2NO + 4\,H_2O$$

$$Sn(\text{metal}) + 4HNO_3 \rightarrow SnO_2(\text{solid}) + 4NO_2 + 2\,H_2O$$

Evaporation with sulfuric acid to the appearance of sulfur trioxide eliminates the excess nitrate, re-dissolves the tin compound, and possibly causes the formation of lead sulfate. The pH is adjusted through the addition of ammonia, followed by acidification with a measured amount of phosphoric acid.

An excess of potassium iodide is added, and the liberated iodine is titrated with standard thiosulfate.

$$2Cu(NO_3)_2 + 4KI \rightarrow 2CuI(\text{solid}) + I_2 + 4KNO_3$$

$$2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$$

PROCEDURE

We use a slightly simplified procedure for alloys with little Sn and Pb.

Weigh (to the nearest 0.1 mg) a 0.4-g sample into 250-mL beaker, introduce 5 mL of 6 M HNO_3; warm *(use the hood)* until solution is complete. Dilute with about 25 mL of distilled water, add 0.3 g urea, and boil briefly to eliminate nitrogen oxides. Rinse the watch glass, collecting the rinsings in the flask. Cool it.

Transfer the resulting solution into a 200-mL volumetric flask and make it to the mark.

If you need to continue the analysis next laboratory period, this is a good place to stop.

Take a 25-mL aliquote and transfer in into a 250 mL conic flask. Add concentrated NH_3 dropwise and with thorough mixing to produce the intensely blue $Cu(NH_3)_4^{2+}$; the solution should smell faintly of ammonia. Make dropwise additions of 3 M H_2SO_4 until the color of the complex just disappears, and then add 1 mL of 85% H_3PO_4. Cool to room temperature.

Treat each sample individually from this point on to minimize the air oxidation of iodide ion.

Add 2.0 g of KI to the sample, and titrate immediately with $Na_2S_2O_3$ until the solution becomes pale yellow. Add starch indicator, and continue the titration until the blue color becomes faint. Complete the titration, using the disappearance of the blue starch/I_2 color as the end point. Perform this titration in triplicate.

1. **Standardization of thiosulfate solution**

Sodium thiosulfate solution (approximately 0.05 M) requires additional standardization. To perform it, a sample of pure copper foil (100% Cu with uncertainty lees than 0.2%) is treated as described above, simultaneously with your sample. From titration of pure copper standard you calculate the precise concentration of thiosulfate:

$$C_{Na_2S_2O_3} = \frac{m_{CuPure} \times 1000}{FW_{Cu} \times V_{Na_2S_2O_3}} \times \frac{V_{aliquote}}{V_{total}}$$

2. **Analysis of Copper-containing alloy**

Run a sample of copper-containing alloy as described above (parallel to a pure copper standard). From the titration results, calculate percentage of Cu in the sample.

$$m_{Cu} = \frac{FW_{Cu} \times C_{Na_2S_2O_3} \times V_{Na_2S_2O_3}}{1000} \times \frac{V_{total}}{V_{aliquote}}$$

$$\%\%Cu = \frac{m_{Cu}}{m_{sample}} \times 100\%$$

Be sure you have an Excel template for calculations ready when you start this experiment.