

LABORATORY EXPERIMENT 7

The Iodometric Determination of Copper in Brass

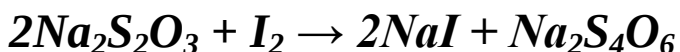
Discussion

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.



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.



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 $\text{Cu}(\text{NH}_3)_4^{2+}$; 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 $\text{Na}_2\text{S}_2\text{O}_3$ 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 less 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:

$$C_{\text{Na}_2\text{S}_2\text{O}_3} = \frac{m_{\text{CuPure}} \times 1000}{FW_{\text{Cu}} \times V_{\text{Na}_2\text{S}_2\text{O}_3}} \times \frac{V_{\text{aliquote}}}{V_{\text{total}}}$$

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.

$$m_{\text{Cu}} = \frac{FW_{\text{Cu}} \times C_{\text{Na}_2\text{S}_2\text{O}_3} \times V_{\text{Na}_2\text{S}_2\text{O}_3}}{1000} \times \frac{V_{\text{total}}}{V_{\text{aliquote}}}$$

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

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