IODOMETRIC DETERMINATION OF IRON

Background
In this experiment an iron ore sample is dissolved and treated to oxidize all iron to the ferric-state, Fe\(^{3+}\). Addition of excess iodide under mildly acidic conditions results in quantitative iron reduction to the ferrous-state, Fe\(^{2+}\), and simultaneous oxidation of the iodide to iodine.

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2\text{Fe}^{3+} + 2\text{I}^- \rightarrow 2\text{Fe}^{2+} + \text{I}_2
\]

Iodine produced in the iron reduction is titrated with standard thiosulfate to a starch end-point.

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\text{I}_2 + 2\text{S}_2\text{O}_3^{2-} \rightarrow 2\text{I}^- + \text{S}_4\text{O}_6^{2-}
\]

The experiment produces no hazardous waste. All solutions may be discarded down the drain.

Procedure

Sample Preparation
Dry the iron ore sample at 110 °C for at least 2 hours; overnight drying is preferred.

**Note:** In this and subsequent instructions, you will be told to dry various materials at 100 °C for 1-2 hours. Recognize that when everyone is opening the oven, it will not be able to stay at its set temperature. Overnight drying is recommended to avoid this problem. Dry everything loosely covered, as with a watch glass. Remember to label all materials as they tend to get moved around.

Standard Preparation
The thiosulfate solution is standardized using *pure* (the assay on the label should be recorded in your notebook) iron wire. Make sure the wire is free of rust; any visible rust may be removed with fine sand paper. Degrease the wire with some methylene chloride or acetone on a tissue.

Weigh out approximately 0.4 g of clean iron wire using weights-by-difference. **The actual value must be accurately known** (weight to 0.1 mg). In a suitable beaker or flask, dissolve the wire in concentrated HCl by addition of 15 mL of acid. Use a graduated cylinder or a dispensing pump. Mark the acid level and, if necessary, return the liquid level to the mark by addition of acid at the completion of the dissolution. Do the digestion in a hood or a well-ventilated area. Heat the solution *nearly* to boiling for 10-15 minutes on a hotplate. Place a watch glass on top of the beaker. When all the wire has dissolved, slowly add 1 mL 30% hydrogen peroxide to the solution and boil it gently for 10 minutes. Adjust the volume as noted above if required.

Carefully transfer the solution to a 100 mL volumetric flask. Wash out solution adhering to the walls of the beaker with distilled water using a wash bottle. Add 1.5 mL additional
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HCl and then make up to volume. This solution will be used to standardize your thiosulfate solution; recheck its volume when it is at room temperature.

Sample Preparation
Weigh out 0.8 g of your unknown. The actual value must be known to high accuracy. Use a weights-by-difference technique. In a beaker flask add 15 mL HCl to the unknown and heat to almost boiling with occasional swirling for about 5 minutes. Cover with a watch glass and place on a hot plate to simmer just below boiling for about 30 minutes. When only silica remains, slowly add 1 mL 30% hydrogen peroxide and then boil gently for at least 10 minutes. As with the standard, the beaker or flask should be marked to indicate the 15 mL HCl level at the outset, and the volume should be adjusted to that level at the conclusion of the dissolution. Cool the solution to room temperature and transfer it with the aid of a wash bottle to a 100 mL volumetric flask. Add an additional 1.5 mL of HCl and make up to volume with distilled water. Make the final volume check when the solution is at room temperature as indicated above for the standard.

Titrant Preparation
A solution approximately 0.05 F in sodium thiosulfate is prepared in the following manner. Using either boiled distilled water or deionized distilled water, dissolve 12.5 g of reagent Na$_2$S$_2$O$_3$·5H$_2$O and 0.1 g sodium carbonate and make up to one liter in a 1 L beaker or flask. If anhydrous Na$_2$S$_2$O$_3$ is used, weigh a smaller number of grams to correct for the formula weight difference.) Store the solution in a container with a tight stopper or cap. (Note: the sodium carbonate renders the solution slightly basic and serves to stabilize the thiosulfate).

Analyses
Both the standard and the unknown will be titrated with your thiosulfate solution by the following method. Each will be done in triplicate. Sample aliquots may be delivered in advance, but the addition of potassium iodide should be made immediately prior to each titration to avoid possible loss of iodine vapor.

Use a transfer pipet to place 25 mL of iron solution in a 250 mL Erlenmeyer flask. Add 3 g of KI, cover the flask with a watch glass, swirl until the solid is dissolved and let stand, preferably in the dark, for five minutes. Add 80 mL of cool distilled water and titrate rapidly with thiosulfate. When the color of iodine becomes indistinct, add drops of starch indicator solution and carefully titrate until the blue color disappears. Cover the flask and wait about 2-3 minutes. If the blue color returns, it should be discharged with about one additional drop of thiosulfate. It is important to titrate until the blue color just disappears; do not over titrate. The titration is repeated until both standard and unknown have been titrated in triplicate.

Results
Calculate the weight percent iron (as the element) in your sample from the results of the titrations. 100% of your grade will be based on reported values and confidence limits.
Reference