Experiment 10a: Preparation of Sample for Measuring Manganese in Steel with Standard Addition

CH2250: Techniques in Laboratory Chemistry, Plymouth State University

Adapted from "27. Mn^{2+} Standardization by EDTA Titration," and "28. Measuring Manganese in Steel by Spectrophotometry with Standard Addition " *Experiments To Accompany* Exploring Chemical Analysis, 4th Edition, Daniel C. Harris (2008), available at http://www.whfreeman.com/exploringchem4e. Originally taken from S. P. Perone, J. Pesek, C. Stone, and P. Englert, *J. Chem. Ed*, **75**:1444 (1998).

Introduction: (Please see the Introduction in Experiment 10, Part B).

For the sake of time, this portion of Experiment 10 will be performed along side Experiment 9.

Start the write-up for this experiment in your notebook on the page **immediately after your pre-lab work for Experiment 9**. You will likely have some pages for Experiment 9 *after* the pre-lab write-up pages for this lab, and then Experiment 10, Part B will start after that. Be sure to cross-reference all these pages, so it is easy to follow the trails of Experiments 9 and 10 through your notebook!

Pre-lab Calculations: Read the procedure carefully, and perform the calculations in the Calculations section of your pre-lab.

Equipment: Read through the procedures and make a list of the equipment you will need.

Safety Considerations:

- Manganese, particularly permanganate (MnO_4^-) is toxic and must be disposed of properly.
- Read through the procedures and note any other safety considerations.

Procedure

1. Obtain an Unknown sample of steel from your instructor. Note the number of the sample.
2. Calculate the mass of steel needed to contain 4 mg of Mn, if the steel contains 0.6 wt% Mn. *Do this calculation before coming to lab.*
3. Weigh out the mass of steel you calculated in Step 2 to the nearest 0.1 mg into a 100-mL beaker.
4. Dissolve steel sample in 75 mL of 3 M HNO_3 by gently boiling in the hood, while covered with a watchglass. Boil until the solid has been dissolved for 5 minutes, but stop after 1 hour, even if undissolved particles remain. Replace the HNO_3 as it evaporates. You may work on Experiment 11 while you wait, but *check your boiling samples often*.
5. Cool the solution for 5 min. Then carefully add ~1.2 g of $(NH_4)_2S_2O_8$ or $K_2S_2O_8$ and boil for 10 min to oxidize carbon to CO_2.
6. If traces of pink color (MnO_4) or brown precipitate (MnO_2(s)) are observed, add 10 drops of 45 wt% NH_4HSO_3 and boil for 5 min to reduce all manganese to Mn(II):

$$2MnO_4^- + 5HSO_3^- + H^+ \rightarrow 2Mn^{2+} + 5SO_4^{2-} + 3H_2O$$

$$MnO_2(s) + HSO_3^- + H^+ \rightarrow Mn^{2+} + SO_4^{2-} + H_2O$$

7. After cooling the solutions to near room temperature, filter each solution quantitatively through #41 filter paper into a 250-mL volumetric flask. To complete a "quantitative" transfer, wash the beaker many times with small volumes of hot 0.05 M HNO_3 and pass the washings through the filter to wash liquid from the precipitate into the volumetric flask.
8. Allow the volumetric flasks to cool to room temperature, dilute to the mark with water, and mix well. Transfer this solution to a clean, dry, labeled 250 mL polyethylene bottle and store it for use in Experiment 10, part B. *This is the Sample solution.*

Analysis and Conclusions: These sections will be completed in Experiment 10, part B.