SIMPLIFIED PROCEDURE (Jan 2020)

Part 1 – Preparation of Analyte Sample ***Partner #1***

1. Obtain the unknown steel sample-bottle corresponding to the number assigned to you and your partner by your instructor. *This is not the same as your locker number.*

2. Using a metal scoopula, accurately weigh into a tared weigh-boat **0.20 to 0.30 g** of the powdered steel at an analytical balance. Seal and return the sample-bottle.

3. Carefully add a few drops of distilled water to the steel powder to moisten it.[1]

Quantitatively transfer the steel into a clean, but not necessarily dry, 250 mL Erlenmeyer flask, using the smallest amount of distilled water as possible, as described below:

> *Begin by squeezing-together two sides of the weigh-boat to create a lip at one end. Carefully tilt the lip of the weigh-boat over the mouth of the Erlenmeyer flask, allowing the wet powder to roll into the flask. Then use a gentle spray of a very small amount of distilled water to rinse any remaining steel from the weigh boat into the flask. It is normal for a small brown stain to remain in the weigh boat; this is not steel.*

4. **WEARING PROTECTIVE GLOVES,** add **40 mL** of **5 M nitric acid** (**HNO_3**) directly to the wet steel powder in the 250 mL Erlenmeyer flask. *A pump-bottle of the acid will be used.*

5. **AT A FUMEHOOD** place the Erlenmeyer flask on a hotplate (already set up for you by the lab technician). Turn the heat-setting on to almost maximum and wait for the liquid in the flask to reach a boil. Once boiling, **continue to heat for 10 minutes**, noting that:

- after about 3 minutes of boiling, ensure that no metal-particles are stuck on the upper-walls of the flask. If some are, rinse them down using a small amount of water.
- **the sample must not be allowed to boil dry as the flask will shatter.** If necessary, make up any lost volume by periodic addition of small quantities of distilled water.
- a toxic brown gas of NO_2 is produced by this reaction. **The fumehood's window-sash must be lowered to the point labeled by a red-arrow on the side of the fumehood's window-frame to ensure no inhalation of this gas**.
- all of the steel in the sample will have dissolved after 10 minutes, resulting in a clear yellow or orange solution due to the presence of Fe^{3+}. Manganese is present as the colourless cation Mn^{2+}. Some black particles of carbon may remain undissolved.

6. Sometime during the prior 10 minute boiling-phase, weigh out approximately **1 g** of ammonium persulphate, **$(NH_4)_2S_2O_8$,** into a plastic weigh-boat using a top-loading balance. Do **not** add it to your flask yet. Set this white powder aside at the fumehood, ready for use later on in step #8. *The mass weighed is not recorded on your report sheet*.

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[1] The electrostatic steel particles tend to fly away when transferred dry, hence the need to moisten them.

7. When the steel sample has boiled for 10 minutes, use your crucible-tongs to move the hot flask onto the silver heat-resistant mat that sits next to the hotplate in the fumehood. *Never place hot glassware on the plastic liner in the fumehood, as it will instantly melt!*

8. While still at the fumehood, use a scoopula to **add approximately half** of the previously-weighed **$(NH_4)_2S_2O_8$** into the hot flask. Using crucible-tongs, carefully give the hot flask a quick swirl to mix the contents. *The ensuing chemical reaction causes the solution to bubble or froth-up as the excess nitric acid and nitric oxides are destroyed.*

Once the chemical reaction has calmed, add the remaining **second-half** of ammonium persulphate, $(NH_4)_2S_2O_8$, to the flask and swirl it as before.

Return the flask to the hot-plate and boil gently for 10 minutes. *Check your flask periodically to ensure it doesn't boil dry; add distilled water if needed to prevent this.*

9. Once the boiling of step #8 is over, use crucible-tongs to set the hot flask onto the heat-resistant mat. **Dilute the sample to ~ 100 mL with distilled water**, using the lines on the side of the flask for guidance.

10. Let the flask cool down a bit until you can hold the neck safely with your gloved-hands.

Take the flask to the fumehood where a pump-bottle of **concentrated (85%) phosphoric acid**, **H_3PO_4** is located. Pump **10 mL** of the **H_3PO_4** directly into your flask. *The acid forms a complex with Fe(III), thereby allowing the purple-colour of Mn^{7+} in step #12 to be visible.*

11. Weigh out approximately **0.5 g** of potassium periodate, **KIO_4**, into a plastic weigh-boat at a top-loading balance. *This chemical is also a white powder; the mass is not recorded.*

12. At the fumehood, pour all of the KIO_4 powder into the flask, swirl to mix, and then return the flask to the hot-plate. Wait for the contents to reach a boil. Once at this temperature, **boil the sample for just 1 minute.**

Reduce the heat-setting on the hotplate to the minimum setting and leave the flask there for 10 minutes to heat gently. *The solution will very gradually change to a purple colour as the manganese oxidizes from Mn^{2+} to Mn^{7+}, in the form of MnO_4^-.*

13. At a sink, **cool the purple solution to room temperature** by running a stream of cold water against the outside of the Erlenmeyer flask, with swirling of the contents.

14. **Refer to Figure 2.3** in the prior Titration Experiment # 2.2 (Part 1 – KHP). Apply all of those techniques to transfer the cooled purple MnO_4^- analyte solution quantitatively from the Erlenmeyer flask into a rinsed 250.00 mL volumetric flask.

Dilute carefully to the 250 mL calibration line with distilled water. DO NOT OVERSHOOT! Cap the flask and mix well to ensure a uniform solution. The MnO_4^- analyte is now ready for colorimetric analysis in Part 3.