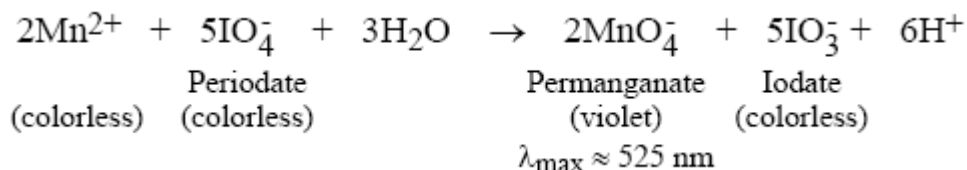


**Lab #6 – Photometric Determination of Mn in Steel using Standard Addition****Introduction:**

Steel Alloys are solid solutions of various metals in iron. These metals must be present in precise amounts in order to achieve the desired strength, hardness, or other properties of the alloy. Many steel alloys contain small amounts of manganese, varying from .1% to 2.0% of the total mass of steel. Manganese can easily be detected by visible absorbance, if it is converted into the purple permanganate ion ( $\text{MnO}_4^-$ ). This ion has a strong absorbance at 525nm.

The procedure given below works for steels that do not have large amounts of chromium. The metal is dissolved in nitric acid, and any carbon present is oxidized with persulfate ions. Interference from iron is eliminated by complexing it with phosphoric acid. The Mn(II) ions are oxidized to permanganate by the following reaction with potassium periodate:



Other metals in the steel may cause interferences that make the use of a calibration curve impractical for this determination. Therefore we will use the method of standard addition to get an accurate measurement of the %Mn in steel.

**Procedure:**

- A standard manganese(II) solution has been prepared to a concentration of ~.1g/L. Be sure to note the exact concentration on the bottle. Throughout the experiment you will find it helpful to keep you concentrations in either g/L or mg/mL (most find the latter to be more convenient).

- *Preparation of the Unknown* - You have been given ~1g of sample, which will allow you to repeat the experiment if necessary.

- Weigh a single sample of ~.5g to the nearest .0001g. No drying is necessary. Place in a 150ml beaker, and add about 50ml of 6M nitric acid. After any violent bubbling subsides, boil gently in the hood for about 10 min.

- Cautiously and slowly add about 1g of ammonium persulfate(a ppt. May appear at this point – don't worry, it will likely dissolve as you boil the solution). Then boil for an additional 10 minutes. If the solution is pink or has a deposit of  $\text{MnO}_2$ , add .1g of  $\text{NaHSO}_3$  and heat for 5 min.

Quantitatively transfer the solution to a 250ml volumetric flask, dilute to volume, and mix well.

Use a 20ml transfer pipet to transfer 3 20ml aliquots to individual 150ml beakers. Number each beaker and prepare as follows:

Aliquot	Volume of 85% H <sub>3</sub> PO <sub>4</sub> , mL	Volume of Std. Mn, mL	KIO <sub>4</sub> , g
1	5	0.00	0.4
2	5	5.00	0.4
3	5	0.00	0.0

Boil each solution for 5 min, cool, and transfer quantitatively to a 50ml volumetric flask. Mix well, then fill to the mark. Use aliquot 3 as the blank (zero the absorbance on the spectrometer with this sample), and measure the absorbance of aliquots 1 and 2. Be sure to rinse the cuvettes with the solution before measuring the absorbance. Use the same cuvette for all 3 samples.

*Calculate the percentage of manganese in the unknown.*