PROTOCOL 3 [CHM112]

- **Determination of Iron in an Ore by Redox titration with potassium dichromate solution**

The percentage of iron in an unknown iron oxide sample needs to be determined. In ore, Fe remains as mixed oxide of Fe(II) and Fe(III). We need to determine the total strength of iron in ore using redox titration with potassium dichromate.

Reagents supplied

Unknown iron ore solution in HCl
Predried primary standard $K_2Cr_2O_7$
Concentrated HCl
$SnCl_2$ solution of 0.125 M
$HgCl_2$ solution
syrupy H_3PO_4
BDS indicator

1. Preparation of Standard sodium dichromate of N/20.
   Dissolve the weighed sample in water

   1.30985

   Calculation

   Table and Calculation

   Strength of permanganate

2. Determine the strength of Fe in ore using dichromate.

   Steps:

   (a). Reduction of Fe(III) to Fe(II)

   The reduction step must be done one trial at a time and the titration of that sample must be done immediately after the reduction. This is because the reduced Fe^{2+} is readily oxidized in air to Fe^{3+}. Note the oxidized form of the iron does not react with $K_2Cr_2O_7$.

   (i) Heat each solution containing the iron sample almost to boiling.
   (ii) Carefully add $SnCl_2$ solution dropwise until the yellow Fe(III) color just disappears. Then add only 2 drops excess of $SnCl_2$ solution. This is to ensure complete reduction of Fe (II).
   (iii) Cool by running the outside of the flask under cold water (until the flask can be comfortably held, <40°C).
   (iv) Next rapidly add 10ml (measured in a graduated cylinder) of the $HgCl_2$ solution to reduce excess Sn(II) which otherwise interfere in redox titration .
   (v) A small quantity of a white precipitate should appear. If no precipitate forms or if the precipitate is gray or black, the trial must be discarded. An absence of precipitate indicates that insufficient $SnCl_2$ was used and the reduction of iron (III) was incomplete. A gray residue indicates the presence of elemental mercury resulting from

the use of too much $SnCl_2$. The sample must be discarded if either of those situations occurs.

Reaction:

$$Sn^{2+} + 2\ Fe^{3+} \rightarrow Sn^{4+} + 2\ Fe^{2+}$$

$$Sn^{2+} + 2\ HgCl_2 \rightarrow Sn^{4+} + Hg_2Cl_2(s) + 2\ Cl^-$$

$$MnO^{4-} + 8\ H^+ + 5\ Fe^{2+} \rightarrow Mn^{2+} + 5\ Fe^{3+} + 4\ H_2O$$

(b). Titration of reduced Fe solution with dichromate

I. After waiting only about 2 to 3 minutes, add to the pre-reduced solution, about 5ml of concentrated sulfuric acid and 7ml of syrupy phosphoric acid (measured in a graduated cylinder).

II. Dilute with distilled water to bring the volume to about 100 ml. Again cool the solution to room temperature by running the outside of the flask under cold water.

III. Add 8 drops of BDS indicator and slowly titrate with your standard $K_2Cr_2O_7$ solution from a blue-green, through a greyish tinge to the first permanent violet, which is the end point. The titration should be conducted dropwise when the grey tinge is noted because the oxidation of the indicator is somewhat slow at this point. Do the trial run first to see the end point and determine the sample weight. Then do three good trials. The average relative deviation should not exceed 2 ppt.

Table and Calculation

Strength of Fe(II)

Write down the precautions you need to use for permanganate as oxidizing agent instead of dichromate and state the reason.