Ferrous Iron Determination

Safety Alert: This method requires the use of concentrated sulfuric and hydrofluoric acids. These acids pose hazards to those using them. Contact with skin or eyes can cause serious burns. Inhaling acid vapors can cause a variety of respiratory and pulmonary ailments. Before beginning this method you first must receive specific authorization, training and supervision from a member of the staff. In addition, the following safety procedures must be followed:

1) To minimize acid vapor hazards all work must be done in a properly functioning fume hood. Please note that one of the five fume hoods in H-105 is the designated FeO titration hood. All the necessary paraphernalia will be found there.

2) To minimize the danger of contact with skin, every effort must be made to avoid having any area of bare skin exposed while working with acids. The nature of street clothing worn while working with acids affects the minimum requirements for additional personal safety equipment. The minimum safety apparel needed for an individual wearing a long sleeve shirt, long pants, socks and closed shoes includes safety glasses or goggles, face shield, gloves and apron. In addition, an individual wearing a short sleeved shirt must wear a sleeved apron/smock to cover bare arms. An individual wearing shorts or a skirt must wear an apron/smock long enough to reach the ankles or an acid resistant jump suit. Individuals wearing sandals or open shoes must wear protective footwear. Required safety apparel is stored in labeled drawers or hangs from hooks in room H-105.

3) Before starting, be sure you know what to do if acid is spilled or comes in contact with your skin or clothing. Know how to use the eye wash sink and HF Burn Kit stored under the fume hood. Report all accidents immediately. Clean up when finished.

Material Safety Data Sheets:
Sulfuric acid: www.fishersci.com/msds
Hydrofluoric acid: www.fishersci.com/msds

A. Ferrous Iron Method- The Details

Safety Warning: Boric acid poses a hazard to skin, eyes, and internal organs. For more information see www.fishersci.com/msds
Potassium dichromate poses fire and serious health hazards. For more information see www.fishersci.com/msds
Phosphoric acid poses a hazard to skin, eyes, and internal organs. For more information see www.fishersci.com/msds
1. Weigh out a 0.5000 +/- 0.0002 grams of sample powder and transfer to a large alumina crucible and cover. Since we have 12 crucibles up to 11 samples and a standard can be analyzed as a batch, taking care not to mix up which sample is in which crucible. Analyze a geochemical rock standard first (or some other rock powder of known FeO concentration) to make sure everything is O.K.

2. Add 5 ml of deionized H2O, 5 ml of concentrated H2SO4, rinse pipette, and 5 ml of HF directly into each alumina crucible; use the pipette and beakers stored in the acid storage cabinet beneath the fume hood. Cover immediately and heat over Meker burner until felt to be boiling.

3. Move covered crucible to hot plate (set at low) and let stand for 10 minutes at very low boil.

4. Add 450 ml of the boric acid solution to an 800 ml plastic beaker, one beaker for each crucible you are analyzing.

5. Add three to five drops of a 3.0% solution of Sodium diphenylamine sulfonate which is an indicator.

6. After the crucible has been heated for 10 minutes, hold down the cover with a stirring rod and pick up the crucible around the outside with a pair of Nichrome tongs; lower the crucible so that it nearly touches the solution in the beaker; tilt the beaker and then let crucible drop so that it falls to the bottom of the beaker and is covered by the solution as quickly as possible. Minimize as much as possible the contact between tongs and boric acid solution.

7. Promptly titrate with a standard solution of K2Cr2O7 (potassium dichromate) to a purple end point. (Unstirred solution left in crucible will be available for mixing if you go past end point).
8. Calculations:

a) % FeO = ml. K2Cr2O7 x 0.8. This value is listed as "FeO" on your chemical analysis summary sheet.

b) To calculate the amount of Fe2O3 in your sample (not Fe2O3T), the computation is as follows:

%Fe2O3T (from whole rock x-ray data) divided by 1.111348 = % FeOT

% FeOT - % FeO (titrated value) = the amount of iron in your sample which really exists as Fe2O3 (ferric iron). This # needs to be multiplied by 1.111348 to re-convert back to the ferric oxide state (Fe2O3) and it should be listed as Fe2O3 on your summary analysis data sheet.

9. Please Note:

a) Note #1: K2Cr2O7 standard solution (2.730 g of K2Cr2O7 in 1 liter of distilled H2O). Due to the light sensitivity of this solution it must always be stored in opaque bottles.

b) Note #2: The boric acid solution is usually made in 15 liter batches. The ingredients are: for each 5 liters of distilled H2O, use 50 grams of H2BO3 (boric acid), 65 ml H3PO4 (phosphoric acid), and 100 ml H2SO4 (sulfuric acid).

c) Note #3: Make sure you detach the plastic pipette tip from the plunger part when you are done titrating and in the process of cleaning up. Rinse tip thoroughly in deionized water.

10. The waste from this procedure is considered hazardous and must be collected and disposed of properly. During the demonstration by your instructor, please note the cupboard beneath the fume hood labeled “Hazardous Waste Storage”. Therein is located a 5 gallon plastic drum in which all the liquid from this technique is gathered. There is a large plastic funnel stored in this same location to facilitate the liquid transfer from the plastic beaker to the large storage container. After emptying the liquid please go to the sink and thoroughly rinse the alumina crucible, the lid, and the beaker in deionized water and set out on paper towels to dry. Lay both the crucibles and beakers on their sides to dry; do not stand them in an upright position.

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