The Flame Photometric Determination of Alkali Metals in Iron and Manganese Ores

Introduction
A PFP7 flame photometer offers a method for the determination of the sodium and potassium content of iron and manganese ores.

Materials Required

Equipment
- Jenway flame photometer
- Accurate balance weighing to 0.0005g
- Volumetric flasks
- Beakers
- Watch glass
- Whatman No. 40 filter papers
- Pestle and Mortar
- Platinum crucibles

Reagents
- Analar Ammonium Chloride
- Analar Calcium Carbonate
- Analar Ammonium Carbonate
- Sodium Standard – 1000ppm (Part number 025 021)
- Potassium Standard – 1000ppm (Part number 025 023)
- Deionised Water

Method

Standard preparation
1. Accurately pipette 10.0ml of the 1000ppm sodium standard and 10.0ml of the 1000ppm potassium standard into a 1.0 L volumetric flask containing 400ml of deionised water.

2. Make up to volume with deionised water. This is a 10ppm sodium and potassium standard solution.

3. From the 10ppm standard solution, prepare 100.0ml of 7.5, 5 and 2.5ppm standard solutions using deionised water as diluent.

Sample preparation
1. Accurately weigh 0.5 g of sample into a mortar.

2. Add 0.5 g of ammonium chloride and 3.0 g of calcium carbonate.

3. Mix thoroughly by grinding.

4. Carefully transfer the mixture to a platinum crucible and heat carefully, using a Bunsen burner, for about ten minutes or until all the ammonia has been removed. Take care to ensure that the crucible does not get hot enough to evolve white fumes of ammonium chloride.

5. When all the ammonia has been evolved, raise the temperature so that the bottom three-quarters of the crucible and no more, are at red heat.
6. Maintain this position for one hour and then allow the crucible to cool.

7. Transfer the sintered cake of material from the crucible to a beaker with deionised water.

8. Continue to add deionised water to a volume of approximately 100ml.

9. Cover the beaker with a watch glass, and boil to disintegrate the solid cake of material and to ensure solution of its soluble constituents.

10. Remove the beaker from the heat and carefully add sufficient powdered ammonium carbonate to precipitate all the calcium in the solution.

11. Bring back to the boil, continue boiling until all the ammonia has been evolved and then allow the precipitate to settle.

12. Decant and filter the solution through a Whatman No. 40 filter paper into a second beaker and wash the paper free of soluble salts with hot deionised water.

13. Carefully evaporate the filtrate down to about 30ml and allow it to cool.

14. Filter the cold solution through a Whatman No. 40 filter paper into a 100.0ml volumetric flask, rinse the filter paper and dilute to volume with deionised water.

Method

1. Set up the flame photometer as detailed in the instruction manual.

2. Aspirate deionised water and set the zero.

3. Aspirate the standards into the flame photometer.

4. Plot a standard curve of sodium/potassium concentration against intensity.

5. Aspirate the sample solution into the flame and record the reading. If the reading is above the value recorded for the 10ppm standard, dilute the sample until the value is within the range of the calibration curve.

Calculation

Using the calibration graph, determine the concentration of the sample from the recorded reading. If required, multiply the determined concentration by the dilution factor.

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\text{Sodium content of ore (\%)} = \frac{\text{sample (ppm)} \times 100 \times 100}{1000000 \times \text{sample weight (g)}}
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\text{Potassium content of ore (\%)} = \frac{\text{sample (ppm)} \times 100 \times 100}{1000000 \times \text{sample weight (g)}}
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