1.0 Purpose

1.1 To verify that the iron content of magnesium sulfate heptahydrate is below the USP limit of 20 ppm.

2.0 Scope

2.1 USP Monograph: Magnesium Sulfate, and General Chapter <241>

3.0 Responsibility

3.1 Quality Associate is responsible for this procedure.

4.0 Safety Considerations

4.1 Wear safety glasses, lab coat, and nitrile gloves. Mixing of chemicals shall be performed in the hood. Safety is a condition of employment. Employees are not authorized to work in an unsafe manner and are prohibited from harming the environment of the facility or community.

5.0 Materials/Equipment

5.1 Mortar and Pestle
5.2 2 50-mL Nessler Low Form Color Comparison Tubes
5.3 Stir Rod or Spatula (long enough for color comparison tubes)
5.4 Balance-Mettler Toledo X5105Du, B13929Z316
5.5 Weigh Paper
5.6 Spatula
5.7 1000-µL Eppendorf Pipette and Tips
5.8 5-mL Eppendorf Pipette and Tips
5.9 10-mL Eppendorf Pipette and Tips
5.10 100-mL Beaker
5.11 2 100-mL Volumetric Flasks with Stoppers
5.12 1000-mL Volumetric Flask with Stopper
5.13 White Sheet of Paper

Reagents:

5.14 Ferric Ammonium Sulfate (Dodecahydrate)
5.15 2 N Sulfuric Acid Solution
5.16 30% Ammonium Thiocyanate Solution
5.17 Concentrated Hydrochloric Acid
5.18 Ammonium Peroxydisulfate

Solutions Preparation:

Standard Iron Solution– Dissolve 0.8634 g of ferric ammonium sulfate dodecahydrate in water *(NOTE: ferric ammonium sulfate dodecahydrate come in large crystals that will have to be ground with a mortar and pestle before weighing. This grinding should be performed gently as frictional heating can cause ferric ammonium sulfate dodecahydrate to melt becoming an orange-brown liquid*), add 10 mL of 2 N sulfuric acid, and dilute with water to 100.0 mL in a clean 100-mL volumetric flask. Pipet 10 mL of this solution into a 1000-mL volumetric flask, add 10 mL of 2 N sulfuric acid, dilute with water to volume, and mix. This solution contains the equivalent of 0.01 mg (10 µg) of iron per mL.

Standard Preparation– Into a 50-mL color-comparison tube pipet 1 mL of Standard Iron Solution (10 µg of Fe), dilute with water to 40 mL, add 2 mL of hydrochloric acid, and mix.

Test Preparation– In a 50-mL color comparison tube dissolve 0.50 g of the magnesium sulfate sample (for magnesium sulfate solution use 1.00 mL) in 40 mL of water. Add 2 mL of hydrochloric acid, and mix.

6.0 Procedure

6.1 Into each of the tubes containing the Standard Preparation and the Test Preparation add 0.050 g of ammonium peroxydisulfate crystals and 3 mL of Ammonium Thiocyanate Solution.

6.2 Dilute with DI H_2O to the 50 ml mark, and mix.

6.3 View downward over white sheet of paper.

If the color of the solution from the Test Preparation is not darker than that of the solution from the Standard Preparation then the iron content of the magnesium sulfate heptahydrate sample is below the USP limit of 20 ppm (or 10 ppm for magnesium sulfate solution).

7.0 Reference Documents

7.1 Laboratory Notebook
7.2 *USP Stability Testing Summary Worksheet* Q12-PR-100-F010

8.0 Amendment Record

Revision Number	Revision Date	Revision Author	Revision Description
0	04/10/12	SB	New Document