

GILES CHEMICAL ~ PREMIER MAGNESIA

Company Procedure

Title: USP Iron: Magnesium Sulfate Number: L12-PR-100-005

Owner: John Safi Revision: 2
Effective Date: 03/31/16 Page: 1 of 3



1.0 Purpose

To describe how to verify that the iron content of magnesium sulfate heptahydrate is below the USP limit of 20ppm following USP Monograph: Magnesium Sulfate, and General Chapter <241>.

2.0 Scope

This procedure applies to USP lot change, stability testing, and any time USP quality needs to be verified. All USP testing is performed in the Quality Assurance laboratory.

3.0 Responsibility

QA Lab personnel are responsible for USP testing.

4.0 Safety Considerations

Wear safety glasses, lab coat, and nitrile gloves. When specified, mixing of chemicals shall be performed in the fume 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

- Mortar and Pestle
- 2 50 ml Nessler Low Form Color Comparison Tubes
- Stir Rod or Spatula (long enough for color comparison tubes)
- Balance-Mettler Toledo X5105Du, B13929Z316
- Weigh Paper
- Spatula
- 1000 µL Eppendorf Pipette and Tips
- 5 ml Eppendorf Pipette and Tips
- 10 ml Eppendorf Pipette and Tips
- 100 ml Beaker
- 2 100 ml Volumetric Flasks with Stoppers
- 1000 ml Volumetric Flask with Stopper
- White Sheet of Paper

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Reagents:

- Ferric Ammonium Sulfate (Dodecahydrate)
- 2N Sulfuric Acid Solution
- 30% Ammonium Thiocyanate Solution
- Concentrated Hydrochloric Acid
- Ammonium Peroxydisulfate

6.0 Procedure

Solutions Preparation

- 1. 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 2N sulfuric acid, and dilute with water to 100 ml in a clean 100 ml volumetric flask. Pipet 10 ml of this solution into a 1000mL volumetric flask, add 10 ml of 2N sulfuric acid, dilute with water to volume, and mix. This solution contains the equivalent of 0.01 mg (10 μg) of iron per ml.
- 2. <u>Standard Preparation</u> 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.
- 3. <u>Test Preparation</u> 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.

Test Procedure

- 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.
- 2. Dilute with DI H₂O to the 50 ml mark, and mix.
- 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).

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7.0 Reference Documents

N/A

8.0 Change Information

Updated using SOP Template Instructions (Q12-PR-100-004) and Document Numbering (Q12-PR-100-003)