1.0 Purpose

To describe how to prepare magnesium sulfate heptahydrate samples for Giles method L13-PR-100-057, *USP ICP-OES Analysis*.

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 sample preparation.

4.0 Safety Considerations

Safety Goggles, Chemical Resistant Gloves, and Lab Coat should be worn.

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

- Balance-Mettler Toledo X5105Du, B13929Z316
- Weigh Paper
- Eppendorf 1000-µl Adjustable Pipette
- Eppendorf 5-ml Adjustable Pipette
- 2000-ml Class A Volumetric Flask
- 1 x 5000-mL Reagent Carboys
- 15-ml Metal Free Centrifuge Tubes (One Per Sample)
- 44 Position Rack for 15-ml Autosampler Tubes

Reagents:

- Nitric Acid, 70%, High Purity, Trace Metals
- Deionized H_2O (ASTM Type II or Better)
- Gallium, 1000 µg/ml ICP Standard in 5% Nitric Acid-Teledyne Leeman
- Yttrium, 1000 µg/ml ICP Standard in 5% Nitric Acid-Teledyne Leeman

6.0 Procedure

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Solutions Preparation:

1. Internal Standard Working Solution—Using a clean 100-ml graduated cylinder, add 57 ml of 70% Nitric Acid to 1000 ml of DI H_2O in a clean 2000-ml class A volumetric flask. Using a 100-ml beaker and the analytical balance, weigh out 61.02 g of gallium standard, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Using a 100-ml beaker and the analytical balance, weigh out 10.17 g of yttrium standard, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Dilute to volume, and mix. Store this solution in a 5000-ml reagent carboy. This Internal Standard Working Solution is 30 ppm gallium and 5 ppm yttrium in 2% nitric acid.

Sample Preparation Procedure:

1. Label a centrifuge tube for each sample to be tested, and place them in order in the 44 position rack.
2. Remove the caps from the centrifuge tubes.
3. Weigh out 3.00 g of each sample into its respective centrifuge tube (for magnesium sulfate solution us 1.00 ml).
4. To each sample add 0.43 ml of 70% nitric acid.
5. Add 1.50 ml of Internal Standard Working Solution to each sample.
6. Add DI H_2O to each centrifuge tube up to the 15-ml mark.
7. Place the caps back onto the centrifuge tubes.
8. Dissolve the samples by shaking them, or using a vortex mixer.

7.0 Reference Documents

USP ICP-OES Analysis (L13-PR-100-057)

8.0 Change Information

New Document

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