1. **­Purpose**

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

1. **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.

1. **Responsibility**

QA Lab personnel are responsible for USP sample preparation.

1. **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.

1. **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 H2O (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

1. **Procedure**

**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 H2O 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 H2O 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.
9. **Reference Documents**

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

1. **Change Information**

New Document