
	GILES CHEMICAL ~ PREMIER MAGNESIA		
	Company Procedure		
	Title: USP ICP-OES Sample Preparation	Number: L13-PR-100-058	
	Owner: John Safi	Revision: 1	
	Effective Date: 05/01/17	Page: 1 of 2	

## 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, B139292316
- 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

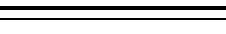
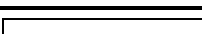
### Reagents:

- Nitric Acid, 70%, High Purity, Trace Metals
- Deionized H<sub>2</sub>O (ASTM Type II or Better)
- Gallium, 1000 µg/ml ICP Standard in 5% Nitric Acid
- Yttrium, 1000 µg/ml ICP Standard in 5% Nitric Acid

## 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<sub>2</sub>O 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<sub>2</sub>O 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

Updated SOP to current Doc System format  
Changed Owner

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