
	GILES CHEMICAL ~ PREMIER MAGNESIA		
	Company Procedure		
	Title: USP Identification of Magnesium Sulfate	Number: L12-PR-100-001	
	Owner: Stephen Ballew	Revision: 1	
	Effective Date: 03/31/13	Page: 1 of 3	

1.0 Purpose

To describe how to identify that magnesium and sulfate are present following USP Monograph: Magnesium Sulfate, and General Chapter <191>.

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

Equipment:



- Balance – Mettler Toledo X5105Du, B13929Z316
- Weigh Paper
- 3 - 15 ml Centrifuge Tubes (Labeled as Tubes 1, 2, and 3)
- 3 - Glass Stir Rods
- Test Tube Rack
- Centrifuge – Drucker 614B
- 3 – 100 ml Volumetric Flasks
- 6 – 100 ml Bottles with Dropper Caps
- 5 ml Eppendorf Pipette and Tip

Reagents:

- Ammonium Chloride
- Dibasic Sodium Phosphate

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- DI H₂O (in Wash Bottle)
- Ammonium Carbonate TS (in Bottle with Dropper Cap)
- Barium Chloride TS (in Bottle with Dropper Cap)
- 6N Ammonium Hydroxide (in Bottle with Dropper Cap)
- Concentrated Hydrochloric Acid (in Bottle with Dropper Cap)

6.0 Procedure

Solutions Preparation:



1. Ammonium Chloride TS— Dissolve 10.5 g of ammonium chloride in DI H₂O and dilute to 100 ml in a 100 ml volumetric flask. Store this solution in a 100 ml bottle with dropper cap.
2. Dibasic Sodium Phosphate TS— Dissolve 12 g of dibasic sodium phosphate in DI H₂O and dilute to 100 ml in a 100 ml volumetric flask. Store this solution in a 100 ml bottle with dropper cap.
3. Test Solution— Dissolve 5.00 g of magnesium sulfate heptahydrate sample in DI H₂O (for magnesium sulfate solution use 25 ml) and dilute to 100 ml in a 100 ml volumetric flask.

Test Procedure

1. Thoroughly clean and dry centrifuge tubes and stir rods with a final rinse before drying in DI H₂O. Place in test tube rack.
2. Add three drops of *Test Solution* into each centrifuge tube. **NOTE:** The rest of the *Test Solution* can be saved for the USP pH test.
3. To centrifuge tube 1 add three drops of *Ammonium Chloride TS* and three drops of *Ammonium Carbonate TS* and stir. No more than a slightly hazy precipitant should form.
4. To the same tube add three drops of *Dibasic Sodium Phosphate TS* and stir. A white crystalline precipitate should form. If it does not, no magnesium is present. **NOTE: The confirmation of magnesium will be performed on this precipitate, so save this centrifuge tube with its contents.**
5. To the centrifuge tube 2 add three drops of concentrated hydrochloric acid and mix. This test is for thiosulfate which is an interferent in the sulfate test. If no precipitate forms then no thiosulfate is present. The contents of centrifuge tube 2 can be disposed of and the tube and stir rod washed and put away.

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6. To centrifuge tube 3 add three drops of *Barium Chloride TS* and stir. A white precipitate should form. If it does not, no sulfate is present. **NOTE: The confirmation of sulfate will be performed on this precipitate, so save this centrifuge tube with its contents.**
7. Add 6 drops of DI H₂O to centrifuge tube 3.
8. Place centrifuge tube 1 and centrifuge tube 3 opposite each other in the centrifuge.
9. Centrifuge for five minutes.
10. Remove the tubes from the centrifuge and pour off the liquid supernatants. **NOTE: All of the contents of centrifuge tube 3 should be poured into a properly labeled barium waste container.**
11. To the separated precipitate in centrifuge tube 1 add 3 drops of 6N ammonium hydroxide and stir. If the separated precipitate is not soluble in 6N ammonium hydroxide then the presence of magnesium in the sample has been confirmed.
12. To the separated precipitate in centrifuge tube 3 add 3 drops of concentrated hydrochloric acid and stir. If the separated precipitate is not soluble in concentrated hydrochloric acid then the presence of sulfate in the sample has been confirmed. **NOTE: All of the contents of centrifuge tube 3 should be poured into a properly labeled barium waste container.**

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)*

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