
	GILES CHEMICAL ~ PREMIER MAGNESIA		
	Company Procedure		
	Title: USP Assay: Magnesium Sulfate	Number: L12-PR-100-008	
	Owner: Stephen Ballew	Revision: 1	
	Effective Date: 03/31/13	Page: 1 of 3	

1.0 Purpose

To describe how to determine % MgSO₄ in Magnesium Sulfate following USP Monograph: Magnesium Sulfate.

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
- Weigh Boat
- Spatula
- Mortar and Pestle
- pH Meter
- 1000 ml Volumetric Flask
- 250 ml Erlenmeyer Flask
- Stir bar
- Stir plate
- Class A 50 ml burette
- Burette Stand
- 1000 µL Eppendorf Pipette and Tips
- 5 ml Eppendorf Pipette and Tips

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Reagents:

- Ammonium Chloride
- Concentrated Ammonium Hydroxide Solution
- DI H₂O
- Eriochrome Black TS
- 0.05M Edetate Disodium (Disodium EDTA) Volumetric Solution
- 3N Hydrochloric Acid Solution (If Needed)
- 1N Sodium Hydroxide Solution (If Needed)

6.0 Procedure

Solutions Preparation:



1. Ammonium – Ammonium Chloride Buffer Test Solution– Dissolve 67.5 g of ammonium chloride in water, add 570 ml of ammonium hydroxide, dilute with DI H₂O to 1000 ml.

Test Procedure

1. Weigh out approximately 0.3 g of magnesium sulfate from Loss on Ignition test into mortar.
2. Grind ignited Magnesium Sulfate with pestle into a fine powder.
3. Place weigh paper on balance and tare balance.
4. Weigh out 0.250 g ground Magnesium Sulfate. *NOTE: This needs to be done quickly because the sample is hygroscopic.*
5. Transfer weighed sample into 250 ml Erlenmeyer flask.
6. Dissolve sample in 100 ml of DI H₂O.
7. If sample solution is not clear, add minimum amount of 3N hydrochloric acid required to make a clear solution.
8. Adjust sample solution, if needed, with 1 N sodium hydroxide to a pH of 7.
9. Using 5 ml Eppendorf pipette, add 5 ml of ammonium-ammonium chloride buffer TS.
10. Using 1000 µL Eppendorf pipette add 150 µL of erichrome black TS. Swirl flask for five seconds to mix sample solution.
11. Fill 50 ml volumetric burette with 0.05M Edetate Disodium Volumetric Solution.
12. Record initial volume mark. For example, the initial volume mark is 2.5 ml.
13. Titrate with 0.05M Edetate Disodium Volumetric Solution until blue endpoint is reached (purple to blue). Swirl flask continuously while titrating.
14. Record the final volume mark of the solution in the burette. For example, the final volume mark is 26.4 ml.
15. Subtract the initial volume mark (Step 12) from the final volume mark (Step 14) to calculate the volume of 0.05M Edetate Disodium Volumetric Solution used for the titration. In this example, the volume is 26.4 ml - 2.5 ml = 23.9 ml. Each ml of 0.05 M Edetate Disodium Volumetric Solution is equivalent to 6.018 mg MgSO₄.

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The % of MgSO₄ in sample solution is calculated using the following formula:

$$\frac{(\text{ml Edetate Disodium Volumetric Solution}) \times (6.018 \text{ mg of MgSO}_4)}{250 \text{ mg ignited Magnesium Sulfate}} \times 100 = \% \text{ MgSO}_4$$

Specifications: 99.0%-100.50%

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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