1.0 Purpose

1.1 To determine % $MgSO_4$ in Magnesium Sulfate.

2.0 Scope

2.1 USP Monograph: Magnesium Sulfate

3.0 Responsibility

3.1 Lab Personnel

4.0 Safety Considerations

4.1 Wear safety glasses, lab coat, and nitrile gloves. Mixing of chemicals shall be performed in the hood.

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

Reagents:

- Ammonium Chloride
- Concentrated Ammonium Hydroxide Solution
- DI H_20
- Eriochrome Black TS
- 0.05 M Edetate Disodium (Disodium EDTA) Volumetric Solution
- 3 N Hydrochloric Acid Solution (If Needed)

- 1 N Sodium Hydroxide Solution (If Needed)

Solutions Preparation:

Ammonium – Ammonium Chloride Buffer Test Solution– Dissolve 67.5 g of ammonium chloride in water, add 570 ml of ammonium hydroxide, dilute with DI H_2O to 1000 ml.

6.0 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 H_20.
7. If sample solution is not clear, add minimum amount of 3 N 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 0.05 M Edetate Disodium Volumetric Solution.
12. Record initial volume mark. For example, the initial volume mark is 2.5 ml.
13. Titrate with 0.05 M 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 0.05 M 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 $MgSO_4$.

The % of $MgSO_4$ in sample solution is calculated using the following formula:

$$\frac{\text{(ml Edetate Disodium Volumetric Solution)} \times \text{(6.018 mg of } MgSO_4\text{)}}{\text{250 mg ignited Magnesium Sulfate}} \times 100 = \%\ MgSO_4$$

Specifications: 99.0%-100.50%

7.0 Reference Documents

7.1 Laboratory Notebook
7.2 *USP Stability Testing Summary Worksheet* Q12-PR-100-F010

8.0 Amendment Record

Revision Number	Revision Date	Revision Author	Revision Description
0	03/09/12	SB	New Document