
	GILES CHEMICAL ~ PREMIER MAGNESIA		
	Company Procedure		
	Title: USP Heavy Metals	Number: L12-PR-100-006	
	Owner: Stephen Ballew	Revision: 0	
	Effective Date: 04/10/12	Page: 1 of 3	

1.0 Purpose

- 1.1 To demonstrate that the content of metallic impurities that are colored by sulfide ion, under specified test conditions, do not exceed the heavy metals limit of 0.001% (w/w) of lead in magnesium sulfate.

2.0 Scope

- 2.1 USP Monograph: Magnesium Sulfate, and General Chapter <231> Method I

3.0 Responsibility

- 3.1 Quality Associate is responsible for this procedure.

4.0 Safety Considerations



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

- 5.1 3 50-mL Nessler Low Form Color Comparison Tubes
- 5.2 3 Stir Rods (long enough for color comparison tubes)
- 5.3 3 Centrifuge Tubes
- 5.4 Balance – Mettler Toledo X5105Du, B13929Z316
- 5.5 Weigh Paper
- 5.6 Spatula
- 5.7 1000-μL Eppendorf Pipette and Tips
- 5.8 5-mL Eppendorf Pipette and Tips
- 5.9 2 100-mL Volumetric Flasks with Stoppers
- 5.10 1000-ml Volumetric Flask with Stopper
- 5.11 250-mL Beaker
- 5.12 Hot Plate
- 5.13 Timer
- 5.14 White Sheet of Paper

Reagents:

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- 5.15 Lead Nitrate
- 5.16 Thioacetamide TS, 4% (w/v) Aqueous Solution
- 5.17 Glycerin Base TS
- 5.18 Nitric Acid
- 5.19 Ammonium Acetate
- 5.20 6 N Hydrochloric Acid
- 5.21 1 N Acetic Acid
- 5.22 6 N Ammonium Hydroxide

Solutions Preparation:

Lead Nitrate Stock Solution– Dissolve 0.1598 g of lead nitrate in 100 ml of water to which 1 ml Nitric Acid has been added in a clean 1000-ml volumetric flask and fill to volume with DI H₂O. Prepare and store this solution in glass that is free from soluble lead salts.



Standard Lead Solution– On the day of use, add 10.0 ml of *Lead Nitrate Stock Solution* to a clean 100-ml volumetric flask and fill to volume with DI H₂O. Each ml of Standard Lead Solution contains the equivalent of 10 µg of lead. A comparison solution prepared on the basis of 100 µg of *Standard Lead Solution* per g of substance being tested contains the equivalent of 1 part of lead per million parts of substance being tested.

pH 3.5 Acetate Buffer– Dissolve 25.0 g of ammonium acetate in 25 ml of water in a clean 100-ml volumetric flask. Add 38.0 ml of 6 N hydrochloric acid. Adjust, if necessary, with 6 N ammonium hydroxide or 6 N hydrochloric acid to a pH of 3.5. Fill to volume with DI H₂O, and mix.

Standard Preparation– Into a 50-ml color-comparison tube pipette 2 ml of *Standard Lead Solution* (20 µg of Pb) and dilute with water to 25 ml. Using a pH meter or short-range pH indicator paper as external indicator, adjust with 1 N acetic acid or 6 N ammonium hydroxide to a pH between 3.0 and 4.0. Dilute with water to 40 ml, and mix.

Test Preparation– In a second 50-mL color-comparison tube, dissolve 2 g of the Magnesium Sulfate Heptahydrate sample (for magnesium sulfate solution use 4 mL) in 25 ml of water. Using a pH meter or short-range pH indicator paper as external indicator, adjust with 1 N acetic acid or 6 N ammonium hydroxide to a pH between 3.0 and 4.0, dilute with water to 40 ml, and mix.

Monitor Preparation– Into a third 50-ml color-comparison tube dissolve 2 g of the Magnesium Sulfate Heptahydrate sample in 25 ml of water and add 2.0 ml of *Standard Lead Solution*. Using a pH meter or short-range pH indicator paper as external indicator, adjust with 1 N acetic acid or 6 N ammonium hydroxide to a pH between 3.0 and 4.0, dilute with water to 40 ml, and mix.

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Thioacetamide-Glycerin Base Test Solution– (CAUTION : thioacetamide is a carcinogen) This will need to be done three times in three different centrifuge tubes at the same time. Mix 0.2 ml of thioacetamide TS and 1 ml of glycerin base TS. Heat the mixture in a boiling water bath (250-mL beaker) for 20 seconds and use immediately.

5.15

6.0 Procedure

6.1 To each of the three tubes containing the Standard Preparation, the Test Preparation, and the Monitor Preparation, add 2 ml of pH 3.5 Acetate Buffer.

6.2 Add 1.2 ml of Thioacetamide-glycerin Base TS to each of the three tubes and dilute with DI H₂O to the 50 ml mark. Mix and allow to stand for 2 minutes.

6.3 View downward over white sheet of paper.

6.4 If the color of the solution from the Monitor Preparation is lighter than that of the solution from the Standard Preparation then this method is not valid for this sample and Method II (USP 34) must be used. If the color of the solution from the Test Preparation is not darker than that of the solution from the Standard Preparation then the content of metallic impurities does not exceed the heavy metals limit of 0.001% (w/w) of lead in the magnesium sulfate heptahydrate sample (or 5 ppm (w/v) for magnesium sulfate solution). **NOTE: Dispose of all waste in a properly labeled waste container.**

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	04/10/12	SB	New Document