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

Personnel responsible:
Lab

Purpose:
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.

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

Equipment:

- 3 50-mL Nessler Low Form Color Comparison Tubes
- 3 Stir Rods (long enough for color comparison tubes)
- 3 Centrifuge Tubes
- Balance – Mettler Toledo X5105Du, B13929Z316
- Weigh Paper
- Spatula
- 1000-µL Eppendorf Pipette and Tips
- 5-mL Eppendorf Pipette and Tips
- 2 100-mL Volumetric Flasks with Stoppers
- 1000-ml Volumetric Flask with Stopper
- 250-mL Beaker
- Hot Plate
- Timer
- White Sheet of Paper

Reagents:

- Lead Nitrate
- Thioacetamide TS, 4% (w/v) Aqueous Solution
- Glycerin Base TS
- Nitric Acid
- Ammonium Acetate
- 6 N Hydrochloric Acid
- 1 N Acetic Acid
- 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 H_20. 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 H_20. 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 H_20, 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.

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.

Procedure:

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.
2) Add 1.2 ml of *Thioacetamide-glycerin Base TS* to each of the three tubes and dilute with DI H_2O to the 50 ml mark. Mix and allow to stand for 2 minutes.
3) View downward over white sheet of paper.

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 that 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.**

Revision Number	Revision Date	Effective Date	Revision Author	Quality Approval	Production Approval	Revision Description
00	04/10/2012	04/10/2012	Stephen Ballew	Deborah Durbin	Jason Bumgarner	New Document