

GILES CHEMICAL CORPORATION		
COMPANY PROCEDURE		
Standard Operating Procedure	Page : 1 of 2	Revision : Date : 3/28/06
Reviewed: Carl Mooney	Title: ASSAY – % MgSO <sub>4</sub> in MgSO <sub>4</sub> ·7H <sub>2</sub> O	

QA-LAB-13

**Safety:** At all times wear the appropriate lab PPE.

**Purpose:** ASSAY – % MgSO<sub>4</sub> in MgSO<sub>4</sub>·7H<sub>2</sub>O

## Procedure:

### Introduction:

Occasionally a customer requests analysis of the crystal producer for “Assay” as listed in the US Pharmacopoeia. The laboratory electronic scale is not sufficiently delicate to weigh the specified amount of salt to conform precisely to the USP method. This section outlines a small but necessary modification to the USP procedure, thus allowing the determination to be performed

### Procedure

A sample weighing ten times the USP specified amount is carefully weighed and dissolved. in DI water in a one liter volumetric flask. A one-tenth portion of the dissolution is then obtained by filling a 100 ml. volumetric flask from the liter flask, and used for EDTA titration, thus measuring the amount of MgSO<sub>4</sub> present.

### Equipment

Use similar equipment as in Determination of %MgSO<sub>4</sub> by EDTA Titration  
100 ml. porcelain crucible.

### Method

1. Place a 100 ml. porcelain crucible on the scale and tare the scale to zero.
2. Add about seven grams of the heptahydrate sample to the crucible.
3. Place the crucible in the 100 °C oven for two hours.
4. Transfer the crucible to the muffle furnace to constant weight, app. one hour.
5. Place the crucible in the dessicator until cool.
6. Place the paper on the scale and tare the scale to 0.
7. Carefully weigh out exactly 2.50 grams of the dessicated sample.
8. Using a dry funnel transfer the weighed sample from the paper to a one liter volumetric flask.  
Rinse the funnel with DI water and add DI water to the flask to about 2/3 full.
9. Swirl the contents of the flask until the salt is completely dissolved.
10. Add DI water to the flask to the mark and invert a few times to mix well.
11. Using a 150 ml. beaker transfer some of the solute to a 100 ml volumetric flask full to the mark.
12. Pour the 100 ml. into a 225 ml. Erlenmeyer flask for titration. Rinse the 100 ml. flask with a few ml. of DI water two times.
13. Using a 2.5 ml. disposable pipette add 5 ml. of ammonium chloride hydroxide buffer, then add 5 drops of Eriochrome Black TS indicator to the contents of the Erlenmeyer flask
14. Titrate to blue end-point with 0.1 N EDTA.

$$\text{ml. EDTA} \times 6.018 \times 2 = \text{grams MgSO}_4$$

$$\text{grams MgSO}_4 \div 0.250 = \text{Assay (Should be 99.0 – 100.5)}$$

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TRAINING DOCUMENTATION

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