

GILES CHEMICAL CORPORATION		
COMPANY PROCEDURE		
Standard Operating Procedure	Page : 1 of 3	Revision : Date : 3/27/06
Reviewed: Carl Mooney	Title: Determination of % MgSO ₄ by EDTA Titration	

QA-LAB-07

Safety: Wear the appropriate safety PPE when working in the lab.

Purpose: Determination of % MgSO₄ by EDTA Titration

Procedure:

Background Information :

The magnesium sulfate that is manufactured at this facility is derived from the reaction of roasted magnesite ore and sulfuric acid to yield magnesium sulfate in aqueous solution. Some customers do not require a dry product for their purpose: therefore, it is convenient to forego the crystallization and drying processes and ship the product in liquid form. Concentrations of 18, 21, 24, and 27% are among the most commonly requested. Therefore, it becomes necessary to routinely monitor the daily shipments of the liquid in terms of concentration of magnesium sulfate. The following procedure accomplishes this objective.

Scope :

A suitable sample of the liquid product is weighed, prepared for titration and titrated with a standard solution of EDTA ((ethylenedinitrilo)tetraacetic acid). By measuring the volume of titrant used to reach the end point, the strength of the solution in terms of % MgSO₄ is calculated.

Equipment :

250-mL Erlenmeyer flask
 100-mL burette with stand
 Weighing Balance -- B440 Sartorius
 1 - mL volumetric pipette
 10 - mL volumetric pipette
 2 1000 - mL volumetric flasks
 100 - mL volumetric flask
 Pipette bulb
 EDTA, disodium salt, dihydrate -- J.T. Baker
 Ammonium Hydroxide AR app. 58 % -- Mallinckrodt
 Ammonium Chloride TAC USP granular -- Mallinckrodt
 Standard Solution -- 0.10 M EDTA
 Standard Solution -- Eriochrome Black TS indicator
 Standard Solution -- Ammonium Chloride - Hydroxide buffer
 Magnetic Stirring Plate -- Thermoline Cimarec 2
 Magnetic Stirring Bars -- 1 1/2 - 2 " length
 De-ionized water -- Stephens Scientific
 Sheet of 8 1/2" x 11" office letter paper

GILES CHEMICAL CORPORATION		
COMPANY PROCEDURE		
Standard Operating Procedure	Page : 2 of 3	Revision : Date : 3/27/06
Reviewed: Carl Mooney	Title: Determination of % MgSO ₄ by EDTA Titration	

QA-LAB-07

Procedures :

1. A clean, dry Erlenmeyer flask is placed on the weighing balance and it is tarred to zero.
2. Using a 1-mL pipette, about that volume of the sample is transferred to the flask and the weight is recorded.
3. 100 mL of de-ionized water is added to the flask
4. Using a 10-mL pipette, **exactly** that volume of standard Ammonium Chloride-Hydroxide buffer is then transferred and added to the contents of the flask.
5. Five (5) drops of Eriochrome Black TS indicator is added.
6. Swirl to mix
7. The graduated burette is filled with the 0.10 M EDTA standard solution to the 100 - mL mark.
8. Titrate to a blue end point.

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

$$\text{mL of EDTA solution} \times 1.2036 / (\text{weight of sample}) = \% \text{MgSO}_4$$

Preparation of Standard Solutions

EDTA

Weigh 0.10 moles (37.22g) EDTA (disodium salt and dihydrate crystal) on the balance and dissolve in about 700 mL of de-ionized water in a 1000-mL beaker. Agitate with mechanical stirring to hasten dissolution. When EDTA has completely dissolved transfer to a 1000-mL volumetric flask. Fill to mark with de-ionized water. Store in a sealable plastic container.

Ammonium Chloride-Hydroxide Buffer Solution

Weigh 33.75g ammonium chloride (NH₄Cl) (anhydrous) and dissolve in about 500 mL of de-ionized water in a 1000-mL volumetric flask. When the NH₄Cl is dissolved add 285 mL of ammonium hydroxide. Fill flask to mark with de-ionized water. Transfer to sealable plastic container for storage until needed.

Eriochrome Black TS Indicator

Weigh 0.5 grams Eriochrome Black TS and dissolve in about 50 mL of de-ionized water in a 100-mL volumetric flask. Swirl to mix. When dissolved, fill to mark with de-ionized water.

GILES CHEMICAL CORPORATION			
COMPANY PROCEDURE			
Standard Operating Procedure		Page : 3 of 3	Revision : Date : 3/27/06
	Reviewed: Carl Mooney Title: Determination of % MgSO ₄ by EDTA Titration		

QA-LAB-07

TRAINING DOCUMENTATION

	EMPLOYEE	TITLE	SIGNATURE	DATE
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				