Assay for magnesium carbonate—Transfer an accurately weighed portion of Magnesium Carbonate and Sodium Bicarbonate for Oral Suspension, equivalent to about 4.2 g of $MgCO_3$, to a 500-mL volumetric flask. Add 200 mL of 1 N hydrochloric acid, and mix. When dissolved, dilute with water to volume, and mix. Transfer 10.0 mL of this stock solution to a suitable container, dilute with water to 100 mL, add 10 mL of ammonia–ammonium chloride buffer TS, 5 mL of triethanolamine, and 0.3 mL of eriochrome black TS, and titrate with 0.05 M edetate disodium VS to a blue endpoint. Each mL of 0.05 M edetate disodium consumed is equivalent to 4.216 mg of $MgCO_3$.

Assay for sodium bicarbonate—

Standard preparations—Dissolve a suitable quantity of sodium chloride, previously dried at 125° for 30 minutes and accurately weighed, in water, and dilute quantitatively with water to obtain a solution having a known concentration of about 600 µg per mL. On the day of use, further dilute this solution quantitatively with water to obtain three solutions containing 6.0, 12.0, and 18.0 µg of sodium chloride per mL, respectively.

Assay preparation—Transfer an accurately measured volume of the stock solution remaining from the *Assay for magnesium carbonate*, equivalent to about 180 mg of $NaHCO_3$, to a 100-mL volumetric flask, dilute with water to volume, and mix. Transfer 10.0 mL of the resulting solution to a 1000-mL volumetric flask, dilute with water to volume, and mix.

Procedure—Concomitantly determine the absorbances of the *Standard preparations* and the *Assay preparation* at the sodium emission line at about 589.0 nm, with a suitable atomic absorption spectrophotometer (see *Spectrophotometry and Light-scattering* ⟨851⟩) equipped with a sodium hollow-cathode lamp and an air–acetylene flame, using water as the blank. Plot the absorbances of the *Standard preparations* versus concentration, in µg of sodium chloride per mL, and draw the straight line best fitting the three plotted points. From the graph so obtained, determine the concentration, in µg per mL, of sodium chloride equivalent in the *Assay preparation*. Calculate the quantity, in g, of $NaHCO_3$ in the portion of Magnesium Carbonate and Sodium Bicarbonate for Oral Suspension taken by the formula:

$$(84.01 / 58.44)(5C / V)$$

in which 84.01 and 58.44 are the molecular weights of sodium bicarbonate and sodium chloride, respectively; *C* is the concentration, in µg per mL, of sodium chloride equivalent in the *Assay preparation;* and *V* is the volume, in mL, of the stock solution remaining from the *Assay for magnesium carbonate* taken.

Magnesium Chloride

$MgCl_2 \cdot 6H_2O$	203.30
Magnesium chloride, hexahydrate [7791-18-6].	
Anhydrous	95.21
[7786-30-3].	

DEFINITION

Magnesium Chloride contains NLT 98.0% and NMT 101.0% of $MgCl_2 \cdot 6H_2O$.

IDENTIFICATION

- **A. IDENTIFICATION TESTS—GENERAL,** *Magnesium* ⟨**191**⟩
  Sample solution: 50 mg/mL
- **B. IDENTIFICATION TESTS—GENERAL,** *Chloride* ⟨**191**⟩
  Sample solution: 50 mg/mL
  [NOTE—Acidify the *Sample solution* with diluted nitric acid before adding 6 N ammonium hydroxide.]

ASSAY

- **PROCEDURE**
  Sample: 450 mg
  Analysis: Dissolve the *Sample* in 25 mL of water, add 5 mL of ammonia–ammonium chloride buffer TS and 0.1 mL of eriochrome black TS, and titrate with 0.05 M edetate disodium VS to a blue endpoint. Each mL of 0.05 M disodium edetate is equivalent to 10.17 mg of $MgCl_2 \cdot 6H_2O$.
  Acceptance criteria: 98.0%–101.0%

IMPURITIES

- **INSOLUBLE MATTER**
  Sample: 20 g
  Analysis: Dissolve the *Sample* in 200 mL of water, heat to boiling, and digest in a covered beaker on a steam bath for 1 h. Filter through a tared filtering crucible, wash thoroughly, dry at 115°, and determine the weight of the residue.
  Acceptance criteria: NMT 0.005%
- **CHLORIDE AND SULFATE,** *Sulfate* ⟨**221**⟩
  Sample: 10 g
  Acceptance criteria: It shows no more sulfate than corresponds to 0.50 mL of 0.020 N sulfuric acid (0.005%).
- **BARIUM**
  Sample: 1 g
  Analysis: Dissolve the *Sample* in 10 mL of water, and add 1 mL of 2 N sulfuric acid.
  Acceptance criteria: No turbidity is produced within 2 h.
- **LIMIT OF CALCIUM**
  [NOTE—A commercially available atomic absorption standard solution for calcium may be used where preparation of a calcium standard stock solution is described below. Concentrations of the *Standard solutions* and the *Sample solution* may be modified to fit the linear or working range of the instrument.]
  Dilute hydrochloric acid: Dilute 100 mL of hydrochloric acid with water to 1000 mL.
  Lanthanum solution: To 58.65 g of lanthanum oxide add 400 mL of water, and add, gradually with stirring, 250 mL of hydrochloric acid. Stir until dissolved, and dilute with water to 1000 mL.
  Standard solutions: Transfer 249.7 mg of calcium carbonate, previously dried at 300° for 3 h and cooled in a desiccator for 2 h, to a 100-mL volumetric flask. Dissolve in a minimum amount of hydrochloric acid, and dilute with water to volume. Transfer 1.0, 5.0, 10.0, and 15.0 mL of this stock solution to separate 1000-mL volumetric flasks, each containing 20 mL of *Lanthanum solution* and 40 mL of *Dilute hydrochloric acid*. Dilute with water to volume. These *Standard solutions* contain 1.0, 5.0, 10.0, and 15.0 µg/mL of calcium, respectively.
  Blank solution: Transfer 4 mL *of Lanthanum solution* and 10 mL of *Dilute hydrochloric acid* to a 200-mL volumetric flask, and dilute with water to volume.
  Sample solution: Transfer 10.0 g of Magnesium Chloride to a 200-mL volumetric flask, and add water to dissolve. Add 4 mL of *Lanthanum solution*, and dilute with water to volume.
  Instrumental conditions
  (See *Spectrophotometry and Light-Scattering* ⟨851⟩.)
  Mode: Atomic absorption spectrophotometry
  Lamp: Calcium hollow-cathode
  Flame: Nitrous oxide–acetylene
  Analytical wavelength: Calcium emission line at 422.7 nm
  Analysis
  Samples: *Standard solutions*, *Blank solution*, and *Sample solution*.
  Determine the concentration, C, in µg/mL, of calcium in the *Sample solution* using the calibration graph.

Calculate the percentage of calcium in the portion of Magnesium Chloride taken:

$$\text{Result} = (V/W \times C \times F) \times 100$$

V = volume of the *Sample solution* (mL)
W = weight of Magnesium Chloride taken (mg)
C = as defined above
F = conversion factor from µg/mL to mg/mL, 0.001

Acceptance criteria: NMT 0.01%

- **POTASSIUM**
  Sample solution: 5 g
  Analysis: Dissolve the *Sample* in 5 mL of water, and add 0.2 mL of sodium bitartrate TS.
  Acceptance criteria: No turbidity is produced within 5 min.
- **ALUMINUM** ⟨**206**⟩ (where it is labeled as intended for use in hemodialysis)
  Test preparation: Prepare as directed in the chapter, using 2.0 g.
  Acceptance criteria: NMT 1 ppm
- **HEAVY METALS** ⟨**231**⟩
  Test preparation: Dissolve 2 g in water, and dilute with water to 25 mL.
  Acceptance criteria: NMT 10 ppm

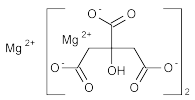
SPECIFIC TESTS

- **pH** ⟨**791**⟩
  Sample solution: 50 mg/mL in carbon dioxide-free water
  Acceptance criteria: 4.5–7.0

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers.
- **LABELING:** Where Magnesium Chloride is intended for use in hemodialysis, it is so labeled.

Magnesium Citrate



$C_{12}H_{10}Mg_3O_{14}$ 451.11
1,2,3-Propanetricarboxylic acid, hydroxy-, magnesium salt (2:3);
Magnesium citrate (3:2) [3344-18-1].

DEFINITION

Magnesium Citrate contains NLT 14.5% and NMT 16.4% of magnesium (Mg), calculated on the dried basis.

IDENTIFICATION

- **A. IDENTIFICATION TESTS—GENERAL,** *Magnesium* ⟨**191**⟩
  Sample: 10 mg/mL
  Acceptance criteria: Meets the requirements
- **B. IDENTIFICATION TESTS—GENERAL,** *Citrate* ⟨**191**⟩
  Sample: 80 mg/mL
  Acceptance criteria: Meets the requirements

ASSAY

- **PROCEDURE**
  Sample: 400 mg
  Analysis: Dissolve the *Sample* in 50 mL of water. Add 20 mL of ammonia–ammonium chloride buffer TS and 0.1 mL of eriochrome black TS. Titrate with 0.05 M edetate disodium VS to a blue endpoint. Perform a blank determination (see *Titrimetry* ⟨541⟩), and make any necessary correction. From the volume of 0.05 M edetate disodium consumed, deduct the volume of 0.05 M edetate disodium corresponding to the amount of calcium in the portion of Magnesium Citrate taken, based on the amount of calcium found in the test for *Limit of Calcium*. Each mg of Ca is equivalent to 0.25 mL of 0.05 M edetate disodium. The difference is the volume of 0.05 M edetate disodium consumed by the magnesium. Each mL of 0.05 M edetate disodium is equivalent to 1.215 mg of Mg.
  Acceptance criteria: 14.5%–16.4% on the dried basis

IMPURITIES

- **CHLORIDE AND SULFATE,** *Chloride* ⟨**221**⟩
  Sample: 300 mg
  Acceptance criteria: It shows no more chloride than corresponds to 0.20 mL of 0.020 N hydrochloric acid (0.05%).
- **CHLORIDE AND SULFATE,** *Sulfate* ⟨**221**⟩
  Sample: 100 mg
  Acceptance criteria: It shows no more sulfate than corresponds to 0.20 mL of 0.020 N sulfuric acid (0.2%).
- **ARSENIC,** *Method I* ⟨**211**⟩**:** NMT 3 ppm
- **HEAVY METALS,** *Method I* ⟨**231**⟩
  Test preparation: Dissolve 0.4 g in 25 mL of water, and proceed as directed in the chapter, except use glacial acetic acid to adjust the pH.
  Acceptance criteria: NMT 50 ppm
- **IRON** ⟨**241**⟩
  Test preparation: Boil 50 mg with 5 mL of 2 N nitric acid for 1 min. Cool, dilute with water to 45 mL, and add 2 mL of hydrochloric acid.
  Acceptance criteria: NMT 200 ppm
- **LIMIT OF CALCIUM**
  [NOTE—A commercially available atomic absorption standard solution for calcium may be used where preparation of a calcium standard stock solution is described below. Concentrations of the *Standard solutions* and the *Sample solution* may be modified to fit the linear or working range of the instrument.]
  Dilute hydrochloric acid: Dilute 100 mL of hydrochloric acid with water to 1000 mL.
  Lanthanum solution: To 58.65 g of lanthanum oxide add 400 mL of water, and add, gradually with stirring, 250 mL of hydrochloric acid. Stir until dissolved, and dilute with water to 1000 mL.
  Standard solutions: Transfer 249.7 mg of calcium carbonate, previously dried at 300° for 3 h and cooled in a desiccator for 2 h, to a 100-mL volumetric flask. Dissolve in a minimum amount of hydrochloric acid, and dilute with water to volume. Transfer 1.0, 5.0, 10.0, and 15.0 mL of this stock solution to separate 1000-mL volumetric flasks, each containing 20 mL of *Lanthanum solution* and 40 mL of *Dilute hydrochloric acid*. Dilute with water to volume. These *Standard solutions* contain 1.0, 5.0, 10.0, and 15.0 µg/mL of calcium, respectively.
  Sample solution: Transfer 250 mg of Magnesium Citrate to a beaker, add 30 mL of *Dilute hydrochloric acid*, and stir until dissolved. Transfer the solution to a 200-mL volumetric flask containing 4 mL of *Lanthanum solution*, and dilute with water to volume.
  Blank solution: Transfer 4 mL *of Lanthanum solution* and 10 mL of *Dilute hydrochloric acid* to a 200-mL volumetric flask, and dilute with water to volume.
  Instrumental conditions
  (See *Spectrophotometry and Light-Scattering* ⟨851⟩.)
  Mode: Atomic absorption spectrophotometry
  Analytical wavelength: Calcium emission line at 422.7 nm
  Lamp: Calcium hollow–cathode
  Flame: Nitrous oxide–acetylene
  Analysis
  Samples: *Standard solutions*, *Sample solution*, and *Blank solution*
  Determine the concentration, C, in µg/mL, of calcium in the *Sample solution* using the calibration graph.