

Lithium Hydroxide

LiOH · H ₂ O	41.96
LiOH	23.95
Lithium hydroxide monohydrate [1310-66-3].	
Anhydrous [1310-65-2].	

DEFINITION

Lithium Hydroxide contains NLT 98.0% and NMT 102.0% of lithium hydroxide (LiOH), calculated on the anhydrous basis.

[CAUTION]—Exercise great care in handling Lithium Hydroxide, as it rapidly destroys tissues.]

IDENTIFICATION

- **A.** When moistened with hydrochloric acid, it imparts an intense crimson color to a nonluminous flame.

ASSAY

• PROCEDURE

Sample solution: Nominally equivalent to 10 mg/mL of anhydrous lithium hydroxide from Lithium Hydroxide in carbon dioxide-free water

Analysis

Preliminary titration: Pipet 50 mL of the *Sample solution* into a 250-mL conical flask. Start the titration by adding 35 mL of 0.5 N hydrochloric acid VS with continuous vigorous stirring. Add 20 mL of 1 N barium chloride and 3 drops of phenolphthalein TS, and allow to stand for 2 min. Continue the titration with 0.5 N hydrochloric acid VS. At the discharge of the pink color of the indicator, record the volume of acid solution consumed.

Final titration: Pipet 50 mL of the *Sample solution* into a 250-mL conical flask. While pipeting and during the subsequent titrations, keep the contents of the flask blanketed with a stream of carbon dioxide-free air.

Start the titration by adding with continuous vigorous swirling a volume of 0.5 N hydrochloric acid VS that is 0.50 mL less than that consumed in the preliminary titration. Add 20 mL of 1 N barium chloride and 3 drops of phenolphthalein TS, and allow to stand for 2 min. Rinse the sides of the flask with carbon dioxide-free water, and continue the titration with 0.1 N hydrochloric acid VS. At the discharge of the pink color of the indicator, record the volume of acid solution consumed. Each mL of 0.5 N hydrochloric acid VS and 0.1 N hydrochloric acid VS is equivalent to 11.975 and 2.395 mg of total alkali, respectively, calculated as lithium hydroxide (LiOH).

Acceptance criteria: 98.0%–102.0% on the anhydrous basis

IMPURITIES

• CHLORIDE AND SULFATE, *Sulfate* (221)

Sample: 2.0 g

Acceptance criteria: It shows no more sulfate than corresponds to 1.0 mL of 0.020 N sulfuric acid (0.05%).

• CALCIUM

Sample solution: Dissolve 3.33 g in 50 mL of 3 N hydrochloric acid. Boil the clear solution to expel carbon dioxide, add 5 mL of ammonium oxalate TS, render alkaline with 6 N ammonium hydroxide, and allow to stand for 4 h. Pass through a filtering crucible, and wash with warm water until the last washing yields no turbidity with calcium chloride TS. Place the crucible in a beaker, cover it with water, add 3 mL of sulfuric acid, and heat to 70°.

Analysis: Titrate the *Sample solution* with 0.10 N potassium permanganate to a pale pink color that persists for 30 s.

Acceptance criteria: NMT 3.34 mL of 0.10 N potassium permanganate is consumed (0.20%).

• CARBONATE

[NOTE]—While pipeting and during the subsequent titrations, keep the contents of the flasks blanketed with a stream of carbon dioxide-free air.]

Analysis: To the flask containing the completed *Final titration* obtained in the *Assay*, add 1 drop of methyl orange TS. Titrate with 0.1 N hydrochloric acid VS until a persistent orange color is produced and no undissolved barium carbonate remains. Perform a blank titration to determine the volume of 0.1 N hydrochloric acid consumed in going from the phenolphthalein endpoint to the methyl orange endpoint. To 100 mL of carbon dioxide-free water in a 250-mL conical flask, add 3 drops of the *Sample solution* from the *Assay*, 20 mL of 1 N barium chloride, and 3 drops of phenolphthalein TS. Allow to stand for 2 min. Titrate this solution with 0.1 N hydrochloric acid. At the discharge of the pink color of the indicator, add 1 drop of methyl orange TS, and titrate with 0.1 N hydrochloric acid VS until a persistent orange color is produced.

Acceptance criteria: The titration shows no more carbon dioxide than corresponds to 1.5 mL of 0.10 N hydrochloric acid (0.7%).

• HEAVY METALS, *Method I* (231)

Sample solution: Dissolve 1.0 g in 15 mL of 3 N hydrochloric acid, and dilute with water to 25 mL.

Acceptance criteria: NMT 20 ppm

SPECIFIC TESTS

Change to read:

• LITHIUM CONTENT

Standard stock solution: 0.3 mg/mL of USP Lithium Carbonate RS prepared as follows. Dissolve first in water using 20% final volume and hydrochloric acid using 0.5% of final volume. Dilute with water to volume.

Standard solution: 6.0 µg/mL of USP Lithium Carbonate RS from the *Standard stock solution* prepared as follows. Pipet a volume of the *Standard stock solution* into a suitable volumetric flask, add water to fill 80% of final volume, and a suitable surfactant solution to fill 2% of final volume. Dilute with water to volume. Measure the pH.

Sample stock solution: 0.4 mg/mL of Lithium Hydroxide in water

Sample solution: Pipet 20 mL of the *Sample stock solution* into a 1000-mL volumetric flask. Add 950 mL of water, 2 mL of 1 N hydrochloric acid, and 20 mL of a surfactant solution, and mix. Adjust with 1 N hydrochloric acid or 1 N sodium hydroxide to the same pH (±0.1 pH unit) as that of the *Standard solution*, and dilute with water to volume.

Instrumental conditions

Mode: Flame photometry

Analytical wavelength: 671 nm. Adjust the instrument with the surfactant solution.

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of lithium (Li) in the portion of Lithium Hydroxide taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times (A_r/M_r) \times F \times 100$$

r_U = photometric reading of the *Sample solution*

r_S = photometric reading of the *Standard solution*

C_S = concentration of USP Lithium Carbonate RS in the *Standard solution* (mg/mL)

2 Lithium

- C_U = concentration of Lithium Hydroxide in the
Sample solution (mg/mL)
 A_r = atomic weight of lithium, 6.94
 M_r = molecular weight of lithium carbonate, 73.89
 F = number of lithium ions per mole of lithium
carbonate, 2

Acceptance criteria: 28.1%–29.9% (RB 1-Jan-2012) on the
anhydrous basis

- **WATER DETERMINATION**, Method III (921)
Analysis: Dry at 135° at a pressure of NMT 5 mm of
mercury for 1 h.

Acceptance criteria: 41.0%–43.5%

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers.
- **USP REFERENCE STANDARDS** (11)
USP Lithium Carbonate RS