

MIL-L-20213E(SH)
 18 June 1980
 SUPERSEDING
 MIL-L-20213D(SHIPS)
 11 December 1959
 (See 6.6)

MILITARY SPECIFICATION

LITHIUM HYDROXIDE (LiOH), TECHNICAL

This specification is approved for use by the Naval Sea Systems Command, Department of the Navy, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers lithium hydroxide, for use in removal of carbon dioxide from air in submarines.

2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieve Test.
 PPP-B-576 - Box, Wood, Cleated, Veneer, Paper Overlaid.
 PPP-B-585 - Boxes; Wood, Wirebound.
 PPP-B-591 - Boxes, Fiberboard, Wood-Cleated.
 PPP-B-601 - Boxes, Wood, Cleated-Plywood.
 PPP-B-621 - Boxes, Wood, Nailed and Lock-Corner.
 PPP-B-636 - Boxes, Shipping, Fiberboard.

MILITARY

MIL-L-10547 - Liners, Case, And Sheet, Overwrap; Water-Vaporproof Or Waterproof, Flexible.
 MIL-C-21004 - Canister, Lithium Hydroxide, Screened Ends, Friction Type Covers.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Sea Systems Command, SEA 3112, Department of the Navy, Washington, DC 20362 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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STANDARDS

MILITARY

- MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.
- MIL-STD-129 - Marking for Shipment and Storage.

(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

- AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)
C136 - Rotap Sieve Test.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

- UNIFORM CLASSIFICATION COMMITTEE, AGENT
Uniform Freight Classification Ratings, Rules and Regulations

(Application for copies should be addressed to the Uniform Classification Committee Agent, Tariff Publication Officer, Room 1106, 222 South Riverside Plaza, Chicago, IL 60606.)

3. REQUIREMENTS

3.1 Qualification. The lithium hydroxide furnished under this specification shall be products which are listed, or are qualified for listing on the applicable qualified products list at the time set for opening of bids (see 4.3 and 6.3).

3.2 Materials. The lithium hydroxide shall be manufactured from lithium hydroxide monohydrate.

3.2.1 Recovered materials. Unless otherwise specified herein, all equipment, material, and articles incorporated in the products covered by this specification shall be new and shall be fabricated using materials produced from recovered materials to the maximum extent practicable without jeopardizing the intended use. The term "recovered materials" means materials which have been collected or recovered from solid waste and reprocessed to become a source of raw materials, as opposed to virgin raw materials. None of the above shall be interpreted to mean that the use of used or rebuilt products is allowed under this specification unless otherwise specifically specified.

3.3 Solubility. Lithium hydroxide shall be completely soluble, giving a clear colorless solution without any sediment when tested as specified in 4.6.2.

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3.4 Apparent density. The apparent density of the lithium hydroxide shall be not less than 0.38 gram per milliliter (g/mL) when tested as specified in 4.6.3.

3.5 Particle size. Lithium hydroxide shall conform to the sieve sizes shown in table I when tested as specified in 4.6.4.

TABLE I. Particle size.

Sieve number	Percentage material retained by weight
4	0 to 5
6	25 to 70
8	20 to 65
14	5 to 45
16	0 to 3
120	0 to 1.5
Pan	0 to 0.5

3.6 Hardness. Lithium hydroxide shall be not less than No. 95 hardness and the amount retained on the No. 14 screen at the end of the test shall be not less than 45 g when tested as specified in 4.6.5.

3.7 Absorption. Initial breakthrough of detectable carbon dioxide in sample effluent must not occur for a minimum of 30 minutes when tested as specified in 4.6.6. The absorption of carbon dioxide shall be not less than 0.70 g of carbon dioxide per gram of lithium hydroxide at an absorption efficiency of 50 percent determined as specified in 4.6.6.

3.8 Chemical composition. The following parameters shall be tested as specified in 4.6.7.

3.8.1 Lithium hydroxide. The lithium hydroxide (LiOH) content shall be not less than 97.6 percent by weight.

3.8.2 Moisture content. The moisture content shall not exceed 0.50 percent by weight.

3.8.3 The constituents shall not exceed the limits listed in table II as specified.

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TABLE II. Chemical composition.

Constituent	Maximum permissible percent by weight
Sulphates and carbonates (calculated as Li_2SO_4 or Li_2CO_3)	1.94 (total)
CaO	0.20
Heavy metals	0.002
Chlorides (see 4.6.7.6)	Trace
Sodium and potassium	0.20 (total)

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Classification of inspections. The inspection requirements specified herein are classified as follows:

- (a) Qualification inspection (see 4.3).
- (b) Quality conformance inspection (see 4.4).

4.3 Qualification tests. Qualification tests shall be conducted at a laboratory satisfactory to the Naval Sea Systems Command. Qualification tests shall consist of the tests specified in 4.6.

4.4 Quality conformance tests. Each sample specimen selected in accordance with 4.4.1.3, shall be subjected separately to the tests specified in 4.6.2, 4.6.4, 4.6.5, 4.6.6, and 4.6.7.2. If any sample specimen fails, this shall be cause for rejection of the entire lot.

4.4.1 Sampling for quality conformance.

4.4.1.1 Lot. For purposes of sampling, a lot shall consist of all lithium hydroxide prepared from one batch of raw material without any change in the processing procedure and presented for acceptance inspection at one time.

4.4.1.2 Sampling for examination of filled containers. A random sample of filled containers shall be selected from each lot offered for examination in accordance with MIL-STD-105 at inspection level I, and

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acceptable quality level (AQL) = 2.5 percent defective to verify compliance with all stipulations of this specification regarding fill, closure, marking and other requirements not involving tests.

4.4.1.3 Sampling for tests. From each lot, two containers shall be selected. From each of the two containers, a 2-pound (lb) specimen shall be taken and placed in a separate, clean, dry, container. The sample container shall have an airtight closure and be labeled in a manner to identify the lot represented. Each container specimen shall be subjected to the tests as specified in 4.6 and the balance of the sample shall be reserved for possible retest until the lot has been accepted by the government.

4.5 Examination of filled containers. Each of the sample filled containers selected in accordance with 4.4.1.2 shall be examined for defects of the container and the closure for evidence of leakage, and for satisfactory markings. Each sample filled container shall be weighed to determine the amount of contents. Any container in the sample having one or more defects or under the required fill (see 5.1) shall be cause for rejection. If the number of defective containers in any sample exceeds the acceptance number for the appropriate sampling plan of MIL-STD-105, this shall be cause for rejection of the lot represented by the sample.

4.6 Inspection test procedures.

4.6.1 Preparation of specimen. Caution is required in handling lithium hydroxide to prevent unnecessary exposure to the air owing to its moisture and carbon dioxide content. Approximately 1 lb of each sample specified in 4.4.1.3 shall be put in a wide-mouth glass bottle of sufficient size to permit homogeneous distribution. The bottle shall be stoppered or sealed to prevent access of air, and shall be rolled or shaken until the particle size distribution of the specimen appears to be homogeneous. The balance of the samples shall be held for duplicating the tests, if necessary.

4.6.2 Solubility. Approximately 10 g (plus or minus 0.1 g) of the prepared specimen (see 4.6.1) shall be placed in a tared weighing bottle with cover and weighed to an accuracy of the nearest milligram. The weighed specimen shall be added to a 1,000 mL flask, dissolved at room temperature in distilled water free from chlorine and diluted to exactly 1,000 mL. The entire sample shall dissolve without any remaining sediment to a clear colorless solution.

4.6.3 Apparent density. From the specimen (see 4.6.1), a weighed quantity of approximately 50 g shall be transferred to a clean, dry 500 mL graduated glass cylinder. The cylinder shall be inclined and rotated until the entire sample flows freely; care shall be taken to avoid any loss. Thereafter, the surface of the sample shall be leveled by tipping the graduated cylinder slightly forward and backward, while held vertically, without tapping or jarring. The volume occupied by the LiOH will be determined in mL. The ratio of weight (in gram) to the volume (in mL) so determined will be reported as "Apparent Density" (see 3.4).

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4.6.4 Sieve test. Sieve tests shall be made by screening 200 g of material, taken from the specimen (see 4.6.1), for 5 minutes on a Rotap machine, conforming to ASTM C 136 run at the rate of 1750 revolutions per minute (r/min) of the driving shaft. The sieves used shall be No. 4, 6, 8, 14, 16 and 120. The sieves shall conform to RR-S-366. The amount held on each sieve shall be in accordance with table I.

4.6.5 Hardness. The hardness number shall be determined by taking approximately 100 g of the specimen (see 4.6.1) and screening as specified in 4.6.4 using only the No. 4 and No. 14 sieves. Approximately 50 g of the material which passes through the No. 4 sieve and remains on the No. 14 sieve shall be taken, weighed to 0.1 g accurately and placed in an 8-inch pan with a concave bottom 5/16 inch thick at the rim and 3/16 inch thick at the center. Fifteen steel ball bearings of 5/16 inch diameter shall be placed in the pan and shaken for 30 minutes with a Rotap sieving machine running at a rate of 1750 r/min of the driving shaft. The contents of the pan shall then be brushed onto an assembly, comprised of a NO. 14 sieve, a No. 30 sieve, and a pan, and shaken for 3 minutes on the Rotap sieving machine. The weight of material retained on each screen shall be recorded and the sum of the weights retained on the screens, multiplied by two, shall be the hardness number (see 3.6).

4.6.6 Absorption. The absorption of carbon dioxide shall be determined by transferring LiOH from the specimen (see 4.6.1) to a weighted absorption tube approximately 3 centimeters (cm) in diameter to a (pre-determined) 15.0 cm depth. The absorption tube with the lithium hydroxide (test bed) shall be weighed, and the weight of the lithium hydroxide determined by difference. An air stream at approximately 20°C Celsius (C) containing 4 percent \pm 0.1 percent carbon dioxide, by volume, and water vapor equivalent to an approximate relative humidity of 85 percent at 20°C, shall be passed vertically from bottom to top through the lithium hydroxide at a velocity of 500 cm per minute, that is, 500 cc/min/sq cm cross-sectional area of absorption tube (approximately 3.5 L/min). The air and carbon dioxide shall be passed through the lithium hydroxide until the effluent air contains one-half the carbon dioxide concentration of the influent air. The 50 percent concentration point shall be determined from examination of a continuous time versus efficiency curve obtained by monitoring the effluent with a carbon dioxide infrared detector. The initial breakthrough point shall be determined by readings of $0.1\% \text{ CO}_2 \pm 0.01\% \text{ CO}_2$ as read on the time versus concentration curve. The CO_2 concentration of the effluent air stream shall be continuously recorded, using an appropriate strip chart recorder. The effluent absorption chart shall be retained for two years and made available to the Naval Sea Systems Command upon request. The amount of carbon dioxide (CO_2) absorbed to the 50 percent efficiency point specified in 3.7 is to be determined from equation (1). The initial gram equivalent of LiOH is derived from equation (2). The value (.976) in equation (2) corresponds to LiOH purity specified in 4.6.7.1. After the 50 percent efficiency level is reached, reweigh the test bed. Then thoroughly mix test bed and extract a 10 g representative sample and dissolve it volumetrically in 1-liter of distilled water per 4.6.2. A 100 mL Aliquot of this solution is then transferred to a 250 mL flask. A neutral 10 percent BaCl_2 solution is slowly added until the white precipitate BaCO_3 is no longer formed. Two drops of phenolphthalein indicator are added and the solution immediately titrated with N-hydrochloric acid of a known normality. To avoid contamination of the sample by atmospheric CO_2 , this analysis must be completed as soon as possible. After

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obtaining the final weight of the test bed and the titration results, calculate the gram equivalents of LiOH remaining with equation (3). The difference between the original and the remaining gram equivalents of LiOH are expressed as gram equivalents LiOH used, and are calculated using equation (4). Using results in equation (4), calculation of CO₂ absorbed in grams is derived using equation (1).

Equation (1)

$$\text{Grams CO}_2 \text{ absorbed} = (\text{gram--equivalents LiOH used}) \left(\frac{.5 \text{ moles CO}_2}{\text{gram equivalent LiOH}} \right) \cdot \left(\frac{44 \text{ grams}}{\text{mole}} \right)$$

Equation (2)

$$\text{LiOH initial gram equivalent} = \left(\frac{\text{grams LiOH in test bed}}{24} \right) \cdot (X)$$

where X = fraction of LiOH in test bed sample (see 4.6.7.1)

Equation (3)

$$\text{LiOH final gram equivalents} = (10) \left(\frac{\text{total final weight of bed}}{\text{weight of sample taken from the test bed}} \right) (\text{HCl normality}) (\text{mL of HCl}) \left(\frac{.001 \text{ liters}}{\text{mL}} \right)$$

Equation (4)

$$\text{gram equivalents LiOH used} = (\text{Equation (2)}) - (\text{Equation (3)})$$

4.6.7 Chemical composition. Reagents used for testing shall be of an analytical grade. The solution specified in 4.6.2 shall be used as the sample for tests of chemical composition, except moisture content.

4.6.7.1 Lithium hydroxide. LiOH shall be determined by titration of 100 mL of the sample solution (see 4.6.2) with N-hydrochloric acid, using phenolphthalein indicator. The content of lithium hydroxide calculated from the titration shall be not less than 97.6 percent of the original sample as specified in 3.8.1. A sample of 97.6 percent LiOH containing no acid-titratable impurities requires 40.77 mL of N-HCl per gram of sample, that is, the amount in 100 mL of sample solution (see 4.6.2).

4.6.7.2 Moisture content. Approximately 1 g of material taken from the specimen (see 4.6.1), shall be placed in a tared weighing bottle without unnecessary exposure to air and weighed in milligrams. It shall be dried at 120°C for 4 hours with the cover removed in an atmosphere free from carbon dioxide. At the conclusion of the drying period, the LiOH shall be placed in a desiccator free of carbon dioxide to cool. After cooling to room temperature, the bottle containing the LiOH shall be weighed again, and the weight of the dried material determined. The moisture content of the original LiOH shall be determined in percent from the difference in weight of the material from before and after drying, and shall conform to 3.8.2.

4.6.7.3 Sulphates and carbonates (as Li₂SO₄ and Li₂CO₃). A standard solution of sulphates and carbonates for comparative purposes shall be prepared as follows: To a liter of distilled water, add 0.109 g of Li₂SO₄ and 0.085 g of Li₂CO₃ and shake until dissolved. This standard solution is equivalent to the 1.94 percent specified in 3.8.3. One hundred mL of this solution and 100 mL of sample solution (see 4.6.2) shall be compared by the following test. Add 25 mL of 5 normal barium chloride solution to each and shake for 1/2 hour. The quantity of sulphate and carbonate permitted is determined by the turbidity in the sample solution which shall not exceed that of the prepared standard (see 3.8.3).

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4.6.7.4 Calcium oxide. A standard lime-water solution shall be made with 0.020 g of calcium oxide in a liter of distilled water. A test shall be made between 100 mL of this standard solution and 100 mL of sample solution (see 4.6.2). The two solutions shall be adjusted with acetic acid to an acidity between pH4 and pH6, and to each shall be added 20 mL of N/10 sodium oxalate solution. The turbidity of the sample solution shall be not greater than that of the standard solution, which does not contain in excess of 0.20 percent calcium oxide (see 3.8.3).

4.6.7.5 Heavy metals. Heavy metal impurities shall be determined by standard gravimetric, colormetric, or spectroscopic methods (see 3.8.3).

4.6.7.6 Chlorides. A standard solution of chlorides shall be prepared containing 0.08 g of KCl per 50 mL of chlorine-free distilled water. Fifty mL of sample solution (see 4.6.2) and 50 mL of standard solution shall be acidified with nitric acid, and a drop of 2 percent silver nitrate solution added to each. The cloudiness of the sample solution shall not exceed that of the standard solution. This does not exceed a trace of chloride as specified in 3.8.3.

4.6.7.7 Sodium and potassium. Sodium and potassium shall be determined using atomic absorption methods or other standard spectroscopic methods.

4.7 Packaging inspection. Sample packages and packs and the packaging inspection for shipment and storage and/or storage shall be in accordance with the requirements of 4.4 and Section 5 herein.

5. PACKAGING

(The preparation for delivery requirements specified herein apply only for direct Government acquisitions. For the extent of applicability of the preparation for delivery requirements of referenced documents listed in section 2, see 6.5.)

5.1 Packaging. -

5.1.1 Levels A and C.

5.1.1.1 Lithium hydroxide shall be packaged in canisters conforming to MIL-C-21004. Each container shall be packaged and marked in accordance with MIL-C-21004. The stock number gross weight when packaged, and net weight when packaged shall be marked on the container in the spaces provided by the container manufacturer. The net weight of lithium hydroxide in each container shall be 6.3 lb minimum and 6.5 lb maximum. The marking shall be colored black (or yellow) and shall be applied by the silk screen process, rubber stamp, or stencil. Waterproof ink shall be used. Minimum height of letters shall be 1/8-inch.

5.2 Packing. (See 6.2)

5.2.1 Level A. Lithium hydroxide, shall be packed in overseas type wood cleated fiberboard, nailed wood, wirebound wood, corrugated or solid fiberboard, wood-cleated paper overlaid, or wood-cleated plywood boxes conforming to PPP-B-591, PPP-B-621, PPP-B-585, PPP-B-636 (class 3),

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PPP-B-576, or PPP-B-601, respectively, at the option of the contractor. Shipping containers shall have caseliners conforming to MIL-L-10547 and shall be closed and sealed in accordance with the appendix thereto. Caseliners for boxes conforming to PPP-B-636 are not required. Fiberboard boxes shall be closed, waterproofed and reinforced in accordance with Method V of the appendix to PPP-B-636. Box closures shall be as specified in the applicable box specification or appendix thereto. The gross weight of wood boxes shall not exceed 200 lb; fiberboard boxes shall not exceed the weight limitations of the applicable box specification.

5.2.2 Level B. Lithium hydroxide shall be packed in domestic type wood-cleated fiberboard, nailed wood, wirebound wood-cleated plywood, or wood-cleated paper overlaid boxes or class 2 fiber boxes conforming to PPP-B-591, PPP-B-621, PPP-B-585, PPP-B-601, PPP-B-576, or PPP-B-636, respectively, at the option of the contractor. Box closures shall be as specified in the applicable box specification or appendix thereto. Fiberboard boxes shall be closed in accordance with Method I of the appendix to PPP-B-636 except that pressure-sensitive tapes shall be used. The gross weight of wood boxes shall not exceed 200 lb; fiberboard boxes shall not exceed the weight limitations of the applicable box specification.

5.2.3 Level C. Lithium hydroxide shall be packed in containers which will insure acceptance and safe delivery at destination. Shipping containers shall comply to the Uniform Freight Classification Rules or regulations or other regulations as applicable to the mode of transportation.

5.3 Marking. In addition to any special marking (see 6.2), each can and exterior pack shall be marked in accordance with MIL-STD-129 and 5.1.1.1.

6. NOTES

6.1 Intended use. Lithium hydroxide will be used as a carbon dioxide absorbent on nuclear powered submarines. Its use is primarily as a back-up system and for emergency absorbent requirements.

6.2 Ordering data. Acquisition documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Whether lithium hydroxide is to be packed in accordance with level A, B, or C (see 5.2).
- (c) Special markings required (see 5.3).

6.3 With respect to products requiring qualification, awards will be made only for products which are at the time set for opening of bids, qualified for inclusion in the applicable Qualified Products List QPL-20213 whether or not such products have actually been so listed by that date. The attention of the contractors is called to these requirements, and manufacturers are urged to arrange to have the products that they propose to offer to the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or orders for the products covered by this specification. The activity responsible for the Qualified

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Products List is Naval Sea Systems Command, SEA 3112, Department of the Navy, Washington, DC 20362, and information pertaining to qualification of products may be obtained from that activity. Application for Qualification tests shall be made in accordance with "Provisions Governing Qualification SD-6" (see 6.4).

6.4 Copies of "Provisions Governing Qualification SD-6" may be obtained upon application to Commanding Officer, Naval Publications and Forms Center, 5801 Tabor Avenue, Philadelphia, PA 19120.

6.5 Sub-contracted material and parts. The preparation for delivery requirements of referenced documents listed in section 2 do not apply when material and parts are acquired by the contractor for incorporation into the equipment and lose their separate identity when the equipment is shipped.

6.6 Changes from previous issue. Asterisks (*) are not used in this revision to identify changes with respect to the previous issue, due to the extensiveness of the changes.

Preparing activity:
Navy - SH
(Project 6810-N801)

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL		OMB Approval No. 22-R255
<p>INSTRUCTIONS: The purpose of this form is to solicit beneficial comments which will help achieve procurement of suitable products at reasonable cost and minimum delay, or will otherwise enhance use of the document. DoD contractors, government activities, or manufacturers/vendors who are prospective suppliers of the product are invited to submit comments to the government. Fold on lines on reverse side, staple in corner, and send to preparing activity. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements. Attach any pertinent data which may be of use in improving this document. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity.</p>		
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