

METRIC

MIL-DTL-12468E

30 June 2009

SUPERSEDING

MIL-DTL-12468D

12 August 1998

DETAIL SPECIFICATION

DECONTAMINATING AGENT, STB

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE. This specification covers one type of decontaminating agent called Super Topical Bleach (STB) for military use.

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3, 4 or 5 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3, 4, or 5 of this specification, whether or not they are listed.

2.2 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those listed cited in the solicitation or contract.

ASTM INTERNATIONAL (American Society for Testing and Materials)

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|------------|--|
| ASTM D1193 | - Standard Specification for Reagent Water |
| ASTM E11 | - Standard Specification for Wire Cloth or Sieves for Testing Purposes |

(Copies of these documents are available from <http://www.astm.org> or ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

Comments, suggestions, or questions on this document should be addressed to Defense Supply Center Richmond (DSCR), ATTN: DSCR-VEB, 8000 Jefferson Davis Highway, Richmond, VA 23297-5610 or e-mailed to STDZNMGT@DLA.MIL . Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at http://assist.daps.dla.mil .
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2.3 Order of precedence. Unless otherwise noted herein or in the contract, in the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Material. The decontaminating agent STB shall be an intimate mixture of calcium oxide and bleaching powder (chlorinated lime). The bleaching powder shall have a maximum moisture content of 1.0 percent by weight. Calcium hypochlorite (high test hypochlorite or HTH) shall not be used as a substitute for bleaching powder in the manufacture of the decontaminating agent STB (see 6.4.2 and 6.5).

3.2 Physical and chemical characteristics. The decontaminating agent STB shall conform to the physical and chemical characteristics of table I when tested as specified therein.

Table I. Physical and chemical characteristics.

Characteristics	Percentage by Weight		Test Paragraph
	Minimum	Maximum	
Available chlorine	28.0	----	4.4.2.1
Water	----	1.0	4.4.2.2
Loss of available chlorine	----	4.0	4.4.2.2
Calcium oxide	3.0	6.6	4.4.2.2
Iron (as ferric oxide)	----	0.2	4.4.2.3
Particle size:			
Through 1.40 mm (No. 14) sieve	98.0	----	4.4.2.4
Through 600 um (No. 30) sieve	60.0	----	4.4.2.4

3.3 Bulk density. The decontaminating agent STB shall have a minimum bulk density of 0.8 gram (g) per milliliter (ml) when tested as specified in 4.4.2.5.

3.4 Setting characteristics. The decontaminating agent STB shall not set when tested as specified in 4.4.2.6 (see 6.4.3).

4. VERIFICATION

4.1 Classification of inspections. The inspection requirements specified herein are classified as a conformance inspection (see 4.3).

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4.2 Inspection conditions. Unless otherwise specified, all inspections shall be performed on a lot. A lot shall consist of the decontaminating agent STB produced by one manufacturer in no more than 24 hours, at one plant, from the same materials, and under essentially the same manufacturing conditions provided the operation is continuous. In the event the process is a batch process, each batch shall constitute a lot (see 6.4.1).

4.3 Conformance inspection. Conformance inspection shall be performed in accordance with inspection provisions set forth herein. The characteristics shown in table I, when tested in accordance with 4.4, shall constitute minimum inspections to be performed by the supplier prior to Government acceptance or rejection by lot. Sampling shall be conducted in accordance with table II. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies, submitted to the Government for acceptance, comply with all requirements of the contract. Failure of any test, by any sample, shall be cause for rejection of the lot represented.

Table II. Sampling.

Number in a Batch or Lot	Number of Samples
2 to 25	2
26 to 150	3
151 to 1,200	5
1,201 to 7,000	8
7,001 to 20,000	10
20,001 to 35,000	15
over 35,000	20

4.4 Test methods and procedures.

4.4.1 Test specimen preparation. See 6.7 for sampling and testing precautions. A representative specimen of approximately 0.45 kg shall be removed from each sample container and placed in a suitable, clean, dry container labeled to identify the lot or batch and the container from which it was taken. Each sample specimen taken shall be tested as specified in 4.4.2.

4.4.2 Tests. See 6.7 for sampling and testing precautions. Water in accordance with ASTM D 1193 and reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections applied where significant. Tests shall be conducted in accordance with paragraphs 4.4.2.1 thru 4.4.2.6.

4.4.2.1 Available chlorine. Place an approximate 5 g specimen in a porcelain mortar and grind with a pestle to obtain a uniform powder. Weigh out approximately 0.25 g of the uniform powder to the nearest milligram into a 250 ml Erlenmeyer flask. First add 25 ml of potassium iodide (KI) solution (made by dissolving 50 g of KI in 1 liter of water) and then add 10 ml of

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glacial acetic acid to the flask. Swirl until all solids have dissolved. Titrate the resulting solution with 0.1 N sodium thiosulfate solution until the solution color is light yellow. Then add approximately 2 ml of starch solution and complete the titration with 0.1 N sodium thiosulfate solution until the solution reaches the clear, colorless endpoint. Calculate the percent by weight available chlorine as follows:

$$\text{Percent available chlorine} = \frac{3.5453A}{W}$$

where: A = Total milliliters of sodium thiosulfate solution added to starch endpoint times the normality of the solution, and

W = Weight of the specimen added to the flask in grams.

4.4.2.2 Sequential determination of water, loss of available chlorine, and calcium oxide.

Weigh, to the nearest milligram, approximately 10 g of specimen into a 50 ml, low-form, preweighed weighing bottle with cover. Record the initial specimen weight (A). Place weighing bottle with cover removed in an oven at 100°C for 2 hours. Always replace the cover before removing the weighing bottle from the oven and allow to cool in a desiccator containing “Drierite” before each weighing. After cooling, weigh again and record the specimen weight after drying for 2 hours at 100°C (B). Remove 0.25 g of specimen and weigh to the nearest milligram (C) and place it in a 250 ml Erlenmeyer flask. Determine the available chlorine (Y) by the procedure specified in 4.4.2.1. Place weighing bottle containing (B-C) g of specimen in an oven at 100°C again, then raise the temperature of the oven slowly to 270° ± 5°C and hold at that temperature for at least 1 hour. Cool in a covered desiccator and weigh. Continue heating at 270° ± 5°C to constant weight and record the weight (D). Add 10 ml of freshly boiled distilled water to the specimen in the 50 ml weighing bottle and stir for at least 15 minutes with a glass rod fitted with a rubber policeman. (Inconsistent results will be obtained if the slurry is not thoroughly stirred and care is not taken to scrape the policeman clean on the weighing bottle and to rinse the rubber policeman with a small amount of water into the weighing bottle.) Evaporate the slurry to dryness in an oven set at 80°C. Transfer the bottle containing the dry slurry to a 270° ± 5°C oven and place on an insulating sheet in the oven to prevent the bottle from cracking. Dry to a constant weight and record the weight (E).

Calculate the percent by weight water as follows:

$$\text{Percent water} = \frac{100(A - B - Z)}{A}$$

Calculate the percent by weight loss of available chlorine as follows:

$$\text{Percent loss of available chlorine} = \frac{100(X - Y)}{X}$$

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Calculate the percent by weight calcium oxide as follows:

$$\text{Percent calcium oxide} = \frac{311.1(E - D)}{B - C}$$

where: A = Weight of original specimen in grams,
 B = Weight of specimen after drying at 100°C in grams,
 C = Weight of 0.25 g of specimen removed for available chlorine analysis in grams,
 D = Weight of dried specimen before slurring in grams,
 E = Weight of dried specimen after slurring in grams,
 X = Percent available chlorine determined on as-received specimen,
 Y = Percent available chlorine after heating at 100°C, and
 Z = A(X - Y).

4.4.2.3 Iron (as ferric oxide).

4.4.2.3.1 Indicator solution. Dissolve 0.32 g of barium diphenylamine sulfonate in 100 ml of water. Add 0.5 g of sodium sulfate, mix, and allow the barium to settle. Decant and use the clear solution as the indicator solution.

4.4.2.3.2 Stannous chloride solution. Dissolve 60 g of stannous chloride in 60 ml of concentrated hydrochloric acid and dilute to 1 liter with water.

4.4.2.3.3 Procedure. Weigh to the nearest 0.01 g approximately 20 g of specimen into a 600 ml beaker. Cautiously add 50 ml of concentrated hydrochloric acid until effervescence ceases. Dilute to 250 ml with water and add 5 ml of concentrated nitric acid. Boil 2 to 3 minutes. Precipitate the iron with concentrated ammonium hydroxide while the solution is still hot. If only a white precipitate of aluminum hydroxide is obtained, the ferric oxide present is negligible (less than 0.01 percent). If a reddish-brown precipitate is obtained, filter through filter paper and wash the precipitate three times with hot water. Dissolve the precipitate in hot 1 to 4 hydrochloric acid and wash the filter paper three times with solution prepared in 4.4.2.3.2; then add five drops in excess and allow to cool. Add 25 ml of a saturated solution of mercuric chloride and 15 ml of a phosphoric-sulfuric acid mixture (150 ml concentrated sulfuric acid and 150 ml concentrated phosphoric acid per liter). Add five drops of the indicator solution prepared in 4.4.2.3.1 and titrate with 0.025 N potassium dichromate solution to a purple end point. Calculate the percent by weight of iron, as ferric oxide, as follows:

$$\text{Percent iron} = \frac{7.984AB}{W}$$

where: A = Milliliters of potassium dichromate used in the titration,
 B = Normality of the potassium dichromate solution, and
 W = Weight of specimen in grams.

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4.4.2.4 Particle size. Use sieves conforming to ASTM E 11. Nest a 1.40 mm (No. 14) sieve in a 600 μm (No. 30) sieve and place a receiving pan at the bottom. Weigh 200 ± 5 g of the specimen into the top sieve, cover, and assemble in a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. Vibrate the sieves for 5 minutes with the tapper in operation. Brush any residue remaining on the sieves with a camel's-hair brush until no appreciable quantity of material passes the sieves. Weigh and calculate the percentage of specimen passing through each sieve.

4.4.2.5 Bulk density. Weigh approximately 40.0 g of the specimen, which has previously passed through a 1.40 mm (No. 14) sieve, into a 100 ml graduated cylinder from which the lip has been removed. Stopper the graduate and pass a closely fitting glass sleeve, approximately 76 mm long, over it. Clamp the sleeve to a ring stand. Place a large rubber stopper under the cylinder and adjust the sleeve so that the graduate will be 100 mm above the rubber stopper when the base of the graduate touches the lower edge of the sleeve. Raise the graduate until it touches the sleeve, then release. Continue raising and dropping the graduate until 100 cycles are completed. Read the volume of the sample and calculate the bulk density in grams per milliliter.

4.4.2.6 Setting characteristics. Place a solution of 0.25 g of citric acid in 75 ml of water in a 250 ml beaker, add 50 g of the specimen with constant stirring during a period of 2 minutes, then stir the mixture constantly for 5 minutes to break up lumps and to insure as uniform a slurry as possible. Place a suitable thermometer in the mixture and place the beaker in an ice-water bath. Stir occasionally until the temperature of the slurry falls to 4°C or less. Hold the slurry below 4°C with occasional stirring for 15 minutes. The specimen shall be considered as failing the test if the slurry sets during any phase of the test (see 6.4.3).

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Services or Defense Agency, or within the military service's system commands. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. The decontaminating agent STB is military unique. It is intended to be used as a decontaminating agent in chemical and biological warfare for the military. It is used in destroying or converting certain chemical and biological warfare agents into harmless or less toxic compounds. No commercial alternative exists.

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6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number, and date of this specification.
- b. Unit quantity required.
- c. Packaging requirements (see section 5).

6.3 Material Safety Data Sheets (MSDS). Contracting officers will identify those activities requiring copies of completed Material Safety data Sheets prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313. It is required by 29CFR 1910.1200 that the Material Safety Data Sheet for each hazardous chemical used in an operation must be readily available to personnel using the material. Contracting officers will identify the activities requiring copies of the Material Safety Data Sheet.

6.4 Definitions.

6.4.1 Batch. A batch is defined as that quantity of material which has been manufactured by some unit chemical process or subject to some physical mixing operation intended to make the final product substantially uniform.

6.4.2 Bleaching powder. Bleaching powder is also known as chlorinated lime, chloride of lime, or calx chloride, basic hypochlorite. The chemical formula for bleaching powder is $\text{Ca}(\text{ClO})_2 \cdot \text{CaCl}_2 \cdot x\text{Ca}(\text{OH})_2 \cdot x\text{H}_2\text{O}$.

6.4.3 Setting. For the purpose of this specification, setting is defined as a thickening of the slurry sufficient to prevent flow. As the slurry is cooled, it will become more viscous but this effect should not be construed as setting so long as the slurry will flow when its container is inclined. The slurry will be considered as having set when its container may be inverted for 1 minute without loss of contents. The slurry should be stirred vigorously immediately before testing for ability to flow.

6.5 Materials. Decontaminating agent STB which has been prepared by mixing approximately 6.6 percent by weight calcium oxide with bleaching powder has been found to be satisfactory. The decontaminating agent STB should not contain the salt $\text{Ca}(\text{OCl})_2 \cdot 3\text{H}_2\text{O}$ as determined by an x-ray diffraction powder pattern of the decontaminating agent STB. This method of preparing decontaminating agent STB is furnished with the distinct understanding that:

- a. the contractor is not required to follow this method,
- b. the requirements of this specification must be met regardless of the method of manufacture, and
- c. the Government makes no warranty that decontaminating agent STB made in accordance with this method will meet the requirements of this specification.

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6.6 Markings. Each container and pallet load will be marked in accordance with MIL-STD-129 and 49 CFR (Code of Federal Regulations). Containers will be marked to show the date of manufacturer, lot or batch number of the decontaminating agent, and the following information:

(a) Head and shell of container. The head and shell of the container will be marked to show the following information:

Decontaminating agent STB.

For storage in all climates.

Inspect in accordance with TB 740-10/DLM 4155.5 - Quality Control Depot Serviceability Standards, Appendix G - General Supplies.

Clean bare spots and breaks in paint film and coat with semi-gloss, rust-inhibiting enamel.

WARNING: HARMFUL IF INHALED OR SWALLOWED

Decontaminating agent STB contains calcium oxide and chlorinated lime. Exposure may cause irritation of the skin, eyes, nose, and throat. Skin burns may result from contact with this chemical. Protective mask or other respiratory protection devices should be worn when preparing slurry. Use face shield or safety goggles with rubber gloves to prevent prolonged contact. Keep away from combustibles. Do not store at high temperatures.

FIRE: Poisonous gases are produced when heated. Wear chemical protective suit including self-contained breathing apparatus.

FIRST AID: Remove contaminated clothing and shoes. Flush affected areas with plenty of water. If irritation persists, call doctor. If in eyes, hold eyelids open and flush with plenty of water. If ingested, give lots of water and call doctor. Do not give acidic antidotes. If victim is unconscious, do nothing except keep victim warm. Do not induce vomiting.

Chlorites, Inorganic, NOS (containing calcium oxide) UN1462 PG II

(b) Shell of container. The midsection of the container will be marked with the DOT oxidizing label and show the following information:

Mixing Instructions:

FOR BUCKET MIXTURE - Dissolve one measuring cup (3 ounces or 85 grams) of anti-setting compound M2 in accordance with MIL-A-51027 in 2-1/4 gallons (18 pounds or 8.2 kilograms) of water. Add three shovel fulls (18 pounds or 8.2 kilograms) of STB. Mix thoroughly. Apply with swab or broom.

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FOR EARTH MIXTURE - Add two shovel fulls of STB to three shovel fulls of earth or sand. Mix thoroughly. Apply with shovel.

FOR POWER-DRIVEN DECONTAMINATING APPARATUS - Complete mixing directions are included in manual furnished with apparatus.

(c) Base of container. The base of the container will be marked using ½ inch letters with the following United Nations performance oriented packaging marking:

y 1A2/Z25/A/*
USA/DOD/AYE

*year packed

6.7 Sampling and testing precautions. Personnel sampling and testing decontaminating agent STB should be adequately protected against the destructive effects of this material. Sampling should be performed as rapidly as possible to prevent the chemical changes which the material undergoes when exposed to the air.

6.8 Subject term (key word) listing.

Bleaching powder
Biological warfare
Calcium oxide
Chlorinated lime
Chemical agent
Chemical warfare

6.9 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to previous issue due to the extent of the changes.

MILITARY INTERESTS:

Custodians:

Army - EA
Air Force - 68

Preparing activity:

DLA – GS3
(Project 6850-12009-007)

Review activity:

Army - MD1

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at <http://assist.daps.dla.mil>.