

MIL-P-150D
17 November 1982
 SUPERSEDING
 MIL-P-150C
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MILITARY SPECIFICATION

POTASSIUM CHLORATE, TECHNICAL

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

1. SCOPE

1.1 Scope. This specification covers three grades and seven classes of technical grade potassium chlorate (KClO₃).

1.2 Classification. Potassium chlorate shall be of the following grades and classes as specified (see 6.2):

- Grade A - Low bromate
- Grade E - High bromate
- Grade C - With magnesium carbonate

- Class 1 - No. 80 sieve, nominal
- Class 2 - No. 100 sieve, nominal
- Class 3 - No. 60 sieve, nominal
- Class 4 - No. 100 sieve, nominal, fine
- Class 5 - No. 50 sieve, nominal
- Class 6 - No. 325 sieve, nominal
- Class 7 - No. 80 sieve, nominal, fine

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications, standards, and handbooks. Unless otherwise specified, the following specifications, standards, and handbooks of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

: Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, US Army Armament Research and Development Command, ATTN: DRDAR-TSC-S, Aberdeen Proving Ground, MD 21010 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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SPECIFICATIONS

FEDERAL

- L-P-378 - Plastic Sheet and Strip, Thin Gauge, Polyolefin
- RR-S-366 - Sieve, Test

MILITARY

- MIL-D-3464 - Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification
- MIL-M-11361 - Magnesium Carbonate

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, handbooks, drawings, and publications required by manufacturers in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.1.2 Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this specification to the extent specified herein.

CODE OF FEDERAL REGULATIONS (CFR)

- 49 CFR 171 to 179 - Department of Transportation Hazardous Materials Regulations

(The Code of Federal Regulations is available from the Superintendent of Documents, US Government Printing Office, Washington, DC 20402. Orders for the above publication should cite "49 CFR 171 to 179.")

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

AMERICAN CHEMICAL SOCIETY (ACS) PUBLICATION:

Reagent Chemicals, American Chemical Society Specifications, Sixth Edition

(Application for copies should be addressed to the American Chemical Society, 1155 Sixteenth Street, N.W., Washington, DC 20036.)

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ASTM STANDARDS

D1193 - Reagent Water

(Application for copies should be addressed to ASTM, 1916 Race Street, Philadelphia, PA 19103.)

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS

3.1 Appearance. Potassium chlorate shall be a white, crystalline powder when tested as specified in 4.2.4.1.

3.2 Chemical characteristics. Potassium chlorate shall conform to the applicable chemical characteristics of table I when tested as specified therein.

3.3 Granulation characteristics. Potassium chlorate shall conform to the applicable granulation characteristics of table II when tested as specified in 4.2.4.14.

3.4 Magnesium carbonate. The magnesium carbonate added to produce grade C potassium chlorate shall conform to grade B of MIL-M-11361.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, 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 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the potassium chlorate produced by one manufacturer, 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 operation, each batch shall constitute a lot (see 6.3).

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TABLE I. Chemical characteristics

Characteristic	Percent by weight			Test paragraph
	Grade A	Grade B	Grade C	
Moisture, maximum	0.05	0.05	0.05	4.2.4.2
Assay (as KClO ₃), minimum	99.5	99.5	96.5	4.2.4.3
Water-insoluble matter, maximum	0.02	0.10	3.20	4.2.4.4
pH of water-soluble matter	5 to 8	5 to 8	-----	4.2.4.5
Hypochlorites	To pass test	To pass test	To pass test	4.2.4.6
Chlorites	To pass test	-----	-----	4.2.4.7
Chlorides (as KCl), maximum	0.10	0.10	0.10	4.2.4.8
Bromates (as KBrO ₃), maximum	0.02	0.10	0.10	4.2.4.9
Heavy metals	To pass test	To pass test	To pass test	4.2.4.10
Alkaline earths	To pass test	To pass test	-----	4.2.4.11
Sodium, maximum	0.04	0.04	0.04	4.2.4.12
Magnesium carbonate	-----	-----	3.0 + 0.2	4.2.4.13

TABLE II. Granulation characteristics

Sieve size	Percent by weight passing						
	Class 1	Class 2	Class 3	Class 4	Class 5	Class 6	Class 7
	Min:Max	Min:Max	Min:Max	Min:Max	Min:Max	Min:Max	Min:Max
No. 50	-- : --	-- : --	-- : --	-- : --	98.0 : --	-- : --	-- : --
No. 60	-- : --	-- : --	99.5 : --	-- : --	-- : --	-- : --	-- : --
No. 80	99.0 : --	-- : --	60.0 : --	-- : --	-- : --	-- : --	99.0 : --
No. 100	-- : --	99.9 : --	-- : --	99.9 : --	85.0 : --	-- : --	-- : --
No. 140	45.0 : 55.0	-- : --	-- : --	45.0 : 55.0	-- : --	-- : --	-- : --
No. 200	17.0 : 23.0	-- : 5.0	-- : --	40.0 : 40.0	70.0 : --	-- : --	90.0 : --
No. 325	-- : --	-- : --	-- : --	-- : --	-- : --	95.0 : --	60.0 : --

4.2.2 Sampling.

4.2.2.1 For examination of packaging. Sampling shall be conducted in accordance with MIL-STD-105.

4.2.2.2 For test (see 6.5). Sampling shall be conducted in accordance with table III. A representative specimen of approximately 1 pound shall be removed from each sample container and placed in a suitable, tightly closed, clean, dry container labeled to identify the lot and container from which it was taken.

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TABLE III. Sampling for test

<u>Number of containers in batch or lot</u>	<u>Number of sample containers</u>
3 to 150	3
151 to 1,200	5
1,201 to 7,000	8
7,001 to 20,000	10
Over 20,000	20

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. The sample unit shall be one filled shipping container, ready for shipment. Sample shipping containers shall be examined for the following defects using an AQL of 2.5 percent defective:

- (a) Contents per container not as specified.
- (b) Container or bag liner not as specified.
- (c) Container or bag liner closure not as specified.
- (d) Container damaged or leaking.
- (e) Desiccant missing (level B only).
- (f) Marking incorrect, missing, or illegible.

4.2.3.2 For test. Each sample specimen taken in 4.2.2.2 shall be tested as specified in the applicable portions of 4.2.4. Failure of any test by any specimen shall be cause for rejection of the lot represented.

4.2.4 Tests (see 6.5). Water in accordance with ASTM D1193 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 as follows:

4.2.4.1 Appearance. Visually examine approximately 50 grams (g) of the specimen for form and color.

4.2.4.2 Moisture. Heat a moisture dish and its stopper in an oven at 100° to 110°C for 1 hour, cool in a desiccator, and weigh to the nearest milligram (mg). Transfer approximately 10 g of the specimen to the dry dish. Stopper and weigh to the nearest mg. Heat unstoppered in an oven at 100° to 110°C for 1 hour. Cool in a desiccator. Replace the stopper and weigh to the nearest mg. Repeat the heating for 1/2-hour periods until successive weighings differ by no more than 1 mg. The total time of heating shall not exceed 5 hours. Calculate the percent moisture as follows:

$$\text{Percent moisture} = \frac{100 A}{W}$$

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where: A = Weight loss in grams after heating, and
W = Weight in grams of specimen.

4.2.4.3 Assay (as KClO₃).

(a) Ferrous ammonium sulfate solution. Dissolve 40 g of ferrous ammonium sulfate [Fe(NH₄)₂(SO₄)₂·6H₂O] in a cold 5 + 95 solution by volume of sulfuric acid and dilute to 1 liter with the sulfuric acid solution. Titrate with 0.1N potassium permanganate solution and calculate the volume in milliliters (ml) of potassium permanganate solution equivalent to 1 ml of the ferrous ammonium sulfate solution. Determine equivalency under the same conditions of test as in (b) except that no potassium chlorate will be present.

(b) Procedure. Weigh to the nearest 0.1 mg approximately 0.8 g of the specimen used in 4.2.4.2. Dissolve in water and dilute to 500 ml in a volumetric flask with water. Transfer a 50-ml aliquot to a 500-ml Erlenmeyer flask and dilute to about 150 ml with water. Add 10 ml of a 1 + 2 solution by volume of sulfuric acid. Insert a Bunsen valve in the flask and heat the contents almost to boiling. Add 50 ml of the ferrous ammonium sulfate solution prepared as specified in (a) by means of a pipet and boil the solution for 2 minutes. Cool the solution, loosen the valve, add 5 ml of concentrated phosphoric acid and 10 ml of a 10-percent solution of manganous sulfate. Titrate the excess of ferrous ion with 0.1N potassium permanganate solution to a faint pink end point. Calculate the percent potassium chlorate as follows:

$$\text{Percent potassium chlorate} = \frac{0.2043 A (B - C)(100 - D)}{W}$$

where: A = Normality of the potassium permanganate solution used,
B = Milliliters of potassium permanganate solution equivalent to the total volume of ferrous ammonium sulfate solution added,
C = Milliliters of potassium permanganate solution required for titration of the excess ferrous ion,
D = Percent moisture calculated in 4.2.4.2, and
W = Weight in grams of specimen.

4.2.4.4 Water-insoluble matter. Dry a filtering crucible of sufficient pore size to prevent clogging to constant weight at 100° to 110°C. Cool in a desiccator and weigh to the nearest 0.1 mg. Weigh to the nearest 0.1 g approximately 50 g of the specimen and dissolve in approximately 800 ml of water. Filter the solution through the weighed crucible and wash the residue with five portions of hot water. Retain the filtrate for subsequent determinations. Dry the crucible and residue for 3 hours at 100° to 110°C. Cool in a desiccator and weigh to the nearest 0.1 mg. Calculate the percent water-insoluble matter as follows:

$$\text{Percent water-insoluble matter} = \frac{100 A}{W}$$

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where: A = Weight in grams of residue, and
W = Weight in grams of specimen.

4.2.4.5 pH of water-soluble matter. Cool the filtrate from 4.2.4.4 to room temperature. Transfer to a 1-liter volumetric flask and dilute with water to the mark. Determine the pH of a portion of this solution by means of a suitable standardized pH meter. Save the remaining solution for later use.

4.2.4.6 Hypochlorites. To a 200-ml aliquot of the solution from 4.2.4.5, add a strip of potassium iodide-starch paper. There shall be no immediate appearance of a blue color which indicates the presence of hypochlorites. If the test for hypochlorites is negative, retain this solution for 4.2.4.7, if required. Disregard the test for chlorites if hypochlorites are present.

4.2.4.7 Chlorites. If a negative test for hypochlorites was obtained in 4.2.4.6, add 5 ml of 0.1N sulfuric acid to the solution and test with a fresh strip of potassium iodide-starch paper. There shall be no immediate presence of a blue color which indicates the presence of chlorites.

4.2.4.8 Chlorides. To a 100-ml aliquot of the solution from 4.2.4.5, add 1 ml of a 5-percent solution of potassium chromate and titrate to a permanent faint blood-red tinge with 0.1N silver nitrate solution. Calculate the percent chlorides as KCl as follows:

$$\text{Percent chlorides} = \frac{7.456 A B}{W}$$

where: A = Milliliters of silver nitrate solution used,
B = Normality of silver nitrate solution, and
W = Weight in grams of specimen in aliquot from 4.2.4.5.

4.2.4.9 Bromates.

(a) Starch indicator solution. Mix 2 g of soluble starch and several milligrams of mercuric iodide, CP grade, (as a preservative) with a little water. Add the mixture slowly to 500 ml of boiling water. Allow the liquid to boil for 5 minutes; then cool to room temperature.

(b) Procedure. To a 200-ml aliquot of solution from 4.2.4.5 in a 500-ml iodine flask, add 5 ml of a freshly prepared 10-percent potassium iodide solution, 10 ml of 1N hydrochloric acid and 15 ml of the starch indicator solution prepared as specified in (a). Stopper the flask, swirl to mix, and set aside in the dark for one hour. Prepare a reagent blank as follows: Place 200 ml of freshly boiled and cooled water in a 500-ml iodine flask. Add 10 ml of 1N hydrochloric acid and 15 ml of the starch solution prepared as specified in (a). Stopper the flask, swirl to mix, and set aside in the dark for one hour. Titrate the specimen and the blank against 0.05N sodium thiosulfate solution to the starch end-point, using a microburet calibrated in 0.01-ml divisions. Calculate the percent bromates as KBrO₃ as follows:

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$$\text{Percent bromates} = \frac{2.78 A (B-C)}{W}$$

where: A = Normality of sodium thiosulfate solution,
 B = Milliliters of sodium thiosulfate solution required to titrate specimen,
 C = Milliliters of sodium thiosulfate solution required to titrate blank, and
 W = Weight in grams of specimen in aliquot from 4.2.4.5.

4.2.4.10 Heavy metals. To 25 ml of the solution from 4.2.4.5, add 1 ml of a 0.1N hydrochloric or sulfuric acid solution, and pass hydrogen sulfide through the solution for 30 seconds. No precipitate or coloration shall result.

4.2.4.11 Alkaline earths. To a 25-ml aliquot of the specimen solution from 4.2.4.5, add 1 ml of a 1 + 9 solution of ammonium hydroxide and 5 ml of a 10-percent solution of ammonium oxalate. Heat the solution nearly to boiling. No precipitate shall be found when the liquid is cooled to room temperature. (Not applicable to grade C).

4.2.4.12 Sodium. Determine the sodium content of the specimen in accordance with the method for determining sodium in potassium chlorate in Reagent Chemicals, American Chemical Society Specifications, except use 0.5 g of specimen instead of 2.0 g.

4.2.4.13 Magnesium carbonate (grade C only).

(a) Standard magnesium solution. Dissolve exactly 0.5000 g of polished magnesium metal ribbon in 50 ml of a 1 + 10 solution of hydrochloric acid and dilute to 500 ml with water.

(b) Buffer solution. Dissolve 60 g of ammonium chloride in about 200 ml of water. Add 570 ml of concentrated ammonium hydroxide and dilute to 1 liter with water.

(c) Indicator solution. Dissolve 0.15 g of Eriochrome Black T and 0.50 g of borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) in 35 ml of methanol and warm to effect solution.

(d) Standard disodium dihydrogen ethylenediaminetetraacetate (EDTA). Dissolve 8.00 g of disodium dihydrogen EDTA in 2 liters of water. Pipet 10 ml of standard magnesium solution prepared as specified in (a) into a 250-ml Erlenmeyer flask. Add 25 ml of water, 10 ml of buffer solution prepared as specified in (b), and 3 or 4 drops of indicator solution prepared as specified in (c). Titrate against a white background under an incandescent light with the disodium dihydrogen EDTA solution to a pure blue end point with no trace of pink. Calculate the molarity of the disodium dihydrogen EDTA solution as follows:

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$$\text{Molarity of disodium dihydrogen EDTA} = \frac{0.4112}{A}$$

where: A = Milliliters of disodium dihydrogen EDTA used to titrate standard magnesium solution.

(e) Procedure. Weigh to the nearest mg approximately 1 g of the specimen. Transfer to a 250-ml Erlenmeyer flask, dissolve in 10 ml of a 1 to 10 solution of hydrochloric acid, add 25 ml of water, 10 ml of buffer solution prepared as specified in (b), and 3 or 4 drops of indicator solution prepared as specified in (c). Titrate as specified in (d) and calculate the percent magnesium carbonate as follows:

$$\text{Percent magnesium carbonate} = \frac{8.433 AB}{W}$$

where: A = Milliliters of disodium dihydrogen EDTA solution used,
 B = Molarity of disodium dihydrogen EDTA solution calculated in (d),
 and
 W = Weight in grams of specimen.

4.2.4.14 Granulation.

(a) Classes 1, 2, 3, 4, 5, and 7. Select sieves conforming to RR-S-366, as applicable (see table II). Weigh to the nearest 0.01 g and nest in order of fineness with the most coarse sieve on top. Place the assembly on a bottom pan. Weigh to the nearest 0.01 g approximately 100 g of the specimen and place it on the first (most coarse) sieve. Break lumps due to moisture by gently brushing with a camel's hair brush. Cover the sieves and shake for 10 minutes by hand or for 5 minutes by means of a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. Weigh the material retained on each sieve and calculate the percent material passing through each sieve as required by table II.

(b) Class 6.

(1) Preparation of isopropyl alcohol wash solution. Transfer a 5 g portion of the specimen to a 250-ml Erlenmeyer flask and add 150 ml of isopropyl alcohol. Place the flask on a shaking machine and shake for 1 hour at a temperature of $25^\circ \pm 5^\circ\text{C}$. Decant and filter the solution through Whatman No. 41 filter paper. Retain the filtrate for use as wash solution.

(2) Procedure. Weigh to the nearest 0.01 g approximately 10 g of the specimen and place on a 3-inch diameter No. 325 sieve conforming to RR-S-366. Wash the material with a steady gentle stream of the isopropyl alcohol wash solution prepared as specified in (b)(1), at a temperature of $25^\circ \pm 5^\circ\text{C}$ from a wash bottle, breaking any lumps formed by touching them with a glass rod. Continue washing until no more specimen passes through the sieve, catching the washings in a beaker. During the washing shake the sieve gently.

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Occasionally tap the bottom of the screen with a glass rod. Dry the sieve on a steam bath until the odor of alcohol can no longer be detected and then dry in an oven at 100° to 105°C for 15 minutes. Cool in a desiccator, weigh the residue, and calculate the percent passing through.

5. PACKAGING

5.1 Packing. Packing shall be level B or C as specified (see 6.2).

5.1.1 Level B. Uniform quantities of 210 to 300 pounds of potassium chlorate shall be packed in a nominal 30-gallon, fully removable head, clean, dry steel drum in accordance with Department of Transportation (DOT) regulations. The drum shall not be one which has been used for hazardous chemicals. The drum shall be fitted with a bag formed from polyethylene of 0.004 inch thickness conforming to type I, grade and finish optional of L-P-378. All seams except that of the closure shall be completely heat sealed and shall meet the heat-sealed seam requirements of L-P-378. The filled bag shall be closed by tying or knotting. Two units of desiccant conforming to MIL-D-3464 shall be placed on top of the closed bag prior to closure of the drum. Drum closure shall be fully gasketed and shall form a tight seal between the rim and top of the drum.

5.1.2 Level C. Potassium chlorate shall be packed as specified in 5.1.1 except that no desiccant shall be required.

5.2 Marking. In addition to the precautionary marking specified in 5.2.1, shipping containers shall be marked in accordance with DOT regulations and MIL-STD-129. In addition, the yellow "OXIDIZING MATERIAL" label specified by DOT regulations shall be applied to each shipping container.

5.2.1 Precautionary marking. The side of each container shall be durably and legible marked as follows:

HAZARD
Strong oxidizer

DANGER! STRONG OXIDIZER

CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE

Keep from contact with clothing and other combustible materials. Do not store near combustible materials. Store in tightly closed container. Remove and wash contaminated clothing promptly.

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6. NOTES

6.1 Intended use. Potassium chlorate is intended for the following uses:

- Grade A - as an ingredient in primer mixtures.
- Grade B - as an ingredient in pyrotechnic mixtures.
- Grade C - as an ingredient in colored smoke mixtures.

6.2 Ordering data. Acquisition documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Grade and class of potassium chlorate required (see 1.2).
- (c) Level of packing required (see 5.1).

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

6.4 Significant places. For the purpose of determining conformance with this specification, an observed or calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM E29.

6.5 Sampling and testing precautions. This specification covers inspection of chemical material which is potentially dangerous to personnel. Potassium chlorate is a strong oxidizer. All applicable safety rules, regulations and procedures must be followed in the handling and processing of this material. When potassium chlorate is rubbed with organic substances, sulfides, or readily oxidizable substances, it may react with explosive violence. The material should be stored in a dry place, and containers should be kept tightly closed.

Custodians:

Army - EA
Navy - OS

Preparing activity:

Army - EA

Project No. 6810-B334

Review activities:

Army - AR, MD
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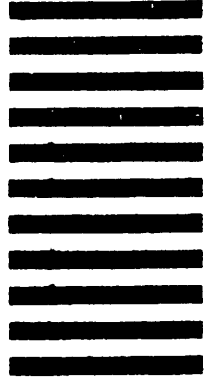


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