INCH–POUND

MIL–DTL–150E
5 August 2008

SUPERSEDING
MIL–P–150D
w/Amendment 1
28 April 1999

DETAIL SPECIFICATION

POTASSIUM CHLORATE, TECHNICAL

Inactive for new design after 19 Apr 1996.

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 ($KClO_3$).

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

Grade A	–	Low bromate
Grade B	–	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

Comments, suggestions, or questions on this document should be addressed to: U.S. Army Edgewood Chemical Biological Center, ATTN: AMSRD–ECB–ENA–S, 5183 Blackhawk Road, Aberdeen Proving Ground, MD 21010–5424 or emailed to SpecsTeam@apgea.army.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.

AMSC N/A FSC 6810

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 or 4 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 insure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3 or 4 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 cited in the solicitation or contract.

AMERICAN CHEMICAL SOCIETY

ACS – Reagent Chemicals, 10th edition.

(Copies of this document are available from http://pubs.acs.org/reagents or American Chemical Society, 1155 Sixteenth Street, N.W., Washington, DC 20036.)

ASTM INTERNATIONAL

D 1193 – Standard Specification for Reagent Water
E 11 – Standard Specification for Wire Cloth and Sieves for Testing Purposes

(Copies of this document are available from www.astm.org or ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428–2959.)

2.3 Order of precedence. 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 First article. When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.2.

3.2 Appearance. Potassium chlorate shall be a white, crystalline powder when tested as specified in 4.4.1.

3.3 Chemical characteristics. Potassium chlorate shall conform to the applicable chemical characteristics of table I when tested as specified therein. For Grade C, Tricalcium Phosphate, in a quantity not exceeding 0.25 percent of the weight of potassium chlorate, may be mixed with potassium chlorate to prevent caking.

TABLE I. Chemical characteristics.

Characteristic	Percent by weight			Test Paragraph
	Grade A	Grade B	Grade C	
Moisture, maximum	0.05	0.05	0.05	4.4.2
Assay (as $KClO_3$), minimum	99.5	99.5	96.5	4.4.3
Water−insoluble matter, maximum	0.02	0.10	3.2	4.4.4
pH of water−soluble matter	5 to 8	5 to 8	−−−−	4.4.5
Hypochlorites	To pass test	To pass test	To pass test	4.5.6
Chlorites	To pass test	−−−−	−−−−	4.5.7
Chlorides (as KCl), maximum	0.10	0.10	0.10	4.5.8
Bromates (as $KBrO_3$), maximum	0.02	0.10	0.10	4.5.9
Heavy metals	To pass test	To pass test	To pass test	4.5.10
Alkaline earths	To pass test	To pass test	−−−−	4.5.11
Sodium, maximum	0.04	0.04	0.04	4.5.12
Magnesium carbonate content	−−−−	−−−−	3.0 ± 0.2	4.5.13

3.4 Granulation characteristics. Potassium chlorate shall conform to the applicable granulation characteristics of table II when tested as specified in 4.4.14.

TABLE II. Granulation characteristics.

Sieve Size No.	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
50	−−	−−	−−	−−	−−	−−	−−	−−	98.0	−−	−−	−−	−−	−−
60	−−	−−	−−	−−	99.5	−−	−−	−−	−−	−−	−−	−−	−−	−−
80	99.0	−−	−−	−−	60.0	−−	−−	−−	−−	−−	−−	−−	99.0	−−
100	−−	−−	99.9	−−	−−	−−	99.9	−−	85.0	−−	−−	−−	−−	−−
140	45.0	55.0	−−	−−	−−	−−	45.0	55.0	−−	−−	−−	−−	−−	−−
200	17.0	23.0	−−	5.0	−−	−−	−−	40.0	40.0	70.0	−−	−−	90.0	−−
325	−−	−−	−−	−−	−−	−−	−−	−−	−−	−−	95.0	−−	60.0	−−

4. VERIFICATION

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

(a) First article inspection (see 4.2)
(b) Conformance inspection (see 4.3)

4.2 First article inspection.

4.2.1 Lotting. The first article lot shall consist of at least 50 kg of technical potassium chlorate manufactured using the same methods, materials, equipment, and processes as will be used during regular production. The first article sample shall be submitted for inspection and approval in accordance with the terms of the contract. If there is any change of vendors, methods, materials, equipment, facilities, or if there is a break in production of 90 days or more, the contracting Officer shall be notified and at the discretion of the Government, a new first article sample may be required.

4.2.2 Sampling. The contractor shall randomly select three representative samples, of approximately one kg each, from the first article lot of potassium chlorate and place each sample in a clean dry container labeled to identify the first article lot.

4.2.3 Inspection procedure. Each first article sample shall be subjected to all of the inspections specified in the classification of characteristics in 4.3.5.

4.2.4 Acceptance criteria. If any first article sample fails to comply with any of the applicable requirements, the first article sample shall be rejected.

4.3 Conformance inspection.

4.3.1 Lotting. A lot shall consist of the potassium chlorate produced by one manufacturer, at one plant, from the same materials, under the same manufacturing conditions, and at the same time. The minimum lot size shall be 500 kg. In the event the lot is a batch operation (see 6.3), several batches may be mixed together to form a lot, provided the batches are subjected to some physical mixing operation intended to make the final product uniform and homogenous.

4.3.2 Sampling. The contractor shall randomly select three representative samples, of approximately one kg each, from the lot of potassium chlorate and place each sample in a clean dry container labeled to identify the lot represented.

4.3.3 Inspection procedure. Each potassium chlorate sample shall be inspected in accordance with the classification of characteristics in 4.3.5. Failure of any sample to conform to any characteristic in the classification of characteristics based acceptance criteria specified therein shall be cause for rejection of the lot represented.

4.3.4 Inspection characteristics. Critical characteristics are characteristics whose nonconformance to specified requirements is likely to result in hazardous or unsafe conditions for individuals who use or maintain the product. Characteristics whose nonconformance to specified requirements is likely to prevent performance of the tactical function of a major end item are also critical characteristics. Major characteristics are characteristics whose nonconformance to specified requirements is likely to result in failure or to reduce materially the usability of the item for its intended purpose. Minor characteristics are characteristics whose nonconformance to specified requirements is not likely to reduce materially the operation or usability of the item for its intended purpose.

4.3.5 Classification of characteristics. Conformance inspections shall be as specified in the following classification of characteristics paragraphs.

CLASSIFICATION OF CHARACTERISTICS

PARAGRAPH	TITLE		DRAWING NUMBER
4.3.5(a)	Potassium Chlorate, Technical	SHEET 1 OF 1	

CATEGORY	CHARACTERISTIC	SAMPLING AND ACCEPTANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD
Critical	None defined			
Major				
101	Appearance	See note 1	3.2	4.4.1
102	Chemical properties	See note 1	3.3	
	a. Moisture			4.4.2
	b. Assay			4.4.3
	c. Water–insoluble matter			4.4.4
	d. pH of water–soluble matter			4.4.5
	e. Hypochlorites			4.4.6
	f. Chlorites			4.4.7
	g. Chlorides			4.4.8
	h. Bromates			4.4.9
	i. Heavy metals			4.4.10
	j. Alkaline Earths			4.4.11
	k. Sodium			4.4.12
	l. Magnesium carbonate			4.4.13
103	Granulation properties	See note 1	3.4	4.4.14

NOTES:

1. Sampling shall be in accordance with 4.3.2. One specimen shall be taken from each sample to perform each test.

4.4 Tests.

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

4.4.2 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:

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

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

4.4.3 Assay (as $KClO_3$).

(a) Ferrous ammonium sulfate solution. Dissolve 40 g of ferrous ammonium sulfate $[Fe(NH_4)_2(SO_4)_2 \bullet 6H_2O]$ 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. 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 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 manganese 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:

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

where: A = Normality of the potassium permanganate solution used,
B = Milliliters of the 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 iron,
D = Percent moisture calculated in 4.4.2, and
W = Weight in grams of specimen.

4.4.4 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 5 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:

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

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

4.4.5 pH of water−soluble matter. Cool the filtrate from 4.4.4 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.

4.4.6 Hypochlorites. To a 200 mL aliquot of the solution from 4.4.5, 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 4.4.7, if required. Disregard the test for chlorites if hypochlorites are present.

4.4.7 Chlorites. If a negative test for hypochlorites was obtained in 4.4.6, 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.

4.4.8 Chlorides. To a 100 mL aliquot of the solution from 4.4.5, 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:

$$\text{Percent chlorides} = \frac{7.456AB}{W}$$

where: A = Milliliters of silver nitrate solution used,
B = Normality of silver nitrate solution, and
W = Weight in grams of specimen in aliquot from 4.4.5.

4.4.9 Bromates.

4.4.9.1 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.

4.4.9.2 Procedure. To a 200 mL aliquot of solution from 4.4.5 in a 500 mL iodine flask, add 5 mL of 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 4.4.9.1. 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 4.4.9.1. 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 $KBrO_3$ as follows:

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

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 4.4.5.

4.4.10 Heavy metals. To 25 mL of the solution from 4.4.5, 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.

4.4.11 Alkaline earths. To a 25 mL aliquot of the specimen solution from 4.4.5, 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).

4.4.12 Sodium. Determine the sodium content of the specimen in accordance with the method for determining sodium in potassium chlorate in the ACS reference titled *Reagent Chemicals*. For Grade C, if Tricalcium Phosphate is used to prevent caking, the sodium level in the Potassium Chlorate should conform to Table I before the Tricalcium Phosphate is added.

4.4.13 Magnesium carbonate (grade C only).

4.4.13.1 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.

4.4.13.2 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 L with water.

4.4.13.3 Indicator solution. Dissolve 0.15 g of Eriochrome Black T and 0.50 g of borax ($Na_2B_4O_7 \bullet 10H_2O$) in 35 mL of methanol and warm to effect solution.

4.4.13.4 Standard disodium dihydrogen ethylenediaminetetraacetate (EDTA). Dissolve 8.00 g of disodium dihydrogen EDTA in 2 L of water. Pipet 10 mL of standard magnesium solution prepared as specified in 4.4.13.1 into a 250 mL Erlenmeyer flask. Add 25 mL of water, 10 mL of buffer solution as prepared in 4.4.13.2, and 3 or 4 drops of indicator solution prepared as specified in 4.4.13.3. Titrate against a white background under an incandescent light with 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:

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

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

4.4.13.5 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 4.4.13.2, and 3 or 4 drops of indicator solution prepared as specified in 4.4.13.3. Titrate as specified in 4.4.13.4 and calculate the percent magnesium carbonate as follows:

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

where: A = Milliliters of disodium dihydrogen EDTA solution used,
B = Molarity of disodium EDTA solution calculated in 4.4.13.4, and
W = Weight in grams of specimen.

4.4.14 Granulation.

4.4.14.1 Classes 1, 2, 3, 4, 5, and 7. Select sieves conforming to ASTM E 11, 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 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.

4.4.14.2 Classes 6. Prepare an isopropyl alcohol wash solution by transferring 5 g 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°± 5°C. Decant and filter the solution through Whatman No. 41 filter paper. Retain the filtrate for use as a wash solution. 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 ASTM E 11. Wash the material with a steady gentle steam of the isopropyl alcohol wash solution at a temperature of 25°± 5°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. 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 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual 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 requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Service 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. 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 Acquisition requirements. Acquisition documents should specify the following:

(a) Title, number, and date of this specification.
(b) Grade and class of potassium chlorate required
(c) First article:

(1) Time allowed for contractor submission of samples for Government test and evaluation after award of contract when testing is performed by the Government.
(2) Name and address of test facility and shipping instructions when testing is per-

formed by the Government.

(3) Time required for the Government to notify the contractor whether or not to proceed with production.

(d) Packaging requirements (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 Changes from previous issues. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

6.5 Subject term (key word) listing.

Colored smoke mix
Primer mix
Pyrotechnic mix
Sieve tray

Custodians:
Army – EA
Navy – OS

Preparing activity:
Army – EA
(Project 6810–2007–005)

Review activities:
Army – AR, MD
Navy – AS
DLA – GS

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.