MIL-P-193A

28 JULY 1958
SUPERSEDING
JAN-P-193
1 MARCH 1945

MILITARY SPECIFICATION

POTASSIUM SULFATE (FOR ORDNANCE USE)

This specification has been approved by the Department of Defense and is mandatory for use by the Departments of the Army, the Navy, and the Air Force.

1. SCOPE

1.1 Scope. This specification covers potassium sulfate intended for Ordnance applications.

1.2 Classification. Potassium sulfate furnished under this specification shall be of the following types:

Type I — for use in the manufacture of propellants.

Type II — for use in flash-reducing mixtures.

Type III — as a filter in thermosetting resin potting applications.

Type IV — for use in ballistic salt rods.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids, form a part of this specification:

SPECIFICATIONS

FEDERAL

RR-S-366 — Sieves, Standard for Testing Purposes.

UU-S-48 — Sacks, Paper, Shipping.

STANDARDS

MILITARY

MIL-STD-129 — Marking for Shipment and Storage.

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

3. REQUIREMENTS

3.1 Chemical requirements. Potassium sulfate, types I or II shall comply with the requirements specified in table I, when determined as specified in the applicable test paragraphs under 4.4.

TABLE I. *Chemical requirements*

Potassium sulfate purity (moisture-free basis):	
Based on sulfate content.	99.0 percent, minimum
Based on potassium content.	99.0 percent, minimum
Moisture	1.0 percent, maximum
Insoluble matter	0.1 percent, maximum
Grit	None
Acidity	0.01 percent, maximum
Alkalinity	0.03 percent, maximum
Chlorides (as potassium chloride).	0.02-percent, maximum

3.2 Granulation requirements. Potassium sulfate shall comply with the granulation re-

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TABLE II. *Granulation requirements*

U. S. Standard sieve [1]	Type I percent		Type II percent		Type III percent		Type IV percent	
	Min	Max	Min	Max	Min	Max	Min	Max
Through No. 4			100					
Retained on No. 12			25	35				
Through No. 50	100							
Retained on No. 70		5		10				5
Through No. 100							90	
Through No. 200					100			
Retained on No. 325						20		

[1] The sieves shall conform to Specification RR-S-366.

quirements specified in Table II, for the applicable type, when determined as specified in 4.4.7.

3.3 Average particle size. The average particle size for Type III shall be 0.5 microns minimum.

4. QUALITY ASSURANCE PROVISIONS

4.1 Lot. A lot shall consist of material from the same batch or blending operation, and subjected to the same processing operations and conditions.

4.2 Sampling. Ten percent of the containers in the lot, but in no case more than 10 nor less than 3 containers, shall be selected so as to be representative of the lot. If there are less than 3 containers in the lot, all the containers shall be sampled. The material shall be mixed to a uniform consistency throughout, and approximately 8 ounces of it shall be taken from each selected container. The primary samples shall be placed in separate airtight containers, labeled so that the container from which each sample was taken can be identified. A composite sample, of approximately 1 pound shall be made from equal portions of the primary samples. The composite sample shall be thoroughly mixed and placed in an airtight container, labeled to show the name of the material, manufacturer, plant, contract or purchase order number, and lot size. All acceptance tests shall be made on the composite sample. However, if it becomes apparent during sampling that the lot is not uniform, it may be required that any primary sample be tested for compliance with the requirements of the specifications. All primary samples shall be held for possible future examination should the composite sample fail to comply with the requirements.

4.3 Inspection.

4.3.1 *General.* All tests and inspections shall be performed by the contractor except those tests and/or inspections specifically reserved for performance by the Government as stated herein and/or by the contract. Certification shall be furnished to the Government inspector, prior to or at the time of delivery, showing the tests and inspections conducted and the results obtained, and shall be signed by a responsible agent of the supplier, and shall be accompanied by evidence of the agents' authority to contractually bind the supplier. The government may, at its option, repeat any or all of the inspections specified herein.

4.3.1.1 *Test equipment and inspection facilities.* The manufacturer shall furnish and maintain all test equipment and inspection facilities for making all inspection, and shall maintain complete inspection records. Test equipment and inspection facilities shall be of sufficient accuracy, quality, and quantity to permit performance of the required inspection. The manufacturer shall establish calibration of test equipment to the satisfaction of the Government.

4.3.1.1.1 *Commercial facilities.* Manufacturers not having test equipment and inspection

facilities satisfactory to the Government may engage the services of a commercial laboratory acceptable to the Government.

4.3.2 *Packing and marking.* It shall be ascertained that the packing of the potassium sulfate and the marking of the container conform to this specification.

4.4 Test procedures.

4.4.1 *Determination of moisture.* A weighed portion of approximately 5 grams (g) of the specimen shall be transferred to a tared moisture dish and heated at 100° to 105° C. for 2 hours. The tared moisture dish and contents shall be cooled in a desiccator and weighed. The loss in weight shall be calculated to percentage of moisture in the sample.

4.4.2 *Determination of potassium sulfate.*

4.4.2.1 *Based on sulfate content.*

4.4.2.1.1 *Preparation of the ion-exchange column.* Fifty milliliters (ml) of water shall be added to 50 g. of Dowex 50 (50–100 mesh) or equivalent cation exchange resin and allowed to stand for one hour or more until the swelling of the resin particles ceases. The resin shall be transferred to a Jones reductor column provided with a glass stopcock. The resin shall rest on a pad of glass wool supported by a Witt plate and the resin bed shall be approximately 8 centimeters (cm) high and 3 cm in diameter. If the resin is in the sodium form, the resin shall be converted to the acid form by passing 500 ml of 6 molar (m.) hydrochloric acid through the column and then washed with water until the effluent shows a pH or 5 or higher. A 0.5 specimen of potassium sulfate shall be dissolved in 50 ml of distilled water and transferred to the ion-exchange column. The solution shall be drained through the column at a rate not exceeding 35 ml per minute. While draining the solution from the column, the liquid level should never be allowed to fall below the top of the resin bed. The column shall then be washed with four 50-ml portions of distilled water. The column is then ready for use. The resin should be kept covered with water when the column is not in use. A column of this size will handle ten 0.5 g. samples of potassium sulfate before regeneration is necessary. Regenerate by passing 50 ml of 6 M. hydrochloric acid through the column, then wash with water until the effluent shows a pH of 5 or higher. The resin bed should be removed from the column, slurried with distilled water, and replaced in the column at this time.

4.4.2.1.2 *Procedure.* An accurately weighed specimen of approximately 0.5 g. of potassium sulfate shall be transferred to a 250-ml. beaker and dissolved in 50 ml of distilled water. The solution shall be transferred to the ion-exchange column. The solution shall be drained through the column at a rate not exceeding 35 ml. per minute passing the effluent through a paper filter and catching it in a 600-ml. beaker. The column shall be washed with four 50-ml. portions of distilled water. One and one-half ml of concentrated hydrocloric acid shall be added to the solution in the 600-ml beaker and heated to boiling. Twenty ml of 5 percent barium chloride solution (5g. of $BaCl_2$. $2H_20$ in 100 ml. of solution) shall be diluted to 50 ml. and this solution added dropwise with good stirring over a period of 5 minutes to the hot sample solution. The precipitate shall be allowed to settle and then tested for complete precipitation by adding a few more drops of the barium chloride solution. The beaker shall be covered with a watch glass, digested on a steam bath for at least 3 hours, and then filtered through a carefully prepared Gooch or fine porosity Selas (porcelain) filtering crucible, which has previously been ignited to constant weight (= 0.2 milligrams (mg.) at 600 degrees centigrade (° C.). If any precipitate runs through the filter, the filtrate shall be poured back through the crucible repeatedly until the solution runs through perfectly clear. After most of the supernatant liquid has been poured through the crucible, the precipitate shall be quantitatively transferred to the crucible. A rubber police-

man shall be used to remove any precipitate adhering to the walls of the beaker or stirring rod. The precipitate shall be washed with water at room temperature until the washings give a negative chloride test with water at room temperature until the washings give a negative chloride test with silver nitrate. The crucible and contents shall be dried for one-half hour at 105° C., then ignited for 1 hour at 600° ± 50° C., cooled in a desiccator and weighed. The ignition shall be repeated for 10 minute periods until constant weight (± 0.2 mg.) is obtained. The percentage of potassium sulfate in the sample shall be calculated on a moisture-free basis as follows:

$$\text{Potassium sulfate, percent} = \frac{74.65A}{W}$$

where:

A = weight of precipitate

W = weight of specimen corrected for moisture.

4.4.2.2 *Based on potassium content.*

4.4.2.2.1 *Preparation of solutions.*

4.4.2.2.1.1 *0.2M Aluminum chloride.* Five g. of aluminum chloride $AlCl_3 \cdot 6H_2O$) shall be dissolved in about 50 ml. of distilled water and sufficient distilled water shall be added to give 100 ml. of solution.

4.4.2.2.1.2 *0.1N Sodium tetraphenylboron.* Thirty-four g. of sodium tetraphenylboron analytical reagent grade shall be dissolved in 900 ml. of distilled water at room temperature and diluted to approximately 1 liter. Four milliliters of 0.2M aluminum chloride solution shall be added. The solution shall be allowed to stand until it clears. The solution shall be filtered and stored in a polyethylene bottle. The solution shall be re-filtered before use if any turbidity or suspended matter is present, but should not be used when older than 48 hours.

4.4.2.2.1.3 *Saturated potassium tetraphenylboron wash solution.* A quantity of potassium tetraphenylboron shall be prepared from reagent grade potassium sulfate using the procedure described in 4.4.2.2.2 except that the precipitate shall be washed with 50 ml. of 0.5 percent acetic acid solution. A 0.35 g. portion of this potassium tetraphenylboron shall be shaken for 2 hours with 300 ml. of distilled water contained in a glass stoppered flask, and the resulting solution filtered through a No. 40 Whatman filter paper.

4.4.2.2.2 *Procedure.* An accurately weighed portion of approximately 0.22 g. of potassium sulfate shall be transferred to a 250-ml beaker and dissolved in 100 ml. of distilled water. The solution shall be adjusted, if necessary, to a pH value of 5 to 6 with diluted acetic acid or sodium acetate. A few drops of 0.2M aluminum chloride solution shall be added and the solution heated to 70° C. Fifty milliliters of 0.1N sodium tetraphenylboron solution shall be added dropwise, with constant stirring. The suspension shall be cooled to room temperature and allowed to stand for 5 minutes. The solution shall be filtered and the precipitate collected on a tared, medium porosity glass filtering crucible. The precipitate shall be washed with five 10 ml. portions of the saturated potassium tetraphenylboron wash solution. The crucible and contents shall be dried at 120° ± 2° C. to constant weight, cooled in a desiccator, and weighed. The purity shall be calculated as follows:

$$\text{Purity, percent} = \frac{24.32A}{W}$$

where:

A = weight of dried percipitate.

W = weight of specimen corrected for moisture.

In case of failure of disagreement, 5 determinations shall be made and the average reported.

4.4.3 *Determination of insoluble matter and grit.* A weighed portion of approximately 50 g. of potassium sulfate shall be dissolved in approximately 500 ml. of hot distilled water. The solution shall be filtered through a tared,

fine porosity filtering crucible. The crucible and residue shall be washed with hot water until the washings no longer give a test for sulfate. The crucible and contents shall be dried at 100° to 105° C. for 1 hour, cooled in a desiccator and weighed. The increase in weight shall be calculated as percentage of insoluble matter in the sample. The material retained in the crucible shall be transferred to a smooth glass slide, and the material shall be pressed and rubbed between glass slides determining the presence of grit by scratching noise and scratches on the glass slides.

4.4.4 *Determination of acidity.* A weighed portion of approximately 20 g. of potassium sulfate shall be dissolved in approximately 250 ml. of freshly boiled, and cooled, distilled water. If no red coloration appears upon addition of a few drops of phenolphthalein indicator, the solution shall be titrated with 0.02N sodium hydroxide. Correction shall be made for the volume of sodium hydroxide used in a blank determination. Any acidity present shall be calculated as percentage of sulfuric acid as follows:

$$\text{Percentage of sulfuric acid} = \frac{4.90VN}{W}$$

where:

V = corrected volume of sodium hydroxide solution.
N = normality of sodium hydroxide solution.
W = weight of specimen.

4.4.5 *Determination of alkalinity.* If the solution obtained 4.4.4 is alkaline to phenolphthalein, the solution shall be titrated with 0.5N sulfuric acid. A blank determination shall be run to correct the volume of acid used. Alkalinity shall be calculated to percentage of potassium hydroxide as follows:

$$\text{Percentage of potassium hydroxide} = \frac{5.61VN}{W}$$

where:

V = corrected volume of sulfuric acid solution used.
N = normality of sulfuric acid solution.
W = weight of specimen.

4.4.6 *Determination of chlorides (as potassium chloride).* A weighed portion of approximately 50 g. of potassium sulfate shall be transferred to a beaker, dissolved in 400 ml. of distilled water, and the solution filtered. Twenty milliliters of 5 percent nitric acid solution and 5 ml. of 5 percent silver nitrate solution shall be added to the filtrate. The solution shall be heated to boiling and agitated vigorously to coagulate the precipitate of silver chloride. Exposure of the precipitate to strong light shall be avoided, as strong light causes the exposed surface to decompose and liberate chlorine. The solution shall be filtered through a tared filtering crucible and the residue washed with 1 percent nitric acid. The crucible and residue shall be dried at 100° to 105° C. then heated 130° to 150° C. The crucible and residue shall be dried at 100° to 105° C. The crucible and residue shall be cooled in a desiccator, and weighed. The increase in weight shall be calculated to percentage of potassium chloride in the sample as follows:

$$\text{Percentage of potassium chloride} = \frac{52.02A}{W}$$

where:

A = weight of silver chloride.
W = weight of specimen.

4.4.7 *Determination of granulation.* An accurately weighed portion of approximately 100 g. of potassium sulfate shall be placed on the specified nest of sieves complying with Specification RR-S-366, properly arranged and assembled with a bottom pan. Two metal washers, weighing not more than 15 gm. each, shall be added to the upper sieve to help break up lumps. The nest of sieves shall be covered and shaken for 5 minutes by hand or mechanically by means of a shaker geared to produce 300 ± 15 gyrations, and 150 ± 10 taps of the striker per minute. If necessary, the material on each sieve, beginning with the top one, shall be brushed lightly with a ½ inch flat bristle brush until no further material passes through each sieve. The portions retained on the various sieves shall be weighed, and the results shall be calculated to a percentage basis as required.

4.4.8 *Determination of average particle size.* The average particle size of Type III only shall be determined on a Fisher Sub-Sieve Sizer or equivalent instrument.

4.4.5 *Acceptance criteria.* A submitted lot shall be acceptable if the sample selected according to paragraph 4.2 meets each requirement of section 3. based on the determinations performed as specified herein. Otherwise, the lot shall be rejected.

4.6 **Rejection and resubmission.** When a lot fails to meet the requirements specified herein, it shall be returned to the manufacturer. If the rejected lot is resubmitted for acceptance, it must be designated a resubmitted lot and evidence of reprocessing furnished the inspection agency. The failure of a resubmitted lot to meet the requirements specified herein permanently rejects the lot unless otherwise specified by the contracting agency.

5. PREPARATION FOR DELIVERY

5.1 Packing (see 6.1).

5.1.1 *Level A.* Fifty pounds of potassium sulfate shall be packed in a shipping sack conforming to type II, style No. 1W, class C, of Specification UU-S-48. The closure and waterproofing of the ends shall be accomplished in accordance with the requirements contained in Specification UU-S-48.

5.1.2 *Level C.* Potassium sulfate shall be packed for shipment in conformance with Consolidated Freight Classification rules and container specifications for rail shipments, or with National Motor Freight classification rules and container specifications for truck shipments.

5.2 **Marking.** Containers shall be marked in accordance with Standard MIL-STD-129.

6. NOTES

6.1 Ordering data. Procurement documents should specify the following:

(a) Title, number, and date of this specification.

(b) Type required (see 1.2).

(c) Levels of protection (see 5.1)

6.2 Classification cross reference. The types of potassium sulfate under this specification are equivalent to the grades covered by Specification JAN-P-193, as follows:

MIL-P-193A	*JAN-P-193*
Type I	Grade A
Type II	Grade B

6.3 All equipment used for specification tests should be of sufficient accuracy and should be standardized according to normal laboratory procedures.

Custodians
Army—Ordnance Corps
Navy—Bureau of Ordnance
Air Force

Other interest:
Army—CM

Preparing activity:
Army—Ordnance Corps

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 119-R004

INSTRUCTIONS

This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity *(as indicated on reverse hereof)*.

SPECIFICATION

ORGANIZATION *(Of submitter)*

CITY AND STATE

CONTRACT NO.

QUANTITY OF ITEMS PROCURED

DOLLAR AMOUNT
$

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ SUBCONTRACT

1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?

A. GIVE PARAGRAPH NUMBER AND WORDING.

B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES.

2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID

3. IS THE SPECIFICATION RESTRICTIVE?

☐ YES ☐ NO IF "YES", IN WHAT WAY?

4. REMARKS *(Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)*

SUBMITTED BY *(Printed or typed name and activity)*

DATE

DD, FORM, APR 62 1426 REPLACES NAVSHIPS FORM 4863. ICH IS OBSOLETE

PLATE NO. 15419