

MIL-P-156B

18 APRIL 1956

SUPERSEDING

JAN-P-156A

9 JULY 1946

MILITARY SPECIFICATION**POTASSIUM NITRATE**

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 nitrate for use in the manufacture of propellants, chemical munitions, and pyrotechnic compositions.

1.2 Classification. Potassium nitrate shall be of the following classes, as specified (see 6.1 and 6.2):

Class 1—for use in black powder and chemical munitions.

Class 2—for use in pyrotechnic compositions.

Class 3—for use in propellants.

2. APPLICABLE DOCUMENTS

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

SPECIFICATION**FEDERAL**

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

MILITARY

JAN-P-113 —Packaging and Packing for Overseas Shipment-Bags, Shipping, Textile, and Paper-Laminated

MIL-G-2550 —General Specification for Ammunition Except Small Arms Ammunition

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.)

2.2 Other publications. The following document forms a part of this specification. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

INTERSTATE COMMERCE COMMISSION

49 CFR 71-78—Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles

(The Interstate Commerce Commission regulations are now a part of the Code of Federal Regulations (1949 Edition—Revised 1950) available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Orders for the above publications should cite "49 CFR 71-78 (Rev 1950).")

FED. SUP. CLASS. 6810

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3. REQUIREMENTS

3.1 **Chemical requirements.** Potassium nitrate shall comply with the chemical requirements specified in table I, when determined as specified in the applicable paragraphs of section 4.

TABLE I. *Chemical requirements*

Property	Class 1	Class 2	Class 3
	Percent	Percent	Percent
Moisture, maximum (max.)	0.2	0.2	0.2
Acidity	None	None	None
Alkalinity	None	None	None
Water insoluble material (max.)	0.1	0.1	0.1
Grit	None	None	None
Chlorides, as potassium chloride (KCl), (max.)	0.07	0.07	0.07
Chlorates and perchlorates, as potassium perchlorate (KClO ₄), (max.)	0.5	0.5	0.5
Iron and aluminum, as oxide, (max.)	0.5	0.5	-----
Calcium and magnesium, as oxides, (max.)	0.5	0.5	0.5
Sodium, as oxides, (max.)	0.25	0.25	-----
Ammonium compounds	None	None	-----
Nitrogen, minimum (min.)	13.77	13.77	13.77

3.2 **Odor.** The potassium nitrate shall be odorless.

3.3 **Color (crystals).** White.

3.4 **Granulation.**

3.4.1 *Class 1.* Class 1 potassium nitrate shall be furnished in lump form.

3.4.2 *Class 2 and 3.* Class 2 and 3 potassium nitrate shall comply with the granulation requirements shown in table II, using United States (U.S.) Standard sieves conforming to Specification RR-S-366, when tested as specified in 4.4.13.

TABLE II. *Granulation requirements (class 2 and 3)*

Sieve number (No.)	Percent, min., through specified sieve	
	Class 2	Class 3
60	99.9	-----
80	-----	99.9
100	40	-----

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 (see 6.3).

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 8 ounces, 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 this specification. 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.* Inspection shall be as specified in Specification MIL-G-2550 and as specified herein.

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

4.4 **Test procedures.** The determinations shall be conducted as follows:

4.4.1 *Determination of moisture.* Dry an accurately weighed portion of approximately 10 grams (gm.) of the sample in a tared

dish at 150 ± 2 degrees ($^{\circ}$) Centigrade (C.) to constant weight, cool in a desiccator and weigh. Calculate the loss in weight as percent moisture.

4.4.2 Determination of acidity. Dissolve a weighed portion of approximately 1 gm. of the sample in 20 milliliters (ml.) of neutral distilled water and determine if sensitive blue litmus paper is changed in color within 3 minutes, when immersed in the solution. The blue litmus paper shall be considered to be sufficiently sensitive if it shows a distinct reddish coloration, after being immersed for 3 minutes in .0001 normal (N) hydrochloric or sulfuric acid.

4.4.3 Determination of alkalinity. Using the solution from the acidity test determine if sensitive red litmus paper is changed in color within 3 minutes, when immersed in the solution. The red litmus paper shall be considered sensitive enough if it shows a distinct bluish coloration, after being immersed for 3 minutes in .0001N sodium hydroxide.

4.4.4 Determination of insoluble material. Dissolve a weighed portion of approximately 10 gm. of the sample in 100 ml. of boiling distilled water and filter the solution through a tared filtering crucible. Wash the insoluble residue on the filter with hot water. Dry the crucible and contents at 100° to 105° C. for 3 hours, cool in a desiccator, and weigh. Calculate the increase in weight as percent insoluble material.

4.4.5 Determination of grit. Dissolve a 25-gm. portion of the sample in water, filter through a medium porosity filter, and wash thoroughly. Transfer any material retained on the filter to a smooth glass slide. Press and rub the material with a smooth steel spatula against the glass slide. The presence of grit is indicated by the persistence of a scratching noise.

4.4.6 Determination of chlorides. Dissolve an accurately weighed portion of approximately 5 gm. of the sample in 100 ml. of distilled water and make the solution slightly acid with nitric acid. Add a few

drops of approximately 0.25N silver nitrate solution. If more than a faint opalescence results, add more silver nitrate solution until precipitation is complete. Boil for 2 minutes with constant stirring, and allow the precipitate to settle overnight in a dark place. Decant the solution through a tared filtering crucible. Wash the precipitate four times by decantation, using 20-ml. portions of water containing 1 ml. of concentrated nitric acid and 2 drops of silver nitrate solution per 100 ml. Transfer the precipitate to the crucible with 20 ml. of the same wash water, wash it with 20 ml. of 1-percent nitric acid solution and with two 20-ml. portions of distilled water. Dry the crucible and precipitate for 4 hours at 135° C., cool in a desiccator, and weigh. Calculate the percent chlorides, as KCl, as follows:

$$\text{Percent KCl} = \frac{52A}{W}$$

where:

A = weight of precipitate, in gm.

W = weight of sample, in gm.

4.4.7 Determination of chlorates and perchlorates. Transfer an accurately weighed portion of approximately 2 gm. of the sample to a platinum crucible, and mix with approximately 10 gm. of anhydrous sodium carbonate. Cover the mixture with a layer of sodium carbonate, fuse, and cool. Dissolve the fused mass in distilled water, render the solution slightly acid with nitric acid, and proceed with the precipitation as described in paragraph 4.4.6. Calculate the percent total chlorates and perchlorates, as KClO_4 , as follows:

$$\text{Percent KClO}_4 = \frac{97(A - 0.019BW)}{W}$$

where:

A = weight of precipitate, in gm.

B = percent KCl as determined in 4.4.6.

W = weight of sample, in gm.

4.4.8 Determination of iron and aluminum (applicable to classes 1 and 2 only). Dissolve a weighed portion of approximately 5 gm. of the sample in distilled water and

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filter the solution. Acidify the filtrate with nitric acid, add a slight excess of ammonium hydroxide, and boil. Catch the precipitate on the ashless filter paper and wash with hot distilled water. Retain the filtrate and washings for further analysis. Ignite the filter paper and precipitate, in a tared porcelain crucible, to constant weight. Cool the crucible and contents in a desiccator, and weigh. Calculate the increase in weight as percent iron and aluminum oxides.

4.4.9 Determination of calcium and magnesium. Dilute the combined filtrate and washings from the determination of iron and aluminum (see 4.4.8) to approximately 200 ml. and boil. Evaporate the filtrate to a volume of about 20 ml., filter, and add 15 ml. of 9N ammonium carbonate solution, and 15 ml. of 95-percent ethyl alcohol. Allow the mixture to stand for at least 30 minutes, with frequent stirring. Filter through paper and wash the precipitate with 9N ammonium carbonate solution. Dissolve the precipitate in dilute hydrochloric acid, dilute to approximately 50 ml., boil, and add 1 ml. of saturated ammonium oxalate solution. Allow the solution to stand for 12 hours, filter through a tared filtering crucible, and wash the precipitate with hot water containing ammonium oxalate. Ignite the precipitate, gently at first and then with a blast lamp, cool the crucible and contents in a desiccator, and weigh. Calculate the increase in weight as percent calcium oxide. Concentrate the filtrate from the calcium determination to approximately 50 ml., add 10 ml. of a 10-percent solution of ammonium phosphate, and 20 ml. of a 10-percent solution of ammonia. Allow the solution to stand for 4 or 5 hours, filter through a tared filtering crucible, and wash with a 2.5-percent solution of ammonia. Dry the precipitate and ignite to constant weight with a blast lamp. Cool the crucible and contents in a desiccator, and weigh. Calculate the increase in weight as percent magnesium oxide, as follows:

$$\text{Percent magnesium oxide} = \frac{36A}{W}$$

where:

A = weight of precipitate, in gm.

W = weight of sample, in gm.

4.4.10 Determination of sodium (applicable to classes 1 and 2 only).

4.4.10.1 Magnesium uranyl acetate reagent. Prepare solution A by dissolving 90 gm. of uranyl acetate, $\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$, and 60 ml. of glacial acetic acid in sufficient distilled water to make a volume of 1 liter by heating to 70°C., and stirring until solution is complete. Prepare solution B by dissolving 600 gm. of magnesium acetate, $\text{Mg}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$, in 60 ml. of glacial acetic acid and sufficient distilled water to make a volume of 1 liter by heating to 70°C., and stirring until solution is complete. Mix together solutions A and B while at 70°C. and cool the mixture to 20°C. After allowing the mixture to stand at this temperature for at least 2 hours, filter it through a dry filter paper into an amber-colored bottle. Store the bottle containing the solution where it is not exposed to direct sunlight. If a precipitate appears on standing, filter the solution again prior to use.

4.4.10.2 Alcohol wash liquid. Prepare an alcohol wash liquid as follows: Shake 1 ml. of 1-percent sodium chloride solution with 25 ml. of magnesium uranyl acetate reagent. Catch the precipitate on a filter paper and wash thoroughly with 95-percent ethyl alcohol. Mix the precipitate with 1 liter of 95-percent ethyl alcohol and allow to stand for 1 hour, with frequent shaking. Filter the solution and use the filtrate as a wash liquid.

4.4.10.3 Procedure. Transfer a weighed portion of approximately 0.2 gm. of the sample to a 150-ml. beaker. Dissolve it in 5 ml. of distilled water and cool the solution to 20°C. Add 100 ml. of the magnesium uranyl acetate reagent which has previously been cooled to 20°C. While maintaining the temperature at 20°C., rapidly stir the contents of the beaker for 1/2 hour by means of a mechanical stirrer. Transfer the precipitate to a tared filtering crucible, using a total of

25 to 30 ml. of the wash liquid for transferring and washing the precipitate. Dry the crucible and precipitate at 100° to 110°C. for ½ hour, cool in a desiccator, and weigh. Calculate the weight of the magnesium sodium uranyl acetate, $MgNa(UO_2)_2(C_2H_3O_2)_6 \cdot 6\frac{1}{2} H_2O$, as percent sodium oxide, as follows:

$$\text{Percent sodium oxide} = \frac{2.06A}{W}$$

where:

A = weight of precipitate, in gm.

W = weight of sample, in gm.

4.4.11 Determination of ammonium compounds (applicable to class 1 and 2 only). Transfer an accurately weighed portion of approximately 1.0 gm. of the sample to a 150-ml. beaker, dissolve in 25 ml. of distilled water, and add sufficient excess of concentrated sodium hydroxide solution to render the potassium nitrate solution alkaline. Heat the mixture and test the vapors with a moist strip of red litmus paper of such sensitivity that it changes color in 30 seconds, when immersed in 0.0005N alkali and stirred continuously. Care should be taken that no spray from the solution touches the test paper. Note if any ammonium compounds are present in the sample, as indicated by the paper becoming blue in color.

4.4.12 Determination of nitrogen.

4.4.12.1 Standardization of the nitrometer.

4.4.12.1.1 Absolute method. Assemble the nitrometer (duPont or equivalent) after thoroughly cleaning and drying all parts. The various parts, with the exception of the

reservoir bulbs, are completely filled with mercury. Place 25 ml. of 94.5 ± 0.5-percent sulfuric acid in the generating bulb, draw air into the bulb to nearly the level of the lower shoulder by lowering the mercury reservoir and opening both stopcocks. Close both stopcocks and shake vigorously for 3 minutes. Connect the generating bulb into the compensating tube by adjustment of the two mercury reservoirs, keeping open the stopcock of the measuring tube. Continue the introduction of the air until the mercury in the reading tube is approximately at the 12.50 mark. Seal the compensating tube by means of a small blowpipe flame. Desiccate similarly, in the generating bulb, another portion of the air and transfer this to the reading tube until the mercury in the reading tube, compensating tube, and their mercury reservoirs is at the same level, the mercury in the reading tube being approximately at the 12.50 mark. (It is not necessary that these gas volumes be exact or equal.) Fill a small U-tube manometer with sulfuric acid and attach to the reading tube. Allow the apparatus to come to constant temperature and then adjust the mercury levels in the reading tube, compensating tube, and their mercury reservoirs so that the air in the reading tube is under atmospheric pressure, as shown by the acid levels in the arm of the manometer tube. Take the reading on the graduated scale of the calibrated reading tube, and at the same time note the temperature and barometric pressure. From these readings calculate the volume of air at 20°C. and 760 millimeter (mm.) pressure in the reading tube, as follows:

$$V = \frac{R P \times 293 (1 - 0.00018t)}{760 (273 + T) (1 - 0.00018 \times 20)} = \frac{0.3869 PR (1 - 0.00018t)}{273 + T}$$

where:

V = ml. of air at 20°C. and 760 mm., barometer at 20°C.

R = ml. of air at the observed temperature (70°C.) and at the observed pressure, P, (in mm.)

t = temperature of the barometer, in °C.

0.00018 = coefficient of expansion of mercury in glass

Adjust the heights of the reading tube, compensating tube, and their reservoirs so that,

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with the mercury in the reading and compensating tubes on a level, the mercury in the reading tube marks the calculated volume of the air. Paste a strip of paper on the tube so as to mark the height of the mercury in the compensating tube. Consider this as the standard volume of air with which every volume of gas, to be measured, is compared.

(Note. The use of dry air tends to form a scum around the reading level of the compensating tube, thus requiring frequent restandardization of the nitrometer. This scum is caused by reaction of oxygen in the air with mercury, or by impurities. Dry nitrogen eliminates the condition and should be used, if available. A half drop of sulfuric acid will also prevent this scum.)

4.4.12.1.2 Potassium nitrate method. Recrystallize three times, from 95-percent alcohol, a sample of reagent grade potassium nitrate, and grind this to pass through a U. S. Standard No. 100 sieve. After drying at 135° to 150°C. for 2 or 3 hours, weigh a portion of exactly 1 gm. into a small weighing bottle and transfer this portion to the cup of the generating bulb. Wash the potassium nitrate into the generating bulb by means of eight to ten portions of 94.5 ± 0.5-percent nitrogen-free sulfuric acid cooled to approximately 5°C., using a total volume of 25 ml. of the acid. With the lower stopcock of the generating bulb open and the mercury reservoir low enough to maintain a slightly reduced pressure in the generating bulb, shake the sample until most of the gas has been generated. Adjust the mercury reservoir until the mercury drops nearly to the lower shoulder of the generating bulb. Close the lower stopcock and shake the bulb vigorously for 3 minutes. Replace the bulb on the rack, open the lower stopcock, and allow the tube to stand for 5 minutes, the levels in the generating bulb and reservoir being approximately the same, having previously introduced desiccated air into the compensating tube as described in 4.4.12.1.1. Close the lower stopcock, and shake the generating bulb for 1 additional minute. Transfer the gas to the reading tube, and adjust the levels of the mercury in the reading and compensating tubes to approximately the same height. Allow the gas to stand for

approximately 20 minutes, to permit equalization of the temperature. With a sulfuric acid U-tube leveling device make careful adjustments, so that the mercury in both tubes is at the same level, and the mark in the reading tube is at the 13.85 mark (the percentage of nitrogen in potassium nitrate). Make a mark on the compensating tube to show the volume occupied by the air equivalent to the volume of nitric oxide gas liberated from exactly 1 gm. of potassium nitrate. (This mark may be made on a strip of gummed paper attached to the compensating tube.)

(Note 1. In order to avoid any doubt regarding the purity of the potassium nitrate, it is desirable to check the standardization obtained by the use of potassium nitrate by means of the absolute method. If the agreement is within one or two hundredths of one percent, the potassium nitrate can be considered as of satisfactory quality and used in subsequent periodic standardizations. It has been found by actual experimentation that the solubility of nitric oxide in sulfuric acid, of the strength prescribed, is so small that no correction need be applied to bring the value in agreement with the mark set by the absolute method.)

(Note 2. If the nitrometer is standardized by both the absolute and potassium nitrate methods, the results may not be in agreement by 0.01 to 0.04-percent nitrogen due to expansion of the brass barometer scale. Such difference may be removed by applying the brass scale correction corresponding to the temperature (see 6.4).)

4.4.12.2 Procedure. Crush a portion of approximately 3 gm. of the sample so that it will pass through a U. S. Standard No. 100 sieve, and dry for 2 hours at 150°C. Accurately weigh approximately 1.0 gm. of the dried sample and transfer it to the cup of a standardized 5-part nitrometer (duPont or equivalent). Measure into a small graduate, 25 ml. of nitrogen-free 94.5 ± 0.5-percent sulfuric acid cooled to approximately 5°C. Wash out the weighing bottle into the nitrometer cup with 5 ml. of the sulfuric acid. Lower the mercury reservoir sufficiently to maintain a reduced pressure in the generating bulb. Open wide the upper stopcock and open the lower stopcock sufficiently to draw the acid mixture into the generating bulb. Make successive 4-ml. rinsings with the rest

of the sulfuric acid until it has all been drawn in. Close the upper stopcock, leaving the lower one open, and adjust the mercury reservoir just low enough to maintain a slightly reduced pressure in the generating bulb. Shake the bulb gently until most of the gas has been generated. Lower the mercury reservoir until the level of the mercury in the generating bulb is at the height of the lower shoulder, then close the lower stopcock and shake vigorously for 3 minutes. Replace the bulb on the rack, open the lower stopcock, and adjust the mercury reservoir until the mercury in the generating bulb is at approximately the same height as the mercury in the reservoir. This will bring the pressure inside the generating bulb to approximately one atmosphere, and the solubility of the nitric oxide gas in the sulfuric acid will be normal. Close the lower stopcock and shake the bulb vigorously for an additional 1 minute. Transfer the gas to the measuring tube and adjust the levels of the mercury in the compensating and measuring tubes to approximately the same height. Let the gas stand for 20 minutes in order to permit equalization of the temperature of the gas in the two tubes, and adjust the mercury levels closely with a sulfuric acid U-tube leveling device, the mercury level in the compensating tube being at the standard volume mark. Calculate the percent nitrogen as follows:

$$\text{Percent nitrogen} = \frac{R}{W} \uparrow$$

where:

R = reading of the measuring tube.

W = weight of sample, in gm.

4.4.13 Granulation. Nest the specified sieve or sieves (see table II) on a bottom pan. Place a weighed portion of 100 gm. of the sample on the upper sieve, cover the sieve and shake the assembly 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 amounts retained or passing through the sieves, and calculate to percentages as required.

4.5 Retest and rejection. If the composite sample or any primary sample subjected to test fails to pass the tests, the lot shall be rejected. The contractor shall have the option of having a partial or complete analysis made on samples taken from all or any of the containers in the lot at no expense to the Government. The contractor may then remove the defective portions of the lot and resubmit the lot for acceptance. The resubmitted lot shall be accepted, provided that the new samples, selected in accordance with 4.2, pass all the tests required by this specification and that three additional primary samples pass any test or tests failed by the material on original submission.

5. PREPARATION FOR DELIVERY

5.1 Packing. Unless otherwise specified in the contract or order, potassium nitrate shall be packed in accordance with best commercial practice, in textile-and-paper laminated shipping bags conforming to Specification JAN-P-113, or in drums or barrels. Drums or barrels of wood construction shall be lined with a water-proof, creped, bag-type liner. All containers shall conform to the requirements of Interstate Commerce Commission (ICC) Regulations for transportation of potassium nitrate as stipulated in Code of Federal Regulations 49 CFR 71-78, so as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery.

5.2 Marking. In addition to any special marking required by the contract or purchase order, shipments shall be marked in accordance with ICC Regulations and 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) Class of material required (see 1.2).

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6.2 Classification cross-reference. The classes of potassium nitrate under this specification are equivalent to the classes covered by Specification JAN-P-156A, as follows (see 1.2) :

<i>MIL-P-156B</i>	<i>JAN-P-156A</i>
Class 1	Class A
Class 2	Class B
Class 3	Class C

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

6.4 An explanation of the procedure for making a brass scale correction will be found in Lange's Handbook of Chemistry, page 1368, Fifth Edition (1944) (see note 2 in paragraph 4.4.12.1.2).

Patent notice. When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

Preparing Activity:

Army—Ordnance Corps

Custodians:

Army—Ordnance Corps
Navy—Bureau of Ordnance
Air Force

Other interest:

Army—C

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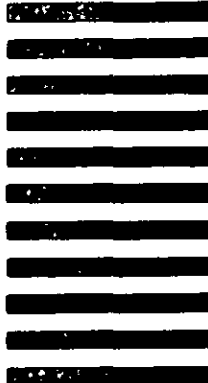
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STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

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1. DOCUMENT NUMBER	2. DOCUMENT TITLE
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