

MIL-P-10830 (ORD)
15 January 1951

MILITARY SPECIFICATION

POTASSIUM PICRATE

1. SCOPE

1.1 This specification covers one type of potassium picrate.

2. APPLICABLE SPECIFICATIONS AND OTHER PUBLICATIONS

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

U. S. ARMY SPECIFICATIONS

- 50-0-1 - General Specification for Ammunition Except Small Arms Ammunition.
- 100-2 - Standard Specification for Marking Shipments by Contractors.

FEDERAL SPECIFICATION

RR-S-366 - Sieves; Standard; Testing.

(Copies of specifications should be obtained from the procuring agency or as directed by that agency. Both the title and identifying number or symbol should be stipulated when requesting copies.) .

2.2 Other publications.— The following publication, of the issue in effect on date of invitation for bids, forms a part of this specification:

INTERSTATE COMMERCE COMMISSION REGULATIONS

Regulations for Transportation of Explosives and Other Dangerous Articles, etc.

(Information as to the availability of Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles etc., may be obtained from the Interstate Commerce Commission, Washington 25, D. C.)

3. REQUIREMENTS

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3.1 Material.— The potassium picrate shall be the anhydrous normal potassium salt of picric acid and shall be a free-flowing crystalline product free from aggregates of crystals and visible foreign materials.

3.2 Color.— Yellow to orange.

3.3 Moisture and other volatile matter.— 0.20 percent, maximum.

3.4 Water insoluble matter.— 0.10 percent, maximum.

3.5 Grit.— None.

3.6 Heavy metals.—None.

3.7 pH of water extract.—6.0 \pm 1.0.

3.8 Purity.—99.0 percent, minimum.

3.9 Granulation.— Potassium picrate shall conform to the following granulation requirements using U. S. Standard sieves conforming to the requirements of Specification RR-S-366.

Through No. 20 sieve (min) _____99.9 percent

Through No. 100 sieve (min) _____45.0 percent

4. SAMPLING, INSPECTION AND TEST PROCEDURES

4.1 Lot.— A lot shall consist of 5,000 pounds, maximum.

4.2 Sampling.— Ten percent but in no case more than ten nor fewer than three of the containers shall be selected by the inspector so as to be representative of the lot. If there are fewer than three containers in the lot, all the containers shall be sampled. The material shall be mixed to a uniform consistency throughout and approximately eight ounces of it shall be taken from each selected container. Each of these primary samples shall be placed in an airtight container and labelled so that the container from which it 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 labelled to show the name of the material, manufacturer, plant, contract or order number, lot 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, the inspector may require that any primary sample be tested for compliance with the requirements of the specification. All primary samples shall be held for possible future examination should the composite sample fail to meet the requirements.

4.3 Inspection.—

4.3.1 Inspection shall be at the place of manufacture unless otherwise specified and shall be as specified in Specification 50-0-1.

4.3.2 The material shall be inspected by the Government inspector to determine compliance with the requirements of this specification for which no tests have been prescribed.

4.4 Determination of moisture and other volatile matter.— Transfer a portion of approximately 5 gm of the sample to a tared low-form weighing dish 2 inches in diameter. Weigh the assembly to obtain the exact weight of the sample. Place the assembly, with cover removed, in an oven maintained at 100° - 105°C for two hours. Replace the cover, cool in a desiccator and weigh. Calculate the loss in weight of the assembly as percent moisture and other volatile matter.

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4.5 Determination of water insoluble matter.- Transfer an accurately weighed portion of approximately 10 gm of the sample to a 400-ml beaker. Add approximately 200 ml of boiling distilled water and place the beaker and contents on a steam bath. Continue heating the beaker and contents for 15 minutes with occasional stirring. Filter the resulting solution, while hot, with the aid of a tared medium porosity sintered-glass-bottom crucible. Wash the beaker and any residue in the crucible with a stream of hot, distilled water until the filtrate shows no trace of potassium picrate (the absence of a yellow color). Dry the crucible for two hours in an oven maintained at 100° - 105°C. Cool in a desiccator and weigh. Calculate the increase in weight of the crucible as percent water insoluble material.

4.6 Grit.-Dissolve 25 gm of the sample in about 600 ml of boiling distilled water, filter, and wash thoroughly. Transfer any material retained on the filter to a smooth glass slide, and press and rub the material with a smooth steel spatula. The presence of grit is indicated by the persistence of a scratching noise.

4.7 Heavy metals.- Transfer 1.0 \pm 0.1 gm of the sample to a clean 600 ml beaker. Add 300 \pm 25 ml of distilled water and heat beaker and contents on a steam bath until all the soluble material is apparently dissolved. Filter if necessary to remove insoluble material. To the solution or filtrate, add 5 ml of 15 N ammonium hydroxide and pass a stream of hydrogen sulfide into the solution at a rate of 2-3 bubbles per second for 10 minutes. Examine the solution for the presence of a precipitate. Report heavy metals to be absent if there is no precipitate present.

4.8 Determination of pH of water extract.- Transfer an accurately weighed portion of approximately 1 gm of the sample to a 150-ml beaker. Add 100 ml of freshly boiled and cooled distilled water having a pH of 6.0 \pm 0.5, and allow the mixture to stand with occasional stirring for 15 minutes. Determine the pH of the mixture at 25° \pm 2°C by means of a pH meter (see 6.1) which reads directly in pH units and is capable of measuring within 0.1 pH unit. The pH meter shall be equipped with a glass electrode and a saturated calomel electrode and shall be calibrated with standard buffer solutions (see 6.2). In case of question or dispute, the standard buffer solutions prescribed by the U. S. Bureau of Standards shall be used to calibrate the pH meter.

4.9 Determination of purity.-

4.9.1 Titanous chloride reduction method.-

4.9.1.1 Preparation of reagents.- Prepare the volumetric solutions that are necessary for this determination with freshly distilled water which is free from atmospheric oxygen. Connect the siphon tubes and burettes in such a way that only carbon dioxide gas, supplied from a Kipp generator, will be drawn into the stock bottles as the solutions are used. Replace the rubber tubing used for connections whenever cracks appear. When no titrations are being made, turn off the stop cock in the tube connecting the Kipp generator with the stock bottle of titanous chloride solution and the burette (Figure 1).

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4.9.1.1.1 Titanous chloride solution.- Prepare a 0.2 normal solution of titanous chloride by mixing 150 ml of 20 percent titanium trichloride solution with 100 ml of 38 percent hydrochloric acid solution and diluting the resulting solution to 1 liter with distilled water. Keep the solution protected from the air as much as possible by means of carbon dioxide. Mix the solution thoroughly with a current of carbon dioxide and store in a bottle covered with black paint or black paper to exclude light.

4.9.1.1.1.1 Standardization.- Transfer an accurately weighed portion of 3.0 to 3.5 gm of pure hexahydrate ferrous ammonium sulfate of known iron content to a 400 ml beaker and dissolve in 50 to 75 ml of water. Add 10 ml of 38 percent hydrochloric acid solution and 0.5 gm of potassium chlorate, then evaporate to dryness on a steam bath. Add 50 ml of water, 5 ml of 38 percent hydrochloric acid solution and evaporate again to dryness. Pass a current of carbon dioxide through a special type titration flask (Figure 2) for five minutes to displace the air. Dissolve the resulting ferric salt in a minimum quantity of water and transfer to the flask. Add 25 ml of 15 percent hydrochloric acid solution and titrate with 0.2 normal titanous chloride solution, keeping a current of carbon dioxide passing through the flask. Near the end point, which can be judged by the gradual disappearance of the reddish-colored ferric salt, add 5 ml of 20 percent ammonium thiocyanate solution. Continue the titration until the blood-red color of ferric thiocyanate just disappears. Apply temperature and burette corrections to the observed reading.

$$\text{Normality of titanous chloride solution} = \frac{W}{0.3921V}$$

where:

W = weight of ferrous ammonium sulfate.

V = corrected volume of titanous chloride solution.

Check the normality of the titanous chloride solution at least once a week by titration with ferric ammonium sulfate solution.

4.9.1.1.2 Ferric ammonium sulfate solution (0.15 N).- Prepare a 0.15 normal ferric alum solution by mixing 75 grams of hydrated ferric ammonium sulfate ($\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$) with 25 ml of 95 percent sulfuric acid solution for each liter of solution and mix thoroughly by means of a current of carbon dioxide. Displace the air in a titration flask with carbon dioxide. Accurately measure 40 to 45 ml of ferric ammonium sulfate solution into the flask, add 25 ml of 15 percent hydrochloric acid solution and 50 ml of water. Titrate with 0.2 normal titanous chloride solution until near the end point, then add 5 ml of 20 percent ammonium thiocyanate solution. Continue the titration until the red color just disappears. Supply temperature and burette corrections to the observed readings and calculate the normality of the ferric ammonium sulfate solution. It can be assumed that the strength of the solution will remain constant for an indefinite period.

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4.9.1.2 Determination.-- Transfer an accurately weighed portion of approximately 0.5 gm of the sample to a 250-ml volumetric flask. Dissolve the sample in the flask with glacial acetic acid, make up to volume with glacial acetic acid and mix thoroughly. Avoid temperature changes on account of the high thermal coefficient of expansion of acetic acid. Displace the air in a carbon dioxide titration flask (Figure 2) by passing in a current of carbon dioxide for five minutes. Rinse a calibrated 25 ml pipette with the acetic acid solution of the sample, then transfer a 25 ml portion of the solution to the flask. Add an accurately measured excess of 0.2 normal titanous chloride solution (2.5 ml for each 5 mg of potassium picrate present) and 25 ml of 15 percent hydrochloric acid solution. Connect the flask to a reflux condenser and boil gently for five minutes on a hot plate. Add a few glass beads to prevent bumping. Increase the current of carbon dioxide, then cautiously immerse the flask in a large beaker of cold water, keeping the index finger over the top of the condenser. Detach the flask from the condenser and insert a one-hole rubber stopper which contains a short piece of glass tubing. Insert the tip of the burette into the glass tubing, add 5 ml of 20 percent ammonium thiocyanate and titrate the cooled solution with 0.15 normal ferric ammonium sulfate solution to the first permanent red color. Run a blank, as follows to correct for reducible impurities in the reagents. Sweep the air from a titration flask, add 25 ml of glacial acetic acid, 25 ml of 15 percent hydrochloric acid solution and about 20 ml of 0.2 normal titanous chloride solution. Connect the flask to the condenser and boil the solution for five minutes on the hot plate. Cool the flask and contents, and titrate with 0.15 normal ferric ammonium sulfate solution adding 5 ml of 20 percent ammonium thiocyanate solution near the end point. Calculate the purity as follows:

$$\text{Percent purity} = \frac{1.484 (V - AF) (N)}{W}$$

where:

- V = volume of titanous chloride solution added
 - N = normality of titanous chloride solution
 - A = volume of ferric ammonium sulfate solution
 - W = weight of sample on moisture free basis
 - F = factor, number of ml of titanous chloride equivalent to 1 ml of ferric ammonium sulfate.
- This is obtained by means of the blank.

4.9.2 Spectrophotometric method (alternative method (see 6.4)).-- Transfer an accurately weighed portion of approximately 0.100 gm of the sample to a clean 1000-ml volumetric flask. Add approximately 500 ml of distilled water to the flask to dissolve the sample and then dilute to the mark with distilled water. Agitate the flask and contents sufficiently to insure uniform mixing. By means of a calibrated pipette,

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transfer a 25-ml aliquot of this solution to a 250-ml volumetric flask and dilute to the mark with distilled water. Transfer a portion of this solution to a corex glass cell having a width of approximately 1 centimeter and determine its optical density at a wavelength of 358 millimicrons. Use a quartz ultra-violet spectrophotometer (see 6.3). Use as a reference, a similar cell containing distilled water. Since the cell which holds the potassium picrate solution is not identical with the reference cell, correct the optical density value obtained as described above for the difference in the amount of light which the two cells scatter and absorb. To do this, fill both cells with distilled water and measure the optical density of the cell, which held the potassium picrate solution, at a wavelength of 358 millimicrons. Subtract the optical density value obtained for the cell when containing distilled water from the value obtained for the cell when containing the potassium picrate solution. Calculate the percentage of potassium picrate in the sample as follows:

$$\text{Percent potassium picrate} = \frac{18.68 A}{DW}$$

where:

- A = corrected optical density of the potassium picrate solution.
 D = width of the corex cell in centimeters.
 W = weight of the sample on a moisture free basis.

4.10 Granulation.- A set of previously tared U. S. Standard stainless steel sieves of the prescribed screen sizes shall be nested in order of decreasing size, placing the largest mesh on top, and fitted with a bottom pan. Place a 50.0 \pm 0.1 gm portion of the sample on the No. 20 sieve, and brush each sieve gently with a camel hair brush until less than 0.1 of a gram of material passes through the sieve during the final 1 minute of brushing. Weigh and calculate the percentage of the material remaining on the No. 20 sieve and that which passes through the No. 100 sieve.

4.11 Resubmission and retests.- 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 provided complete replacement of the defective portions can be made to the satisfaction of the inspector. The resubmitted lot shall be accepted provided that new samples, selected in accordance with 4.2, pass all tests required by this specification.

5. PREPARATION FOR DELIVERY

5.1 Packing.-

5.1.1 For domestic shipment.-

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5.1.1.1 Unless otherwise specified, potassium picrate shall be packed in nailed wood boxes, so constructed as to insure acceptance by common or other carriers for safe transportation, at the lowest rate to the point of delivery (see 6.5). The containers shall conform to the requirements of the Interstate Commerce Commission regulations. All containers shall be able to withstand storage, rehandling, and reshipment without the necessity of repacking. All proposed types of containers shall be submitted to the contracting agency for approval prior to use. The net weight shall be 100 pounds.

5.1.1.2 Box liners.- Boxes shall be completely lined with a box liner made from at least a 35-pound, basis weight, crinkled kraft paper, moisture-proofed with at least a 10 percent solution of paraffin wax. Liners prepared for insertion in the box shall be 32 inches across by 28 inches deep with a seam straight across the bottom and up one side. Closure of the liner shall be effected by bringing together and folding over and creasing several times from the sides first and then finally from the ends.

5.1.1.3 Optional closure of wood boxes.- When specified in the contract or order, the box lid shall be secured by means of two 3/8 - by 0.015 inch steel bands, by two steel wire bands, of 0.0625-inch diameter (16 gage) having a tensile strength of 140,000 pounds per square inch, or two wire bands of 0.0720-inch diameter (15 gage) having a tensile strength of 100,000 pounds per square inch applied girthwise at approximately 3 inches from each end of the box. All bands shall be treated to prevent corrosion. To prevent sliding of the lids when boxes are closed as described above, either of the following methods shall be used: (see 6.6)

One nail, three - or four - penny, shall be driven in each of two diagonal corners of the lid in such a manner that the movement of the lid shall be restricted to 1/4 inch in either direction, or

One wooden cleat shall be secured to the underside of the lid near each end, with at least four clinched nails. Cleats shall be 3/8 x 3/4 inch and of such length and positioned in such a manner that movement of the lid shall be restricted to 1/4 inch in either direction.

5.2 Labeling and marking.- All boxes shall be marked in accordance with the requirements of the Interstate Commerce Commission regulations for the transportation of dangerous explosives, and in addition to any special marking required by the contract or order, shipments shall be in accordance with Specification 100-2.

6. NOTES

6.1 A beckman pH meter, Model H-2, was found to be satisfactory for the determination of pH. (see Fisher Scientific Company Catalogue 90, P577).

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6.2 Satisfactory methods of preparing buffer solutions are indicated below:

6.2.1 Prepare solutions of National Bureau of Standards pH standards number 185, 186I, 186II and 187 according to the description prescribed on their respective certificates.

6.2.2 Prepare buffer solutions having the pH values listed below in the following manner:

Stock solution.— Prepare the following stock solutions for use in the preparation of standard buffer solutions.

Solution A.— Prepare a 0.1 molar solution of potassium dihydrogen phosphate by dissolving 13.61 gm of the material, ACS grade, in distilled water and diluting to a liter.

Solution B.— Prepare a 0.05 molar solution of sodium borate (borax) by dissolving 19.10 gm of the material, ACS grade, in distilled water and diluting to 1 liter.

Solution C.— Prepare a 0.05 molar succinic acid solution by dissolving 5.9 grams of succinic acid, which has been purified by recrystallizing twice from distilled water and dried to constant weight in a desiccator over calcium chloride, in distilled water and diluting the resulting solution to 1 liter.

Buffer solutions.— Prepare buffer solutions of the following pH at 25°C by mixing the above mentioned stock solutions in the proportions indicated.

<u>pH of solution</u>	<u>Stock solution, ml</u>		
	<u>A</u>	<u>B</u>	<u>C</u>
4.0	-	1.78	8.22
5.0	-	3.68	6.32
6.0	8.77	1.23	-
7.0	6.23	3.77	-
8.0	4.65	5.35	-
9.0	1.75	8.25	-

6.3 Quartz ultraviolet spectrophotometer as described in the Journal of American Optical Society, 31, 683 (1941) has been found to be satisfactory.

6.4 The titanous chloride reduction method should be used in case the results obtained for the purity of the potassium picrate are in question, disagreement or dispute or if a spectrophotometer is not available.

6.5 A satisfactory shipping container for potassium picrate is a style I nailed wood box having the following dimensions:

Outside ——— L = 24-1/2 inches; W = 14-1/4 inches; D = 12-1/2 inches

Inside ——— L = 22 inches; W = 13 inches; D = 11-1/4 inches

Volume of contents ——— 2.52 cubic feet

Space occupied ——— 2.71 cubic feet.

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6.6 Requests, requisitions, schedule, and contracts or orders should contain the following information:

- (a) Title, number and date of the specification.
- (b) The type of packing box and the method of closure.

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 invention that may in any way be related thereto.

Custodian:

Army -- Ordnance Corps.

APPARATUS FOR STORING AND USING TITANOUS CHLORIDE SOLUTION.

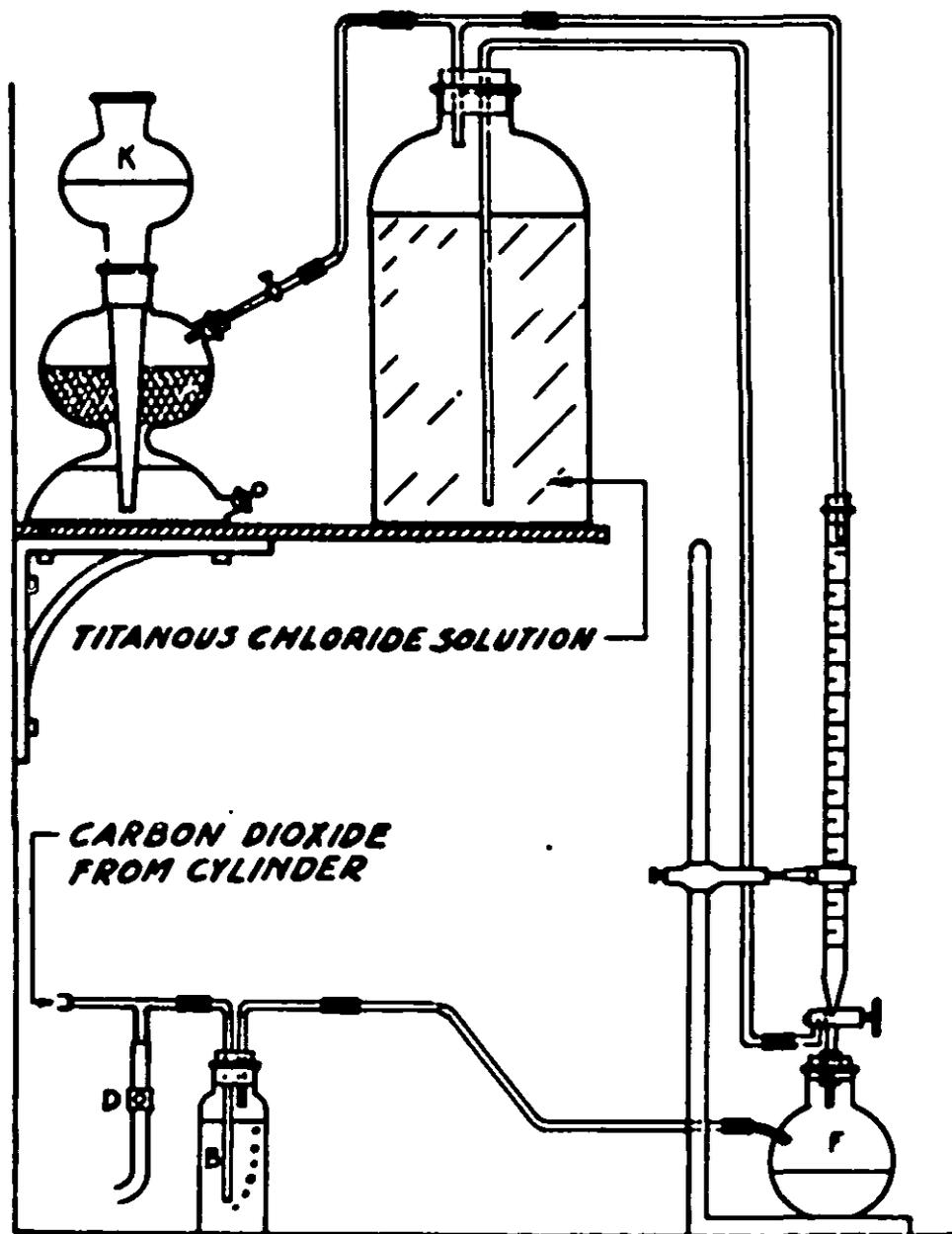
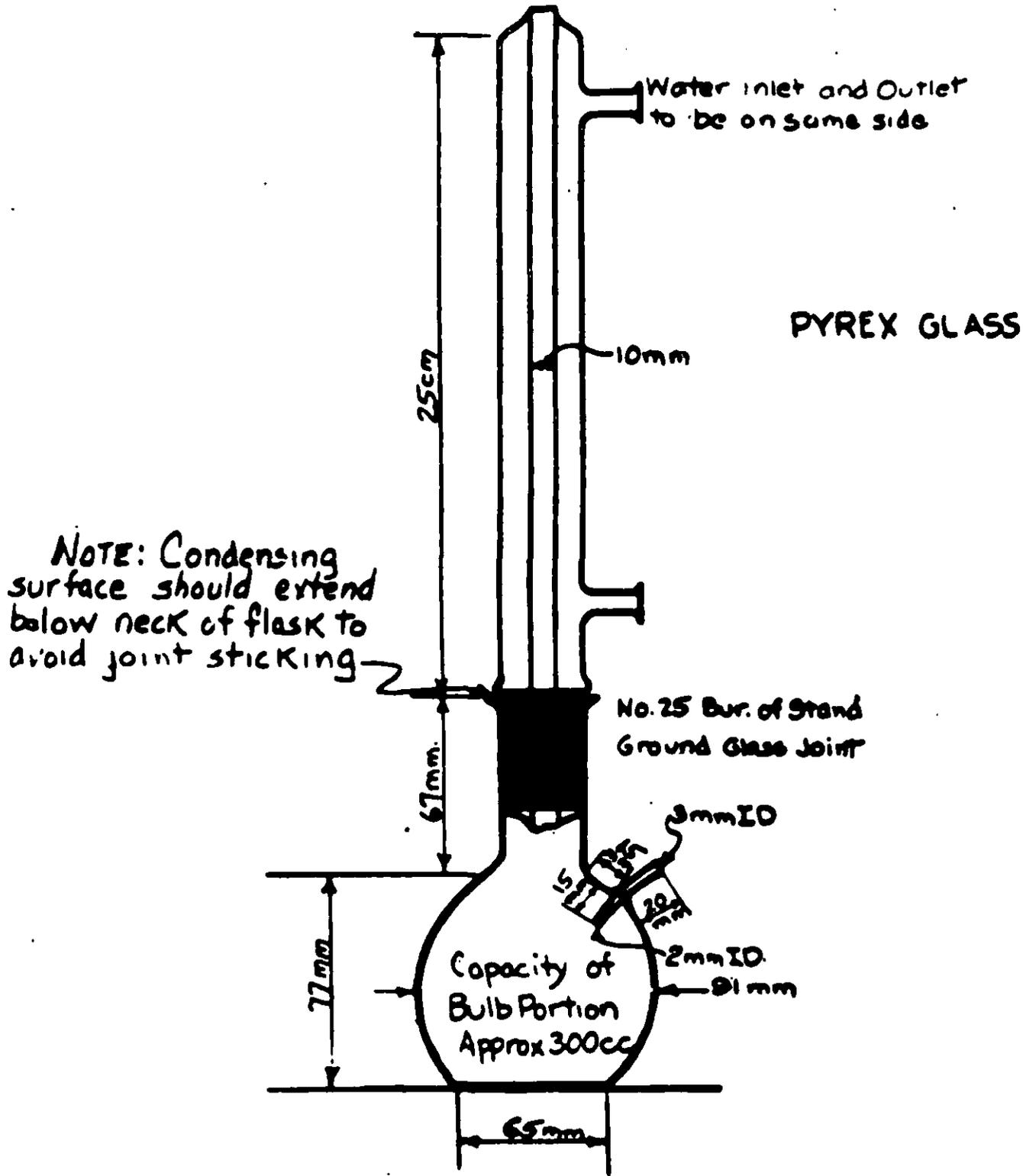


FIGURE 1

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TITRATION FLASK



All Dimensions Are Approximate

FIGURE 2

GPO 83-11197