DEPARTMENT OF COMMERCE
BUREAU OF STANDARDS
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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR TRISODIUM PHOSPHATE, TECHNICAL (PHOSPHATE CLEANER)

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 558

This specification was officially promulgated by the Federal Specifications Board on May 22, 1928, for the use of the departments and independent establishments of the Government in the purchase of technical trisodium phosphate (phosphate cleaner).

[The latest date on which the technical requirements of this specification shall become mandatory for all departments and independent establishments of the Government is August 22, 1928. They may be put into effect, however, at any earlier date after promulgation]

CONTENTS

I. GENERAL SPECIFICATIONS

There are no general specifications applicable to this specification.

II. GRADE

Phosphate cleaner shall be of one grade only, as hereinafter described.

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III. MATERIAL

See General requirements, Section IV.

IV. GENERAL REQUIREMENTS

Phosphate cleaner shall be a white, uniform product in finely granulated form, and shall contain not less than 95 per cent of crystalline trisodium phosphate ($Na_3PO_4.12H_2O$).

V. DETAIL REQUIREMENTS

Phosphate cleaner as received shall conform to the following requirements:

1. It shall be a white, uniform, finely granulated product.
2. Total alkalinity to methyl orange indicator, calculated as Na_2O, shall be not less than 15.5 per cent or more than 20 per cent.
3. Phosphoric anhydride (P_2O_5) shall be not less than 17.7 per cent.
4. Matter insoluble in distilled water shall not exceed 0.1 per cent.
5. No residue shall be retained on a No. 10 sieve (sieve opening = 0.0787 inch) and the residue retained on a No. 100 sieve (sieve opening = 0.0059 inch) shall be not less than 50 per cent.

VI. METHODS OF SAMPLING, TESTING, AND BASIS OF PURCHASE

I. SAMPLING

(*a*) No samples shall be submitted with bids.

(*b*) When Packed in Cans or Cartons.—One can or carton shall be taken at random from not less than 1 per cent of the sellers' shipping containers, provided such containers contain not less than 50 pounds each. In the case of smaller containers a can or carton shall be taken at random from each lot of containers totaling not to exceed 5,000 pounds. The total sample shall in all cases consist of not less than three cans or cartons taken at random from separate containers. With very large lots, where the sample drawn as above will amount to more than 20 pounds, the percentage of packages sampled shall be reduced, so that the amount drawn shall not exceed 20 pounds. Wrap the individual cans or cartons tightly in paraffined paper at once and seal by rubbing the edges with a heated iron. The inspector should accurately weigh each wrapped can or carton, record its weight and the date of weighing on the wrapper, place the wrapped cans or cartons in an air-tight container, which should be nearly filled, seal, mark, and send to the laboratory for test. Samples should be kept cool until tested. The seller shall have the option of being represented at the time of sampling, and when he so requests shall be furnished with a duplicate sample.

(*c*) When in Bulk.—A grab sample of not less than one-half pound shall be taken at random from not less than 1 per cent of the sellers' shipping containers, provided such containers contain not less than 100 pounds each. In case of smaller containers a grab sample of not less than one-half pound shall be taken at random from each lot of containers totaling not to exceed 10,000 pounds. The total sample shall in all cases consist of not less than three grab portions taken at random from separate containers. With very large lots, where the sample drawn as above will amount to more than 20 pounds, the percentage of packages sampled shall be reduced, so that the amount drawn shall not exceed 20 pounds. The inspector should rapidly mix the sample, place in an air-tight container, which shall be filled, seal, mark, accurately weigh, record its weight and date of weighing on the package, and send to the laboratory for test. Samples should be kept cool until tested. The seller shall have the option of being represented at the time of sampling, and when he so requests shall be furnished with a duplicate sample.

2. TESTING

(*a*) Preparation of Sample.—Note the color and the condition of the sample. Rapidly mix the sample; if desired, quarter down to about 1 pound. Weigh out all portions for analysis at once. Unused portions of the sample used for analysis shall be preserved in an air-tight container in a cool place.

When a determination shows nonconformity with specification, a duplicate shall be run.

(*b*) Total Alkalinity.—Transfer 2 g of the sample to a 600 ml beaker, dissolve in about 400 ml of distilled water, add 3 drops of methyl orange solution, and titrate to the end point with 0.5 *N* H_2SO_4. Calculate the percentage of total alkalinity as Na_2O (1 ml 0.5 *N* $H_2SO_4 = 0.0155$ g Na_2O).

(*c*) Phosphoric Anhydride.—Transfer 5 g of the sample to a 1-liter volumetric flask, dissolve in distilled water, dilute to the mark with distilled water, and mix. Pipette a 50-ml aliquot of this solution (corresponding to 0.25 g of the sample) into a 300-ml Erlenmeyer flask, add a small piece of litmus paper and carefully acidify with dilute HNO_3, then add 25 ml of HNO_3 (sp. gr. 1.42) and 25 ml of distilled water. Add to the solution 40 ml of NH_4OH (sp. gr. 0.96) and 50 ml of molybdate solution, stopper, shake for 10 minutes, and let stand at room temperature until the precipitate settles. Filter through an 11-cm filter paper of close texture, wash the flask and precipitate five times with 15 to 20 ml portions of a 1 per cent solution of potassium nitrate; then wash the filter paper containing most of the precipitate, five times with like portions of the same solution. The paper should, of course, be carefully washed each

time from the rim downwards and then allowed to drain completely before washing with the next portion of solution. The wash solution, as well as all others subsequently used, must be free from CO_2. Return the paper and precipitate to the flask, add enough 0.3 *N* NaOH to decompose the precipitate and then approximately 2 ml in excess. Dilute with 25 ml of distilled water, stopper, and shake until the precipitate is dissolved. Wash off the stopper with distilled water, dilute the solution to about 150 ml with distilled water, add 6 drops of a 1 per cent solution of phenolphthalein, and discharge the pink color with 0.3 *N* HNO_3. Finish the titration by adding 0.3 *N* NaOH until the reappearance of the pink color. Subtract the volume of 0.3 *N* acid used from the total volume of 0.3 *N* NaOH used and multiply the remainder by the titer of the 0.3 *N* NaOH solution. Calculate the percentage of P_2O_5. (1 ml 0.3 *N* NaOH = 0.00093 g P_2O_5. $P_2O_5 \times 2.309 = Na_3PO_4$. $P_2O_5 \times 5.352 = Na_3PO_4 \cdot 12H_2O$).

(*d*) Matter Insoluble in Distilled Water.—Transfer 10 g of the sample to a 600 ml beaker, add about 400 ml of distilled water, and boil the solution for 10 minutes. Filter on a weighed Gooch crucible with an asbestos mat, wash thoroughly with hot distilled water, dry the crucible and residue at 105 to 110° C. for three hours, cool and weigh. Calculate the percentage of matter insoluble in water.

(*e*) Sieve Test.—Transfer 100 g of the well-mixed sample, without previous drying, to a dry No. 10 sieve and sift, tapping the sieve frame from time to time and catching all of the material passing through the No. 10 sieve on a dry No. 100 sieve. The sifting on the No. 10 sieve is continued until the weight of the residue retained on the No. 10 sieve is not reduced by more than 0.1 g on further sifting for one minute, tapping the sieve frame as before. Calculate the final weight of residue to percentage retained on the No. 10 sieve. Sift the material on the No. 100 sieve, tapping the sieve frame from time to time, until the weight of the residue retained on the No. 100 sieve is not reduced by more than 0.1 g on further sifting for one minute, tapping the frame as before. Add the final weight of the residue retained on the No. 10 sieve to the final weight of the residue retained on the No. 100 sieve and calculate the sum to percentage of residue retained on the No. 100 sieve.

3. REAGENTS

(*a*) Standard Sulphuric Acid Solution.—0.5 *N* or about 25.8 g strong sulphuric acid (sp. gr. 1.84) diluted to 1 liter. Standardize against standard sodium hydroxide solution that has been standardized against Bureau of Standards standard acid potassium phthalate.

(*b*) Ammonium Hydroxide Solution (Specific Gravity 0.96, or Approximately 6 *N*).—Mix 400 ml of NH_4OH (sp. gr. 0.90) with 600 ml of distilled water. Check with a hydrometer or by titration.

(*c*) Molybdate Solution.—Mix 100 g of pure molybdic anhydride or 118 g of 85 per cent molybdic acid with 400 ml of distilled water and add 80 ml of NH_4OH (sp. gr. 0.90). When solution is complete, filter and pour the solution slowly and with constant stirring into a mixture of 400 ml of HNO_3 (sp. gr. 1.42) and 600 ml of distilled water. Let settle for 24 hours and use the clear supernatant liquid.

(*d*) Potassium Nitrate Solution (1 Per Cent).—Dissolve 10 g of KNO_3 in recently boiled distilled water, dilute to 1,000 ml with recently boiled distilled water and mix.

(*e*) Standard Sodium Hydroxide Solution (Approximately 0.3 *N*).—Prepare a saturated solution of NaOH in a stoppered flask, let settle overnight, and pipette a clear 20 ml portion into a 1 liter volumetric flask. Dilute to 1,000 ml with recently boiled distilled water and mix, cool to room temperature, and standardize against Bureau of Standards standard acid potassium phthalate, using phenolphthalein indicator. Dilute, if desirable, restandardize, and protect the solution from CO_2.

(*f*) Standard Nitric Acid Solution (Approximately 0.3 *N*).—Mix 20 ml of HNO_3 (sp. gr. 1.42) and 1,000 ml of recently boiled distilled water, cool to room temperature, and standardize against the standard NaOH solution (*e*), using phenolphthalein indicator. Adjust the solution until it is exactly equivalent.

(*g*) Phenolphthalein Indicator.—Dissolve 1 g of pure phenolphthalein in 100 ml of 85 to 95 per cent ethyl alcohol.

(*h*) Methyl Orange Indicator.—Dissolve 1 g of methyl orange in 1 liter of distilled water.

4. BASIS OF PURCHASE

Phosphate cleaner shall be purchased by net weight.

VII. PACKING AND MARKING OF SHIPMENTS

Shall be in accordance with the best commercial practice unless otherwise specified.

VIII. NOTES

1. Phosphate cleaner is suitable for a variety of cleaning purposes where abrasive action is not desired, such as typewriter parts, typewriter type, floors, most metals, and painted surfaces. It should be used in most cases in dilute solution (about 1 to 2 tablespoonfuls to 2 to 3 gallons of warm water).

2. Hot solutions of phosphate cleaner may attack and discolor aluminum. Cold solutions if too strong may act similarly. The material should not be used for cleaning aluminum motor parts.

3. After using any alkaline cleaner, the surface being cleaned should be washed or rinsed off thoroughly with water.

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