IS 12557 (1988): Sodium pyrophosphate for electroplating [CHD 5: Electroplating Chemicals and Photographic Materials]

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Indian Standard

SPECIFICATION FOR SODIUM PYROPHOSPHATE FOR ELECTROPLATING

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Indian Standard

SPECIFICATION FOR SODIUM PYROPHOSPHATE FOR ELECTROPLATING

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 12 October 1988 after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

0.2 Sodium pyrophosphate is used in solutions for electroplating of metals and alloys, for electroless nickel plating as well as phosphatizing. Certain alkaline soak or electrolytic cleaners also contain sodium pyrophosphate. The salt is industrially produced by thermal dehydration and decomposition of disodium orthophosphate. The salt is also known as tetrasodium pyrophosphate.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements, methods of sampling and tests for sodium pyrophosphate for electroplating.

2. REQUIREMENTS

2.1 Sodium pyrophosphate shall be in the form of colourless crystals or white powder, effloresing in air, soluble in about 10 parts of cold water or 2 parts of boiling water, insoluble in alcohol and less toxic and shall correspond essentially to the formula $Na_4 P_2O_7{\cdot}10H_2 O$.

2.2 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Appendix A. References to relevant clauses of Appendix A are given in col 4 of Table 1.

TABLE 1 REQUIREMENTS FOR SODIUM PYROPHOSPHATE FOR ELECTROPLATING

Sl No.	Characteristic	Requirements	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Sodium pyrophosphate. (as $Na_4P_2O_7{\cdot}10H_2O$) percent by mass, *Min*	97·0	A-2
ii)	Matter insoluble in water percent by mass, *Max*	0·01	A-3
iii)	Chloride (as Cl), percent by mass, *Max*	0·005	A-4
iv)	Sulphate (as SO_4), percent by mass, *Max*	0·05	A-5
v)	Iron (as Fe), percent by mass, *Max*	0·003	A-6
vi)	Heavy metals (as Pb), *Max*	0·002	A-7
vii)	Arsenic (as AS_2O_3) percent by mass, *Max*	0·0 005	A-8
viii)	Orthophosphate (as PO_4) percent by mass, *Max*	0·3	A-9
ix)	Sodium carbonate (as Na_2CO_3), percent by mass, *Max*	0·3	A-10

3. PACKING AND MARKING

3.1 Packing

3.1.1 The product shall be packed in multiwall paper bags/drums or polyethylene bags not exceeding 50 kg in mass when packed.

3.2 Marking

3.2.1 The containers shall be marked with the following particulars:

a) Name of the product;
b) Name of the manufacturer and/or his recognized trade mark, if any;
c) Net mass of the product;
d) Date of manufacture; and
e) Batch number.

3.2.2 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of preparing representative samples of the material and criteria for its conformity with the specification shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2 and *Table* 1)

METHODS OF TEST FOR SODIUM PYROPHOSPHATE FOR ELECTROPLATING

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977[*]) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of tests.

A-2. DETERMINATION OF SODIUM PYROPHOSPHATE

A-2.1 Reagents

A-2.1.1 *Hydrochloric Acid* — 1 N.

A-2.1.2 *Bromophenol Blue Indicator* — prepared by mixing 0·1 g of the indicator compound with 1·5 ml of 0·1 N sodium hydroxide and diluting to 100 ml with water.

A-2.2 Procedure — Dissolve 50 g of the material accurately weighed in water in a 500 ml volumetric flask and make up to the mark. Pipette a 50 ml portion of the solution into a 250 ml conical flask, add about 20 ml of water, and a few drops of bromophenol blue indicator solution. Titrate the test solution to a *p*H of 3·8 against 1 N hydrochloric acid in a burette. A change in colour of the test solution from blue violet to yellow denotes the end point.

A-2.3 Calculation

1 ml of 1 N Hydrochloric acid = 0·223 0 g of $Na_4P_2O_7 \cdot 10\,H_2O$

Sodium Pyrophosphate content (as $Na_4P_2O_7 \cdot 10\,H_2O$), percent by mass

$$= \frac{22{\cdot}3027 \times V}{M}$$

where

M = mass in g of the material taken for test.

V = volume of the acid consumed.

A-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure — Dissolve 10 g of the material accurately weighed in about 100 ml water in a 250 ml glass beaker and heat on a steam bath for 1 hour with occasional stirring. Filter through a weighed sintered glass crucible (Porosity No. 4) and wash several times with hot water. Dry at 105°C. Cool it in a desiccator and weigh.

A-3.2 Calculation — Matter insoluble in water, percent by mass = $M \times 10$

where

M = the mass in g of the water insoluble residue.

A-4. DETERMINATION OF CHLORIDE

A-4.1 Reagents

A-4.1.1 *Concentrated Nitric Acid* — (*see* IS : 264-1976[*]).

A-4.1.2 *Silver Nitrate Solution* — 0·2 M or 3·4 percent (*m/v*).

A-4.1.3 *Standard Chloride Solution* — (0·002 mg/ml) prepared by dissolving 0·33 g of dried sodium chloride or 0·421 g of potassium chloride in water in a one-litre volumetric flask and make up the volume to the mark. Pipette out 10 ml of the solution, dilute with water and make up the volume to 1 000 ml. One millilitre of this solution contains 0·002 mg of Chloride (as cl).

A-4.2 Procedure — Pipette out 10 ml of sodium pyrophosphate solution prepared for test (*see* **A-2.2**) in a 50 ml Nessler cylinder and add 3 ml of concentrated nitric acid. To this add 1 ml of silver nitrate solution. Dilute to 50 ml with water, and mix well. Carry out a control test in another Nessler cylinder using 25 ml of standard chloride solution and the same quantities of the reagents.

The material shall be taken as not having exceeded the limit prescribed in Table 1, if the turbidity produced with the material is not greater than that produced in the control test.

A-5. DETERMINATION OF SULPHATE

A-5.1 Reagents

A-5.1.1 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976[†].

A-5.1.2 *Barium Chloride Solution* — Approximately 0·2 M or 5 percent (*m/v*).

A-5.2 Procedure — Pipette the solution prepared for Test (*see* **A-2.2**) two times with a 50 ml pipette into a 400 ml glass beaker, add 5 ml of concentrated hydrochloric acid and dilute to about 200 ml. Heat the solution to boiling and introduce dropwise with a pipette, 10 ml of barium chloride solution, stirring the solution constantly during addition. Cover the solution with a watch glass and keep it hot without boiling for 1 hour. Filter the supernatent liquid using gentle suction through a dried and weighed sintered glass crucible (Porosity No. 4).

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[*] Specification for water for general laboratory use (*second revision*).

[*] Specification for nitric acid (*second revision*).

[†] Specification for hydrochloric acid (*second revision*).

Transfer the precipitate and wash with warm water until the washings give a negative reaction for chloride. Dry the crucible and precipitate in an oven at 105 ± 2°C until consecutive weighings give constant values. The mass of the precipitate shall not be more than 0·012 15 g when determined with a microbalance.

A-6. DETERMINATION OF IRON

A-6.1 General — 4, 7 Diphenyl-1, 10-phenanathroline, also called bathophenanthroline has a highly sensitive colour reaction with ferrous ions in solution. The main advantage of this compound is that it does not react with cations or anions commonly encountered.

A-6.2 Reagents

A-6.2.1 *Concentrated Hydrochloric Acid* — (*See* IS : 265-1976*).

A-6.2.2 *Hydroxylamine Hydrochloride Solution* — 10 percent (*m/v*).

A-6.2.3 *Sodium Acetate Solution* — 10 percent (*m/v*).

A-6.2.4 *Bathophenanthroline* — 0·082 5 percent (*m/v*) solution in ethanol (absolute alcohol) or iso-amyl alcohol.

A-6.2.5 *Standard Iron Solution* — Dissolve 7·021 5 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4.\ 6H_2O$] or 4·978 1 g of ferrous sulphate [$FeSO_4.\ 7H_2O$] with 5 ml of 20 percent (*v/v*) sulphuric acid solution and dilute with water to exactly 1 000 ml. Pipette out 10 ml of the solution and dilute to 1 000 ml with water. One millilitre of this solution contains 0·01 mg of iron (Fe).

A-6.3 Procedure — Take a 25 ml portion of the solution prepared for test (*see* **A-2.2**) in a 250 ml glass beaker and adjust its *p*H to 4–5 with concentrated hydrochloric acid added carefully dropwise. Add 1 ml of hydroxylamine hydrochloride and 2 ml of sodium acetate. Introduce 5 ml of bathophenanthroline. Make up to 50 ml in a volumetric flask with ethanol and mix. Read at 530 nm against a reagent blank. Determine iron content of the solution by reference to a calibration curve prepared with known quantities of the standard iron solution under similar conditions.

A-6.4 Calculation

$$\text{Iron (as Fe) percent by mass} = \frac{M \times 100}{2{\cdot}5}$$

where

M = mass in g or iron in the aliquot solution taken for test.

A-7. DETERMINATION OF HEAVY METALS PRECIPITATED BY HYDROGEN SULPHIDE

A-7.1 Reagents

A-7.1.1 *Dilute Hydrochloric Acid* — 20 percent (*v/v*).

A-7.1.2 *Dilute Ammonia* — 25 percent (*v/v*).

A-7.1.3 *Standard Lead Solution (1 ml = 0·01 mg Pb)* — Prepared by dissolving 0·18307 g of lead acetate trihydrate in water in a 100 ml volumetric flask, making up to the mark and subsequently diluting it exactly 100 times in a 1-litre volumetric flask.

A-7.1.4 *Hydrogen Sulphide Gas* — Produced by reaction of ferrous sulphide with dilute sulphuric acid [10 percent (*v/v*)] in a Kipp's apparatus and then passed through water.

A-7.2 Procedure — Pipette out 25 ml of the solution prepared for test (*see* **A-2.2**), into a 250 ml glass beaker, add 5 ml of dilute hydrochloric acid, boil gently for 5 minutes and cool. Add 10 ml of dilute ammonia solution to the beaker. Pass hydrogen sulphide for a few seconds. No turbidity shall be produced and any colour obtained not deeper than that observed when to a mixture of 5 ml of the standard lead acetate solution and 3 ml of dilute acetic acid standard iron solution containing the same quantity of iron as found in test **A-6** is added along with 10 ml of dilute ammonia and water to bring the volume to the same level, and hydrogen sulphide passed through for a few seconds.

NOTE — Transfer all the solutions quantitatively to Nessler cylinder for colour comparison.

A-8. DETERMINATION OF ARSENIC

A-8.1 General — An accurate method of arsenic determination is the silver diethyldithiocarbamate method of colorimetric estimation. The red addition compound formed is measured photometrically at any wavelength between 525 and 540 nm.

A-8.2 Reagents

A-8.2.1 *Standard Arsenic Solution* — Bring 0·132 0 g of arsenious oxide into solution with 20 ml of a 10 percent (*m/v*) sodium hydroxide solution in a 100 ml glass beaker. Transfer to a 1 000 ml volumetric flask, dilute to the mark with water and mix well by shaking the solution (1 ml = 0·1 mg As). Introduce 10 ml of this solution into a 1 litre volumetric flask, add 10 ml of concentrated hydrochloric acid, make up to the mark with water and thoroughly mix. One millilitre of this solution contains 0·001 mg Arsenic (as As).

A-8.2.2 *Ferric Ammonium Sulphate Solution* — Prepare a solution of 34 g of ferric ammonium

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*Specification for hydrochloric acid (*second revision*).

sulphate [$Fe(NH_4)(SO_4)_2 . 12H_2O$] in water. Make additions of 2 ml of concentrated sulphuric acid and 1 g of sodium chloride and dilute to 1 litre with water. Mix well by agitation with a glass rod.

A-8.2.3 *Potassium Permanganate Solution* — Potassium permanganate solution 2 percent (*m/v*).

A-8.2.4 *Stannous Chloride Solution* — Dissolve 80 g of stannous chloride ($SnCl_2 . 2H_2O$) in a mixture of 25 ml of water and 75 ml of concentrated hydrochloric acid.

A-8.2.5 *Lead Acetate Solution* — Dissolve 1 g of lead acetate in 100 ml of water. Clear the solution by dropwise addition of concentrated acetic acid.

A-8.2.6 *Lead Acetate Paper* — Saturate 15 cm qualitative grade filter paper with lead acetate solution and place on a watch glass in an oven at 100-105°C to dry. Cut the papers into quarters and store in an airtight container.

A-8.2.7 *Zinc Shot* — Zinc shot of uniform size is to be used. Though shot of different sizes (3-20 mesh) has been successfully used, more uniform gas flow is obtained with 3-6 mesh over a wider range of acid concentration. Treat the shot with 1 : 1 hydrochloric acid so that it assumes a dull grey appearance. Decant the acid and wash zinc particles with acid-free water. Store the clean shot under distilled water.

A-8.2.8 *Silver Diethyldithiocarbamate Solution* — Dissolve 8·26 g of silver diethyldithiocarbamate in 100 ml of pure pyridine.

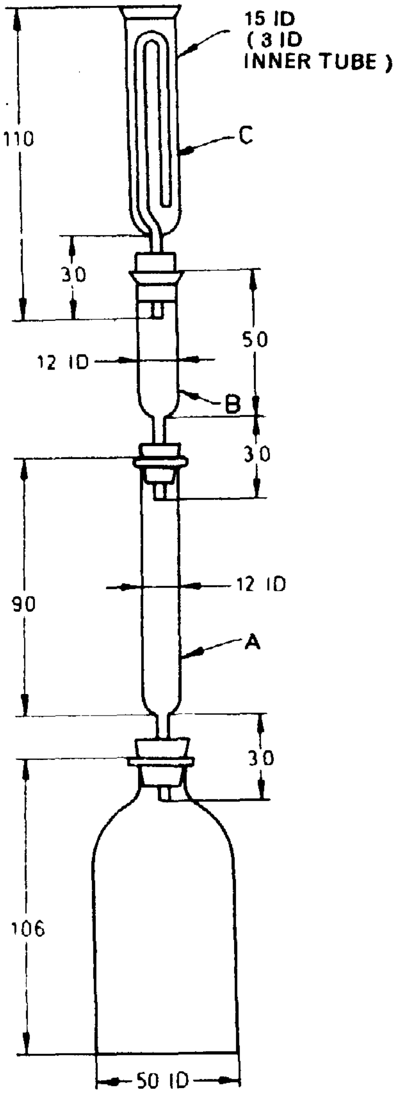
A-8.2.9 *Sodium Chloride* — *See* IS : 797-1982*.

A-8.3 Procedure — Take a 5 ml portion of the solution prepared for test (*See* **A-2.2**), alongwith 3-4 ml of concentrated sulphuric acid or 7-8 ml of perchloric acid in the apparatus bottle (*See* Fig. 1), and dilute to 30-40 ml with water.

A-8.3.1 Introduce 10 ml of ferric ammonium sulphate solution and then permanganate solution dropwise to a permanent pink colour. Add 1·5 g of sodium chloride and stir. Heat nearly to boiling, remove from the heat and add stannous chloride solution in order to reduce ferric ions to the ferrous state as shown by disappearance of yellow colour, and then 2 drops in excess. Cool and dilute to 60 ml.

A-8.3.2 Set up the apparatus as shown in Fig. 1. Fold a piece of lead acetate paper into a fluted form and insert it into the lower scrubber *A* such that its lower end is nearly 1 cm above the constriction in the tube. Loosely pack the second scrubber *B* with glass wool and moisten the wool with a few drops of lead acetate solution. Remove the excess solution by blowing for a few seconds a stream of compressed air under moderate pressure through the scrubber *B*. Assemble parts *A*, *B* and *C*. Fill a dry 50 ml graduated cylinder up to 5 ml with zinc shot, pour it into the apparatus bottle and fix the scrubbers and adsorber in position. Pipette without loss of time a 5 ml portion of silver diethyldithiocarbonate into the scrubber *C*. Keep the bottle immersed to the 60 ml mark in a water bath maintained at 25 ± 3°C for 20 minutes, allowing the reaction to proceed. Remove the adsorber and either transfer to a 3 ml microcell or dilute with a measured volume of pyridine and measure photometrically at 530 nm.

A-8.3.3 Plot a calibration curve in the usual manner and determine the amount of arsenic present.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR SILVER DIETHYLDITHIOCARBAMATE METHOD

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*Specification for common salt for chemical industry (*first revision*).

A-8.4 Calculation

$$\text{Arsenic (as As), percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g of arsenic in the test solution, and

M = mass in g of the material in the aliquot solution taken for test.

A-9. DETERMINATION OF ORTHOPHOSPHATE

A-9.1 General — An orthophosphate suitably treated with ammonium molybdate gives rise to $(NH_4)_3PO_4 \cdot 12MoO_3$ and the latter after reduction can be volumetrically estimated by titration against a standard potassium permanganate solution.

A-9.2 Reagents

A-9.2.1 *Molybdate Reagent* — Dissolve 10 g of molybdic anhydride or 11·8 g of molybdic acid in a mixture of 40 ml of water and 8 ml of concentrated ammonia solution; filter if necessary. Slowly add the molybdate reagent with stirring into a solution containing 40 ml of concentrated nitric acid and 60 ml of water; the end of the tube with the molybdate solution should dip under the surface of the dilute nitric acid. Store the mixture in a warm place for many days or until a portion heated to 40-45°C deposits a yellow precipitate. Decant the solution to remove any sediment.

A-9.2.2 *Standard Sodium Phosphate Solution* — Dry Na_2HPO_4 for 1 hour at 120°C and cool in a desiccator. Prepare a standard solution of disodium hydrogen phosphate Na_2HPO_4 (1 ml = 0·50 mg PO_4) by weighing out 0·747 4 g of the anhydrous salt, dissolving it in water and making up to the mark in a 1 000 ml volumetric flask. Shake well.

A-9.2.3 *Ammonium Nitrate Solid*

A-9.3 Procedure — Pipette 20 ml of the solution prepared for test (*see* **A-2.2**) into a 50 ml conical flask, or test tube, add 1 g of ammonium nitrate and 5 ml of molybdate reagent. Warm the mixture to 40-45°C and shake the whole solution continuously for 10 minutes and allow to stand for 10 minutes. The intensity of yellow colour of the solution shall not be greater than in the case of a solution containing 6·0 mg of orthophosphate as the standard sodium salt solution (*see* **A-9.2.2**), and treated similarly, keeping the total volume constant with water as when treating the test solution.

A-10. DETERMINATION OF CARBONATE

A-10.1 General — Carbonate in a compound can be determined by subjecting to steam distillation a mixture of the material and dilute sulphuric acid, absorbing the evolved carbon dioxide in sodium hydroxide and titrating the excess hydroxide against a standard acid solution.

A-10.2 Reagents

A-10.2.1 *Standard Sodium Hydroxide Solution* — 0·05 N.

A-10.2.2 *Standard Hydrochloric Acid Solution* — 0·05 N.

A-10.2.3 *Phenolphthalein Indicator* — 1 percent (*m/v*) solution in 1 : 1 ethanol water mixture prepared by firstly bringing the solid into solution in ethanol, mixing with an equal volume of water and if necessary, filtering.

A-10.2.4 *Methyl Orange Indicator* — 0·05 percent (*m/v*) solution prepared by dissolving 50 mg of the free acid or solution salt in water, adding 15·2 ml of 0·01 N sodium hydroxide subsequently if the salt is used, diluting to 100 ml and filtering to remove any precipitate formed.

A-10.2.5 *Dilute Sulphuric Acid* — 10 percent (*v/v*).

A-10.3 Procedure — Pipette out 50 ml of solution prepared for test (*see* **A-2.2**) into a 500 ml flat bottomed distilling flask carrying a thistle funnel and suitable attachments for steam distillation. Add about 40 ml of dilute sulphuric acid and steam distil. Cool the mixture of carbon dioxide and steam by cold water circulation in the condenser and receive the condensate in 20 ml of sodium hydroxide solution. Steam distil for 30 minutes. Remove the vessel containing sodium hydroxide and carry out titration with standard hydrochloric acid solution first to phenolphthalein end point with addition of phenolphthalein and then to methyl orange end point with methyl orange addition.

A-10.4 Calculation

$$\text{Sodium Carbonate (as } Na_2CO_3 \text{), percent by mass} = \frac{0{\cdot}53 \times V}{5}$$

where

V = volume of acid consumed beyond the phenolphthalein end point.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF SODIUM PYROPHOSPHATE FOR ELECTROPLATING

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the materials being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.2 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.3 The samples shall be placed in suitable, clean, dry and airtight glass or other suitable containers on which the material has no action.

B-1.4 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number (*n*) of containers to be chosen from a lot shall depend on the size of the lot (*N*) and shall be in accordance with Tabte 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

LOT SIZE (*N*)	NUMBER OF CONTAINERS TO BE SELECTED (*n*)
(1)	(2)
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

B-2.3 All the containers shall be selected at random and in order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all characteristics given under **2** and shall not exceed 500 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 200 g. This composite sample shall be divided into three equal parts, one for the purchaser, second for the manufacturer and the third to be used as referee sample.

B-3.1.3 The remaining portions of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 50 g. These parts shall be immediately transferred to thoroughly dried bottle which are then sealed air tight with stoppers and labelled with all the particulars of sampling given under **B-1.4**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the manufacturer and the third shall be used as referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (*see* **B-3.1.2**) and a set of individual samples (*see* **B-3.1.3**) marked for this purpose. It shall also bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the manufacturer and shall be used in case of dispute between the two.

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*Methods for random sampling.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of sodium pyrophosphate shall be conducted on each of the individual samples for all the grades.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For individual samples

B-5.1.1 *For Sodium Pyrophosphate* — The test results for sodium pyrophosphate shall be recorded, and the mean and range for these test results shall be calculated as follows:

Mean ($\bar{X}$) = Sum of the test results divided by the number of test results,

Range ($\bar{R}$) = The difference between the maximum and minimum values of the test results.

The value of expression ($\bar{X} - 0{\cdot}6\ R$) shall be calculated. If the value of this expression is greater than or equal to limits specified in Table 1, the lot shall be declared to have satisfied the requirements for these characteristics.

B-5.1.1.1 In case of sample of size 10, the first five test results may be taken in one group and the next five in another group. R shall be calculated for each of the groups and the average value of the two shall be calculated as R. If $\bar{X} - 0{\cdot}6\ R$ is greater than or equal to the limits specified in Table 1, then the lot shall be declared to have satisfied the requirements for these characteristics.

B-5.2 For Composite Sample — The test results on the composite samlpe shall meet the corresponding requirements specified in Table 1.

B-5.3 A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics listed in Table 1.