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Mazdoor Kisan Shakti Sangathan
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“पुराने को छोड़ नये के तरफ”
Jawaharlal Nehru
“Step Out From the Old to the New”

IS 5670 (1984): Lead thiocyanate for explosive and pyrotechnic compositions [CHD 26: Explosives and Pyrotechnics]
Indian Standard

SPECIFICATION FOR
LEAD THIOCYANATE FOR EXPLOSIVE AND
PYROTECHNIC COMPOSITIONS

(First Revision)

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May 1984
Indian Standard

SPECIFICATION FOR

LEAD THIOCYANATE FOR EXPLOSIVE AND
PYROTECHNIC COMPOSITIONS

( First Revision )

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(Continued on page 2)
(Continued from page 1)

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SPECIFICATION FOR

LEAD THIOCYANATE FOR EXPLOSIVE AND
PYROTECHNIC COMPOSITIONS

( First Revision )

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 9 March 1984, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally issued in 1970. The Sectional Committee responsible for its preparation decided to revise it in the light of experience gained since its publication. In the present revision changes have been made in the method of determination of grit. An alternate method for the determination of chloride has been included.

0.3 The principal use for lead thiocyanate is as an ingredient of primer compositions for rifle and gun ammunition, and in squib composition for electric detonators. It is suitable for use in furnishing fuel for the burning of the primer as it allows the primer compositions to burn more slowly.

0.4 This standard contains clauses 2.2.1 and 3.1 which call for agreement between the purchaser and the supplier.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or 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.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and tests for lead thiocyanate for explosive and pyrotechnic compositions.

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

2.1 Description — The material shall consist of a uniform free-flowing crystalline powder white or light yellow in colour and free from foreign matter and visible impurities. It shall not blacken on exposure to daylight.

2.2 Particle Size — The material shall be of following particle size:

- **Type A** — All passing through 150-micron IS Sieve.
- **Type B** — All passing through 53-micron IS Sieve.
- **Type C** — All passing through 150-micron IS Sieve; not less than 50 percent to be retained on 90-micron IS Sieve and not less than 90 percent to be retained on 63-micron IS Sieve.

2.2.1 In addition to the above, the material may be of other particle size as agreed to between the purchaser and the supplier.

2.3 The material shall comply with the requirements specified in Table I when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

**TABLE 1 REQUIREMENTS FOR LEAD THIOCYANATE FOR EXPLOSIVE AND PYROTECHNICAL COMPOSITIONS**

<table>
<thead>
<tr>
<th>Sl No.</th>
<th>Characteristic</th>
<th>Requirement</th>
<th>Method of Test (Ref to Cl No. in Appendix A)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td>(2)</td>
<td>(3)</td>
<td>(4)</td>
</tr>
<tr>
<td>i)</td>
<td>Moisture, percent by mass, Max</td>
<td>0.05</td>
<td>A-3</td>
</tr>
<tr>
<td>ii)</td>
<td>Matter insoluble in ammonium acetate solution, percent by mass, Max</td>
<td>0.20</td>
<td>A-4</td>
</tr>
<tr>
<td>iii)</td>
<td>Grit, percent by mass, Max</td>
<td>0.05</td>
<td>A-5</td>
</tr>
<tr>
<td>iv)</td>
<td>Chlorides (as PbCl₂), percent by mass, Max</td>
<td>0.20</td>
<td>A-6</td>
</tr>
<tr>
<td>v)</td>
<td>Lead thiocyanate, percent by mass, Min</td>
<td>98.0</td>
<td>A-7</td>
</tr>
<tr>
<td>vi)</td>
<td>Lead (as lead thiocyanate) after correcting for chloride, percent by mass, Min</td>
<td>98.0</td>
<td>A-8</td>
</tr>
<tr>
<td>vii)</td>
<td>pH (of aqueous solution)</td>
<td>4.5 to 5.5</td>
<td>A-9</td>
</tr>
<tr>
<td>viii)</td>
<td>Bulk density, g/ml, Min</td>
<td>0.8 to 1.1</td>
<td>A-10</td>
</tr>
</tbody>
</table>

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in air-tight amber coloured glass bottles or polyethylene bags packed in paper or cartons, or as agreed to between the purchaser and the supplier. If packed in polyethylene bags, the polyethylene sheet shall be not less than 0.05 mm thick.
3.2 Marking — Each package shall be legibly and indelibly marked with the following information:
   a) Name of the material;
   b) Mass of the material in the package;
   c) Manufacturer's name and/or recognized trade-mark, if any;
   d) Years of manufacture; and
   e) Lot number to enable the batch of manufacture to be traced from records.

3.3 BIS Certification Marking
The product may also be marked with Standard Mark.

3.3.1 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 details of conditions under which the licence for the use of 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 drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

A P P E N D I X A
(Clause 2.3, and Table 1)

METHODS OF TEST FOR LEAD THIOCYANATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

A-1. QUALITY OF REAGENTS
A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1977*) shall be employed in tests.

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

A-2. PREPARATION OF SAMPLE
A-2.1 Mix the sample well by rotating the bottle several times and

*Specification for water for general laboratory use (second revision).
transfer a portion immediately into an amber coloured wide-mouthed bottle and stopper it. Take care that no pieces of cork or sealing wax get mixed with the sample. Do not expose the sample to light or to an atmosphere containing acid fumes or alkaline vapours.

A-3. DETERMINATION OF MOISTURE

A-3.1 Procedure — Heat a flat-bottomed clean glass dish with a cover in the boiling water oven for about 30 minutes, cover the dish and cool it in a desiccator to room temperature and weigh accurately. Place about 5 g of the prepared sample and weigh the dish with the cover accurately. Place the dish and cover separately inside a vacuum desiccator. Evacuate and maintain the desiccator at a reduced pressure of 3 mm head of mercury at room temperature for 24 hours. Admit dry air slowly into the desiccator. Remove the dish, replace its cover and weigh.

A-3.2 Calculation

Moisture, percent by mass = \frac{100 (M_2 - M_1)}{M_2 - M_1}

where

M_2 = mass in g of the dish with the material before drying,
M_3 = mass in g of the dish with the material after drying, and
M_1 = tare in g of the dish.

A-4. DETERMINATION OF MATTER INSOLUBLE IN AMMONIUM ACETATE SOLUTION

A-4.1 Reagents

A-4.1.1 Ammonium Acetate Solution — 20 percent (m/v) slightly acidified with a drop or two of glacial acetic acid.

A-4.1.2 Rectified Spirit — See IS : 323-1959*.

A-4.2 Procedure — Weigh about 2 g of the prepared sample into a 250-ml beaker. Add 100 ml of ammonium acetate solution at 40 to 50°C, stir, allow to settle and transfer the supernatent liquid to a tared sintered glass crucible No. G 4 fitted to a suitable filter flask. Apply gentle suction. Add 20 ml portions of the ammonium acetate solution, stir, allow to settle and filter. Repeat this operation until about 200 ml of the ammonium acetate have all been used. Finally, transfer the contents of the beaker, if any, to the crucible by means of a jet of warm water (50°C). Wash the crucible and its contents with 20 ml of the warm water twice and finally with 10 ml of rectified spirit. Dry at 100 ± 1°C to constant mass. Reserve the residue for the determination of grit in A-5.

*Specification for rectified spirit (revised).
A-4.3 Calculation

Matter insoluble in ammonium acetate, percent by mass = \(100 \times \frac{M_2}{M_1}\)

where

\[M_2 = \text{mass in g of the residue, and}\]
\[M_1 = \text{mass in g of the material taken for the test.}\]

A-5. DETERMINATION OF GRIT

A-5.1 General — Grit is that portion of the material which is insoluble on aqua regia and 50 percent sodium hydroxide solution and is retained in 125-micron IS Sieve and is capable of scratching soda glass.

A-5.1.1 Aqua Regia — Prepared by mixing 3 volumes of concentrated hydrochloric acid with one volume of concentrated nitric acid.

A-5.1.2 Dilute Hydrochloric Acid — 2 percent (v/v).

A-5.2 Procedure — Brush the residue obtained in A-4.2 on a sintered glass crucible into a small beaker and boil with 25 ml of aqua regia. Filter back through the crucible, wash with dilute hydrochloric acid and dry.

A-5.2.1 Brush the matter insoluble in aqua regia into a beaker and boil for about 10 minutes with 20 ml of sodium hydroxide solution. Dilute immediately with 200 to 300 ml of water, wash the gritty matter three times with water by decantation and then once with dilute hydrochloric acid. Filter on Whatman filter paper No. 40 or equivalent. Wash, ignite, cool and weigh till constant mass is obtained.

A-5.2.2 Brush the ignited matter on 125-micron IS Sieve and weigh the portion retained. Carry out the soda glass scratching test as prescribed in A-5.4. If the residue scratches soda glass, express the mass (previously weighed) as percentage grit.

A-5.3 Calculation

Grit, percent by mass = \(100 \times \frac{M_1}{M}\)

where

\[M_1 = \text{mass in g of the grit retained on the sieve, and}\]
\[M = \text{mass in g of the material taken for the test in A-4.2.}\]

A-5.4 Soda Glass Scratching Test — Take a portion of the residue, retained on 125-micron IS Sieve in between two clean glass slides and
press the slides by hand along the length and breadth of the glass. Examine whether any scratch in the glass is obtained. If there is a scratch in the glass, then the residue is termed as grit.

A-6. DETERMINATION OF CHLORIDES

A-6.0 Two methods are prescribed, namely, volumetric method and automatic chloride titrator method. In case of dispute, the volumetric method shall be the referee method.

A-6.1 Volumetric Method

A-6.1.0 Outline of the Method — Thiocyanates are removed by bismuth nitrate reagent and chlorides are determined by volumetric Volhard method.

A-6.1.1 Reagents

A-6.1.1.1 Bismuth nitrate reagent — Warm together about 10 g of bismuth nitrate and 50 ml of glacial acetic acid (see IS: 695-1975*), add 30 ml of water gradually until solution is complete. Dilute to 500 ml with water.

A-6.1.1.2 Dilute nitric acid — 0.1 N.

A-6.1.1.3 Standard silver nitrate solution — 0.1 N.

A-6.1.1.4 Standard ammonium thiocyanate solution — 0.1 N.

A-6.1.1.5 Ferric alum indicator — saturated solution in water.

A-6.1.1.6 Nitrobenzene

A-6.1.2 Procedure — Weigh accurately about 5 g of the prepared sample, add 100 ml of water, stir and allow to stand for one hour with occasional stirring. Add 10 ml of bismuth nitrate reagent, stir well and allow to stand for 15 minutes. Filter off the precipitate and dissolve it in dilute nitric acid. Add 25 ml of silver nitrate solution and 5 ml of nitrobenzene. Shake vigorously till spongy flakes of silver chloride are obtained. Add 2 ml of ferric alum indicator and titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change.

A-6.1.3 Calculation

Chlorides, percent by mass = \( \frac{13.9(25N_1 - VN_2)}{M} \)

where

\( N_1 = \) normality of silver nitrate solution,

\( V = \) volume in ml of ammonium thiocyanate solution, and

\( N_2 = \) normality of ammonium thiocyanate solution, and

\( M = \) mass in g of the sample taken for the test.

*Specification for acetic acid (second revision).*
A-6.2 Automatic Chloride Titrator Method

A-6.2.1 Reagents

A-6.2.1.1 Nitric acid acetic acid reagent — To about 800 ml chloride-free double distilled water, add 6.3 ml of concentrated nitric acid (see IS : 264-1976*) and 100 ml of glacial acetic acid (see IS : 695-1975†). Dilute with the same water to 1 litre and mix thoroughly.

A-6.2.1.2 Gelatin reagent — Weigh accurately about 0.62 g of gelatin reagent (60 parts of gelatin, 1 part thymol blue and 1 part thymol) and dissolve in hot water, cool and dilute to 100 ml. Keep always under refrigeration and take out only desired quantity in a test tube.

A-6.2.1.3 Standard sodium chloride solution — Weigh accurately about 0.23 g of dry sodium chloride, dissolve in water and dilute to 100 ml.

A-6.2.2 Procedure — Clean the electrodes of the instrument and rinse with water thoroughly. Weigh accurately 0.1 to 0.5 g of lead thiocyanate sample directly in the titration vessel. Add 4 ml of nitric acid-acetic acid reagent and 0.2 ml gelatin reagent.

A-6.2.2.1 Prepare standard by taking 0.1 ml of standard sodium chloride solution and 4 ml of nitric acid-acetic acid reagent and 0.2 ml of gelatin reagent. Also prepare blank solution.

A-6.2.2.2 Titrate the sample solution, standard solution as prepared in A-6.2.2.1 and the blank according to the instructions of the instrument and record the time.

A-6.2.3 Calculation

Chlorides (as NaCl), percent by mass = \( \frac{(B - C) \times M_1 \times 100}{(A - C) \times M} \)

where

- \(B\) = time in seconds for lead thiocyanate sample solution,
- \(C\) = time in seconds for blank,
- \(M_1\) = mass in g of sodium chloride present in 0.1 ml of standard sodium chloride solution,
- \(A\) = time in seconds for standard solution (A-6.2.2.1), and
- \(M\) = mass in g of lead thiocyanate sample taken for the test.

A-7. DETERMINATION OF LEAD THIOCYANATE

A-7.1 Reagents

A-7.1.1 Dilute Nitric Acid — 1 percent (v/v).

---

*Specification for acetic acid (second revision).
†Specification for nitric acid (second revision).
A-7.1.2 *Standard Silver Nitrate Solution* -- 0.1 N.

A-7.1.3 *Ferric Alum Indicator* — saturated solution.

A-7.1.4 *Standard Potassium Thiocyanate Solution* — 0.1 N.

A-7.2 Procedure Weigh accurately about 0.7 g of the prepared sample. Dissolve it in about 150 ml of dilute nitric acid. Solution shall be affected by very gentle warming at about 50°C. Hot water decomposes lead thiocyanate and hence a higher temperature shall be avoided.

A-7.2.1 Add 50 ml of silver nitrate solution from a pipette. Then add 2 ml of ferric alum indicator. Determine the excess silver nitrate by titrating with standard potassium thiocyanate solution to the first permanent appearance of a faint reddish colouration due to the formation of ferric thiocyanate complex. Carry out a blank determination at the same time.

A-7.3 Calculation

\[
\text{Lead thiocyanate, percent by mass} = \frac{(X - V) \times N \times 16.17}{M} - 2.0 \times P
\]

where

\[X = \text{volume in ml of potassium thiocyanate solution required for the blank},\]

\[V = \text{volume in ml of potassium thiocyanate required for the sample},\]

\[N = \text{normality of potassium thiocyanate solution},\]

\[P = \text{percentage of sodium thiocyanate (see A-7.4), and}\]

\[M = \text{mass in g of the material taken for the test}.\]

A-7.4 Determination of Sodium Thiocyanate — For applying the correction for sodium thiocyanate determine the percentage of sodium thiocyanate as in A-7.4.2.

A-7.4.1 Reagents

A-7.4.1.1 *Concentrated nitric acid* — See IS: 264-1976*.

A-7.4.1.2 *Ammonium hydroxide* — 25 percent (m/m).

*Specification for nitric acid (second revision).*
A-7.4.1.3 Magnesium uranyl acetate reagent — Prepare the reagent by mixing the following:

**Solution A**
- Crystallized uranyl acetate: 85 g
- Glacial acetic acid (see IS: 695-1975*): 60 g
- Water: 1,000 ml

**Solution B**
- Crystallized magnesium acetate: 500 g
- Glacial acetic acid (see IS: 695-1975*): 60 g
- Water: 1,000 ml

Heat the solution A and B separately, to about 70°C, and maintain at this temperature until the ingredients have dissolved. Mix the two solutions and cool to 20°C. Place the vessel containing the mixed reagent in water at 20°C and keep at this temperature for hours.

A-7.4.1.4 Wash liquid — Shake one millilitre of 1 percent sodium chloride solution with 25 ml of magnesium uranyl acetate reagent. Wash the resulting precipitate thoroughly with 95 percent alcohol and then suspend in 1,000 ml of 95 percent alcohol. After standing for an hour with frequent shaking, filter the mixture and use the filtrate as the wash liquid.

A-7.4.2 Procedure — Take accurately weighed about 3 g of the sample in a 300-ml beaker, add 100 ml of water and then 10 ml of concentrated nitric acid. The reaction between the thiocyanate and the nitric acid, when once begun, is extremely vigorous and shall be carried out under a hood.

After the vigorous reaction has subsided, evaporate the solution to one half of its original volume and filter. Wash the residue, the beaker and the filter with about 50 ml of water. In a 150-ml beaker, carefully evaporate the filtrate and washings nearly to dryness. Dissolve this residue in 25 ml of water and neutralize with ammonium hydroxide. Warm to hasten solution of soluble salts, and filter into a 100-ml beaker using small amounts of water for washings. Evaporate to a volume of 5 ml or less. Add 25 ml of magnesium uranyl acetate reagent to the beaker and place in a 1,000-ml beaker containing water to a depth of 10 mm to maintain a temperature of 20°C. Stir the contents of the small beaker for 30 minutes at fairly rapid rate with a mechanical stirrer. Transfer the precipitate of sodium magnesium uranyl acetate to tared sintered glass crucible No. G 4 using a total of 25 to 30 ml of the wash liquid for transferring and washing. Dry the washed precipitate and crucible in an oven at 100 to 110°C, cool in a desiccator and weigh. Carry out a blank determination at the same time.

*Specification for acetic acid (second revision).*
A-7.4.3 Calculation

Sodium thiocyanate, percent by mass = \( \frac{5.4 \times (A - B)}{M} \)

where

\( A \) = mass in g of sodium magnesium uranyl acetate obtained from the sample,
\( B \) = mass in g of the sodium magnesium uranyl acetate from the blank, and
\( M \) = mass in g of the material taken for the test.

A-7.4.4 Flame photometer may be used alternatively for the determination of sodium wherever such facilities exist.

A-8. DETERMINATION OF LEAD

A-8.1 Reagents

A-8.1.1 Concentrated Nitric Acid — See IS : 264-1976*.

A-8.1.2 Dilute Sulphuric Acid — 50 percent and 3 percent by volume.

A-8.1.3 Ethyl Alcohol — 50 percent by volume.

A-8.2 Procedure — Accurately weigh about 0.5 g of the material into 400-ml beaker, add 25 ml of water, then 3 ml of concentrated nitric acid and cover with a watch glass. Warm carefully and when the reaction ceases, add 10 ml of 50 percent sulphuric acid and evaporate to sulphur trioxide fumes. Cool and cautiously add 75 ml of water and warm to dissolve soluble salts. Add 75 ml of cold water and allow to stand for 30 minutes. Filter the lead sulphate on a weighed filtering crucible. Wash 5 times with 50 percent alcohol. Dry the crucible at 105 to 110°C for about 30 minutes. Cool in a desiccator and weigh to constant mass.

A-8.3 Calculation

Lead (as lead thiocyanate), percent by mass = \( \frac{106.6 \times A}{M} - 1.16 \times B \)

where

\( A \) = mass in g of lead sulphate,
\( B \) = percent by mass of lead chloride, and
\( M \) = mass in g of the material taken for the test.

*Specification for nitric acid (second revision).
A-9. DETERMINATION OF pH

A-9.1 Procedure — Dissolve 3 g of the prepared sample in 100 ml of freshly boiled and cooled water. Determine pH of the solution with a suitable pH meter using glass electrodes.

A-10. DETERMINATION OF BULK DENSITY

A-10.1 Procedure — Support a freshly calibrated glass measuring cylinder of 7·5 mm internal diameter and of 10 ml capacity with graduations at 0·05 ml in a vertical position on a bench free from vibrations. Add normal butyl alcohol into the cylinder in just sufficient quantity to bring the liquid level to about 2·5 ml. Weigh 2 g of the prepared sample passing through 150-micron IS sieve accurately on a tared paper scoop and add it in small quantities to the butyl alcohol already added to the cylinder. Wash down any particles of lead thiocyanate adhering to the inner surface of the walls of the cylinder with butyl alcohol conveniently added from a drawn out tube until the total volume of butyl alcohol used is 5 ml. In doing so the jet of butyl alcohol shall be moved round the circular wall without disturbing the cylinder. Allow the cylinder to stand without interference or movement for 1½ hours and read the volume of the settled lead thiocyanate without disturbing the cylinder.

A-10.2 Calculation

\[ \text{Bulk density} = \frac{M}{V} \]

where

\( M \) = mass in g of the lead thiocyanate added to the cylinder, and
\( V \) = volume in ml occupied by lead thiocyanate in the cylinder.

APPENDIX B

(Clause 4.1)

SAMPLING OF LEAD THIOCYANATE FOR EXPLOSIVE AND PYROTECHNICAL COMPOSITIONS

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions (see B-1.1 to B-1.5) shall be observed.

B-1.1 Samples shall not be drawn in an exposed place.
Precaution shall be taken to protect the samples, the material being sampled and the sampling instrument from adventitious contamination.

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

The sampled material shall be placed in clean, dry, airtight amber coloured glass or other suitable containers which have no action on the material.

Each sample container shall be sealed airtight with a stopper after filling and shall be marked with all particulars of the material (see 3.2) and the date of sampling.

**B.2. SCALE OF SAMPLING**

**B.2.1 Lot** — In a single consignment of the material all the containers of the same type and size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different types and sizes of containers, the containers belonging to the same batch, type and size shall be grouped together and each such group shall constitute a separate lot.

**B.2.1.1** For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

<table>
<thead>
<tr>
<th>LOT SIZE</th>
<th>SAMPLE SIZE</th>
</tr>
</thead>
<tbody>
<tr>
<td>$N$</td>
<td>$n$</td>
</tr>
<tr>
<td>(1)</td>
<td></td>
</tr>
<tr>
<td>Up to 25</td>
<td>3</td>
</tr>
<tr>
<td>26 ,, 50</td>
<td>4</td>
</tr>
<tr>
<td>51 ,, 100</td>
<td>5</td>
</tr>
<tr>
<td>101 and above</td>
<td>7</td>
</tr>
</tbody>
</table>

In order to ensure randomness of selection, random number tables (see IS: 4905-1968*) shall be used. In case such tables are not available, the following procedure is recommended for use:

Arrange the containers in the lot in a systematic order and starting from any one count them as 1, 2, 3,..., etc., up to $r$ and so on where $r$ is the integral part of $N/n$ ($N$ and $n$ being the lot size and sample size respectively). Every $r$th container thus counted shall be withdrawn to constitute the sample.

*Methods for random sampling.
B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected, draw a small representative portion of the material approximately 100 g in mass.

B-3.2 Out of these portions, small but equal quantity of the material shall be taken and mixed thoroughly to form a composite sample of about 150 grams. The composite sample shall be divided into 3 equal parts, one for the purchaser, one for the supplier and the third to be used as a referee sample.

B-3.3 From the remaining portions of the material from each container, draw about 60 g of the material. Divide it into three equal parts and transfer the material to three separate bottles giving full identification particulars of the sample on the bottles. The material in each bottle constitute an individual sample. One of these three sets (each set containing \( N \) bottles representing \( n \) containers sampled) shall be for the purchaser, another for the supplier and the third for the referee.

B-3.4 The referee samples consisting of a composite sample, and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier and be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of moisture content shall be performed on each of the individual samples.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Moisture Content — From the set of individual test results for moisture content the average (\( X \)) and the ranges (\( R \)) of the test results shall be computed (range \( R \) is defined as the difference between the maximum and the minimum value of the test results).

B-5.1.1 A lot shall be declared as conforming to the requirements of moisture content if \( (X + 0.6R) \) is less than or equal to 0.05.

B-5.2 For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test results shall meet the corresponding values specified in the table of requirements.
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(First Revision)

(Page 4, clause 2.2) — Substitute the following for the existing:

'Particle Size — All the material shall pass through 90 micron IS Sieve and not

Reprography Unit, BIS, New Delhi, India.