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भारतीय मानक मसौदा
इलेक्ट्रोप्लेटिंग के लिए कैडमियम ऑक्साइड — विशिष्टि
(IS 5003 का पहला पुनरीक्षण)

Draft Indian Standard

Cadmium Oxide for Electroplating — Specification
(First Revision of IS 5003)

(ICS 25.220.40)

Electroplating Chemicals and Photographic Materials Sectional
Committee, CHD 5

Last Date for Comments: **5 July 2024**

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

FOREWORD

(formal clauses will be added later)

This standard was originally published in 1968. In the plating industry the cyanide bath, which is made up from sodium cyanide and to which wetted cadmium oxide is added, is used almost exclusively. The exact composition of cadmium complex is not known though it is usually considered to be $\text{Na}_2\text{Cd}(\text{CN})_4$. Stable, noncritical additives consisting of organic compounds like dextrin and cellulose derivatives, used together with traces of dissolved nickel, make possible highly lustrous cadmium coating.

In this revision, alternative instrumental test methods AAS, ICP-MS and ICP-OES have been incorporated for the determination of lead, iron, zinc and copper, and the standard has been updated based on the experience of last five decades and on the currently available data.

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: 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of these specified value in this standard.

Draft Indian Standard
CADMIUM OXIDE FOR ELECTROPLATING — SPECIFICATION
(*First Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for cadmium oxide used in electroplating.

2 REFERENCES

The standards listed in Annex A contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 REQUIREMENTS

3.1 Description

The material shall be in the form of brown powder insoluble in water and readily soluble in acids. It shall correspond in composition essentially to CdO. It shall not contain free metal particles and shall be free from dirt and visible impurities.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex B. Reference to the relevant clauses of Annex B are given in col 4 of Table 1.

Table 1 Requirements for Cadmium Oxide for Electroplating
(*Clause 3.2, B-5.2.3.4, B-6.2.3.1, B-7.2.3.1, B-8.2.3.2*)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Clause No. in Annex B)
(1)	(2)	(3)	(4)
i)	Cadmium oxide (as CdO), percent by weight, <i>Min</i>	99.0	B-2
ii)	Insoluble matter (in cyanide), percent by weight, <i>Max</i>	0.1	B-3
iii)	Loss on ignition, percent by weight, <i>Max</i>	0.5	B-4
iv)	Zinc (as Zn), percent by weight, <i>Max</i>	0.005	B-5
v)	Lead (as Pb), percent by weight, <i>Max</i>	0.03	B-6
vi)	Copper (as Cu), percent by weight, <i>Max</i>	0.000 5	B-7
vii)	Iron (as Fe), percent by weight, <i>Max</i>	0.000 5	B-8

3.3 It shall be free from mercury when tested by the method given in Annex C.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier.

4.2 Marking

The containers shall be marked with the following:

- a) Name of the material;
- b) Net weight;
- c) Name of the manufacturer and his recognized trade-mark, if any; and
- d) Lot number and date of manufacture.

4.2.1 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

5 SAMPLING

The method of preparing representative samples of the material and the criteria for conformity of the material to this specification shall be as prescribed in Annex D.

ANNEX A

(Clause 2)

(LIST OF REFERRED STANDARDS)

<i>IS No.</i>	<i>Title</i>
IS 323: 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
IS 799: 2023	Ammonia liquor — Specification
IS 1070: 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 3025 (Part 2) : 2019 ISO 11885 : 2007	Methods of sampling and test (Physical And Chemical) for water and wastewater: Part 2 Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (<i>first revision</i>)
IS 3025 (Part 65) : 2022 ISO 17294-2 : 2016	Methods of sampling and test physical and chemical for water and wastewater Part 65 Application of Inductively Coupled Plasma Mass spectrometry (ICP-MS) — Determination of selected elements including Uranium Isotopes (<i>first revision</i>)
IS 4161: 2023	Nessler cylinder — Specification (<i>first revision</i>)
IS 9958 : 1981	Atomic absorption spectrophotometric method — For determination of zinc
IS 11123 : 1984	Method for determination of copper by atomic absorption spectrophotometry
IS 12074 : 1987	Method for determination of lead by atomic absorption spectrophotometer
IS 13320 : 1992	Determination of iron by atomic absorption spectrophotometry — Test method

ANNEX B

(Clause 3.2)

ANALYSIS OF CADMIUM OXIDE

B-1 QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

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

B-2 DETERMINATION OF CADMIUM OXIDE

B-2.1 General

Cadmium oxide is determined employing electro-gravimetric method.

B-2.2 Reagents

B-2.2.1 *Dilute Sulphuric Acid* — 5 N approximately.

B-2.2.2 *Phenolphthalein Indicator Solution*

Prepared by dissolving 0.1 g in 100 ml of 60 percent (v/v) of rectified spirit conforming to IS 323.

B-2.2.3 *Sodium Hydroxide Solution* — 0.1 N approximately.

B-2.2.4 *Potassium Cyanide Solution* — 20 percent (w/v).

B-2.3 Procedure

Dissolve about 0.5 g of the sample in just sufficient quantity of dilute sulphuric acid, and filter through a sintered-glass crucible (G No. 4) washing with minimum quantity of warm water. Add a drop of phenolphthalein indicator and neutralize excess acid with sodium hydroxide solution. Then add potassium cyanide solution, dropwise, with constant stirring, till the precipitated cadmium hydroxide just dissolves. Avoid a large excess of potassium cyanide. Dilute the solution to 150 ml and electrolyse the cold solution, using tared platinum wire gauze cathode and a current of 0.5 to 0.7 A at 4.8 to 5.0 V. At the end of 6 hours, increase the current to 1.0 A to 1.2 A and continue the electrolysis for another hour. Wash the split clock glass and sides of the beaker with about 20 ml of water and continue electrolysis for about 15 minutes. If the newly exposed surface of the cathode remains bright, thus indicating that the deposition of cadmium is complete, remove the electrolyte from the electrodes, rinse immediately, with water, stop the current and rinse the cathode with alcohol. Dry at 100 °C, cool and weigh. Test the residual electrolyte for cadmium, if any.

B-2.4 Calculation

$$\text{Cadmium oxide (as CdO), percent by weight} = 114 \times \frac{W_1}{W}$$

where

W_1 = weight in grams of the cadmium metal deposited, and

W = weight in grams of the material taken for the test.

B-3 DETERMINATION OF INSOLUBLE MATTER (IN CYANIDE)

B-3.1 Reagents

B-3.1.1 *Potassium Cyanide* — solid.

B-3.2 Procedure

Weigh accurately about 20 g of sample and 30 g of potassium cyanide and mix by coning or repeated stirring (there shall be no grinding). Introduce the mixture into a 100 ml conical flask which is half filled with distilled water. Heat the mixture from 43 °C to 48 °C, swirl for 5 minutes, and then maintain the flask at 44.0 °C ± 0.5 °C for four hours. Dilute to one litre, shake, set aside overnight and then filter through a sintered glass crucible (G No. 4). Wash the residue with cold water, dry the crucible and reweigh.

B-3.3 Calculation

$$\text{Insoluble matter, percent by weight} = \frac{W_1 \times 100}{W}$$

where

W_1 = weight in grams of the residue, and

W = weight in grams of the material taken for the test.

B-4 DETERMINATION OF LOSS ON IGNITION

B-4.1 Procedure

Weigh accurately about 1 g of the sample and heat for two hours in an open muffle furnace at 800 °C. Determine the loss in weight after cooling the residue in a desiccator.

B-4.2 Calculation

$$\text{Loss on ignition, percent by weight} = \frac{W_1 \times 100}{W}$$

where

W_1 = loss in weight in grams, and

W = weight in grams of the material taken for the test.

B-5 DETERMINATION OF ZINC

B-5.1 General

Five methods for the determination of zinc are described. Either of these may be used for general routine purposes, but in case of a dispute Method D shall be the referee method.

B-5.2 Method A

B-5.2.1 General

Zinc is determined colorimetrically using methyl violet as indicator. It is also determined colorimetrically using p-dimethyl aminostyryl- β -naphthiozole methiodide.

B-5.2.2 Reagents

B-5.2.2.1 Ammonium thiocyanate solution — 60 percent (w/v) and 10 percent (w/v) solutions in water.

B-5.2.2.2 Ammonium chloride solution — 20 percent (w/v).

B-5.2.2.3 Ammonium hydroxide — conforming to IS 799.

B-5.2.2.4 Ammonium hydroxide ammonium chloride solution

Prepared by mixing 15 ml of ammonium hydroxide and 15 ml of ammonium chloride solution with 200 ml of water.

B-5.2.2.5 Dilute sulphuric acid — approximately 18 N.

B-5.2.2.6 Dilute hydrochloric acid — approximately 3 N.

B-5.2.2.7 Standard hydrochloric acid — 0.5 N.

B-5.2.2.8 Tartaric acid solution — 25 percent (w/v).

B-5.2.2.9 Methyl orange indicator

Dissolve 0.1 g of the indicator in 100 ml of water.

B-5.2.2.10 Methyl violet

Dissolve 0.1 g of the indicator in 200 ml of water.

B-5.2.2.11 Standard zinc solution

Dissolve 4.240 g of zinc sulphate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) in water and dilute to 1 000 ml. Further dilute 10 ml of this solution to 1 000 ml. One millilitre of the solution contains 0.01 mg of zinc (as Zn).

B-5.2.2.12 Wash mixture

Mix 50 ml of 60 percent (w/v) ammonium thiocyanate solution, 200 ml of water and 1 ml of sulphuric acid.

B-5.2.2.13 Isoamyl alcohol

B-5.2.3 Procedure

Weigh accurately about 1 g of the material and dissolve in dilute hydrochloric acid. Transfer the solution to a separating funnel of 50 ml capacity. Add 3 ml of 60 percent w/v ammonium thiocyanate solution and 0.1 ml of dilute sulphuric acid. Mix and add 10 ml of isoamyl alcohol. Shake the contents of the flask for 2 minutes. Allow the layers to separate and reject the lower aqueous layer. Wash the isoamyl alcohol portion thrice, with 10 ml portions of the wash mixture, by shaking each time for 2 minutes. Transfer the isoamyl alcohol layer into another separating funnel with 10 ml of ammonium hydroxide and add the washings to the contents of the second separating funnel. Shake the contents of the second separating funnel briskly for 2 minutes and allow the layers to separate. Transfer the lower aqueous layer into a 25 ml measuring flask and make up to volume (solution A).

B-5.2.3.1 Preparation of the standard solution

Take 10 ml of water into a separating funnel and treat in the same way as the test sample. Dilute to 25 ml in a measuring flask (solution B).

B-5.2.3.2 Determine the quantity of the acid necessary to neutralize the solutions as follows.

B-5.2.3.2.1 Take 2 ml of the solution A in one test tube and 2 ml of the solution B in another. Neutralize the test solutions with standard hydrochloric acid with methyl orange as indicator, using a microburette. Note the volume of acid used for neutralization of each solution.

B-5.2.3.3 Take 2 ml of solution A (corresponding to 0.08 g of the material) in a Nessler cylinder. Add the known quantity of standard hydrochloric acid, necessary for neutralization of solution A (*see B-5.2.3.2.1*) and water to make up the volume to 4 ml. Stir carefully and add 1.5 ml of standard hydrochloric acid, 0.5 ml of methyl violet indicator, 0.25 ml of tartaric acid and 0.3 ml of 10 percent (w/v) ammonium thiocyanate solution. Carry out a control test in another Nessler cylinder using 0.4 ml of standard zinc solution and 2 ml of solution B and other reagents in the same quantities as with the material.

B-5.2.3.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced by the sample is not more intense than that produced in the control test.

B-5.3 Method B

B-5.3.1 Apparatus

B-5.3.1.1 *Nessler cylinders* — 50 ml capacity (*see IS 4161*).

B-5.3.2 Reagents

B-5.3.2.1 *Dilute hydrochloric acid* — 5 N approximately.

B-5.3.2.2 *Ammonium thiocyanate solution* — 57 percent (w/v).

B-5.3.2.3 *Dilute sulphuric acid* — 1 N approximately.

B-5.3.2.4 *p-dimethyl-aminostyryl- β -naphthiazole methiodide solution* — 0.05 percent (w/v) in absolute industrial methylated spirit.

B-5.3.3 Procedure

Weigh accurately 0.5 g of the material and dissolve in required quantity of dilute hydrochloric acid, filter through a sintered-glass crucible (G No. 4), wash with water and make up the filtrate to 200 ml. To 10 ml of this solution add 0.05 ml of ammonium thiocyanate solution, 0.25 ml of dilute sulphuric acid and 0.4 ml of *p*-dimethyl-aminostyryl- β -naphthiazole methiodide solution. Carry out a control test using 10 ml of water and reagents in the same quantities as used in the test.

B-5.3.3.1 The material shall be taken as having satisfied the requirements of the test if the solution containing the material does not show any pink or orange colour in comparison to the solution made for control test.

B-5.4 Method C

Determine zinc content by atomic absorption spectrophotometer (AAS) in accordance with the method prescribed in IS 9958.

B-5.5 Method D

Determine zinc content by ICP-MS in accordance with the method prescribed in IS 3025 (Part 65).

B-5.6 Method E

Determine zinc content by ICP-OES in accordance with the method prescribed in IS 3025 (Part 2).

B-6 DETERMINATION OF LEAD

B-6.1 General

Four methods for the determination of lead are described. Either of these may be used for general routine purposes, but in case of a dispute Method C shall be the referee method.

B-6.2 Method A

B-6.2.1 Apparatus

B-6.2.1.1 Nessler Cylinders — two, of 50 ml capacity (see IS 4161).

B-6.2.2 Reagents

B-6.2.2.1 *p*-Nitrophenol indicator solution

Dissolve 0.2 g of *p*-nitrophenol in hot water and dilute to 100 ml.

B-6.2.2.2 Dilute ammonium hydroxide — 1: 9.

B-6.2.2.3 Dilute hydrochloric acid — 1: 99.

B-6.2.2.4 Hydrogen sulphide solution — saturated and freshly prepared.

B-6.2.2.5 Standard lead solution

Dissolve 1.60 g of lead nitrate [Pb (NO₃)₂] in water and make up the solution to 1 000 ml with water. Pipette out 10 ml of this solution and dilute again to 100 ml with water. One millilitre of this solution contains 0.1 mg of lead (as Pb).

B-6.2.3 Procedure

Weigh accurately 2 g of the material and dissolve in 20 ml of dilute (1: 99) hydrochloric acid in a Nessler cylinder and add one drop of *p*-nitrophenol indicator solution. Add dropwise dilute ammonium hydroxide until the solution becomes colourless and then add 0.5 ml of alkali in excess. Add 5 ml of hydrogen sulphide solution, dilute to the mark and mix well. Carry out a control test in a similar manner in another Nessler cylinder using 6 ml of standard lead solution.

B-6.2.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

B-6.3 Method B

Determine lead content by atomic absorption spectrophotometer (AAS) in accordance with the method prescribed in IS 12074.

B-6.4 Method C

Determine lead content by ICP-MS in accordance with the method prescribed in IS 3025 (Part 65).

B-6.5 Method D

Determine lead content by ICP-OES in accordance with the method prescribed in IS 3025 (Part 2).

B-7 DETERMINATION OF COPPER

B-7.1 General

Four methods for the determination of copper are described. Either of these may be used for general routine purposes, but in case of a dispute Method C shall be the referee method.

B-7.2 Method A

B-7.2.1 Apparatus

B-7.2.1.1 Nessler cylinders — two, of 50 ml capacity (see IS 4161).

B-7.2.2 Reagents

B-7.2.2.1 Sodium diethyldithiocarbamate solution

Dissolve 1.0 g of sodium diethyldithiocarbamate in 1 000 ml of copper-free water. Filter and keep in an amber bottle and protect from strong light.

B-7.2.2.2 *Dilute hydrochloric acid* — approximately 3 N.

B-7.2.2.3 *Sodium pyrophosphate solution* — 10 percent (w/v).

B-7.2.2.4 *Standard copper solution*

Dissolve 0.392 8 g of copper sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in copper-free water and make up the volume to 1 000 ml. Dilute 100 ml of this solution again to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of copper (as Cu).

B-7.2.2.5 *Isoamyl alcohol*

B-7.2.2.6 *Ethyl alcohol* — redistilled.

B-7.2.2.7 *Disodium ethylene diamine tetra acetate (EDTA) solution* — 5 percent (w/v).

B-7.2.3 *Procedure*

Weigh accurately about 2 g of the sample into a 100 ml flask and moisten with 10 ml of water. Add 5 ml of dilute hydrochloric acid. After the sample has completely dissolved, transfer the solution to a 50 ml Nessler cylinder. Make up the volume to 40 ml with water. Add 1 ml of EDTA solution, 2 ml of sodium pyrophosphate solution and mix. Add 7 ml of *isoamyl alcohol*, 1 ml of sodium diethyldithiocarbamate solution (two portions of 1 ml each) and quickly stir the solution after each addition. Carry out a control test in a similar manner in another Nessler cylinder, containing the same volume of water and dilute hydrochloric acid, 1 ml of standard copper solution, 1 ml of EDTA solution, 2 ml of sodium pyrophosphate, 7 ml of *isoamyl alcohol* and 2 ml of sodium diethyldithiocarbamate solution, added likewise in two portions.

B-7.2.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour of the alcohol layer of the test liquid is not more intense than the colour of the alcohol layer in the control test.

B-7.3 Method B

Determine copper content by atomic absorption spectrophotometer (AAS) in accordance with the method prescribed in IS 11123.

B-7.4 Method C

Determine copper content by ICP-MS in accordance with the method prescribed in IS 3025 (Part 65).

B-7.5 Method D

Determine copper content by ICP-OES in accordance with the method prescribed in IS 3025 (Part 2).

B-8 DETERMINATION OF IRON

B-8.1 General

Four methods for the determination of iron are described. Either of these may be used for general routine purposes, but in case of a dispute Method C shall be the referee method.

B-8.2 Method A

B-8.2.1 Apparatus

B-8.2.1.1 *Nessler cylinders* — 50 ml capacity (*see* IS 4161).

B-8.2.2 Reagents

B-8.2.2.1 *Dilute hydrochloric acid* — 4 N approximately.

B-8.2.2.2 *Potassium permanganate solution* — 0.01 N approximately.

B-8.2.2.3 *Ammonium thiocyanate solution* — 50 percent (w/v).

B-8.2.2.4 *Extraction solvent*

Prepared by mixing equal volumes of amyl alcohol and amyl acetate, both of reagent quality.

B-8.2.2.5 *Standard iron solution*

Dissolve 0.702 g of ferrous ammonium sulphate $[\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}]$ in water containing 10 ml of dilute sulphuric acid (10 percent *v/v*) and dilute with water to 1 000 ml. Before use, again dilute 100 ml of this solution to 1 000 ml. One millilitre of this diluted solution is equivalent to 0.01 mg of iron (as Fe).

B-8.2.3 *Procedure*

B-8.2.3.1 Weigh accurately 1 g of the material and dissolve in 20 ml of water and just sufficient hydrochloric acid. Add 1 ml more of dilute hydrochloric acid. Add 1 drop of potassium permanganate solution and mix. Add 5 ml of ammonium thiocyanate solution and 10 ml of extraction solvent. Shake vigorously and allow the layers to separate. Carry out a control test in a similar manner in another Nessler cylinder, containing in the same volume, 0.5 ml of standard iron solution in place of the material and the same amounts of reagents.

B-8.2.3.2 The material shall be taken to have not exceeded the limit prescribed in Table 1 if the intensity of colour obtained with the material is not greater than that obtained in the control test.

B-8.3 Method B

Determine iron content by atomic absorption spectrophotometer (AAS) in accordance with the method prescribed in IS 13320.

B-8.4 Method C

Determine iron content by ICP-MS in accordance with the method prescribed in IS 3025 (Part 65).

B-8.5 Method D

Determine iron content by ICP-OES in accordance with the method prescribed in IS 3025 (Part 2).

ANNEX C

(Clause 3.3)

DETECTION OF MERCURY IN CADMIUM OXIDE

C-1 REAGENTS

C-1.1 Sodium Cyanide — solid.

C-1.2 Alkaline Stannous Chloride Solution

Dissolve 1.2 g of stannous chloride in 20 ml of water disregarding the precipitate formed by hydrolysis. Pour while stirring into of 20 percent sodium hydroxide solution and continue stirring until the precipitate is dissolved.

C-2 PROCEDURE

C-2.1 Dissolve 15 g of sodium cyanide and 6 g of material in water, filter and dilute to 100 ml in a Nessler cylinder. Add 2 ml of the alkaline stannous chloride and mix.

C-2.2 A turbidity indicates the presence of mercury and is visible if 0.000 2 g or more of mercury is present.

ANNEX D

(Clause 5)

SAMPLING OF CADMIUM OXIDE FOR ELECTROPLATING

D-1 GENERAL REQUIREMENTS OF SAMPLING

D-1.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

D-1.2 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

D-1.3 The sampling instrument shall be clean and dry.

D-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

D-1.6 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers in which the material has no action.

D-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

D-1.8 Each sample container shall, be sealed air-tight after filling, and marked with full details of sampling, and the date of sampling.

D-2 SCALE OF SAMPLING

D-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

D-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested 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 2 Number of Containers to be Selected from Lots of Different Sizes

Lot Size	Sample Size
<i>N</i>	<i>N</i>
(1)	(2)
3 to 15	3
16 to 40	4
41 to 110	5
111 to 180	6
181 to 300	7
301 to 500	8
501 to 800	9
801 and above	10

D-2.3 In order to ensure randomness of selection, random number tables shall be used. If such tables are not available, the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1,2,3.....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 rth container thus counted shall be withdrawn to constitute the sample.

D-3 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

D-3.1 From each of the containers selected according to **D-2.3**, a representative portion of the material sufficient for carrying out the tests specified under 2 shall be drawn and these shall constitute the individual samples.

D-3.2 From each of the individual samples, a small but equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample.

D-3.3 The material in the individual and composite samples shall be transferred to separate bottles and labelled with full identification particulars of the samples.

D-4 NUMBER OF TESTS

D-4.1 Tests for the determination of cadmium oxide content shall be carried out on each of the individual samples.

D-4.2 Tests for the remaining characteristics shall be done on the composite sample.

D-5 CRITERIA FOR CONFORMITY

D-5.1 For Individual Samples

For cadmium oxide content the mean and range of test results shall be computed as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{sum of individual test results}}{\text{number of tests}}$$

Range (R) = difference between the maximum and minimum value of the test results

D-5.1.1 The lot shall be declared to have satisfied the requirements for cadmium oxide content if $\bar{X} - 0.6 R \geq 99.0$.

D-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all other characteristics, the test results on the composite sample shall meet the corresponding requirements specified.