भारतीय मानक
Indian Standard

IS 17444 : 2020

कोबाल्ट एसीटेट — विशिष्टि

Cobalt Acetate — Specification

ICS 71.060

भारतीय मानक ब्यूरो
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Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Cobalt acetate is generally in the form of reddish pink crystalline powder when present as cobalt acetate tetrahydrate and intense red powder in anhydrous form. Cobalt acetate has vinegar like odour. The standard prescribes the requirements, method of test and sampling for cobalt acetate.

It finds application as precursor to various oil drying agents and catalysts for paints and varnishes.

The list of experts who had made significant contribution to the formulation of this standard is given at Annex C.

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

Indian Standard
COBALT ACETATE — SPECIFICATION

1 SCOPE

The standard prescribes the requirements, method of test and sampling for cobalt acetate.

2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision 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 standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

IS No.	*Title*
264 : 2005	Nitric acid — Specification (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)

3 DESCRIPTION

The material is generally in the form of reddish pink crystalline powder when present as cobalt acetate tetrahydrate and intense red powder in anhydrous form. Cobalt acetate has vinegar like odour.

4 REQUIREMENTS

The material shall be of such a composition as to satisfy the requirements of this standard. The material shall comply with the requirements given in Table 1.

5 PACKAGING AND MARKING

5.1 Packaging

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

5.2 Marking

The packages shall be securely closed and bear legibly and indelibly the following information:

a) Name and grade of the material;
b) Name of the manufacturer and his recognized trade mark, if any;
c) Gross and net mass;
d) Date of manufacture; and
e) Batch number.

5.2.1 *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.

Table 1 Requirements for Cobalt Acetate

(*Clause* 4)

Sl No.	Characteristic	Requirements	Method of Test Annex A
(1)	(2)	(3)	(4)
i)	Cobalt, percent by mass (*Min*)	99	A-2 and A-13
ii)	*p*H value (5 perent solution)	6-8	A-3
iii)	Matter insoluble in water (percent by mass)	$\leq$0.01	A-4
iv)	Lead (percent by mass, *Max*)	0.000 5	A-5, A-12 and A-13
v)	Sulphate (percent by mass, *Max*)	0.005	A-6 and A-11
vi)	Iron (percent by mass, *Max*)	0.00 1	A-7, A-12 and A-13
vii)	Chloride (percent by mass, *Max*)	0.00 1	A-8 and A-11
viii)	Copper (percent by mass, *Max*)	0.000 5	A-9, A-12 and A-13
ix)	Sodium (percent by mass, *Max*)	0.01	A-10, A-12 and A-14
x)	Calcium (percent by mass, *Max*)	0.005	A-14
xi)	Magnesium (percent by mass, *Max*)	0.005	A-14

ANNEX A

(*Clause* 4, *Table* 1)

A-1 QUALITY OF REAGENTS

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

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

A-2 DETERMINATION OF PURITY OF COBALT ACETATE

A-2.1 Outline

Determination of cobalt acetate (as Co) can be done by titrating it with chelating ligand, such as ethylene diamine tetra acetic acid [$(HO_2CCH_2)_2$ NCH_2CH_2N $(CH_2CO_2H)_2$, EDTA]. The reaction of disodium salt of EDTA with cobalt can be represented by the equation;

$$\mathrm{Co\ (CH_3CO_2)_2 + (NaO_2CCH_2)_2\,NCH_2\,CH_2\,N \longrightarrow (CH_2CO_2H)_2\ [Co(EDTA)] + NaO_2CCH_3]}$$

A-2.2 Procedure

A-2.2.1 *EDTA Preparation*

0.01 M EDTA solution is prepared by dissolving 1.9 g of EDTA, disodium salt in 500 ml deionized water (disodium salt of EDTA can dissolved in water with thorough mixing). Store the solution in plastic bottle.

A-2.2.2 *Standardization of EDTA*

Pipette out 10 ml of cobalt solution in four 250 ml beakers. Add 50 ml of deionized water to all the cobalt samples. Add 4 M sodium acetate (10 ml) to the beaker and adjust the *p*H to 5.8 using 3M sodium hydroxide. Calibrate the *p*H meter with buffer solutions of 4.0 and 7.0. Heat the cobalt solution to 90°C on a hot plate. Do not boil the solution. Add 5 drops of xylenol orange indicator (0.2g/100 ml alcohol). Titrate with EDTA solution. End point is sudden colour change from violet to yellow pink.

Calculate the molarity of EDTA titrant using the exact molarity of cobalt solution.

$$\mathrm{M_1V_1\ (cobalt\ solution) = M_2V_2\ (EDTA)}$$

$$\mathrm{M_2\ (EDTA)}\ \frac{M_1\,V_1}{V_2}$$

A-2.2.3 *Titration of Cobalt Solution*

Add 4 M sodium acetate (10 ml) to each unknown Co^{2+} solution and set the *p*H to 5.8 with 3M NaOH.

Heat solution to approximately 95°C using hot plate. Add xylenol orange (4-5 drops) in solution [concentration to be kept for xylenol is 0.2 g/100 ml, 50 percent alcohol] and titrate it against EDTA solution. Continue the titration till we observe a end point colour change from violet to yellow pink.

$$\mathrm{M_2V_2\ (EDTA) = M_3V_3\ (cobalt\ sample\ solution)}$$

$$\mathrm{M_3\ (Cobalt\ sample\ solution)} = \frac{M_2\,V_2}{V_3}$$

Estimate the cobalt content by converting it into percentage: $\frac{x}{1000}\times 100$

A-3 *p*H VALUE OF 5 PERCENT SOLUTION

A-3.1 Apparatus

A-3.1.1 *p*H *Meter* — A suitable *p*H meter fitted with glass and calomel electrodes.

A-3.2 Procedure

Weigh 2.50 g of the material in a beaker, add 50 ml of water and stir to dissolve. Measure the *p*H of the solution with the help of *p*H meter.

A-4 MATTER INSOLUBLE IN WATER

A-4.1 Reagents

A-4.1.1 *Concentrated Sulphuric Acid*

A-4.2 Procedure

Weigh accurately about 10 g of the test sample and dissolve in 100 ml of water. Add 3 ml of sulphuric acid and stir thoroughly at room temperature. Filter through a tared filter paper or tared Gooch or sintered glass ncrucible (G No. 4). Wash the residue with water till it is free from acid. Dry the filter paper or crucible in an oven maintained at a temperature of 105 to 110°C till constant mass is obtained. For pure grade, use about 20 g of the test sample.

A-4.3 Calculation

$$\text{Insoluble matter percent by mass} = \frac{M_1}{M_2}\times 100$$

where

M_1 = mass in g, of the residue obtained; and

M_2 = mass in g, of the material taken for the test.

A-5 DETERMINATION OF LEAD

A-5.1 Principle

A sample containing microgram quantities of lead is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammoniacal citrate-cyanide reducing agent (*p*H 10 to 11.5). The quantity of lead present in the sample is determined spectrophotometrically by measuring the absorbance at 510 nm in chloroform extract containing the lead dithizonate complex.

A-5.2 Minimum Detection Limit

1.0 µgPb/10 ml dithizone solution (extract).

A-5.3 Apparatus

A-5.3.1 Spectrophotometer for use at 510 nm with a path length of 1 cm or longer.

A-5.3.2 P*h meter*

A-5.3.3 *Standard Volumetric Glasswares*

A-5.3.4 *TEF Beaker,* 100 ml for acid digestion.

A-5.3.5 *Separatory Funnels,* 250 ml and 500 ml.

All glasswares are to be cleaned with 1:1 HNO_3, and rinsed thoroughly with distilled water.

A-5.4 Reagents

A-5.4.1 *Quality of Reagents*

Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

A-5.4.2 *Stock Lead Solution*

Dissolve 0.159 9 g lead nitrate [$(Pb(NO_3)_2$, minimum purity, 99.5 percent (*w/w*)] in about 200 ml of water. Add 10 ml concentrated HNO_3 and dilute to 1 000 ml with water, 1.0 ml of this solution will contain 100 µg of Pb.

A-5.4.3 *Standard Lead Solution*

Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 µg of Pb.

A-5.4.4 *Stock Dithizone Solution*

Dissolve 25 mg dithizone in about 50 ml chloroform ($CHCl_3$) taken in a 200 ml beaker and filter through Whatman No. 42 (or equivalent) filter paper. Collect the filtrate and two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a 500 ml separatory funnel. Add about 100 ml 1 percent (*v/v*) solution, shake moderately for about 1 min. Transfer the $CHCl_3$ layer to another 250 ml separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the $CHCl_3$ layer) with 100 ml of 1 percent (*v/v*) NH_4OH solution, transfer the $CHCl_3$ layer to another 250 ml separatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out. To the combined aqueous extract in the 500 ml separatory funnel add 1:1 HC1 in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizonewith three 25 ml portions of $CHCl_3$. Dilute the combined extract to 250 ml with $CHCl_3$, 1 ml of this solution will contain 100 µg of dithizone.

A-5.4.5 *Working Dithizone Solution*

Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with $CHCl_3$, 1 ml of this solution will contain 40 µg of dithizone.

A-5.4.6 *Nitric Acid* — Concentrated (18 N).

A-5.4.7 *Nitric Acid — Dilute* —20 percent, *v/v*.

A-5.4.8 *Ammonium Hydroxide* — Concentrated (14 N).

A-5.4.9 *Ammonium Hydroxide* — Dilute 10 percent, *v/v* and 1 percent, *v/v.*

A-5.4.10 *Citrate-Cyanide Reducing Solution* — Dissolve 200 g anhydrous ammonium citrate [$(NH_4)_2$ $HC_6H_5O_7$] 10 g anhydrous sodium sulphite (Na_2SO_3), 5 g hydroxylamine hydrochloride, 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with one litre of concentrated NH_4OH.

> **CAUTION** — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth

A-5.5 Procedure

A-5.5.1 *Sample Digestion*

Digest all samples for lead as per standard digestion procedure using minimum amount of HNO_3, H_2SO_4, and HNO_3-HC_lO_4. Dilute the acidified sample (*p*H = 2) to 100 ml and add 20 ml of dilute (20 percent, *v/v*) HNO_3, filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30 s, allow to stand (to get two separate layers). Discard 1-2 ml $CHCl_3$ layer and then fill the absorption cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

A-5.5.2 *Calibration Curve*

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

A-5.6 Calculation

mgPb/litre = µg (in 10 ml extract obtained from calibration curve)/ Volume of sample (ml)

A-6 DETERMINATION OF SULPHATE

A-6.1 Method A

A-6.1.1 *Outline*

Sulphate is determined by spectrophotometer, developing the turbidity of barium sulphate. The turbidity of a dilute barium sulphate suspension is difficult to reproduce. It is, therefore, essential to adhere rigidly to the conditions of experimental procedure.

A-6.1.2 *Reagents*

A-6.1.2.1 *Standard sulphate solution* — 1 ml equivalent to 1 mg H_2SO_4. Transfer 50 ml of this stock solution to a 250 ml volumetric flask and dilute to the mark. Five ml of this solution is equivalent to 1 mg H_2SO_4.

A-6.1.2.2 *Conditioning reagent* — Mix 50 ml of glycerine with a solution containing 30 ml of concentrated hydrochloric acid, 300 ml of water and 75 g of sodium chloride.

A-6.1.2.3 *Barium chloride* — Use crystals of barium chloride that pass through 850-micron IS Sieve and retain on 500-micron IS Sieve.

A-6.1.3 *Calibration Curve*

Transfer 5, 10, 15, 20, 25 and 30 ml of the standard sulphate solution (5 ml = 1 mg H_2SO_4) from a calibrated burette into separate 100 m1 volumetric flasks. To each flask add 5 ml of the conditioning reagent and dilute to 100 ml with water. Mix well. Add 0.3 g of solid barium chloride to each flask, and shake for 1 min by inverting each flask once per second; all the barium chloride should dissolve. Allow to stand for 4 minutes. Prepare blank for reagents with water. Measure the percent transmission (*T*) on spectrophotometer at 420 nm wavelength adjusting the blank at 100 percent transmission. Obtain log *T* and plot a calibration of mg H_2SO_4 versus log *T* passing through origin.

A-6.1.4 *Procedure*

Transfer an aliquot of the solution containing 2 to 4 mg of sulphate to a 100 ml volumetric flask. Add 5 ml of the conditioning reagent and dilute to the mark with water. Mix well. Add 3 g of barium chloride crystals and shake for 1 min by inverting the flask once per second. Allow to stand for four minutes. Prepare a reagent blank. Obtain percent transmission (*T*) on spectrophotometer at 420 nm wavelength adjusting blank at 100 percent transmission. From the log T, obtain the mg of H_2SO_4 from the calibration curve.

A-6.1.5 *Calculation*

$$\text{Sulphate, percent by mass} = \frac{M_1 \times 100 \times F\ (0.979.5)}{1000 \times M}$$

where

M_1 = mass in mg, of H_2SO_4 obtained from the calibration curve;

F = dilution factor; and

M = mass of the material taken for the test.

A-6.2 Method B

A-6.2.1 *Reagents*

A-6.2.1.1 *Dilute hydrochloric acid*, approximately 10 percent (*v/v*).

A-6.2.1.2 *Barium chloride solution*, approximately 10 percent (*m/v*).

A-6.2.2 *Procedure*

Weigh accurately about 5 g of the material and add 30 ml of dilute hydrochloric acid. Filter, if necessary, and wash the filter paper thoroughly. Heat the solution to boiling and add drop by drop 10 ml of hot barium chloride solution. Boil the solution for 15 min and then allow to stand for 4 h. Filter through a tared Gooch or a sintered glass crucible (GNo. 4). Wash the precipitated barium sulphate with hot water till it is free from chlorides and dry to constant mass at about 110°C.

A-6.2.3 *Calculation*

$$\text{Sulphates percent by mass} = 74.63 \times \frac{M_1}{M}$$

where

M_1 = mass of barium sulphate, and

M = mass of the material taken for the test.

A-7 TEST FOR IRON

A-7.1 Principle

The dissolved iron under slightly acidic medium is reduced using hydroxyl ammonium chloride and the colour is developed with 2,2’bipyridyl solution [iron (II)-2,2’ bipyridyl complex]. The absorbance of the colour is measured by a spectrophotometer at 522 nm.

A-7.2 Apparatus

A-7.2.1 *Spectrophotometer,* suitable for measurement at 522 nm, with accuracy of 1 nm.

A-7.2.2 *Standard Laboratory Glass Apparatus*

A-7.3 Reagents

A-7.3.1 *Distilled Water*

A-7.3.x2 *Hydrochloric Acid,* approximately 6 mol/l (1:1 v/v).

A-7.3.3 *Hydrochloric Acid,* approximately 1 mol/l to 910 ml of distilled water, add 90 ml of concentrated hydrochloric acid and mix.

A-7.3.4 *Hydroxylammonium chloride*

Dissolve l0 g of hydroxylammonium chloride ($NH_2OH.HCI$) in water and dilute to 100 ml.

A-7.3.5 *Ammonium Acetate Solution, 30 percent (m/v).*

A-7.3.6 *2,2' — Bipyridyl Solution* — Dissolve 1 g of 2,2'-Bipyridyl in 10 ml of hydrochloric acid solution (1 mol/l) and dilute to 100 ml.

A-7.3.7 *Standard Iron Solution A*

Dissolve 0.7022 g of ferrous ammonium sulphate hexahydrate [$FeSO_4NH_4SO_46H_2O$] in water in a 250 ml beaker, add 50 ml of sulphuric acid (about 1 mol/l) and mix. Transfer quantitatively into a one-litre volumetric flask, make up to the mark and mix well. One ml of this solution contains 0.1 mg iron (as Fe).

A-7.3.8 *Standard Iron Solution B*

Dilute 100 ml of the above solution (solution A) and dilute to 1 litre in a volumetric flask, mix well. One millilitre of this solution contains 10 µg of iron (as Fe).

A-7.4 Procedure

A-7.4.1 *Calibration*

Into each of a series of 100 ml volumetric flasks, place the quantities of standard iron solution B as given below:

Standard Iron Solution. B (ml)	*Corresponding Iron Content* as Fe µg
0 (Compensation)	0
5.0	50
10.0	100
15.0	150
20.0	200
25.0	250
30.0	300
35.0	350
40.0	400
45.0	450
50.0	500

Add to each volumetric flask an amount of water sufficient to dilute to 50 ml, then 2 ml of hydrochloric acid and 2 ml of hydroxylammonium chloride solution and after 5 min, add 5 ml of ammonium acetate solution and 1 ml of 2,2 bipyridyl solution. Dilute to mark, mix well, and wait for 10 min. Measure absorbance using spectrophotometer with 1-cm cell at 522 nm against reagent blank solution (containing equal amount of added reagent except in standard solution and make up the volume to 100 ml).

A-7.4.1.1 *Preparation of calibration graph*

Prepare a calibration graph with iron as Fe in l.0 µg/ 100 ml as abscissa and absorbance as ordinates.

A-7.4.2 *Determination of Iron*

Weigh accurately to the nearest 1 mg, an amount of cobalt acetate containing 100 to 1 000 µg of iron (as Fe) into a platinum dish. Wet it in minimum amount of distilled water and neutralize with 6 mol/l hydrochloric acid solution and add a few drops in excess. Evaporate to dryness on a water bath. Wet it with a few drops of dilute hydrochloric acid and then dissolve in 20 to 30 ml of distilled water and quantitatively transfer into a l00 ml one-mark volumetric flask, dilute to mark and mix well. If the solution is turbid filter through a Whatman No. 541 filter paper and discard 10 to 15 ml of the initial filtrate.

Transfer a suitable aliquot volume (or the filtrate if filtered) containing 50 to 500 µg of Fe into a l00 ml one-mark volumetric flask. Dilute to around 50 ml (if less), add 2 ml hydrochloric acid and 2 ml of hydroxylammonium chloride. Mix well and after 5 min add 5 ml of ammonium acetate solution followed by 1 ml of 2,2'-bipyridyl solution. Dilute to mark, mix well and allow to stand for 10 min. Measure the absorbance using spectrophotometer at 522 nm wave length as described for calibration. Use the same cell size as the one used for calibration and use blank test solution as reference.

A-7.4.3 *Blank Test*

Prepare a blank test solution using same procedure as used for determination of iron but excluding cobalt acetate.

A-7.5 Calculation and Expression of Results

By means of the calibration graph determine the concentration of iron in micrograms corresponding to the absorbance value of the test solution.

A-8 TEST FOR CHLORIDE

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — Two of 50 ml capacity.

A-8.2 Reagents

A-8.2.1 *Concentrated Nitric Acid* — *See* IS 264

A-8.2.2 *Silver Nitrate Solution* — 1.7 percent (*m/v)* in water.

A-8.2.3 *Standard Chloride Solution* — Dissolve 0.165 g of sodium chloride (NaCl) in water, and dilute with water up to the mark in a 1 000 ml volumetric flask. Dilute 10 ml of this solution in l00 ml volumetric flask. One millilitre of this diluted solution is equivalent to 0.01 mg of chloride (as Cl).

A-8.3 Preparation of the Test Solution — Take 1 g of the sample and dissolve it in 100 ml water. Transfer it to a 250-ml volumetric flask and dilute with water up to the mark.

A-8.4 Procedure

Transfer 25 ml aliquot of the prepared sample solution to a 250 ml volumetric flask and dilute to 250 ml. Take 25 ml of the diluted solution in a 50-ml Nessler cylinder, add 1 ml of concentrated nitric acid followed by 1 ml of silver nitrate solution. Make up to the mark with water, mix and allow the turbidity to develop. Carry out a control test in another Nessler cylinder using 1 ml of standard chloride solution and the same quantities of other reagents in the same total volume as with the material and compare the turbidity produced in two cylinders against a black background.

A-8.4.1 The material shall be taken as having satisfied the requirement of the test if turbidity produced with the material is not greater than that produced in the control test.

A-9 DETERMINATION OF COPPER

A-9.1 Method A

A-9.1.1 *Outline of Method*

Copper is determined calorimetrically by measurement of the colour produced by the material with zinc dibenzyl-dithiocarbomate. The value is read off from a standard calibration graph.

A-9.1.2 *Apparatus*

A-9.1.2.1 *Separating funnels* — 100 ml capacity. Rinse the funnel with dilute hydrochloric acid and with water before use. Allow to drain, then dry the stems with pure absorbent cotton wool and place a small plug of cotton wool in each stem.

A-9.1.2.2 *Photoelectric Absorptiometer* — with 1 cm cells.

A-9.1.3 *Reagents*

A-9.1.3.1 *Ammonium hydroxide* — 10 N approximately.

A-9.1.3.2 *Dilute hydrochloric acid* — 5 N.

A-9.1.3.3 *Zinc dibenzyldithiocarbomate solution* — 0.04 percent solution in carbon tetrachloride.

A-9.1.3.4 *Standard copper solution* — Weigh 0.491 g of copper sulphate pentahydrate ($CuSO_4$, $5H_2O$) in a weighing bottle. Wash into a 250 m1 volumetric flask. Add 3 ml of 18 N sulphuric acid, dilute to about 100 ml, and swirl until the salt has completely dissolved. Dilute to the mark and mix well. This is the stock solution, one millilitre of which contains 0.5 mg of copper (Cu). Take 3 ml of 18 N sulphuric add and about 200 ml of water in a 1 000 ml volumetric flask. Carefully pipette 5 ml of the stock copper solution into the flask. Dilute to the mark and mix well. One millilitre of this solution contains 2.5 µg of copper (Cu).

A-9.1.4 *Procedure*

A-9.1.4.1 Weigh accurately 5 g of the material into a 250 m1 beaker. Take a smaller mass if the copper is expected to exceed 3 parts per million. Add 30 ml of dilute hydrochloric acid and boil gently for 15 min. Place 30 ml of dilute hydrochloric acid in a second beaker as blank and continue with both. Allow to cool, then add ammonium hydroxide until the solution is alkaline to litmus paper. Add dilute hydrochloric acid dropwise until the solution is acidic, then add 3 ml in excess.

A-9.1.4.2 Transfer to a 100-m1 separating funnel and, if necessary, dilute to 50 ml. Add 10 ml of zinc dibenzyl dithiocarbomate solution and shake vigorously for at least 2 min. Allow the layers to separate, then unstopper the funnel, and run the lower layer through a plug of cotton wool into a l-cm cell, using, the first few drops to rinse out the cell. Adjust the photometer at zero at 440 μm (blue photocell, plain glass filter) with a cell of zinc dibenzyldithiocarbomate solution in the light path, then measure the sample and the blank. Read off parts per million copper from the standard calibration graph.

A-9.1.4.3 *Standard calibration graph* — Take five 100 m1 separating funnels, and wash thoroughly with hydrochloric acid and then with water. In each funnel place 3 ml of dilute hydrochloric acid (5 N). Using pipettes or a micro burette, measure respectively: 0, 2, 5, 10 and 15 ml of standard copper solution. Dilute to 50 ml. Subtract a constant from each observed colorimeter reading so that the blank solution with no added copper corresponds to a reading of 0. Convert volumes of standard copper solution to micrograms of copper by multiplying by 2.5. Plot a graph with micrograms as ordinates and adjusted colorimeter readings as abscissae. Draw a line through the points and through the origin. Label the graph with particulars of cells and filters.

A-9.1.5 *Calculation*

$$\text{Copper (Cu), parts per million} = \frac{M_1}{M}$$

where

M_1 = mass in micrograms, of copper read off from the graph; and

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

A-9.2 Method B

A-9.2.1 *Principle*

Copper present in the sample is reduced with the help of ascorbic acid and by addition of 2,2’-biquinolyl a violet coloured complex is formed.

A-9.2.2 *Reagents*

Sodium sulphate, hydrochloric acid solution, amyl alcohol, (+) tartaric acid, sodium hydroxide, L-ascorbic acid (100 g/l) , 2-2'biquinolyl (0.5 g/l), bromine water-saturated solution, copper (0.1 g/l), standard solution.

Copper standard solution (0.1 g/l), indicator paper (narrow range) *p*H 5.5 to *p*H 7.0, methyl orange indicator (0.5 g/l) aqueous solution.

A-9.2.3 *Apparatus*

Spectrophotometer, photoelectric absorptiometer with maximum transmission at a wavelength of 545 nm, optical cells, 4 cm optical path.

A-9.2.4 *Procedure*

A-9.2.4.1 *Test portion*

Weigh about 10g of the sample to the nearest 0.1 g.

A-9.2.4.2 *Blank test*

Carry a test with a blank as the determination follows the procedure.

A-9.2.4.3 *Preparation of the calibration graph*

Take six 500 ml seperatory funnel and introduce the standard copper solutions.

Standard Copper Solutions

Standard Copper Solutions (ml)	Mass of Copper in µg
0	0
2	20
4	40
6	60
8	80
10	100

To all the funnels add 400 ml water and 2 ml of tartaric acid. *p*H of the solution is adjusted around 6.0 using sodium hydroxide. Add about 2 ml of ascorbic acid and mix the contents thoroughly. Allow the mixture to stand for 5 min. Add 2,2'-biquinolyl solution (10 ml) and mix well. Now try to extract the copper complex using two 20 ml of amyl alcohol portions. Transfer the extract to 100 ml beaker. Add sodium sulphate (2 g) to extract and remove water traces by stirring thoroughly.

In the next step, filter dry extract into 50 ml one mark volumetric flask. Wash residual sodium sulphate using 2 ml two portions of amyl alcohol. Dilute the mark using amyl alcohol by transferring to volumetric flask and mix well.

Spectrophotometric Measurements

Measurements are made using spectrophotometer or photoelectric absorptiometer at a wavelength of 545 nm. Measurements are made using filters and adjusting the instruments properly to zero absorbance using amyl alcohol as blank solution.

A-9.2.4.4 *Determination*

Preparing Test solution

To a 400 ml beaker, transfer the test portion. Add 1 drop of methyl orange and 100 ml water. Using Hydrochloric acid, neutralize the solution adding 5 ml in excess. Add bromine water (10 ml) in excess and boil the solution till it is free from bromine. Allow the solution to cool. To a 500 ml seperatory funnel fitted with stopper, transfer the contents of the beaker and add 1 ml of HCl solution to it. After the sample preparation and colour development, photometric measurements are made on the test samples using blank test solution (by adjusting the instrument to zero absorbance).

A-9.2.4.5 *Calibration graph*

The graph for the above standard solutions is plotted between absorbance *versus* concentration. Take the absorbance of all the prepared solutions and then deduce the concentration of copper in the unknown solution using a calibration graph.

A-9.3 Expression of Results

Determine the concentration of copper corresponding to the absorbance value. The copper content expressed as mg/kg is given by:

$$\frac{m_1 - m_2}{1000} \times \frac{1000}{m_o} = \frac{m_1 - m_2}{m_o}$$

m_o = the mass of the test sample;
m_1 = the mass of copper in test solution (in µg); and
m_2 = copper in blank solution.

A-10 DETERMINATION OF SODIUM

A-10.1 Apparatus

EA-10.1.1 *Flame Photometer* — quipped with interference filter.

NOTE — Sodium filter to be used should be of preselected or metal filter type, since ordinary sodium filters (for example, gelatine fillers) have only 1 percent selectivity and work properly when the ratio of Na : K is 100 : 1, whereas in potassium carbonate and potassium hydroxide, the ratio is exactly reverse. Hence results with gelatine filter are erratic.

A-10.2 Reagent

A-10.2.1 *Standard Sodium Solution*

Weigh exactly 2.542 g of sodium chloride, dissolve in deionized water and dilute to 1 000 ml in a measuring flask. Dilute 10 ml of this solution to 1 000 ml. 1 ml of the diluted solution contains 0.01 mg of sodium (as Na).

A-10.3 Sample Solution

Dissolve exactly 10 g of the pure grade of material in deionized water and dilute to 1 000 ml in a volumetric flask. Dilute 100 ml of this solution to 1 000 ml in a volumetric flask. Use this solution for galvanometer readings. If needed, further dilute the solution.

A-10.4 Calibration Graph

Take 10, 20, 40, 60 and 80 ml of standard sodium solution in different measuring flasks and dilute to 100 ml with deionized water. Each flask now contains 0.1, 0.2, 0.4, 0.6 and 0.8 mg of sodium per 100 ml. Use these diluted solutions to obtain corresponding galvanometer readings as given in the procedure (**A-9.5.2**) and plot the concentration against galvanometer reading in a rectangular coordination graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.1 to 1.0 mg of sodium per 100 ml.

A-10.5 Procedure

A-10.5.1 Use a flame photometer equipped with atomizer, burner, optical selective device consisting of reflectors, lenses and diaphragms; and measuring instrument consisting of a photocell, an amplifier and a sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element.

A-10.5.2 Insert the sodium filter corresponding to wavelength 767 nm. Light the burner fed by illuminating gas (laboratory gas) and adjust the specified air pressure between 0.5 to 0.7 kg/cm_2 and maintain the control knob. First spray deionised water and adjust the pointer to zero in galvanometer scale by adjustment knob. Then spray the standard sodium solution and adjust the deflection to maximum (100) by using sensitivity control knob. Again spray deionised water to see that pointer comes to zero; then spray standard solution to indicate 100. Repeat till deionized water reads zero and standard solution reads 100 with the same adjustment during both the operations. Reading zero by deionized water and with the same adjustment 100 by standard solution, indicates that the instrument is now ready for measurement.

A-10.5.3 Without altering the earlier adjustment of the instrument, spray various diluted solutions prepared in **A-9.3** and obtain a calibration graph in the range of 0.1 to 1.0 mg sodium per 100 ml. After washing with deionised water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of sodium (say A).

A-10.6 Calculation

$$\text{Sodium (as Na), percent by mass} = \frac{10A}{M}$$

where

A = concentration of sodium in mg, per 100 ml corresponding to the galvanometer reading (*see* **A-10.5.3**), and

M = mass in g, of the material taken for preparing the sample solution.

A-11 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-11.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the pH of the column. Similarly in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

A-11.2 Equipments

Anion Guard Column : A protector of the separator column.

Anion Separator Column : Suitable for selective separation of ions under analysis.

Anion Suppressor Device : Anion micromembrane suppressor is used to analyse the data.

Detector : Conductivity detector.

Software : Software suitable for control of various operating parameters, receiving inputs and analysis of all data

Sample loop of 100 µl, 200 µl, 500 µl or 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

A-11.3 Reagents

Glass or polyethylene sample bottles.

Distilled water or deionized water free from the anions of interest.

Eluent: 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.2856 g of sodium bicarbonate and 0.3816g of sodium carbonate is dissolved in 2L of water.

Micromembrane suppressor solution: (0.025 N of sulphuric acid)-Dilute 2.8 ml of conc. Sulphuric acid in 4 L of water

A-11.4 Standard Solutions

Chloride: Dissolve NaCl, 1.6485 g in 1 L of reagent water

Sulphate: Dissolve 1.81 g of potassium sulphate in 1 L of reagent water

A-11.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1-1.0 mL injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-11.6 Procedure

Dissolve between 1 to 5 grams sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1-1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyse in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used.

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 μ membrane. This membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-11.7 Data analysis and Calculations

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor. Report results in mg/L or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

A-12 DETERMINATION OF SODIUM, LEAD AND IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-12.1 The solution under analysis is passed with the help of a peristaltic pump through a nebulizer inside a spray chamber. The aerosol formed is aspirated in the form of argon plasma where the atoms are excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and wavelength/intensity of emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

A-12.2 Reagents and Solutions

A-12.2.1 *Magnesium Nitrate*, 6.67 percent -w/v: Add 66.7 g of magnesium nitrate hexahydrate in 1 000 ml of deionized water and mix well.

A-12.2.2 *HCl Solution* : Dilute 83.0 ml conc. HCl to 1 000 ml deionized water.

A-12.2.3 *50 Percent Nitric Acid* : Dilute 500 ml of Nitric Acid in 500 ml of deionized water.

A-12.3 Standards

A-12.3.1 Yttrium Internal Standard (to infuse in the instrument T junction), 2 µg/ml-Prepare 1 000 µg/ml of Yttrium stock standard. Transfer 2 ml of this standard stock solution in 1 L volumetric flask and dilute the solution using 1N HCl.

A-12.3.2 Yttrium Working internal standard: Pipette out 25 ml of 1 000 µg/ml Yttrium stock standard into 50 ml volumetric flask and dilute using 1N HCl.

If Yttrium Internal Standard is used to infuse in the instrument, then there is no need to add Yttrium during the preparation of calibration standards.

Sodium-Calibration standard 0 µg/ml - Add 0 µl of 10 000 µg/ml sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium- Calibration standard 6 µg/ml - Add 30 µl of 10 000 µg/ml of sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium-Calibration standard 12 µg/ml- Add 60 µl of 10 000 µg/ml of sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium-Calibration standard 24 µg/ml- Add 120 µl of 10000 µg/ml of sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium-Caibration standard 48 µg/ml- Add 240 µl of 10000 µg/ml of sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Similar calibration standard solutions can be prepared for iron and lead analysis.

A-12.4 Sample Preparation

Process the sample until homogeneous.

A-12.5 Procedure

Take sample in a platinum crucible. Add magnesium nitrate (2 ml) to the sample and gently swirl it. Place the sample in a cool muffle furnace with temperature less than 80 deg celcius. Set the furnace program as:

Step 1 Ramp = 3°C/min Level = 100°C Dwell = 360 min

Step 2 Ramp = 3°C/min Level = 150°C Dwell = 60 min

Step 3 Ramp = 3°C/min Level = 500°C Dwell = 480 min

Step 4 Ramp = end

Else, heat the sample in the furnace gradually as per the above steps by controlling the sample through the thermostat. The sample must not be heated so rapidly that it ignites. Remove the sample and cool the furnace at room temperature. Add 2 ml 50 percent aqueous HNO_3 and wash with it the sides of the crucible. Dissolve all the ash and transfer in the beaker. Remove excess of acid by slow heating of the sample on a hot plate. Make sure that the sample is free from any black precipitate. If any precipitate is found, again transfer the dry mass of the beaker into the crucible using little water and evaporate the water and heat at 500-550°C for 1 h. Again repeat this procedure till the sample becomes free from black precipitate. Remove the sample from furnace and cool it to room temperature. Add HCl (5 ml, 1 N) and dissolve the ash/mass. Transfer the above solution to 50 ml graduated flask and make up the final volume to 50 ml using 1 N HCl. Fortify the sample with 200 µl of Yttrium working standard and mix it. This step is omitted if T junction is used on the instrument. Analyse the sample using ICP/OES

A-12.6 Instrument Settings

Set up the instrument as per the manufacturer's instructions. Relative to the internal standard concentration, the quantification of the ions is done by the system software. Calibrate the instrument using ICP reagent blank and the calibration standard. Monitor the wavelengths for the respective metal ions.

Na = 589.592 nm, Pb = 220nm, Fe = 238.2 nm

A-12.7 Manual Calculation

Use linear regression analysis to determine a standard curve (emission *versus* concentration)

$$\text{ppm (analyzed)} = \frac{\mu\text{g/ml (evaluated from calibration curve)} \times \text{ml (final volume)}}{\text{g (sample weight)}}$$

A-13 ATOMIC ABSORPTION SPECTROSCOPY FOR IRON, COBALT, LEAD AND COPPER DETECTION

A-13.1 Solutions to be Prepared

A-13.1.1 Iron, Fe (III): Dissolve 0.500 g of iron wire in 20 ml of 1:1 HCl and 5 ml of concentrated nitric acid to oxidize all the iron. Dilute the contents to 1 L with water.

A-13.1.2 Cobalt, Co (III): 1.0 g of cobalt metal salt is heated and dissolve it with 20 ml of 1:1 Nitric acid and dilute it with water to 1 000 ml after it has cooled.

A-13.1.3 Lead, Pb: 1 g of Salt of lead is heated and dissolved with 30 ml of nitric acid (1:1) and dilute it with water to 1000 ml accurately.

A-13.1.4 Copper, Cu : 1.0 g of copper salt is heated and dissolved with 30 ml of nitric acid (1:1) and is diluted to 1 000 ml accurately. Add water after it has cooled.

A-13.2 Procedure

A-13.2.1 *Preparing Standard Solution*

Add 250 ml of deionized water in 400 ml of dry beakers. Pipette out 0, 0.5, 1.0, 1.5, 2.0 and 2.5 ml of the stock solution of the corresponding metal salt and transfer to these beakers. Mix in deionized water and shake thoroughly.

A-13.2.2 *Setting up the Instrument*

Set up the Atomic Absorption instrument as per the operating instructions.

A-13.2.3 *Measurement of Absoption Intensity*

Make measurements for the set of prepared standards and unknown samples. Dilute the unknown samples if the absorbance measured is too large i.e. outside standards range. Make measurements for both the set of standards and unknown.

In order to calculate the final results, the sample dilution factor should be taken into account. Plot standard absorbance versus concentration for each analyte and measure the absorbance of the unknown solutions under the same conditions.

A-14 DETERMINATION OF SODIUM, CALCIUM AND MAGNESIUM SEPARATELY

A-14.1 AES Measurements of Magnesium and Calcium

A-14.1.1 *Magnesium Stock Solution*

Weigh 0.101 g of dry magnesium oxide and rinse in a 1L volumetric flask with deionized water. Add 6 M HCI (a few mL) then dilute using deionized water upto the mark. Mix the contents thoroughly.

A-14.1.2 *Calcium Stock Solution*

Weigh 0.252 g of standard calcium carbonate ($CaCO_3$, FW = 100.087). Rinse in 100 mL flask using deionized water. Add 6 M HCI (a few mL) and dilute using deionized water.

A-14.1.3 *Sodium Stock Solution*

Accurately weigh 0.510 g of sodium chloride and transfer it to 200 ml volumetric flask. Dissolve in deionized water and mix thoroughly.

A-14.2 Preparing Standard Solution

Add 100 ml of deionized water in 250 ml of graduated flasks. Pipette out 0, 0.5, 1.0, 1.5, 2.0 and 2.5 ml of the MgO stock solution and transfer to these flasks. Mix and make up the volume to 250 ml water and shake thoroughly.

As for MgO solution, in the similar way, standard calcium and sodium solution can be prepared.

A-14.3 Setting up the Instrument

Set up the instrument as per the operating instructions. The measurements be made with the long axis of the flame both parallel and perpendicular to the light path. After the measurements are made in one flame position, turn the burner head 90 degrees and continue to make the measurements.

A-14.4 Measurement of Emission Intensity

By aspiring deionized water, set the instrument to read zero emission of respective characteristic light. Then measure the emission intensity of all the standards and the samples. Make the measurements with the flame in parallel as well as perpendicular position depending on concentration.

Tabulate and plot emission *versus* concentration graph for sodium, calcium and magnesium. Derive the calibration equation for both burner orientations. Use polynomial equation to fit the data if calibration curves are nonlinear. By comparing the data of emitted light intensity of sample, calculate metal ion concentration of the unknown samples. If a linear calibration curve is used, report the standard error in the calibrated results for the unknown.

It is likely that the calibration curve with the flame in parallel position will have larger degree of non-linearity and high sensitivity in comparison to the perpendicular flame position.

ANNEX B

SAMPLING OF COBALT ACETATE

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that an ullage of about 5 percent is left after pouring in the sample.

B-1.7 Each sample container shall be sealed air tight after filling, and marked with full details of sampling and the date of sampling.

B-2 SCALE OF SAMPLING

B-2.l Lot

All 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.

B-2.2 Samples shall be tested from each lot separately for judging the conformity of the material to the requirements or the specification. For this purpose 5 containers shall be selected at random from each lot.

> NOTE — If the number of containers in the lot is five or less, the number of containers to be selected and the criteria for conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

B-2.3 The containers shall be selected at random and to ensure randomness of selection, the following procedure is recommended for use.

Starting from any container in the lot, count them 1,2,……………… up to r and so on, where r is the integral part of N/5 (N being the number of containers in the lot). Every rth container thus counted shall be withdrawn to constitute a sample till the required number of 5 containers is obtained.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 Sampling Tube-The sampling tube shall be made of glass and shall be 20 to 40 mm in diameter and 350 to 750 mm in length. The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing sample, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

B-3.1.1 For small containers, the size of the sampling tube may be altered suitably.

B-3.2 From each of the containers selected according to **B-2.3** a small representative portion of the material, about 200 ml, shall be taken out with the help of the sampling tube after thoroughly stirring the acid with the help of a glass rod.

B-3.3 Out of these portions, a small but equal quantity of the material shall be taken out and thoroughly mixed to form a composite sample not less than 600 ml. 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-3.4 The remaining portion of the material from each container shall be divided into 3 equal parts, each forming an individual sample. One set of individual samples representing the 5 containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.5 All the individual and composite samples shall be transferred to separate bottles. These bottles shall be sealed and labelled with full identification particulars.

B-3.6 The referee samples consisting of composite sample and a set of 5 individual samples shall bear the seals of the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier to be used in the case of a dispute between the two.

B-4 NUMBER OF TESTS

Tests for the determination of cobalt acetate shall be performed on each of the 5 individual samples.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For individual samples from the 5 test results for cobalt acetate, the mean (×) and range (R) of test results shall be computed (range being defined as the difference between the maximum and minimum values of test results).

B-5.1.1 The lot shall be declared as conforming to the requirements of cobalt acetate if the value of the expression (x-0.6 R) as calculated from the relevant test results is greater than or equal to the minimum value specified in Table 1.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE DEVELOPMENT OF THIS STANDARD

Inorganic Chemicals Sectional Committee, CHD 01

Organization	*Representative(s)*
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Member Secretary

Sagar Singh
Scientist 'C' (CHD), BIS

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