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SPECIFICATION FOR ALUMINIUM SULPHATE FOR POTABLE WATER
SUPPLIES

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This draft should not be regarded or used as a Sri Lanka Standard.

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Comments to be sent to: SRI LANKA STANDARDS INSTITUTION, 17 VICTORIA PLACE,
ELVITIGALA MAWATHA, COLOMBO 08.

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SPECIFICATION FOR ALUMINIUM SULPHATE FOR POTABLE WATER
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17, Victoria Place,
Elvitigala Mawatha,
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SRI LANKA

Draft Sri Lanka Standard
SPECIFICATION FOR ALUMINIUM SULPHATE FOR POTABLE WATER
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FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of Sri Lanka Standards Institution on

Aluminium sulphate (commonly called as alum) is a chemical compound. It is soluble in water and mainly used as a flocculating agent in the purification of drinking water.

All Standard values given in this Specification are in SI units.

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 an analysis, shall be rounded off in accordance with **SLS 102**. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this Standard.

In the preparation of this Specification, the assistance obtained from the following publications are gratefully acknowledged:

IS	260	Aluminium sulphate, non- ferric Specification (Second Revision)
JIS K	1450	Aluminium sulphate foe water works

1 SCOPE

- 1.1** This Specification prescribes the requirements, methods of sampling and test for Aluminium sulphate used in potable water supplies.
- 1.2** This Specification does not cover Aluminium sulphate (Technical grade).

2 REFERENCE

ASTM E	300	Standard practice for sampling industrial chemicals
SLS	102	Presentation of numerical values
SLS	124	Test sieves
SLS	312	Determination of Arsenic
SLS	428	Random sampling methods
SLS	614	Potable water
SLS	722	Tolerance limits for inland surface waters and used as raw water for public water supply
SLS	894	Bottled drinking water
SLS	1211	Hygienic practice for bottled (packaged) drinking water

3 TYPES

Aluminium sulphate covered under this Specification shall be supplied either in solid (Type 1) or liquid (Type 2) types. Type 1 Aluminium sulphate shall be in lump, powder or flaked (chips) forms.

4 REQUIREMENTS

4.1 General requirements

4.1.1 The colour of Type 1 Aluminium sulphate shall be white and free from visible impurities. The Type 2 Aluminium sulphate shall be colourless to yellowish light brown and transparent.

4.1.2 Supplier shall provide documentary evidence of compliance with the International Standards to the effect that Aluminium sulphate is of food grade quality.

4.2 Other requirements

4.2.1 Particle size of the lump, flaked and powder form Aluminium sulphate shall be less than 40 mm, 25 mm and 5 mm respectively, when tested as prescribed in Appendix B.

4.2.2 Aluminium sulphate shall also conform to the requirements given in Table 1 when tested in accordance with the relevant methods given in Column 5 of the table.

TABLE 1 – Requirements for Aluminium sulphate

SI. No.	Characteristic	Requirement		Method of test
		Solid (Type 1)	Liquid (Type 2)	
(1)	(2)	(3)	(4)	(5)
i)	Water soluble Aluminium sulphate (as Al ₂ O ₃) per cent by mass, min	16.0	8.0	Appendix C
ii)	Water insoluble matter, per cent by mass, max	0.5	-	Appendix D
iii)	pH at 27 ± 2 °C, min	2.8	2.8	Appendix E
iv)	Arsenic (as As) mg/kg, max	4	2	Appendix F
v)	Lead (as Pb) mg/kg, max	10	5	Appendix F
vi)	Manganese (as Mn) mg/kg, max	25	15	Appendix F
vii)	Cadmium (as Cd) mg/kg, max	2.0	1.0	Appendix F
viii)	Mercury (as Hg) mg/kg, max	0.2	0.1	Appendix F
ix)	Chromium (as Cr) mg/kg, max	10	5	Appendix F
x)	Iron (as Fe) mg/kg, max	700	300	Appendix G
xi)	Ammoniacal nitrogen, per cent by mass, max	300	100	Appendix H

5 PACKAGING

Aluminium sulphate shall be supplied in bulk or in packages as agreed to between the purchaser and the supplier. Type 1 Aluminium sulphate shall be packed in 25 kg to 50 kg HDPE bags with inner liner. Type 2 Aluminium sulphate shall be supplied in bulk tanker or in 200 litre drums.

6 MARKING

6.1 Each package shall be marked legibly and indelibly with the following information:

- a) Name of the product;
- b) The manufacturer's and supplier's or distributor's name and address including country of origin;

- c) Registered trade mark if any;
- d) Net mass, in kilograms (kg);
- e) Date of manufacture; and
- f) Batch or code number.

7 METHODS OF TEST

7.1 Tests shall be carried out as prescribed in Appendices **B** to **H** of this Specification.

7.2 During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

APPENDIX A COMPLIANCE OF A LOT

The sampling scheme given in this Appendix shall apply where compliance of a lot to the requirements of this Specification has to be assessed based on statistical sampling and inspection.

A.1 LOT

In any consignment all the packages and containers of the same size, same type and belonging to one batch of manufacture or supply shall constitute a lot.

A.2 GENERAL REQUIREMENTS OF SAMPLING

In drawing, handling, and preparing samples, the following precautions shall be observed.

A.2.1 The samples shall not be taken from broken packages.

A.2.2 The sampling instruments shall be clean and dry when used.

A.2.3 The samples shall be placed in clean, dry and air-tight glass or suitable containers.

A.2.4 The material being sampled, the sampling instruments and the containers for samples shall be protected from adventitious contamination.

A.2.5 The sample containers shall be air-tight after filling and marked with necessary details of sampling. Samples shall be stored in the shade.

A.3 SAMPLING INSTRUMENTS

A.3.1 Shovel or other effective device shall be used for drawing samples from solid bulk.

A.3.2 Hand scoop or other effective device shall be used for drawing samples of powder materials from containers or conveyors.

A.3.3 Sampling tube or other effective device shall be used for sampling liquids in drums and cans. Tube shall be made of either stainless steel or other suitable material. The tube shall be designed so that it will reach to within about 3 mm of the bottom of the container and have a capacity of approximately 0.5 litre or 1.0 litre. A metal tube suitable for sampling 207 litre drums is shown in Figure 1. Two rings, attached to opposite sides of the tubes at the upper end, are convenient for holding it by slipping two fingers through the rings – thus leaving the thumb free to close the opening. An alternative tube sampling apparatus is shown in Figure 2, also designed to reach within 3 mm of the bottom.

A.3.4 Drawing liquid samples from bulk tankers, a suitable sampling bottle, is shown in Figure 3. The diameter of the openings in the bottles shall be 19 mm.

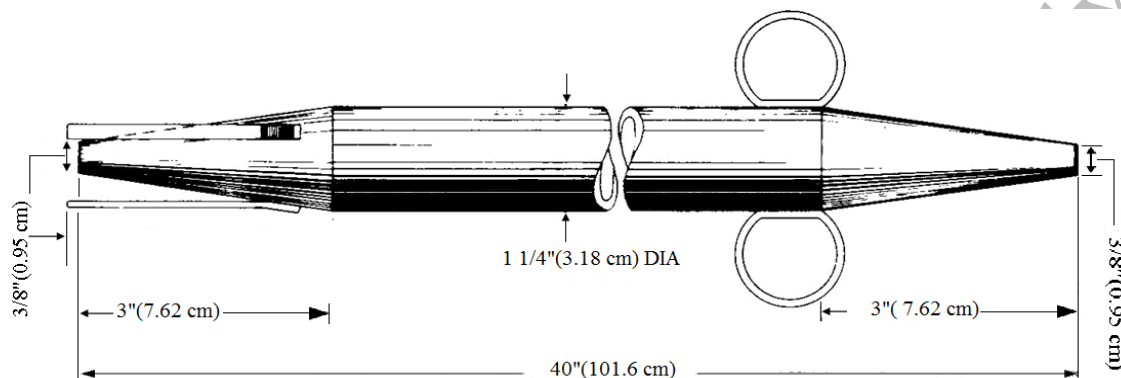


FIGURE 1- Sampling tube

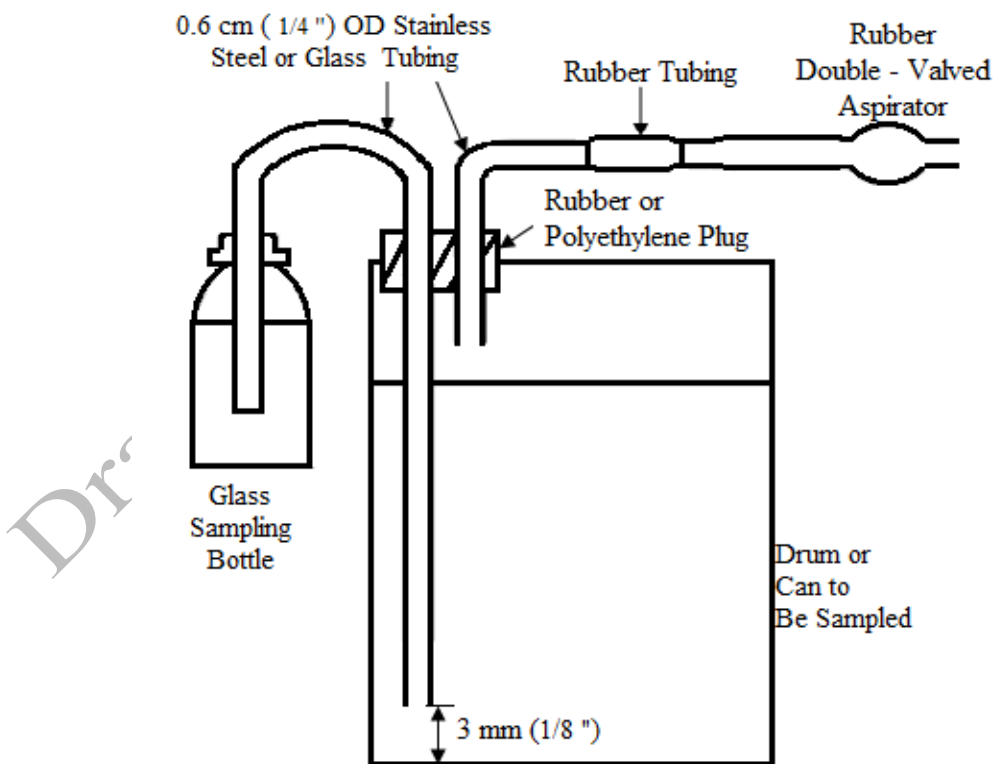


FIGURE 2- Alternative tube sampling assembly

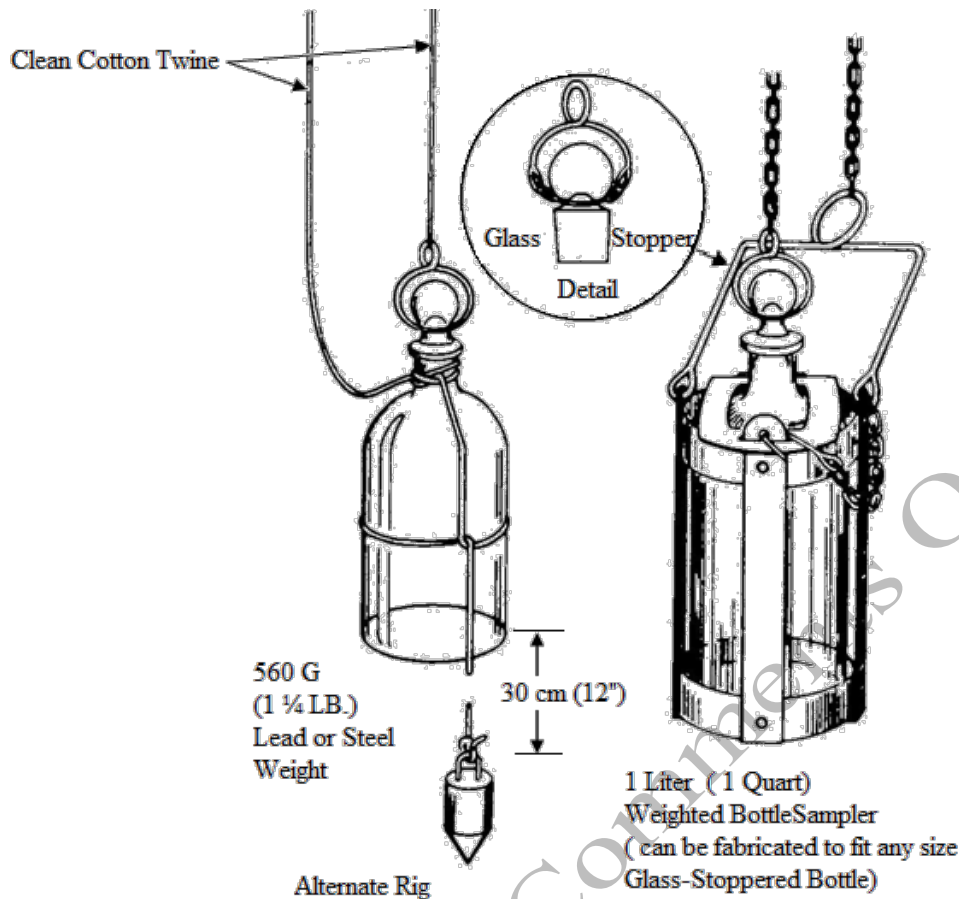


FIGURE 3- Assembly for bottle sampling

A.4 SCALE OF SAMPLING

A.4.1 Samples shall be tested from each lot for ascertaining its conformity of the material to the requirements of this Specification.

A.4.2 Sampling from liquid material supplied in bulk tankers

A.4.2.1 Each tanker shall be sampled separately from the lot.

A.4.2.2 Bottle sampling procedure shall be used to prepare single-tank composite sample/ or compartment-tank composite sample by mixing portions of the upper, middle, and lower samples as prescribed in **A.4.2.3** or portions of all-levels samples as prescribed in **A.4.2.4** during the unloading of the bulk tankers.

A.4.2.3 Single-tank composite sample shall be prepared as a blend of the upper, middle and lower samples (Figure 4). For a tank of uniform cross section, such as an upright cylindrical tank, the blend shall consist of equal parts of the three samples. For a horizontal cylindrical tank, the blend shall consist of the three samples in the proportions shown in Table 2. Drawing the upper, middle, and lower samples through bottle sampling procedure is prescribed in **A.4.2.5**.

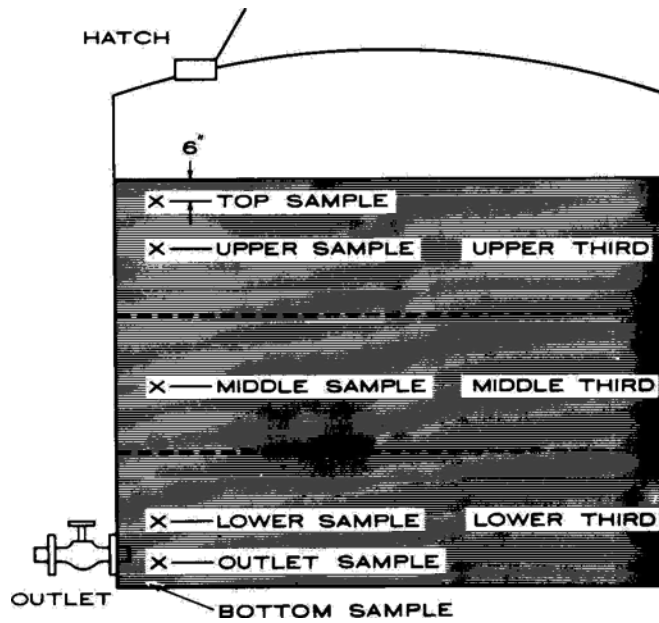


FIGURE 4- Sampling depths

TABLE 2 – Sampling instructions for horizontal cylindrical tanks

SI. No.	Liquid depth percent of diameter	Sampling level, per cent of diameter above bottom			Composite sample proportionate parts of		
		Upper	Middle	Lower	Upper	Middle	Lower
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	100	80	50	20	03	04	03
ii)	90	75	50	20	03	04	03
iii)	80	70	50	20	02	05	03
iv)	70	-	50	20	01	05	04
v)	60	-	50	20	-	05	05
vi)	50	-	40	20	-	04	06
vii)	40	-	-	20	-	-	10
viii)	30	-	-	15	-	-	10
ix)	20	-	-	10	-	-	10
x)	10	-	-	05	-	-	10

A.4.2.4 Compartment-tank composite sample (ship, barge, etc.) shall be prepared as a blend of individual all-levels samples from each compartment, which contains the product

being sampled, in proportion to the volume of material in each compartment. Collecting the all-levels sample through bottle sampling procedure is prescribed in **A.4.2.6**

A.4.2.5 Sampling of upper, middle and lower samples shall be carried out by lowering the weighted, stoppered bottle to the proper depths (Figure 4). Pull out the stopper with a sharp jerk of the twine or chain (spark-proof) attached to the stopper and allows the bottle to fill completely at the selected level, as evidenced by the cessation of air bubbles. When full, raise the bottle, pour off a small amount, and stopper immediately.

A.4.2.6 Sampling of all-levels sample shall be carried out by lowering the weighted, stoppered bottle as near as possible to the draw-off level, pull out the stopper with a sharp jerk of the twine or chain (spark proof) attached to the stopper, and raise the bottle at such a rate that it is about three-fourths full as it emerges from the liquid.

A.4.2.7 The composite sample of liquid material shall be thoroughly mixed and reduced to obtain a test sample. The total sample volume shall equal 2 litre or as agreed.

A.4.3 Sampling from solid materials supplied in packages and liquid materials supplied in drums

A.4.3.1 The number of packages to be selected from a lot shall be in accordance with Table 3.

A.4.3.2 The packages shall be selected at random. In order to ensure randomness of selection, random number tables as given in **SLS 428** shall be used.

A.4.3.3 In case of lump material or flaked (chips) material, each package selected as in **A.4.3.1** shall be emptied and a shovel shall be used to take sufficient quantity of material drawn from various parts of the mass being sampled.

A.4.3.4 In case of powder material, a hand scoop shall be inserted into each package being sampled as in **A.4.3.1**, so that it will take a core of material from substantially the entire length of the package.

A.4.3.5 In case of liquid material supplied in 200 litre drums, a sampling tube shall be inserted into each drum being sampled as in **A.4.3.1**, so that it will take a core of material from substantial the entire volume of the drum. Tube sampling procedure for drums can be performed as described in **A.4.3.6**.

A.4.3.6 Sampling procedure shall be initialized by stand the drum upright and sample from top. If the drum does not have a top bung, place the drum on its side with the bung facing upwards. Close the upper end of the clean, dry sampling tube with the thumb, and lower the tube into the liquid for a depth of about 300 mm. Remove the thumb, allowing the liquid to flow into the tube. Again close the upper end with the thumb and withdraw the tube. Rinse the tube with the liquid by holding it nearly horizontal and turning it so that the liquid comes in contact with the part of the inside surface which will be immersed when the sample is taken. Insert the tube into the liquid again, holding the thumb against the upper end. (If an all-levels sample is desired, insert the tube with the upper end open.) When the tube reaches the bottom, remove the thumb and allow the tube to fill. Replace the thumb, withdraw the tube quickly, and transfer the contents to the sample container.

A.4.3.7 The material obtained from each package shall be thoroughly mixed to make the composite sample and reduced to obtain a test sample.

A.4.3.8 In using the alternative sampling device, the sample shall be pumped directly into the sample bottle by means of double-valve aspirator bulb. Sample at various levels may be obtained by adjusting the depth of the tube in the drum.

**TABLE 3 – Scale of sampling
(For solid materials and liquid supplied in drums)**

Number of packages in the lot (1)	Number of packages to be selected (2)
Up to 50	5
51 to 150	20
151 to 280	32
281 to 500	50
501 to 1200	80
1201 to 3200	125
3201 to 10000	200
10001 to 35000	315
35001 and above	500

A.5 NUMBER OF TESTS

A.5.1 Each bulk tanker sampled as in **A.4.2** or each package selected as in **A.4.3** shall be inspected for marking requirements. This shall be done at the place of sampling.

A.5.2 Each sample selected as in **A.4.2.7** or **A.4.3.7** shall be examined for each relevant requirement given in Clause 4.

A.6 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this Specification, if the following conditions are satisfied.

A.6.1 Each bulk tanker or package inspected for marking as in **A.5.1** satisfies the relevant requirements.

A.6.2 The test results on each sample tested as in **A.5.2** satisfy the relevant requirements.

APPENDIX B DETERMINATION OF PARTICLE SIZE

B.1 APPARATUS

Test sieves, of aperture size, 40 mm, 5 mm, 1 mm conforming to **SLS 124**.

B.2 PROCEDURE

B.2.1 Lump form

Weigh, to the nearest 0.1 g, about 100 g of the sample and place it on the sieve of aperture size 40 mm. Shake the sieve till no more material passes through the sieve.

B.2.2 Flaked form

Weigh, to the nearest 0.1g, about 100 g of the sample and place it on the sieve of aperture size 5 mm. Shake the sieve till no more material passes through the sieve.

B.2.3 Powder form

Weigh, to the nearest 0.1g, about 100 g of the sample and place it on the sieve of aperture size 1 mm. Shake the sieve till no more material passes through the sieve.

APPENDIX C DETERMINATION OF WATER SOLUBLE ALUMINIUM SULFATE

Two methods have been prescribed for the determination of water soluble aluminium sulphate. The method prescribed in **C.1** shall be the reference method and shall be carried out in case of any dispute.

C.1 METHOD 1

C.1.1 Reagents

C.1.1.1 *Concentrated hydrochloric acid, relative density = 1.18*

C.1.1.2 *Concentrated nitric acid, relative density = 1.42*

C.1.1.3 *Methyl red indicator, 1 g of methyl red in 500 ml of 95 per cent ethanol.*

C.1.1.4 *Concentrated ammonium hydroxide, relative density = 0.88*

C.1.1.5 *Ammonium nitrate, Dissolve 2 g in 100 ml of water.*

C.1.2 Procedure

C.1.2.1 Weigh, to the nearest 0.01 g, about 5 g of sample and dissolve in 100 ml of water. Digest on a steam bath for 5 minutes. Stir and filter hot through a tared sintered glass crucible. Wash with hot water until a washing is free of sulphate (approximately 10 separate washings). Retain the residue for the water insoluble matter determination in Appendix D. Transfer the filtrate and washings into a 500-ml volumetric flask, cool to room temperature, and make up to the volume with water. Transfer a 50 ml aliquot to a 400-ml beaker and dilute with 150 ml of water. Add 10 ml of concentrated Hydrochloric acid (C.1.1.1) and a few drops of concentrated Nitric acid (C.1.1.2) and heat to boiling.

C.1.2.2 Add 3 drops of methyl red indicator (C.1.1.3) and a small amount of ashless filter paper pulp. Neutralize with concentrated Ammonium hydroxide solution (C.1.1.4) and add 3 drops in excess. Boil for 2 minutes. Allow the precipitate to settle and filter quantitatively through Whatman No. 41 filter paper (or equivalent). Return the filter paper and contents to the 400-ml beaker, add 10 ml of concentrated Hydrochloric acid, and macerate the paper to a pulp with the aid of a stirring rod. Add 150 ml of hot water and 3 drops of the methyl red indicator and heat to boiling. Neutralize with 1:1 Ammonium hydroxide solution and add 3 drops in excess. Boil for 2 minutes. Allow the precipitate to settle and quantitatively filter through Whatman No. 41 filter paper (or equivalent). Wash the filtrate six times with hot ammonium nitrate solution (C.1.5). Ignite the filter paper and precipitate in a tared crucible at 1200 °C. Cool and weigh. Repeat the process until the difference in mass between two successive weighing does not exceed 1 mg.

C.1.3 Calculation

$$\text{Water soluble aluminium sulfate, as Al}_2\text{O}_3, \text{ per cent by mass} = \frac{m_1 \times 1000}{m_2} - A$$

where,

m_1 is mass, in g, of residue;

m_2 is mass, in g, of sample; and

A is per cent, by mass of iron determined in Appendix G.

C.2 METHOD 2

C.2.1 Reagents

C.2.1.1 0.05 mol/L EDTA solution

Weigh 18.61 g of Dihydrogen disodium ethylenediaminetetraacetate dehydrate, add 1000 ml of water to dissolve it, and store it in an air-tight polyethylene bottle.

C.2.1.2 Sodium acetate buffer solution

Dissolve 272 g of sodium acetate trihydrate in water to make 1000 ml.

C.2.1.3 Xylenol orange solution (1 g/L)

Dissolve 0.1 g of xylenol orange in water to make 100 ml.

C.2.1.4 Aluminium standard solution (1 mg Al/ml)

Weigh 1.000 g of aluminium (purity 99.99 % or more), put it in a 100-ml beaker, cover its upper with a watch glass, add carefully a small quantity of Nitric acid (1:1) and dissolve it by heating. After allowing it to cool transfer whole liquid into a 1000-ml measuring flask and add Nitric acid(1:30) up to the marked line.

C.2.1.5 0.02 mol/L Zinc solution

Weigh 1.308 g of zinc, put it in a 100-ml beaker, add 6 ml to 7 ml of Hydrochloric acid and small quantity of water and dissolve it by heating. Evaporate water on a water bath nearly to dry up, add water to dissolve it, transfer it into a 1000-ml measuring flask and add water up to the marked line.

Take 20 ml of 0.05 mol/L EDTA solution into a 200 ml beaker, add 2 ml of Nitric acid (1:12), carry out the procedures shown in **C.2.1.3** and **C.2.1.4** in below **C.2.2** and the used quantity (ml) of 0.02 mol/l Zinc solution shall be made (V_1). Take 20 ml of Aluminium standard solution and 20 ml of 0.05 mol/L EDTA solution into another 200 ml beaker, add 2 ml of Nitric acid (1:12), cover it with a watch glass, boil it for 1 min, allow it to cool, carry out the procedures shown in **C.2.1.3** and **C.2.1.4** in below **C.2.2** and the used quantity in millilitre of 0.02 mol/L Zinc solution shall be made (V_2).

Calculate amount of Aluminium, equivalent to 1 ml of 0.02 mol/L Zinc solution, according to the following formula:

$$W = \frac{0.001 \times 20}{V_1 - V_2}$$

where,

W is amount, in g, of Aluminium equivalent to 1 ml of 0.02 mol/L Zinc solution.

C.2.2 Procedure

C.2.2.1 Weigh sample by about 5 g of solid or about 10 g of liquid to the nearest 1 mg, put it in a 200-ml beaker, and add about 100 ml of water to dissolve it. Filter it if necessary, transfer it into a 500-ml measuring flask, and add water up to the marked line.

C.2.2.2 Take 20 ml of this solution into a 200-ml Erlenmeyer flask, add 20 ml of 0.05 mol/L EDTA solution, and after boiling for 1 minute, allow it to cool.

C.2.2.3 Add about 10 ml of Sodium acetate buffer solution and 2 drops to 5 drops of xylenol orange solution (1 g/L).

C.2.2.4 Titrate it with 0.02 mol/L Zinc solution, and make it an end point when color of the

solution turns faint red.

C.2.2.5 Take 20 ml of 0.05 mol/L EDTA solution, transfer it into another 200-ml Erlenmeyer flask, add about 20 ml of water and 2 ml of Nitric acid (1:12), and thereafter carry out procedures as shown in **C.2.1.3** and **C.2.1.4**.

C.2.3 Calculation

$$\text{Concentration of Aluminium oxide, Per cent by mass} = \frac{0.01 \times 20}{V_1 - V_2}$$

where,

W is amount, in g, of Aluminium equivalent to 1 ml of 0.02 mol/L Zinc solution;

V_3 is volume, in ml, of 0.02 mol/L Zinc solution consumed at **(C.2.2.5)** ;

V_4 is volume, in ml, of 0.02 mol/L Zinc solution consumed for sample solution;

C is concentration. In ml, of iron obtained at Appendix **G**;

m is mass, in g, of sample;

1.8895 is conversion factor from 1g of Aluminium to Aluminium oxide; and

0.9129 is conversion factor from iron to Aluminium.

APPENDIX D DETERMINATION OF WATER INSOLUBLE MATTER

D.1 PROCEDURE

Dry the sintered glass crucible and residue retained from Appendix **C**. Cool and weigh. Repeat the process until the difference in mass between two successive weightings does not exceed 1 mg.

D.2 CALCULATION

$$\text{Water insoluble matter, per cent by mass} = \frac{m_1}{m_2} \times 100$$

where,

m_1 is mass, in g, of insoluble matter; and

m_2 is mass, in g, of sample in Appendix **C**

APPENDIX E DETERMINATION OF PH

E.1 PROCEDURE

Weigh 5.0 ± 0.1 g of the solid material or 2.0 g liquid material and dissolve in 100 ml of freshly boiled and cooled distilled water. Measure the pH of the solution at 27 ± 2 °C by a suitable pH meter, using a glass electrode.

APPENDIX F DETERMINATION OF HEAVY METALS

Atomic Absorption Spectroscopy (AAS) methodology shall be used for determination of heavy metals.

APPENDIX G DETERMINATION OF IRON

Two methods have been prescribed for the determination of Iron. The method prescribed in G.1 shall be the reference method and shall be carried out in case of any dispute.

G.1 METHOD 1 (BIPYRIDAL METHOD)

G.1.1 Reagents

G.1.1.1 *Concentrated sulfuric acid, relative density = 1.84*

G.1.1.2 *Phosphoric acid, 25 per cent (V/V) solution, prepared by using concentrated phosphoric acid, relative density = 1.69*

G.1.1.3 *Sodium diphenylamine sulfonate indicator, 2 g/L*

G.1.1.4 *Potassium dichromate, standard volumetric solution, $C(K_2Cr_2O_7) = 0.017$ mol/L*

G.2 PROCEDURE

G.2.1 Weigh, to the nearest milligram, about 10 g of the sample and dissolve in 100 ml of hot water. After stirring for a few minutes, digest on a hotplate for 30 minutes just below the boiling point. Filter through a Whatman No. 42 filter paper (or equivalent). Wash with hot water until the washing is free of sulphate (approximately 10 separate washings). Collect the filtrate and washings into a 250-ml Erlenmeyer flask and close with a stopper fitted with a bunsen valve. Add approximately 3 g of granulated Zinc and 8 ml to 10 ml of concentrated Sulphuric acid, (G.1.1.1). After the Zinc has dissolved, boil the solution for 2 to 3 minutes. Cool under running tap water.

G.2.2 Add 40 ml of Phosphoric acid (G.1.1.2) and 4 drops of Sodium diphenylamine sulfonate indicator (G.1.1.3). Titrate slowly with Potassium dichromate solution (G.1.1.4), while stirring constantly, until the colour of the solution changes from green to grey-green.

Continue the titration until the first tinge of purple or violet-blue appears. Run a blank determination.

G.3 CALCULATION

$$\text{Iron, as Fe}_2\text{O}_3, \text{ per cent by mass} = \frac{47.91 C (V_1 - V_2)}{m}$$

where,

C is concentration, in mol/l, of the potassium dichromate solution used;

V_1 is volume, in ml, of potassium dichromate solution required for the sample;

V_2 is volume, in ml, of potassium dichromate solution required for the blank; and

m is mass, in g, of the sample.

G.2 METHOD 2 (AAS METHOD)

Atomic Absorption Spectroscopy (AAS) methodology shall be used for determination of iron.

APPENDIX H DETERMINATION OF AMMONIACAL NITROGEN

H.1 REAGENTS

H.1.1 Sodium carbonate solution (30 g/L)

Dissolve 3 g of Sodium carbonate (anhydride) in water to make 100 ml solution.

H.1.2 Sodium hydroxide solution (10 g /L)

Dissolve 1 g of Sodium hydroxide in water to make 100 ml solution.

H.1.3 Sodium hypochlorite solution (1 g/ L)

Dissolve 100/ Concentration of available chlorine (ml) of sodium hypochlorite and 15 g of Sodium hydroxide in water to make 1000 ml solution.

H.1.4 EDTA – Sodium hydroxide mixed solution

Dissolve 0.93 g of Disodium ethylenediaminetetraacetate dihydrate in Sodium hydroxide solution (40 g/L) to make 250 ml solution.

H.1.5 1 - Naphthol solution

Dissolve 1.6 g of 1– naphthol in acetone– ethyl alcohol solution (15:85) to make 100 ml solution

H.1.6 Ammonical nitrogen standard stock solution (0.1 mgN /ml)

Keep Ammonium chloride in a desiccator (keeping manganese perchlorate) for at least 16 h, Weigh its 0.382 g, put it in a 100 ml beaker and dissolve it in a small quantity of water. transfer it into a 1000 ml measuring flask, and add water up to the marked line.

H.1.7 Ammonical nitrogen standard solution (5 µgN/ml).

Take 50 ml of Ammoniacal – nitrogen standard stock solution (0.1 mgN/ml), transfer it into a 1000 ml measuring flask, and add water up to the marked line. Prepare this solution when it is used.

H.2 PROCEDURE

H.2.1 Weigh to the nearest 1 mg, about 5 g of the solid sample or 10 g of liquid sample, put it in a 200-ml beaker and add about 100 ml of water to dissolve it. Transfer it in to the 500-ml measuring flask and add water up to the marked line. Pipette 25 ml of this solution into a 100-ml measuring cylinder with a stopper, Add 5 ml of sodium carbonate solution (**H.1.1.1**) and add water up to the marked line.

H.2.2 Gently shake the measuring cylinder and stand to precipitate Hydroxide. Take 50 ml of the supernatant, transfer liquid in to a 100 ml color caparison tube with a ground stopper. Add sodium hydroxide solution (**H.1.1.2**) to control its pH and make its pH about 11 (See **Note 1**).

H.2.3 Add 1 ml of Sodium hypochlorite solution (available chlorine 1 g/l) (**H.1.1.3**) mix thoroughly after 2 minutes and within 5 minutes add 1ml of EDTA sodium hydroxide mixed solution and mix it again. After 1 minute and within 5 minutes (see **Note 2**) add 5 ml of 1 – naphthol solution, mix well and add water upto the marked line and stand for 15 minutes at 25 °C to 30 °C .

H.2.4 Take its portion into a 10 mm absorption cell, and measure its absorbance at wavelength of 720 nm.

H.2.5 Preparation of working curve

Transfer 0, 1 up to 7 ml of Ammonical nitrogen Standard solution (5 µgN/ml) into 100 ml measuring flasks and add water to make 50 ml solution. Then carry out the procedure described in (**H.1.2.3**) and (**H.1.2.4**) and plot the working curve making use of the relation between the quantity of Ammonical nitrogen and the absorbance.

NOTES

- 1) *The blank for working curve and standard solution have alow buffer action, so pH adjustment is not needed.*
- 2) *The time control needed when reagents are added shall be carried out accurately, including working curve preparation.*

H.3 Calculation

On the working curve prepared at (H.1.3) , find the amount (µg) of Ammonicalnitrogen which is equivalent to absorbance measured at (H.1.2.4) and calculate the concentration (wt ppm) of Ammonical nitrogen in the sample according to the following formula:

$$C = \frac{W}{m \times \frac{25}{500} \times \frac{50}{100}}$$

where,

C is concentration. in ppm, of Ammonical nitrogen in sample;

W is amount, in µg, of Ammonical nitrogen and

m is mass, in g, of the sample.

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