

**SRI LANKA STANDARD 701: 2017**  
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**SPECIFICATION FOR ALUMINIUM SULPHATE**  
**(TECHNICAL GRADE)**  
*(First Revision)*

**SRI LANKA STANDARD INSTITUTION**



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**SLS 701: 2017**

**Gr. 8**

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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 2017-12-04.

This Standard was first published in 1985 covering the “Technical grade” of Aluminium sulphate used in industries and for the treatment of potable water. In this Revision Aluminium sulphate used for the treatment of potable water has been excluded from the scope. This Revision introduces two types of Aluminium sulphate, limits and methods to determine particle size, water soluble Aluminium sulphate, Ammonical nitrogen and Iron content.

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:

ASTM E 300-03: 2017	Standard practice for sampling industrial chemicals
IS 260: 2001	Aluminium sulphate, non- ferric Specification (Second Revision)
JIS K 1450: 1996	Aluminium sulphate for water works
SABS 1241: 2010	Specification- Aluminium sulphate

## 1 SCOPE

**1.1** This Specification prescribes the requirements, method of sampling and test for Aluminium sulphate, suitable for use in industries.

**1.2** This Specification does not cover Aluminium sulphate used for purification of drinking water supplies.

## 2 REFERENCE

SLS 102	Rules for rounding off numerical values
SLS 124	Test sieves
SLS 312	Determination of Arsenic
SLS 428	Random sampling methods

### 3 TYPES

Aluminium sulphate covered under this Specification shall be of the following types:

- a) Ferric Aluminium Sulphate; and
- b) Non-Ferric Aluminium Sulphate.

### 4 REQUIREMENTS

**4.1** Aluminium sulphate shall be either in the form of lumps, flakes or powder. Particle size of the lumps, flakes and powder form Aluminium sulphate shall be less than 40 mm, 5 mm and 1 mm respectively, when tested as prescribed in Appendix B.

#### NOTE

*Any other particle size as agreed to between the Purchaser and Supplier.*

**4.2** 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
		Ferric	Non Ferric	
(1)	(2)	(3)	(4)	(5)
i)	Water soluble Aluminium sulphate (as Al <sub>2</sub> O <sub>3</sub> ), per cent by mass, min	15	15	Appendix C
ii)	Water insoluble matter, per cent by mass, max	0.5	0.3	Appendix D
iii)	pH at 27 ± 2 °C, min	2.8	2.8	Appendix E
iv)	Arsenic (as As) mg/kg, max	4	4	Appendix F
v)	Lead (as Pb) mg/kg, max	10	10	Appendix F
vi)	Manganese (as Mn) mg/kg, max	25	25	Appendix F
vii)	Cadmium (as Cd) mg/kg, max	2.0	2.0	Appendix F
viii)	Mercury (as Hg) mg/kg, max	0.2	0.2	Appendix F
ix)	Chromium (as Cr) mg/kg, max	10	10	Appendix F
x)	Iron (as Fe) per cent by mass, max	0.7	0.01	Appendix G
xi)	Ammoniacal nitrogen, mg/kg, max	300	-	Appendix H

## **5 PACKAGING**

The material shall be supplied in bulk or in packages as agreed to between the Purchaser and the Supplier.

## **6 MARKING**

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

- a) Name and type 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) Date of manufacture and best before;
- e) Net mass, in kilograms (kg); 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, only reagents of analytical grade and only distilled water shall be used.

## **8 SAMPLING**

Representative samples of the product for ascertaining conformity to the requirements of this Specification shall be drawn as prescribed in Appendix **A**.

## **APPENDIX A COMPLIANCE OF A LOT**

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

Where compliance with this Specification is to be assured, appropriate schemes of sampling and inspection shall be adopted based on manufacturer's control systems coupled with Type Tests and Testing Procedures.

### **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, Hand Scoop, Sampling Tube or other effective device shall be used for drawing samples from containers or conveyors. A Sampling Tube having a core diameter of not less than 25 mm shall be used for drawing samples of powder materials.

### **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** The number of packages to be selected from a lot shall be in accordance with Table 2.

**A.4.3** 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.4.4** In case of lump material or flaked (chips) material, each package selected as in **A.4.2** 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.4.5** In case of powder material, a hand scoop or sampling tube shall be inserted into each package being sampled as in **A.4.2** so that it will take a core of material from substantially the entire length of the package.

**A.4.6** The material obtained from each package shall be thoroughly mixed and reduced by coning and quartering to obtain test sample.

**TABLE 2 – Scale of sampling**

<b>Number of packages in the lot (1)</b>	<b>Number of packages to be selected (2)</b>
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 package selected as in **A.4.2** shall be inspected for marking requirements. This shall be done at the place of sampling.

**A.5.2** Test sample selected as in **A.4.6** 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.1** Each bulk tanker or package inspected for marking as in **A.5.1** satisfies the relevant requirements; and

**A.2** The test results on test 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 with material pass through the sieve until no visible particles retain on the sieve.

#### **B.2.2 Flaked form**

Weigh, to the nearest 0.1 g, about 100 g of the sample and place it on the sieve of aperture size 5 mm. Shake the sieve with material pass through the sieve until no visible particles retain on the sieve.

#### **B.2.3 Powder form**

Weigh, to the nearest 0.1 g, about 100 g of the sample and place it on the sieve of aperture size 1 mm. Shake the sieve with material pass through the sieve until no visible particles retain on the sieve.

## **APPENDIX C DETERMINATION OF WATER SOLUBLE ALUMINIUM SULPHATE**

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** 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.1.1.2** *Sodium acetate buffer solution*

Dissolve 272 g of Sodium acetate trihydrate in water to make 1000 mL.

**C.1.1.3** *Xylenol orange solution (1 g/L)*

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

**C.1.1.4** *Aluminium standard solution (1 mg Al/mL)*

Weigh 1.000 g of Aluminium (purity 99.99 %), in to 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.1.1.5** *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 given in **C.1.2.3** and **C.1.2.4** and the used quantity (mL) of 0.02 mol/L Zinc solution shall be obtained ( $V_1$ ). 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 given in **C.1.2.3** and **C.1.2.4** in below **C.1.2** and the used quantity in millilitre of 0.02 mol/L Zinc solution shall be obtained ( $V_2$ ).

Amount of Aluminium, equivalent to 1 mL of 0.02 mol/L Zinc solution, is given by the following formula:

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

where,

$m$  is the amount, in g, of Aluminium equivalent to 1 mL of 0.02 mol/L Zinc solution.

$V_1$  is the volume, in mL, of 0.02 mol/L Zinc solution consumed at **C.1.2.4** for sample solution;

$V_2$  is volume, in mL, of 0.02 mol/L Zinc solution consumed for Aluminium standard solution.

**C.1.2 Procedure**

**C.1.2.1** Weigh, to the nearest 1 mg about 5 g of sample in to 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.1.2.2** Take 20 mL of this solution into a 200-mL Erlenmeyer flask, add 20 mL of 0.05 mol/L EDTA solution (**C.1.1.1**) and after boiling for 1 minute, allow it to cool.

**C.1.2.3** Add about 10 mL of Sodium acetate buffer solution (**C.1.1.2**) and 2 drops to 5

drops of xylenol orange solution (1 g/L) (C.1.1.3).

**C.1.2.4** Titrate it with 0.02 mol/L Zinc solution (C.1.1.5) and make it an end point when color of the solution turns faint red.

**C.1.2.5** Take 20 mL of 0.05 mol/L EDTA solution (C.1.2.1) transfer it into another 200-mL Erlenmeyer flask, add about 20 mL of deionized water and 2 mL of Nitric acid (1:12), and thereafter carry out procedures as shown in C.1.2.3 and C.1.2.4.

### C.1.3 Calculation

Aluminium sulphate,

$$\text{as Al}_2\text{O}_3, \text{ per cent by mass} = \left[ \left\{ \frac{1.8895 \times (V_3 - V_4) m_1}{m_2 \times \frac{20}{500}} \right\} \times \{100 - (A \times 0.9129)\} \right]$$

where,

- $V_3$  is the volume, in mL, of 0.02 mol/L Zinc solution consumed at C.1.2.5;  
 $V_4$  is the volume, in mL, of 0.02 mol/L Zinc solution consumed for sample solution;  
 $m_1$  is the mass, in g, of Aluminium equivalent to 1 mL of 0.02 mol/L Zinc solution;  
 $A$  is the per cent, by mass of iron determined in Appendix G;  
 $m_2$  is the mass, in g, of sample.

### NOTE

1.8895 and 0.9129 are conversion factors from 1g of Aluminium to Aluminium oxide and iron to Aluminium oxide respectively.

## C.2 METHOD 2

### C.2.1 Reagents

**C.2.1.1** *Concentrated Hydrochloric acid*, relative density, 1.18

**C.2.1.2** *Concentrated Nitric acid*, relative density, 1.42

**C.2.1.3** *Methyl red indicator*, 1 g of methyl red in 500 mL of 95 per cent ethanol

**C.2.1.4** *Concentrated Ammonium hydroxide*, relative density, 0.88

**C.2.1.5** *Ammonium nitrate*, Dissolve 2 g in 100 mL of water

### C.2.2 Procedure

**C.2.2.1** 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 while hot through a tarred sintered glass crucible of pore size between 5 µm to 15 µm (porosity no. 4). 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.2.1.1) and a few drops of concentrated Nitric acid (C.2.1.2) and heat to boiling.

**C.2.2.2** Add 3 drops of methyl red indicator (C.2.1.3) and a small amount of ashless filter paper pulp. Neutralize with concentrated Ammonium hydroxide solution (C.2.1.4) 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.2.1.5). Ignite the filter paper and precipitate in a tarred platinum crucible at 1200 °C for 1 hour. Cool and weigh. Repeat the process until the difference in mass between two successive weighings does not exceed 1 mg.

### C.2.3 Calculation

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

where,

$m_1$  is the mass, in g, of residue;

$m_2$  is the mass, in g, of sample; and

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

## APPENDIX D DETERMINATION OF WATER INSOLUBLE MATTER

### D.1 PROCEDURE

Dry the sintered glass crucible and the residue retained from Appendix C (C.2.2.1) at 105 °C for 2 hours. 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 the mass, in g, of insoluble matter; and

$m_2$  is the mass, in g, of sample in Appendix C

## **APPENDIX E DETERMINATION OF pH**

### **E.1 PROCEDURE**

Weigh 1.0 g of solid material and dissolve in 100 mL of freshly boiled and cooled distilled water. Measure the pH of the solution at  $27 \pm 2$  °C by a suitable pH meter, using a glass electrode.

## **APPENDIX F DETERMINATION OF HEAVY METALS**

Atomic Absorption Spectroscopy (AAS) methodology or Inductively Coupled Plasma Mass Spectrometry (ICP-MS) shall be used for the 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 2 (AAS METHOD)**

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

### **G.2 METHOD 1**

#### **G.2.1 Reagents**

**G.2.1.1** *Concentrated Sulfuric acid*, relative density, 1.84

**G.2.1.2** *Phosphoric acid, 25 per cent (V/V) solution*, prepared by using concentrated Phosphoric acid, relative density, 1.69

**G.2.1.3** *Sodium diphenylamine sulphonate indicator*, 2 g/L

**G.2.1.4** *Potassium dichromate*, standard volumetric solution,  $C(K_2Cr_2O_7)$ , 0.017 mol/L

#### **G.2.2 Procedure**

**G.2.2.1** 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 Zinc has dissolved, boil the solution for 2 to 3 minutes. Cool under running tap water.

**G.2.2.2** Add 40 mL of Phosphoric acid (**G.1.1.2**) and 4 drops of Sodium diphenylamine sulphonate indicator (**G.1.1.3**). Titrate slowly with Potassium dichromate solution (**G.1.1.4**), while stirring continuously, 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.2.3 Calculation

$$\text{Iron, as Fe, per cent by mass (A)} = \frac{33.51 \times C (V_1 - V_2)}{m}$$

where,

$C$  is the concentration, in mol/L, of the Potassium dichromate solution used;  
 $V_1$  is the volume, in mL, of Potassium dichromate solution required for the sample;  
 $V_2$  is the volume, in mL, of Potassium dichromate solution required for the blank; and  
 $m$  is the mass, in g, of the sample.

## 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, available chlorine 1 g/ L

Dissolve the volume of 100/ Concentration of available chlorine 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 mg /mL**

Keep Ammonium chloride in a desiccator (keeping manganese perchlorate) for at least 16 h, weigh 0.382 g and 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 µg/ml**

Take 50-mL of Ammoniacal nitrogen standard stock solution (0.1 mg/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, 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**) 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, and transfer it into a 100 mL color comparison tube with a ground stopper. Add sodium hydroxide solution (**H.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.1.3**) 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 up to the marked line and allow it to 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, 2... up to 7 mL of Ammonical nitrogen Standard solution (**H.1.7**) into 100-mL measuring flasks and add water to make 50 mL solution. Then carry out the procedure described in (**H.2.3**) and (**H.2.4**) and plot the working curve making use of the relation between the quantity of Ammonical nitrogen and the absorbance. On the working curve find the amount of Ammonical nitrogen (µg) which is equivalent to absorbance measured at (**H.2.4**).

## **NOTES**

- 1) *The blank for working curve and standard solution has a low 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

Concentration of Ammonical nitrogen in sample, mg/kg  $= \frac{40 m_1}{m_2}$

where,

$C$  is the concentration, in mg/kg, of Ammonical nitrogen in sample;  
 $m_1$  is the amount, in  $\mu\text{g}$ , of Ammonical nitrogen; and  
 $m_2$  is the mass, in g, of the sample.

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## **SRI LANKA STANDARDS INSTITUTION**

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