SRI LANKA STANDARD 647 : 1994

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SPECIFICATION FOR SULFURIC ACID

(FIRST REVISION)

SRI LANKA STANDARDS INSTITUTION

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SLS 647 : 1994

Gr. 8

SRI LANKA STANDARDS INSTITUTION
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SPECIFICATION FOR SULFURIC ACID
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FOREWORD

This Standard was approved by the Sectoral Committee on Chemicals and Chemical Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 1994-03-31.

This specification covers four grades of sulfuric acid, namely technical, battery, general purpose reagent and analytical reagent. Technical grade acid is intended for use in industry for purposes such as pickling, galvanizing and plating ferrous metals. Battery grade acid is intended for use in lead-acid batteries only. Purity of general purpose reagent grade acid is higher than the technical grade acid and battery grade acid and it could be used in industries and laboratories. Analytical reagent grade acid is the most pure grade and is intended for use in analytical work where a high degree of purity is required.

This standard was first published in 1984. In this revision, requirements for battery grade acid in diluted form have been included. The requirements for antimony and platinum for battery grade acid have been deleted.

Guidelines for the determination of a compliance of a lot with the requirements of this standard based on statistical sampling and inspection are given in Appendix A.

For the purpose of deciding whether a particular requirement of this specification 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 specification.

In the preparation of this standard, the assistance derived from the following publications is gratefully acknowledged :

IS 266 : 1977 Sulfuric acid (Second Revision)
SS 130 : 1975 (Confirmed 1981) Sulfuric acid

1 SCOPE

This specification prescribes the requirements and methods of test for sulfuric acid.

2 REFERENCES

ISO	910	Determination of total acidity and calculation of free sulfur trioxide content of oleum-Titrimetric method.
ISO	913	Determination of ash - Gravimetric method.
ISO	915	Determination of iron content in sulfuric acid - 2,2'-bipyridyl spectrophotometric method.
ISO	2717	Determination of lead content in sulfuric acid -Dithizone photometric method.
ISO	2899	Determination of ammoniacal nitrogen content in sulfuric acid - Spectrophotometric method.
ISO	3423	Determination of sulfur dioxide content in sulfuric acid - Iodometric method.
ISO	5792	Determination of arsenic content - Silver diethyldithiocarbamate photometric method.
SLS	102	Presentation of numerical values.
SLS	301	Determination of copper.
SLS	428	Random sampling methods.
SLS	692	Safety colours and safety signs.

3 GRADES

The following grades of sulfuric acid are covered in this specification :

a) Technical grade;
b) Battery grade concentrated acid and battery grade diluted acid
c) General purpose reagent grade; and
d) Analytical reagent grade.

4 REQUIREMENTS

4.1 General requirements

4.1.1 *Technical grade*

Sulfuric acid of technical grade shall be a clear to slightly cloudy liquid, yellow to light brown in colour and free from visible impurities.

4.1.2 *Battery grade - concentrated and diluted*

Sulfuric acid of battery grade shall be a colourless liquid. The concentrated acid on dilution with an equal volume of water and the diluted acid as received, shall be free from suspended matter and other visible impurities.

4.1.3 *General purpose reagent grade and analytical reagent grade*

Sulfuric acid of general purpose reagent and analytical reagent grades shall be a clear and colourless liquid free from suspended matter and other visible impurities.

4.2 Other requirments

Sulfuric acid of different grades shall also comply with requirements given in Table 1 when tested by the relevant methods given in Column 8 of the table.

TABLE 1 - Requirements for sulfuric acid

Sl. No.	Characteristic	Requirement					Method of test
		Technical grade	Battery grade		General purpose reagent grade	Analytical reagent grade	
			Concentrated	Diluted			
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Total acid content, as H_2SO_4, per cent by mass, min.	98.0	95.0	30.0	98.0	98.0	ISO 910
ii)	Residue on ignition, per cent by mass, max.	0.05	0.01	0.003	0.01	0.002	ISO 913
iii)	Iron, as Fe, mg/kg, max.	100	46	14	20	1	ISO 915
iv)	Lead, as Pb, mg/kg. max.	10	-	-	20	1	ISO 2717
v)	Ammoniacal nitrogen, as NH_3, mg/kg, max.	-	20	6	-	2	ISO 2899
vi)	Sulfur dioxide, as SO_2, mg/kg,max.	20	10	3	-	2	ISO 3423
vii)	Arsenic, as As, mg/kg, max.	2	1	0.3	2	0.05	ISO 5792
viii)	Nitrate, as NO_3, mg/kg, max.	-	40	12	2	0.2	Appendix B
ix)	Copper, as Cu, mg/kg, max.	10	10	3	-	0.1	SLS 301
x)	Chlorides, as Cl, mg/kg, max.	-	10	3	90	0.2	Appendix C
xi)	Selenium, as Se, mg/kg, max.	-	20	6	-	10	Appendix D

5 PACKAGING AND MARKING

5.1 The acid shall be packed in clean, dry, strong and moisture proof containers that are resistant to the corrosive property of the acid.

5.2 For bulk orders, the arrangement for packaging and transportation shall be subject to agreement between the purchaser and the supplier.

5.3 Each container shall be legibly and indelibly marked or labelled with the following information:

a) Name and grade of the acid;
b) Strength of the acid;
c) Name and address of the manufacturer and/or supplier (including country of origin);
d) Net content, in kilograms or litres;
e) Safety sign to indicate corrosive nature (see the Figure) with colours and dimensions as given in SLS 692; and
f) Batch identification mark.

FIGURE – Safety sign to indicate corrosive nature

NOTE

Attention is drawn to the certification marking facilities offered by the Sri Lanka Standards Institution. See the inside back cover of this standard.

6 METHODS OF TEST

6.1 Tests shall be carried out as prescribed in Appendices B to D of this specification and the relevant standards specified in Column 8 of Table 1.

6.2 Unless otherwise specified, reagents of analytical grade and distilled water or water of equivalent purity shall be used.

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 standard is to be assessed based on statistical sampling and inspection.

Where compliance with this standard is to be assured based on manufacturer's control systems coupled with type testing and check tests or any other procedure, an appropriate scheme of sampling and inspection should be adopted.

A.1 LOT

In any consignment of sulfuric acid, all the containers of the same size, containing acid of the same grade and strength and belonging to one batch of manufacture or supply shall constitute a lot.

A.2 SAMPLING INSTRUMENTS

A.2.1 *Glass sampling tube*

A.2.2 *Pipette*

A.3 GENERAL REQUIREMENTS OF SAMPLING

In drawing samples, the following precautions and directions should be observed :

A.3.1 Precautions should be taken to prevent the samples from adventitious contamination.

A.3.2 Sampling instruments, when used, should be clean and dry.

A.3.3 To draw a representative sample, the contents of each container should be mixed thoroughly by rolling, shaking or stirring by suitable means and with necessary caution.

A.3.4 Samples should be placed in suitable, clean, dry and air-tight glass containers.

A.3.5 Each sample container should be sealed air-tight after filling and should be marked with necessary details of sampling.

A.3.6 Samples should be stored in such a manner that the conditions of storage do not affect the quality of the material.

A.4 SCALE OF SAMPLING

A.4.1 Samples should be tested from each lot for ascertaining its conformity to the requirements of this specification.

A.4.2 The number of containers to be selected from a lot should be in accordance with Table 2.

TABLE 2-Scale of sampling

Number of containers in the lot	Number of containers to be selected
Up to 25	2
26 to 100	3
101 to 150	5
151 to 300	8
301 to 500	10
501 and above	13

A.4.3 The containers should be selected at random. In order to ensure randomness of selection, tables of random number as given in SLS 428 should be used.

A.5 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

A.5.1 Individual samples

An equal quantity of the material should be drawn from top, middle and bottom portions of containers, using a glass sampling tube. The material so obtained should be mixed well to form an individual sample of not less than 600 ml. Individual samples should be obtained from each container selected as in A.4.2.

A.5.2 Composite sample

An equal quantity of material should be drawn from top, middle and bottom portions of each container selected as in A.4.2 and material so obtained should be mixed well to form a composite sample of not less than 600 ml to represent the lot.

A.6 NUMBER OF TESTS

A.6.1 Each container selected as in A.4.2 should be examined for packaging and marking requirements. (This may be done at the place of inspection).

A.6.2 Each individual sample obtained as in **A.5.1** should be tested for total acid content.

A.6.3 The composite sample obtained as in **A.5.2** should be tested for the requirements given in Table **1**, except for the acid content.

A.7 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this specification if the following conditions are satisfied :

A.7.1 Each container examined as in **A.6.1** satisfies the relevant requirements.

A.7.2 Each individual sample tested as in **A.6.2** satisfies the relevant requirements.

A.7.3 The composite sample tested as in **A.6.3** satisfies the relevant requirements.

APPENDIX B
TEST FOR NITRATE CONTENT

B.1 FOR ANALYTICAL GRADE

B.1.1 Reagents

B.1.1.1 *Hydrochloric acid,* concentrated, 32 per cent m/m.

B.1.1.2 *Diphenylamine solution*
Mix 90 mg of diphenylamine with 60 ml of nitrogen-free sulfuric acid and add the mixture to 20 ml of water.

B.1.2 Procedure

To 6 ml of the sample add 2 ml of water, cool to 60 °C and add one drop of hydrochloric acid (**B.1.1.1**) and 1 ml of diphenylamine solution (**B.1.1.2**).

B.1.3 Interpretation of results

The limit prescribed in Table 1 shall be taken as not having being exceeded if no blue colour is produced.

B.2 FOR BATTERY GRADE AND GENERAL PURPOSE REAGENT GRADE

B.2.1 Principle

Nitrate ion reacts with brucine in strong sulfuric acid solution to develop a yellow colour. The colour developed is measured at 410 nm and the nitrate ion concentration is determined using a standard calibration curve.

NOTES

1) The colour does not follow the Bear-Lambert relationship. However, a plot of absorbence versus concentration produces a smooth curve.

2) The rate of colour development varies directly with the temperature and the intensity varies inversely with the temperature.

B.2.2 Interferences

Nitrite interference up to 7 mg/kg is eliminated by the sulfanilic acid in the brucine reagent solution.

B.2.3 Apparatus

B.2.3.1 *Photometer*, a spectrophotometer or filter photometer suitable for measurements at 410 nm with a light path of 10 mm to 50 mm.

B.2.4 Reagents

B.2.4.1 *Potassium nitrate, standard solution*
Dry potassium nitrate (KNO_3) in an oven at 105 °C for 24 hours. Dissolve 1.631 g in 20 ml of water and dilute to one litre. (One millilitre of this solution contains 1 mg of NO_3.)

B.2.4.2 *Brucine-sulfanilic acid solution*
Dissolve 1 g of brucine sulfate (see Note) and 0.1 g of sulfanilic acid in approximately 70 ml of hot water. Add 3 ml of hydrochloric acid (rel. den.=1.19), cool, and dilute to 100 ml with water.

This solution is stable for several months. The pink colour that develops slowly does not affect its usefulness.

NOTE

Brucine sulfate is very toxic. Take care to avoid ingestion.

B.2.4.3 *Sulfuric acid*

Carefully add 500 ml of sulfuric acid of analytical grade to 75 ml of water. Cool and keep in a tightly stoppered container to prevent absorption of moisture.

B.2.5 Calibration and standardization

B.2.5.1 Prepare a blank and a series of standard solutions using the standard nitrate solution (**B.2.4.1**). The standard solutions should cover the range from 0 mg/kg to 50 mg/kg of nitrate as NO_3. Pipette 5.0 ml portions of each standard solution into 25-ml volumetric flasks. Then add 1 ml of brucine-sulfanilic acid reagent (**B.2.4.2**) and 5 ml of distilled water to each flask. Proceed as given in B.2.5.2 to B.2.5.5.

B.2.5.2 Add 10 ml of sulfuric acid (B.2.4.3) to a second 25-ml volumetric flask.

B.2.5.3 Mix the contents of the two volumetric flasks by carefully adding the solution with the brucine-sulfanilic acid reagent (**B.2.5.1**) to the flask containing the acid. Pour from one flask to the other six times to ensure mixing. Set aside for 10 ± 1 minutes in a dark place.

B.2.5.4 Dilute to the mark with distilled water. Keep for 20 minutes to 30 minutes in the dark to allow to cool to room temperature.

B.2.5.5 Measure the absorbance of the treated solution at 410 nm against the blank treated similarly to the standard solution except for omitting the addition of the brucine-sulfanilic acid reagent.

B.2.5.6 Plot the absorbance versus the nitrate concentration in milligram per kilogram.

NOTE

A calibration curve shall be prepared with each batch of samples analysed.

B.2.6 Analysis of sample

B.2.6.1 Add 5.0 g of sulfuric acid sample under test, into a 25-ml volumetric flask. Add 7 ml of sulfuric acid (**B.2.4.3**) to the flask.

B.2.6.2 Add 1 ml of brucine-sulfanilic acid reagent and 5 ml of distilled water to a second 25-ml volumetric flask.

B.2.6.3 Treat in accordance with B.2.5.3 to B.2.5.5.

B.2.6.4 Determine the concentration of nitrate in the sample from the calibration curve (see B.2.5.6), in milligrams per kilogram.

APPENDIX C
TEST FOR CHLORIDE CONTENT

C.1 APPARATUS

Nessler cylinders, 50-ml capacity.

C.2 REAGENTS

C.2.1 *Dilute nitric acid,* approximately 4 mol/l.

C.2.2 *Silver nitrate,* approximately 5 g per 100 ml solution.

C.2.3 *Standard chloride solution*

Dissolve 1.648 g of sodium chloride, previously dried,in 1 000 ml of water. Dilute 10 ml of this solution again to 1000 ml in a volumetric flask. One millilitre of this solution contains 0.01 mg of chloride.

C.3 PROCEDURE

C.3.1 Weigh the relevant mass of sample given in Column 2 of Table 3 and dilute to 50 ml with water in a Nessler cylinder (C.1). Add 1 ml of dilute nitric acid (C.2.1) and 1 ml of silver nitrate solution (C.2.2) and mix. Carry out a control test in another Nessler cylinder in the same manner using the relevant quantity of standard chloride solution given in Column 3 of Table 3.

TABLE 3 - Quantities to be taken when testing for chloride content

Sl. No. (1)	Grade of the acid (2)	Mass of the sample to be taken, g (3)	Volume of standard chloride solution to be taken, for the control test, ml (4)
i)	Battery grade, concentrated	2.0	2.0
ii)	Battery grade, diluted	20.0	6.0
iii)	General purpose reagent grade	2.0	18.0
iv)	Analytical reagent grade	25.0	0.5

C.3.2 Interpretation of results

The limit prescribed for the appropriate grade in Table 1 shall be taken, as not having being exceeded if the turbidity produced in the test solution is not greater than that produced in the respective control test.

APPENDIX D
TEST FOR SELENIUM CONTENT

D.1 REAGENTS

D.1 Standard selenium solution

Dissolve 0.100 g of selenium in 5 ml of concentrated nitric acid and 10 ml of concentrated hydrochloric acid. Evaporate to dryness and take up the residue with 10 ml of 1 : 1 sulfuric acid (selenium-free) by slight warming. Make up the volume to 1 000 ml (One millilitre of this solution contains 0.1 mg of selenium).

D.1.2 *Sulfuric acid,* concentrated (rel. den.= 1.84), selenium free.

D.1.3 *Sodium sulfite,* 5 g per 100 ml solution, freshly prepared.

NOTE

The reagent must be freshly prepared and used within one hour.

D.2 PROCEDURE

Evaporate almost to dryness, in three glass dishes, volumes of selenium standard solutions given in Column 3 of Table 4, relevant to the grade of the acid under test, on a water bath. Stir well with a glass rod to dissolve the selenium and transfer (without any washing) to three 150 mm x 16 mm test tubes. Transfer a relevant mass of the test sample as given in Column 4 of Table 3 to another test tube.

Carefully add 5 ml of the sodium sulfite solution (D.1.3) to each test tube. At the end of 2 hours, compare the red selenium rings near the zone of contact, viewing them transversely against a white piece of filter paper in a good source of light.

NOTE

In case of doubt, leave overnight, for a broader ring to develop for easier comparison.

TABLE 4 - Quantities to be taken when testing
for selenium content

Sl. No. (1)	Grade of the acid (2)	Volumes of selenium standard solution to be used, ml (3)	Mass of the test sample to be used, g (4)
i)	Battery grade, concentrated	1.5 ml, 2.0 ml and 2.5 ml	10.0
ii)	Battery grade, dilute	1.0 ml, 1.5 ml and 2.0 ml	25.0
iii)	Analytical reagent grade	0.5 ml, 1.0 ml and 1.5 ml	25.0

D.3 INTERPRETATION OF RESULTS

The limits prescribed in Table 1 for different grades of sulfuric acid shall be taken as not having been exceeded if the intensities of the colour of the rings produced by the test samples are not greater than those produced by the control tests with relevant amount of selenium standard solutions indicated in Column 3 of Table 5.

TABLE 5 - Control tests to be compared with

Sl. No. (1)	Grade of the acid (2)	Amount of selenium standard solution in the control test to be compared with, ml (3)
i)	Battery grade, concentrated	2.0
ii)	Battery grade, dilute	1.5
iii)	Analytical reagent grade	1.0

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