

भारतीय मानक मसौदा
मैग्नीशियम सल्फेट (एप्सॉम लवण) — विशिष्टि
(IS 2730 का दूसरा पुनरीक्षण)

Draft Indian Standard
Magnesium Sulphate (Epsom Salts) — Specification
(*Second Revision of IS 2730*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

(Formal clauses will be added later)

This standard was originally published in 1964 and subsequently revised in 1977. Two Indian Standard specifications had originally been published for magnesium sulphate, IS 257 for the technical grade and IS 377 for the pharmaceutical grade. These two specifications were later on amalgamated as IS 2730. In the first revision, changes were made in the limit of magnesium sulphate content and the limits of chlorides and iron for technical grade of the material for use in leather industry. Also, the requirement of pharmaceutical grade was deleted.

In this revision, instrumental test methods for the determination of arsenic, chlorides, iron and lead have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Amendment No. 1 and Reference clause have been incorporated. Further, Packing and Marking clause has been updated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Draft Indian Standard
MAGNESIUM SULPHATE (EPSOM SALTS) — SPECIFICATION
(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for magnesium sulphate (epsom salts).

2 REFERENCE

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

| <i>IS No</i> | <i>Title</i> |
|-----------------------------------|--|
| IS 323 : 2009 | Rectified spirit for industrial use — Specification (<i>second revision</i>) |
| IS 1070 : 2023 | Reagent grade water — Specification (<i>fourth revision</i>) |
| IS 2088 : 2023 | Methods for determination of arsenic (<i>second revision</i>) |
| IS 3025(Part 2) : 2019 | Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP – OES) (<i>first revision</i>) |
| IS 3025 (Part 65) : 2022 | Methods of sampling and test physical and chemical for water and wastewater part 65 application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (<i>first revision</i>) |
| IS 4905: 2015/ ISO 24153: 2009 | Random sampling and randomization procedures (<i>first revision</i>) |

3 GRADES

3.1 The material shall be of the following two grades:

- a) Pure, and
- b) Technical.

3.1.1 Pure grade of the material is used in cosmetics, mineral waters, explosives and weighting of silk. The technical grade of the material is used in textile and leather industries where it is used as a tan precipitant for the purpose of fixing tan. It is also used as a micronutrient in fertilizers to meet magnesium deficiency in plants.

4 REQUIREMENTS**4.1 Description**

The material shall be in the form of colourless crystals, soluble in water and sparingly soluble in ethyl alcohol (90 percent v/v).

4.1.1 The crystals are liable to become powdery on efflorescence and, unless otherwise agreed to between the purchaser and the supplier, such a condition shall not render the material unacceptable.

4.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of the Annex is given in col 5 of the table.

5 PACKING AND MARKING**5.1 Packing**

The material shall be packed in air-tight containers as agreed to between the purchaser and the supplier. The containers used for packing the pure grade shall be polyethylene lined.

5.2 Marking

Each container shall be marked with the following information:

- Name and grade of the material;
- A statement if the material is intended for use in leather industry;
- Net mass of the material;
- Month and year of packing;
- Manufacturer's name and/or his recognized trade-mark, if any; and
- Lot number to enable the date of manufacture to be traced from records.

Table 1 Requirements for Magnesium Sulphate (Epsom Salts)

(Clauses 4.2, A-3.4, A-4.3.1, A-6.3.1, B-5.1.1 and B-5.2)

| Sl No. | Characteristic | Requirement for Grade | | Method of Test (Ref to Cl No. in Annex A) |
|--------|--|-----------------------|--------------------|---|
| | | Pure | Technical | |
| (1) | (2) | (3) | (4) | (5) |
| i) | Magnesium sulphate (as MgSO ₄), (calculated with reference to the material dried at 300 °C), percent by mass | 99.5 to 100.5 | 98.0 to 100.5 | A-2 |
| ii) | Chlorides (as Cl), percent by mass, <i>Max</i> | 0.12 | 1.0* | A-3 or A-11 |
| iii) | Lead (as Pb), ppm, <i>Max</i> | 5 | — | A-4 or A-10 |
| iv) | Arsenic (as As ₂ O ₃), ppm, <i>Max</i> | 2 | — | A-5 or A-10 |
| v) | Iron as (Fe), percent by mass, <i>Max</i> | 0.002 (20 ppm) | 0.007* (70 ppm) | A-6 or A-10 |
| vi) | Zinc | To pass the test | — | A-7 |
| vii) | Matter insoluble in water, percent by mass, <i>Max</i> | — | 0.20 | A-8 |
| viii) | Acidity or alkalinity | To pass the test | — | A-9 |

*For technical grade of the material when required for leather industry, the limit for chloride (as Cl) shall be 0.6 percent by mass. *Max*, and the limit for iron (as Fe) shall be 0.001 percent by mass, *Max*.

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 there under, and the products may be marked with the Standard Mark.

6 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A

(Clause 4.2)

METHODS OF TEST FOR MAGNESIUM SULPHATE (EPSOM SALTS)

A-1 QUALITY OF REAGENTS

A-1.1 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 MAGNESIUM SULPHATE

A-2.1 Reagents

A-2.1.1 Patton and Reeders' Indicator

Mix 0.1 g of 2-hydroxy 1-(2-hydroxy-4-sulpho-1-naphthylazo)-3-naphthoic acid with 10 g of sodium sulphate powder.

A-2.1.2 Ammonium Hydroxide-Ammonium Chloride Buffer Solution

Mix 350 ml of ammonium hydroxide (20 percent *m/m*) with 54 g of ammonium chloride. Dilute with water and make up the volume to 1 000 ml. (The *pH* of the solution should be not more than 10.)

A-2.1.3 Standard Calcium Solution — 0.01 M.

Dissolve 0.500 5 g of calcium carbonate (CaCO_3) in dilute hydrochloric acid; when effervescence ceases, dilute with water to 500 ml.

A-2.1.4 Ethylenediamine Tetra-Acetate (EDTA) Solution — 0.01 M.

Dissolve 3.72 g of disodium ethylenediamine tetra-acetate dihydrate in water and make up the volume to 1 litre.

A-2.2 Procedure

A-2.2.1 Standardization of EDTA Solution

Take 10 ml of standard calcium solution in a conical flask. Add 20 ml of water, 1 ml of Patton and Reeders' indicator and 25 ml of ammonium hydroxide-ammonium chloride buffer solution. Heat to 40 °C to 50 °C and then titrate with EDTA solution, maintaining the temperature between 40 °C and 50 °C until the colour changes from wine red to distinct blue.

$$\text{A-2.2.1.1 Molarity of EDTA solution} = \frac{10 \times M_1}{V_1}$$

where

M_1 = molarity of standard calcium solution, and

V_1 = volume in ml of EDTA solution used for titration.

A-2.2.2 Weigh accurately about 1 g of the material, previously dried at 300 °C and dissolve in 100 ml of water in a volumetric flask. Take 10 ml of this solution in a conical flask. Add 20 ml of water, about 0.5 g of Patton and Reeders' indicator and 25 ml of ammonium hydroxide ammonium chloride buffer solution. Heat to 40 °C to 50 °C and titrate with standard EDTA solution, maintaining the temperature between 40 °C and 50 °C, until the colour changes from wine red to distinct blue.

A-2.3 Calculation

$$\text{Magnesium sulphate (MgSO}_4\text{), percent by mass} = \frac{120.4 \times V_2 \times M_2}{M}$$

where

V_2 = volume in ml of standard EDTA solution used in **A-2.2.2**.

M_2 = molarity of standard EDTA solution as determined in **A-2.2.1**, and

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

A-3 TEST FOR CHLORIDES

A-3.1 Apparatus

A-3.1.1 Nessler Cylinders — 50 ml capacity.

A-3.2 Reagents

A-3.2.1 Dilute Nitric Acid — approximately 5 N.

A-3.2.2 Silver Nitrate Solution — approximately 5 percent.

A-3.2.3 Standard Sodium Chloride Solution — 0.01 N.

A-3.3 Procedure

A-3.3.1 For Pure Grade

Dissolve 0.30 g of the material in 10 ml of water. Add 10 ml of dilute nitric acid and 1 ml of silver nitrate solution. Transfer the solution completely into a Nessler cylinder. Dilute the solution with water to 50 ml. Carry out a control test in another Nessler cylinder using 1 ml of standard sodium chloride solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Stir both the solutions with a glass rod and compare the turbidities after 5 min.

A-3.3.2 For Technical Grade

In the case of technical grade, use 0.035 g of the material and follow the same procedure as given in **A-3.3.1**. For the material for use in leather industry, 0.06 g of the material shall be taken.

A-3.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced in the test with the material is not greater than that produced in the control test.

A-3.5 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed at **A-11**.

A-4 TEST FOR LEAD

A-4.1 Apparatus

A-4.1.1 Nessler Cylinders — 50 ml capacity.

A-4.2 Reagents

A-4.2.1 Acetic Acid — approximately 35 percent (v/v).

A-4.2.2 Dilute Ammonium Hydroxide — approximately 10 percent.

A-4.2.3 Potassium Cyanide Solution

Dissolve 10 g of potassium cyanide in 90 ml of water, add 2 ml of hydrogen peroxide [approximately 6 percent (v/v)], allow to stand for 24 h and make up to 100 ml with water.

A-4.2.4 Standard Lead Solution

Dissolve 0.160 g of lead nitrate in water, add 1 ml of concentrated nitric acid and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 100 ml with water. One millilitre of this solution contains 0.01 mg of lead (as Pb).

A-4.2.5 Sodium Sulphide Solution — approximately 10 percent.

A-4.3 Procedure

Weigh 12 g of the material into a Nessler cylinder and dissolve in 30 ml of hot water. Add 5 ml of acetic acid. Make it alkaline to litmus by gradual addition of dilute ammonium hydroxide and add 1 ml of potassium cyanide solution. Carry out a control test in the other Nessler cylinder using 2 g of the material, 5 ml of standard lead solution and the same quantities of other reagents. Filter both the solutions if they are turbid and, if the colours of the solutions differ, equalize by the addition of a few drops and a highly diluted solution of burnt sugar or other non-reactive substance. Dilute both the solutions with water and make up the volume to 50 ml. Add 2 drops of sodium sulphide solution to each cylinder, mix thoroughly and compare the colours developed in the two cylinders.

A-4.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-4.4 Alternative Method

Lead may alternatively be determined by ICP-OES instrumental test method as prescribed at **A-10** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-5 TEST FOR ARSENIC

A-5.1 Preparation of Solution

Dissolve 1 g of the material in 10 ml of water.

A-5.2 Procedure

Carry out the test for arsenic with the solution prepared in **A-5.1**, as prescribed in IS 2088, using for comparison a stain obtained with 0.002 mg of arsenic trioxide (as As_2O_3).

A-5.3 Alternative Method

Arsenic may alternatively be determined by ICP-OES instrumental test method as prescribed at **A-10** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-6 TEST FOR IRON

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 100 ml capacity.

A-6.2 Reagents

A-6.2.1 *Citric Acid Solution* — 20 percent (*m/v*).

A-6.2.2 *Thioglycollic Acid*

A-6.2.3 *Ammonium Hydroxide* — 20 percent (*m/m*).

A-6.2.4 *Standard Iron Solution*

Dissolve 0.702 g of ferrous ammonium sulphate in water and add 10 ml of dilute sulphuric acid (10 percent *m/m*). Dilute the solution to 1 litre. One millilitre of the solution contains 0.1 mg of iron (as Fe).

A-6.3 Procedure

Dissolve 10 g of the material, accurately weighed, in 50 ml of water and transfer quantitatively to a Nessler cylinder. Add 10 ml of citric acid solution and five drops of thioglycollic acid, mix and make alkaline with ammonium hydroxide. Dilute with water to 100 ml mark and allow to stand for 5 min. Carry out a control test using the following quantities of standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture:

- a) 2 ml in case of pure grade;
- b) 7 ml in case of technical grade; and
- c) 1 ml in case when technical grade is required for leather industry.

A-6.3.1 Compare the colour produced in the two cylinders. The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the test with the material is not darker than the colour produced in the control test.

A-6.4 Alternative Method

Iron may alternatively be determined by ICP-OES instrumental test method as prescribed at **A-10** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-7 TEST FOR ZINC

A-7.1 Reagents

A-7.1.1 *Dilute Acetic Acid* — approximately 53 percent (v/v).

A-7.1.2 *Potassium Ferrocyanide Solution* — approximately 5 percent.

A-7.2 Procedure

Dissolve 2 g of the material in 20 ml of water. Acidify the solution with dilute acetic acid and then add a few drops of potassium ferrocyanide solution.

A-7.2.1 The material shall be taken to have satisfied the requirement of the test if no turbidity is produced.

A-8 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-8.1 Procedure

Accurately weigh about 10 g of the material. Dissolve it in 100 ml of water by stirring the solution well, warming, if necessary. Filter through a tared filter paper or a tared sintered glass crucible (G No.4) or a Gooch crucible; wash thoroughly the residue till it is free from all soluble compounds and dry at 105 °C to 110 °C to constant mass.

A-8.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{100 M_1}{M}$$

where

M_1 = mass in g of the residue, and

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

A-9 TEST FOR ACIDITY OR ALKALINITY

A-9.1 Reagents

A-9.1.1 *Phenol Red Indicator Solution*

Warm 50 mg of phenol red with 2.85 ml of N/20 sodium hydroxide and 5 ml of rectified spirit (*see* IS 323) and dilute to 250 ml.

A-9.1.2 *Standard Hydrochloric Acid* — 0.01 N.

A-9.1.3 *Standard Sodium Hydroxide Solution* — 0.01 N.

A-9.2 Procedure

Weigh 1 g of the material and dissolve in 10 ml of water. Add a few drops of phenol red indicator.

A-9.2.1 The material shall be taken to have passed the test if the solution does not require more than 0.2 ml of either 0.01 N hydrochloric acid or 0.01 N sodium hydroxide solution to change its colour.

A-10 DETERMINATION OF ARSENIC, LEAD AND IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-10.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get 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 intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

A-10.2 Recommended Wavelength, limit of quantification and important spectral interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, Table 2 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(Clauses A-10.2 and A-10.4)

| Sl No. | Element | Wavelength (nm) | Approximately Achievable Limits | | Interfering Elements |
|--------|---------|--------------------|--|---------------------------------------|----------------------|
| | | | Radial Viewing (μg) | Axial Viewing (μg) | |
| (1) | (2) | (3) | (4) | (5) | (6) |
| i) | As | 188.979 | 18 | 14 | Al, Cr, Fe, Ti |
| | | 193.696 | 5 | 14 | Al, Co, Fe, W, V |
| | | 197.197 | (100) | 31 | Al, Co, Fe, Pb, Ti |
| ii) | Fe | 238.204 | 14 | (3) | Co |
| | | 259.940 | 6 | 2 | Co |
| | | 271.441 | - | - | - |
| iii) | Pb | 220.353 | 14 | 5 | Al, Co, Fe, Ti |
| | | 283.305 | (70) | | Cr, Fe |
| | | 217.00 | | (20) | |

A-10.3 Reagents and Solutions

A-10.3.1. Nitric Acid (65 percent) Suprapure

A-10.3.2 Standard Stock Solution

Either Prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1 000 $\mu\text{g}/\text{ml}$ of arsenic, iron, and lead in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-10.3.3 Standard Solution

Pipette out 5 ml from 100 $\mu\text{g}/\text{ml}$ standard stock solution into a 100 ml volumetric flask & make up volume with 2 percent nitric acid to prepare 5 $\mu\text{g}/\text{ml}$ solution. From this 5 $\mu\text{g}/\text{ml}$ solution, an aliquot of 1.0 ml, 3.0 ml and 5.0 ml

taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml solution of respective elements under reference.

A-10.3.4 Sample Preparation

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

NOTE — Sample should be clear before injecting to the instrument.

A-10.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluoroethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

A-10.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

A-10.5 Procedure**A-10.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-10.3.5). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

A-10.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in µg/ml of the lead (and/or Iron, arsenic) in the sample solution.

NOTE — It is recommended that IS 3025 (Part 2) /ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

A-10.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

- i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.
- ii) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).

A-11 ION CHROMATOGRAPHY FOR CHLORIDES

A-11.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides. 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, 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 Equipment

A-11.2.1 Anion Guard Column — a protector of the separator column.

A-11.2.2 Anion Separator Column — suitable for selective separation of ions under analysis.

A-11.2.3 Anion Suppressor Device

Anion micromembrane suppressor is used to analyse the data.

A-11.2.4 Detector — conductivity detector.

A-11.2.5 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 and Standards

A-11.3.1 Glass or Polyethylene Sample Bottles.

A-11.3.2 Distilled Water or Deionized Water free from the Anions of interest.

A-11.3.3 Eluent

1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used.

For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 L of water.

A-11.3.4 Micromembrane suppressor solution (0.025 N of sulphuric acid)

Dilute 2.8 ml of concentrated Sulphuric acid in 4 litre of water

A-11.3.5 Standard solutions

A-11.3.6 Chloride

Dissolve NaCl, 1.648 5 g in 1 litre of reagent water

A-11.4 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 ml to 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-11.5 Procedure

Dissolve between 1 g to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 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 and deviation of retention force shall not exceed ± 10 percent of RT of calibration. 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 reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. 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-11.6 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.

ANNEX B

(Clause 6)

SAMPLING OF MAGNESIUM SULPHATE (EPSOM SALTS)

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material of one grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 3.

Table 3 Number of Containers to be Selected

(Clause B-2.2)

Lot Size

Number of Containers to

| N (1) | be Selected n (2) |
|--------------|---------------------------|
| Up to 50 | 3 |
| 51 to 200 | 4 |
| 201 to 400 | 5 |
| 401 to 650 | 6 |
| 651 to 1 000 | 7 |

B-2.3 The containers shall be chosen at random from the lot and in order to ensure randomness of selection, a random number table shall be used. In case such tables are not available, the following procedure may be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any container, count them 1, 2, 3, ... , up to r and so on, where r is the integral part of N/n . Every r^{th} container thus counted shall be taken out for drawing samples.

NOTE — For details of this procedure as well as other methods of random selection, reference may be made to IS 4905.

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to make triplicate determination for all the characteristics given under 3 and shall not exceed 1 kg.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, and another for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.5**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample

The referee sample shall consist of the composite sample (*see B-3.1.2*) and a set of individual samples (*see B-3.1.3*) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of magnesium sulphate shall be conducted on each of the individual samples for both the grades.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

B-5.1.1 For Magnesium Sulphate

The test results for magnesium sulphate shall be recorded, and the mean and the range for these test results shall be calculated as follows:

Mean \bar{x} = Sum of the test results divided by the number of test results, and

Range (R) = The difference between the maximum and the minimum values of the test results.

The value of expression ($\bar{x} \pm 0.6 R$) shall be calculated. If the values of this expression lie within the limits specified for the relevant grade in Table 1, the lot shall be declared to have satisfied the requirements for this characteristic.

B-5.2 For Composite Sample

The lot shall be deemed to have satisfied the remaining requirements (*see* **B-4.2**) if all the test results on the composite sample shall meet the corresponding requirements specified in Table 1 for the relevant grade.

B-5.3 The lot shall be declared as conforming to the requirements of the specification if **B-5.1** and **B-5.2** are satisfied.