
ऊर्जा संग्रहण प्रौद्योगिकियों के
लिए इलेक्ट्रोलाइट फॉर्म्युलेशन
— विशिष्ट

**Electrolyte Formulations for
Energy Storage Technologies
- Specification**

FOREWORD

This Indian Standard _____ was adopted by the Bureau of Indian Standards, after the draft finalized by the _____ (Name of Committee) had been approved by the Chemical Department (CHD) Division Council.

An Electrolyte in a battery is a conductive pathway that facilitates movement of ions between positive and negative electrodes, allowing the flow of electric current. Attributes of Electrolyte Formulations govern many aspects such as ionic conductivity, stabilization of cathode & anode, polarization, rate capability, performance over temperature and battery life.

Optimizing EF is essential for the development of high-performance batteries (lithium-ion batteries, sodium-ion batteries, and others) with an extended longevity and enhanced safety. Appropriate viscosity and density of the electrolyte is desirable to ensure better ion mobility, greater charge acceptance, diffusivity, mitigation of concentration polarization and reduced metal deposition at high charges rates.

Customized formulation of Electrolytes requires the presence of multiple solvents and mixed salts, including but not limited to Lithium Hexafluorophosphate (LiPF₆), Sodium Hexafluorophosphate (NaPF₆), Ethylene Carbonate (EC), Diethyl Carbonate (DEC) Dimethyl Carbonate (DMC), etc. Based on the specifications of the constituents; different physical, thermodynamic, flammable, dielectric and thermo-physical properties of the electrolyte solution get defined.

Metallic and other impurities such as moisture and Hydrofluoric acid have to be minimized, as they shorten the battery life span.

Formulation of appropriate electrolytes enable the manufacturers to commercialize new energy storage technologies across various industries like Electric Vehicles (EV), Electronics, Medical Equipment, Aviation and others. Specialized formulations are used for extreme temperatures or rapid charge / discharge applications, ensuring optimal performance, longevity, and safety in demanding operational environment.

To determine compliance with a specific requirement of this standard, the final value observed or calculated, expressing the result of a test or analysis, should 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.

Indian Standard (Proposed)

ELECTROLYTE FORMULATIONS FOR ENERGY STORAGE TECHNOLOGIES - SPECIFICATION

1 SCOPE

This Indian Standard outlines the requirements, testing methods, and application guidelines for Electrolyte Formulations (EFs), a solution that facilitates ion conductivity between negative and positive electrodes to ensure superior battery performance.

This standard applies to EFs used in various applications, in energy storage including operationalization of Lithium-ion batteries, Sodium-ion batteries, etc.

The objective of this standard is to define the material properties and quality requirements of EFs, establish standardized methods for testing and evaluating EFs, and provide guidelines for the packing, marking, transportation, storage, and safe handling.

2 REFERENCES

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

<i>IS / Other Publications</i>	<i>Title</i>
ASTM D 1193-99	Standard Specification for Reagent Water
ASTM D-1209-05	Standard Test Method for Color of Clear Liquids
1260 (Part 1): 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (First revision)
18149: 2023	Transportation of Dangerous Goods — Guidelines

3 TERMS AND DEFINITIONS

For the purpose of this standard the following definitions shall apply.

3.1 Acid (as HF): It refers to presence of acids in a sample estimated as hydrofluoric acid.

3.2 Electrolyte Formulations: These are solutions or mixtures of salts, solvents, and additives.

3.3 ICP-MS: Inductively Coupled Plasma Mass Spectrometry Testing Method for elemental impurities.

3.4 Ion Chromatography: Methodology used for separation and detection of anions like Chloride, Sulphate, Nitrate, etc. and organic anions.

3.5 Turbidity: It refers to the haziness or cloudiness of the fluid caused by the presence of suspended particles in the formulations, which can indicate contamination or presence of impurities.

4 REQUIREMENTS**4.1 Description**

4.1.1 The electrolyte formulations should be clear and free from any turbidity and cloudiness. The color should be consistent throughout the formulation without any variation, indicating uniform mixing.

4.1.2 High purity of ingredients is crucial to prevent contamination and ensure the formulation's effectiveness.

4.1.3 The formulations shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in the table.

5 PACKING, TRANSPORTATION, STORAGE, MARKING AND HANDLING**5.1 Packaging**

5.1.1 Electrolyte Formulations shall be packed in containers in the form of drums, and having dimensions as mentioned in Table 2.

5.1.2 The construction of the drum and lid should be of stainless steel, ensuring durability and corrosion resistance.

5.1.3 The drums should have an electro-polished finish to prevent contamination of the material. It should be air-tight to prevent moisture ingress, with a silicone gasket to provide and effective seal.

5.1.4 The drums shall be filled with Electrolyte under nitrogen blanketing with nitrogen purity being atleast 99.99% and maintained under nitrogen to ensure stability of the contents.

5.2 Transportation

5.2.1 The transportation of Electrolytes must adhere to stringent regulations due to its hazardous nature. If required by a purchaser, a UN certification of SS Drum shall be conducted.

5.2.2 A Safety Data Sheet (SDS) of Electrolytes shall be included with each shipment for reference in case of an emergency. The SDS shall identify the constituents (salts and solvents) and the indicative ranges of each.

5.3 Storage

5.3.1 The formulations should be stored in tightly sealed containers within a well-ventilated, cool, and dry room. The storage temperature will vary depending upon the formulation.

5.3.2 It must be kept away from heat, hot surfaces, sparks, open flames, and other ignition sources. EFs should be separated from incompatible materials such as oxidizing agents, strong acids, and glass, and should not be stored in glassware.

5.3.3 The storeroom should be equipped with proper facilities for managing accidental leakage and fire, and the electrolyte formulations should be kept away from food containers.

5.4 Marking

5.4.1 The label on the packages of the material shall indicate the name of material; the UN-No. according to the hazard classification of the Electrolytes; and details of manufacturer.

5.4.2 The label shall also display:

- Dangerous goods signs (as shown in fig. 2);
- Hazard statements relating to the product;
- Production date;
- Drum number;
- Batch number; and
- Mass of the material

5.4.3 The container shall prominently display the words ‘CORROSIVE AND FLAMMABLE, HANDLE WITH CARE’.

5.4.4 The package labels shall be in compliance with IS 1260 (Part 1): 1973.

5.5 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5.6 Handling

5.6.1 For safe handling of EFs, inhalation of dusts or mists should be avoided, and contact with the skin should be minimized by thoroughly washing hands.

5.6.2 Consuming the substance in any form while handling the product should be avoided to prevent accidental ingestion.

5.6.3 Direct contact with the substance should be avoided by wearing appropriate protective gloves, clothing, and eye or face protection.

5.7 General

The packaging, labelling, handling, and transportation of EFs shall be in accordance with IS 18149:2023 due to its hazardous nature.

6 SAMPLING

Representative sample of the material shall be drawn in a nitrogen protected environment to prevent contamination and degradation.

Fig. 1: Label of Electrolyte Formulation

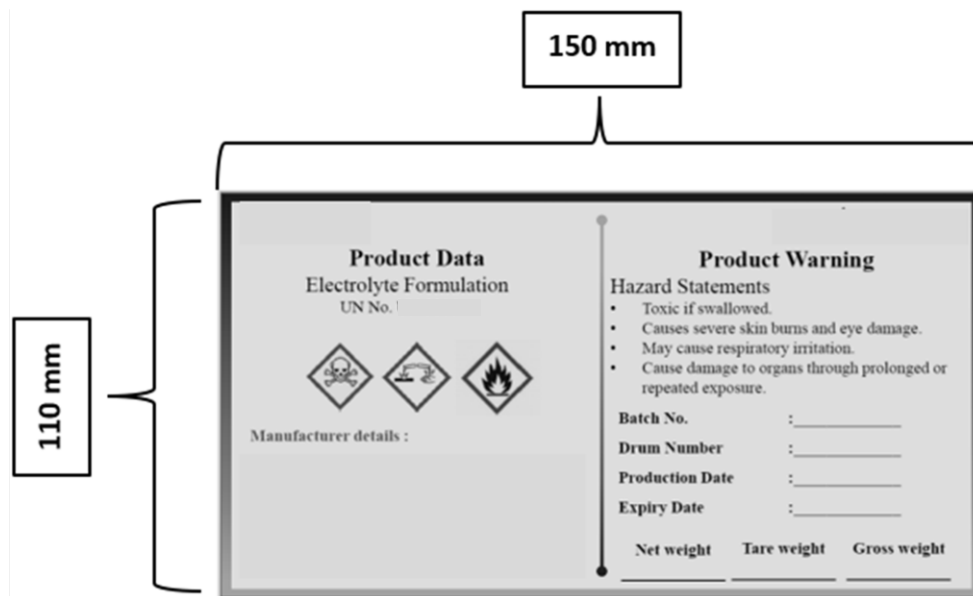


Fig. 2: Dangerous Goods Signs

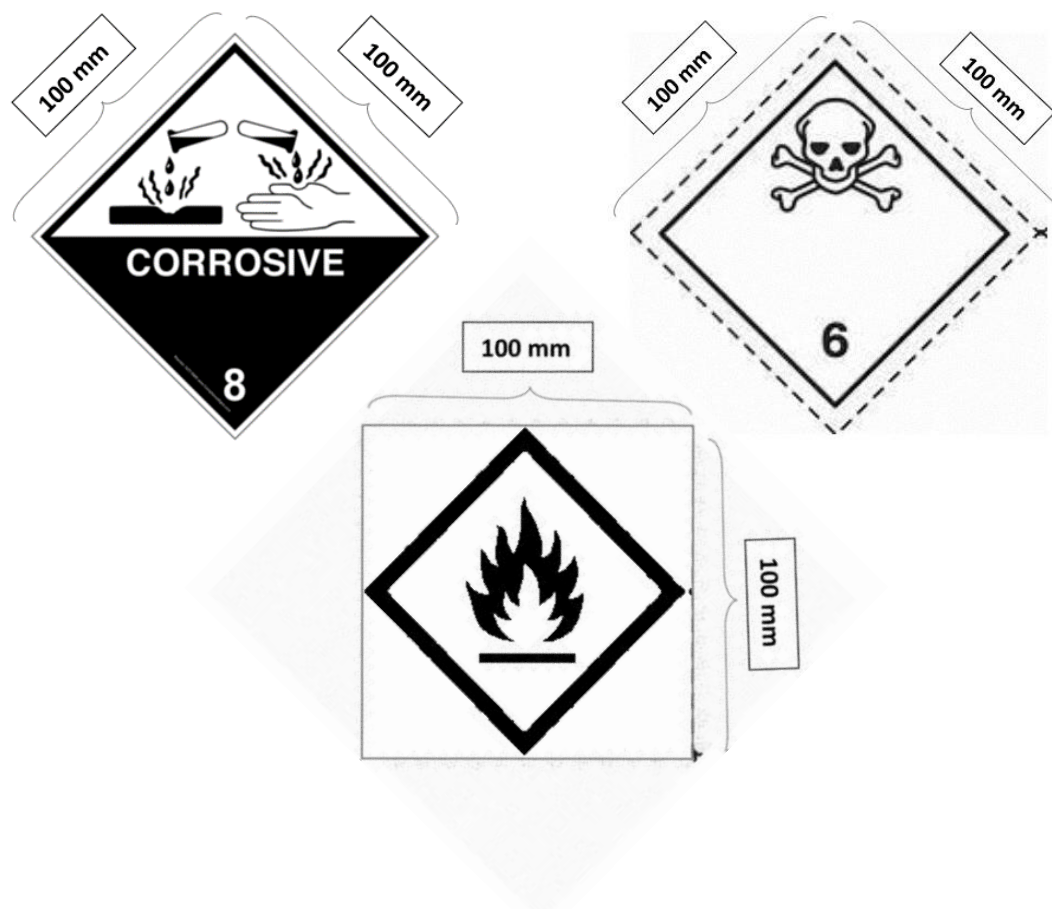


Table 1: Requirements for Electrolyte Formulations

Sl. No	Characteristic	Unit	Requirements	Nature of Testing	Methods of Test (Ref to Annex)
(1)	(2)	(3)	(4)	(5)	(6)
(i)	Colour	APHA	≤ 50	Quality	A
(ii)	Free Acidity (as HF)	ppm	≤ 50	Quality	B
(iii)	Moisture (Water Content) (H ₂ O)	ppm	≤ 20	Quality	C
(iv)	Density	gm/cc	1.2 to 1.5	Quality	D
(v)	Ion Conductivity	mS/cm	≥ 8	Quality	E
(vi)	Viscosity	mPa.s	≤ 8	Quality	F
(vii)	Sulfate	ppm	≤ 5	Quality	G
(viii)	Chloride	ppm	≤ 2	Quality	H
(ix)	Total Metal Impurities	ppm	≤ 10	Quality	I

Table 2: Packaging Requirements

Sr. No	Parameter	Unit	Specification	Test Method
1	Volume	L	200 ± 2	-
2	Cap type	-	Airtight	Visual
3	Color	-	Silver	Visual
4	Design type	-	Round	Visual
5	Material of Construction Body	-	Stainless Steel Grade SS304	Visual
6	Material of Construction Lid	-	Stainless Steel Grade SS304	Visual
7	Material of Construction Gasket	-	Silicone	Visual
8	Finish	-	Electro Polished	Visual
9	Overall Height (Body & Lid)	mm	1010 ± 5	Measuring Tape
10	Body Height	mm	1010 ± 5	Measuring Tape
11	Maximum Body Diameter	mm	487 ± 3	Measuring Tape
12	Lid Diameter	mm	150 ± 2	Measuring Tape
13	Nominal Weight (body, lid, clamp & gasket)	kg	54 - 55	Weighing Scale

ANNEX A

DETERMINATION OF COLOR

A-1 OUTLINE OF THE METHOD

This test method describes the procedure for measuring the color (APHA color or PCU) of an electrolyte formulation.

A-2 APPARATUS

A-2.1 UV Visible Spectrophotometer

A-3 PROCEDURE

The procedure laid down in ASTM D1209-05 shall be followed for determination of color.

A-4 DETERMINATION

As prescribed in ASTM D1209-05

ANNEX B

DETERMINATION OF FREE ACIDITY

B-1 OUTLINE OF THE METHOD

This test method describes the procedure for analyzing free acidity as HF in Electrolyte by non-aqueous titration.

B-2 APPARATUS

B-2.1 Analytical Balance (Readability 0.0001 g).

B-2.2 Burette (25 mL burette with 0.05 mL least count) and Conical Flask (250 mL).

B-3 REAGENTS

B-3.1 Acetonitrile (ACN) (AR grade)

B-3.2 Tripropylamine (TPA) (AR grade $\geq 98\%$)

B-3.3 Methyl Red indicator (2 % in Methanol)

B-3.4 Propylene carbonate (PC) (AR grade)

B-4 SPECIMEN

Electrolyte Formulations are a mixture of salts, solvents and additives.

B-5 PROCEDURE

B-5.1 TPA-PC standard solution (0.01 M)

preparation: Take 0.364 g (0.48 mL) of TPA in 250 ml volumetric flask and makeup with PC up to the mark, where density of TPA is 0.754 g/mL.

B-5.2 Cleaning of conical flask: Rinse the interior of conical flask twice; each time with about 10 mL

ACN. The rinsed ACN is to be discarded. Further add 10 mL ACN into the same flask and add 2-3 drops of methyl red indicator.

If the acetonitrile solution turns yellow, it indicates that the conical flask is qualified for use. If not, repeat rinsing step.

B-6 DETERMINATION OF OUTCOME

B-6.1 Take 10 to 20 mL of ACN in the cleaned conical flask, weigh accurately about 10-15 g of the test sample into the same flask and add 2-3 drops of methyl red indicator. If the solution shows orange color, it indicates absence of HF. If the solution shows red color, it indicates that HF is present. If HF is present, follow next step.

B-6.2 Titrate the test sample with 0.01 M TPA-PC standard solution. The titration end point is when the color of the solution changes from red to yellow (and/or orange). Record the consumed volume of the TPA-PC standard solution.

$$\text{Free Acidity as HF in ppm} = \frac{C \times B.R. \times 20.01 \times 1000}{W}$$

where,

C = Concentration of TPA-PC solution

$B.R.$ = Titrated Volume of 0.01 M TPA-PC solution

W = Weight of Sample (g)

20.01 = Molar Mass of HF

ANNEX C

DETERMINATION OF MOISTURE

C-1 OUTLINE OF THE METHOD

This test method describes the procedure for analyzing the moisture (presence of water) in an Electrolyte using coulometric Karl Fischer (KF) Titration.

C-2 APPARATUS

C-2.1 Analytical Balance (Readability 0.0001 g)

C-2.2 KF Coulometer

Electrode 1: Generator Electrode with diaphragm

Electrode 2: Double Pt wire Indicator Electrode

C-2.3 Plastic BD Syringe - With long needle of approximately 5 inches - 5 ml

C-2.4 Glove Box - with nitrogen atmosphere (moisture < 5 ppm)

C-3 REAGENTS

KF Reagents: Alcohol free anolyte and catholyte reagents for coulometry.

C-4 PROCEDURE

C-4.1 The analytical procedure shall be carried out in a glove box. Transfer about 100 mL of anolyte KF

reagent in glass assembly of the coulometric cell and one vial of 5 mL of catholyte KF reagent in Generator Electrode with diaphragm.

Activate the instrument and condition the device.

C-4.2 Draw a sample into a BD syringe up to 2 mL and tare it on analytical balance.

Once the conditioning is complete, initiate the titration, which will be indicated by a 10 second timer.

C-4.3 Inject about 0.5 to 1.0 mL (0.6 to 1.23 g) of the sample from the syringe into the KF coulometer and reweigh the syringe using the analytical balance.

Enter the sample weight (gms) in coulometric device. The determination curve will be visible on display.

C-4.4 Upon completion of the coulometric titration, the result will appear on device screen.

C-5 COULOMETER PARAMETERS

The Start Criteria i.e., "Drift" is 20 µg/min. for conditioning.

The Stop Criteria i.e., "Relative Drift" is 5 µg/min.

ANNEX D

DETERMINATION OF DENSITY

D-1 OUTLINE OF THE METHOD

This test method describes the procedure for measuring the density of an Electrolyte.

D-2 APPARATUS

Density Meter: A density meter based on oscillating U tube principle with accuracy of 0.001 g/cm³ and resolution of 0.0001 g/cm³ is to be used.

D-3 REAGENTS

D-3.1 Water – Deionized Water Type 1 (as per ASTM D 1193-99)

D-3.2 Acetone - (AR Grade)

D-4 SPECIMEN

Electrolyte Formulations are a mixture of salts, solvents and additives.

D-5 PROCEDURE

While the operating procedures of different instrument makers could be different, it is necessary to rinse clean with acetone and do a water check before testing the sample.

D-5.1 Press the filling pump of the density meter to empty the measuring cell and fill the measuring cell with acetone. Pump the acetone through the whole measuring system two times and empty the measuring cell for avoiding any contamination.

D-5.2 Perform a water check before starting the measurements to verify that the instrument is measuring with adequate accuracy.

Rinse the system with water at temperature between 20 - 25 °C. Do the Water Check with Type 1 water. After the procedure has finished, the water check result will be displayed.

Water Density Limit 1.0000 ± 0.01.

D-5.3 Rinse the measuring cell with the sample, at least three to four times, for density measurement. Fill the measuring cell with sample for density measurement by pressing and releasing filling pump with no air bubbles in measuring cell.

D-6 DETERMINATION OF OUTCOME

Results of density and temperature will be displayed on the screen.

ANNEX E

DETERMINATION OF IONIC CONDUCTIVITY

E-1 OUTLINE OF THE METHOD

This method is conducted to test the quality aspect of the electrolyte formulations through the measurement of ionic conductivity.

E-2 APPARATUS**E-2.1 Conductivity Meter**

E-2.2 Conductivity Electrode: The electrode shall have a range atleast from 0.01 to 50 mS/cm and operating temperature between 0 to 50 °C.

E-3 REAGENTS

E-3.1 Water: Deionized Water - Type 1 (as per ASTM D 1193-99)

E-3.2 Calibration Standard: The specified limit for this calibration standard is 12.88 mS/cm, with an allowable variation of $\pm 1.5\%$. The temperature of shall be maintained at 25 °C.

E-4 PROCEDURE

E-4.1 Ensure the calibration of conductivity meter is as per instrument supplier's manual. The calibration is to be done before every measurement of sample / set of samples.

E-4.2 Rinse the electrode with Deionized Water Type-1 to remove any impurities. Gently shake off the excess water droplets.

E-4.3 Immerse the probe into the sample to fully contact the electrode surface.

E-4.4 Upon stabilization of the reading, record it as displayed.

E-5 DETERMINATION OF OUTCOME

The result will be displayed on the screen of the instrument.

ANNEX F

DETERMINATION OF VISCOSITY

F-1 OUTLINE OF THE METHOD

This method is conducted to test the quality aspect of the electrolyte formulations through the measurement of viscosity.

F-2 APPARATUS

Viscometer: with PTD 100 Cone-Plate or any suitable instrument capable of measuring a minimum low viscosity of 1 mPa.s

F-3 REAGENTS

F-3.1 Water: Deionized Water - Type 1 (as per ASTM D1193-99)

F-3.2 Acetone

F-4 PROCEDURE

To be carried out as per the supplier's instrument manual.

ANNEX G

DETERMINATION OF ANIONIC IMPURITIES

G-1 OUTLINE OF THE METHOD

The chloride ion and sulfate ion, in Electrolyte Formulations, are separated by ion chromatographic column; detected by conductivity detector. The content of detected anion is calculated by external standard method.

G-2 APPARATUS

G-2.1 Ion chromatographs: An analytical system to separate and detect ions in a solution.

G-2.2 Anion exchange column: A resin based chromatographic column that can separate anions.

G-2.3 Conductivity detector: A detector that measures ionic conductivity of solutions.

G-2.4 Membrane Filter: 0.45µm Nylon 66 membrane filter

G-2.5 Plastic BD Syringe: 10 mL

G-2.6 Syringe filter: 0.2µm Nylon non-sterile syringe filter

G-3 REAGENTS

G-3.1 Water: Deionized Water - Type 1 (as per ASTM D1193-99)

G-3.2 Chloride standard solution: 1000 ppm

G-3.3 Sulfate standard solution: 1000 ppm

G-3.4 Sodium Carbonate: Analytical Reagent (AR) Grade

G-3.5 Sodium Bicarbonate: AR Grade

G-3.6 Acetonitrile: AR Grade

G-3.7 Regeneration solution: As per Ion Chromatograph supplier's manual

G-4 PROCEDURE**G-4.1 Mobile Phase Preparation:**

Eluent: 3.2 mmol/L Na₂CO₃, 1.0 mmol/L NaHCO₃ in Acetonitrile + water mixture (25 : 75 v/v).

Other eluents such as KOH may also be used depending on Instrument manufacturer's manual.

Preparation of Eluent: Weigh 678 mg Na₂CO₃ and 168 mg of NaHCO₃ in 2000 mL volumetric flask and add 1500 mL Deionized Water Type 1 to dissolve in the same flask. Add 500 mL Acetonitrile to make it a 2000 mL solution.

Filter the mobile phase with 0.45-micron Nylon 66 membrane filter. Sonicate for 10 minutes.

Diluent: DI water-Type 1 (ASTM D1193-99)

Blank: DI water-Type 1 (ASTM D1193-99)

G-4.2 Rinsing Solution: DI water Type 1 (ASTM D1193-99)

G-4.3 Standard Preparation

Standard Solution: Prepare chloride and sulphate working standard solutions from certified standard materials as shown in Table 3.

Condition the column for 30 minutes as instructed in the Instrument supplier's manual. The conductivity of mobile phase shall be limited to below 1 µs/cm. Inject Blank as Deionized Water Type 1 followed by five different linearity level of standard injections and check the correlation coefficients, which should not be less than 0.999.

G-4.4 Sample Preparation:

Weigh accurately about 0.5g of sample in 50 mL volumetric flask and add 10-20 mL Deionized Water Type 1. Sonicate to dissolve and make up to volume with Deionized Water. Shake well.

Filter the solution through 0.45-micron nylon membrane filter, this sample to be used for analysis.

G-5 DETERMINATION OF OUTCOME

Inject appropriate volumes of the working standard solutions and the sample solution, according to the instrument's operation procedure. Use the measured anion concentration, of the working standard solutions, as the abscissa, and the corresponding peak areas as the ordinate, to draw the working curve. According to the peak area of the tested solution, check the concentration of each anion (ppm), on the working curve.

The outcome will be shown on the instrument's display for easy reference and analysis.

Table 3

Sr No.	Standard Cl (ppm)	Standard SO ₄ ²⁻ (ppm)
01	0.1	0.5
02	0.5	1.0
03	1.0	2.0
04	2.0	5.0
05	5.0	10.0

ANNEX H

DETERMINATION OF ELEMENTAL IMPURITIES

H-1 OUTLINE OF THE METHOD

This test method describes the procedure for determining elemental impurities (Calcium, Cadmium, Chromium, Copper, Iron, Lead, Magnesium, Nickel, Potassium) in Electrolyte Formulation.

H-2 APPARATUS

H-2.1 Inductively Coupled Plasma Mass Spectrometer (ICP – MS).

H-2.2 Volumetric flasks (50 ml)

H-2.3 Tarson tube (50 ml)

H-2.4 Micropipette (10-100 µl, 250 µl, 100-1000 µl)

H-2.5 Microtips

H-2.6 Analytical balance (0.1 mg readability)

H-3 REAGENTS

H-3.1 Water - Deionized Water - Type 1 (as per ASTM D1193-99)

H-3.2 23 multi-element standard (1000 ppm)

H-3.3 Scandium, Germanium and Terbium (1000 ppm) internal standard

H-3.4 Ultrapure / Suprapur Nitric Acid (HNO₃)

H-4 PROCEDURE

H-4.1 Elemental Standard Stock Solution: Take 50 µL of 1000 ppm 23 multi elements standard solution in 50 ml Tarson tube and make up with DI water to get 1 ppm Elemental Standard Stock Solution.

H-4.2 Internal Standard Stock Solution: Take 50 µl of 1000 ppm of each (Sc, Ge, Tb) in 50 ml Tarson tube and make up with DI water to get 1 ppm internal standard stock solution.

H-4.3 Calibration Standard Preparation: To prepare 0.001, 0.002, 0.005, 0.008, 0.010, 0.012, 0.015, and 0.025 ppm solution from 1 ppm Elemental standard stock Solution and 1 ppm Internal Standard Stock Solution, as represented in Table 4.

H-4.4 Sample Preparation: Take about 0.1 gram accurately weighed of sample in 50 ml Tarson tube and dissolve it with 500 µl of ultrapure HNO₃ and 1000 µl of the 1 ppm Internal Standard stock solution. Finally, make up to mark with Deionized water. (Refer Table 5).

Run the sample as per the manufacturer instrument manual.

H-5 DETERMINATION OF OUTCOME

The outcome will be shown on the instrument's display for easy reference and analysis.

Table 4

STD Solution (ppm)	Volume of Elemental Std Stock Solution	Volume of IS Stock Solution	Volume of HNO ₃ Ultrapure (µl)	Final Volume (ml)
0.001	50	1000	500	50
0.002	100	1000	500	50
0.005	250	1000	500	50
0.008	400	1000	500	50
0.010	500	1000	500	50
0.012	600	1000	500	50
0.015	750	1000	500	50
0.025	1250	1000	500	50

Table 5

Electrolyte Formulation (gm)	Volume of IS Stock solution-C (µl)	Volume of HNO ₃ Suprapur/ Ultrapure (µl)	Final Volume (ml)
2.5	1000	500	50

Table 6: List of Abbreviations and Symbols

Full Form	Abbreviation / Symbol
Degree Celsius	°C
Annexure	annex
American Public Health Association	APHA
Centimeters	cm
Column	col
Deionized Water	DI water
Figure	Fig
Grams	gm
Hydrofluoric acid	HF
Hour	hr
Liters	L
Lithium Hexafluorophosphate	LiPF ₆
Milliliter	mL
Millimeter	mm
Millimole	mmol
Platinum cobalt unit	PCU
parts per million	ppm
Polytetrafluoroethylene	PTFE
Reference	Ref
Micro Siemens	μs
Microliters	μl