

भारतीय मानक मसौदा
लिथियम हेक्साफ्लोरोफॉस्फेट — विशिष्टि

(IS XXXX)

Draft Indian Standard

Lithium Hexafluorophosphate — Specification

(IS XXXX)

(Not to be reproduced without the permission of BIS or used as an Indian Standard)

ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 30 March 2025

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(Formal clauses will be added later)

Lithium Hexafluorophosphate (LiPF_6) is a salt in the form of a white crystalline powder, used in production of electrolytes for lithium-ion batteries (LIBs). Liquid electrolyte solutions are crucial in LIBs, acting as carriers of lithium ions between the cathode and anode; hence, high purity and battery-grade electrolyte solutions are essential for optimal performance. The most common LIB electrolytes are derived from solutions of lithium salts like LiPF_6 in non-aqueous solvents, such as alkyl carbonates or solvent blends.

LiPF_6 is produced by the reaction of phosphorus pentachloride with hydrogen fluoride and lithium fluoride. Specifically, solutions of LiPF_6 in carbonate blends (e.g., ethylene carbonate, dimethyl carbonate, diethyl carbonate, and/or ethyl methyl carbonate) with additives like fluoroethylene carbonate and vinylene carbonate, serve as electrolytes in lithium-ion batteries.

LiPF_6 -based electrolyte is suitable for all LIB chemistries, including Lithium Cobalt Oxide (LCO), Lithium Manganese Oxide (LMO), Lithium Nickel Manganese Cobalt Oxide (NMC), Lithium Nickel Cobalt Aluminum Oxide (NCA), Lithium Iron Phosphate (LFP), and Lithium Manganese Iron Phosphate (LMFP), used in various applications like 3C digital devices, power tools, electric vehicles, and energy storage systems. Since the commercialization of LIBs, LiPF_6 has been a leading conducting salt in non-aqueous liquid electrolytes due to its superiority over other competitors. Its unique abilities include passivating the aluminum (Al) current collector of the cathode, excellent anti-oxidation durability for 4-V-class cathodes, and high ionic conductivities ($\sim 10 \text{ mS/cm}$ at room temperature).

Additionally, LiPF_6 with carbonate solvents can be electrochemically reduced before the intercalation of lithium ions (Li^+) into graphite layers during the first cycle, forming relatively stable Li^+ ion conducting; but electronically insulating solid-electrolyte-interphase (SEI) films on the graphite anode. This prevents further decomposition of the electrolyte on the electrode in subsequent cycles. This results in enhanced battery life and reliability of products that consumes EV chemicals.

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.

Draft Indian Standard
LITHIUM HEXAFLUOROPHOSPHATE — SPECIFICATION

1 SCOPE

This Indian Standard outlines the requirements, testing methods, and application guidelines for Lithium Hexafluorophosphate (LiPF₆), a commonly used lithium salt in the electrolyte solutions of lithium-ion batteries.

This standard applies to LiPF₆ used in various applications, including electrolyte solutions for lithium ion batteries, energy storage systems, and portable electronic devices.

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 No.</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 1260 (Part 1): 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (<i>first revision</i>)
IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater
(Part 2) : 2019/ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP – OES) (<i>first revision</i>)
(Part 65) : 2022/ 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (<i>first revision</i>)
IS 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 hydrofluoric acid (HF) in a sample.

3.2 ICP-MS — Inductively coupled plasma mass spectrometry testing method for elemental impurities.

3.3 Ion Chromatography

Methodology used for separation and detection of anions like chloride, sulphate, nitrate, etc and organic anions.

4 REQUIREMENTS**4.1 Description**

4.1.1 The material must be a white crystalline powder with a solid appearance. Any discoloration is unacceptable, as it indicates contamination or degradation, potentially impairing the battery's performance.

4.1.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in col 6 of Table 1.

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

5.1.1 The material shall be packed in containers in the form of drums, having appropriate capacity to accommodate the required amount of the material, and having dimensions as shown 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 airtight to prevent moisture ingress, with a silicone gasket to provide an effective seal.

5.1.4 The drums shall be filled with nitrogen (or argon) with at least 99.99 percent purity, and maintained under controlled conditions, with a pressure range of 0.5 kg/cm² to 1.0 kg/cm² (0.05 to 0.10 MPa), to ensure stability of the contents.

5.2 Transportation

The transportation of LiPF₆ must adhere to stringent regulations due to its hazardous nature. If required by a purchaser, a UN certification of SS Drum shall be conducted. A Safety Data Sheet (SDS) of LiPF₆ shall be included with each shipment for reference in case of an emergency.

5.3 Storage

Due to the hazardous nature of lithium hexafluorophosphate, its storage requires stringent precautions. The material should be stored in a dry, cool, and well-ventilated environment to prevent degradation and ensure safety.

5.4 Marking

5.4.1 The label on the packages of the material shall indicate the name of the chemical; the CAS No.; the UNNo.; and details of manufacture.

5.4.2 The container shall prominently display the words 'CORROSIVE, HANDLE WITH CARE'.

5.4.3 The label shall also display:

- a) Dangerous goods signs (as shown in fig. 2);
- b) Hazard statements relating to the product;
- c) Production date;
- d) Expiry date;
- e) Drum number;
- f) Batch number; and
- g) Mass of the material.

Fig. 1 shows the details to be displayed in the label.

Fig. 3 shows the safety data sheet label to be displayed on the packages.

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

5.4.5 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 LiPF₆, contact with eyes, skin, and clothing should be avoided. The material should be used under a chemical fume hood and glove box with personal protective equipment. Ingestion and inhalation should be prevented.

5.6.2 Handling should take place in a well-ventilated area, and thorough washing should be done after handling. Contaminated clothes should be removed and washed before reuse.

5.7 General

The packaging, labelling, handling, and transportation of LiPF₆ shall be in accordance with IS 18149 due to its hazardous nature.

6 SAMPLING

6.1 The representative samples of the material shall be drawn in a nitrogen (or argon) protected environment to prevent contamination and degradation.

6.2 The sampling bottles shall be made of high-density polyethylene (HDPE) and must be dried prior to use to ensure that no moisture is present. Proper drying procedures should be followed to maintain the integrity of the samples.

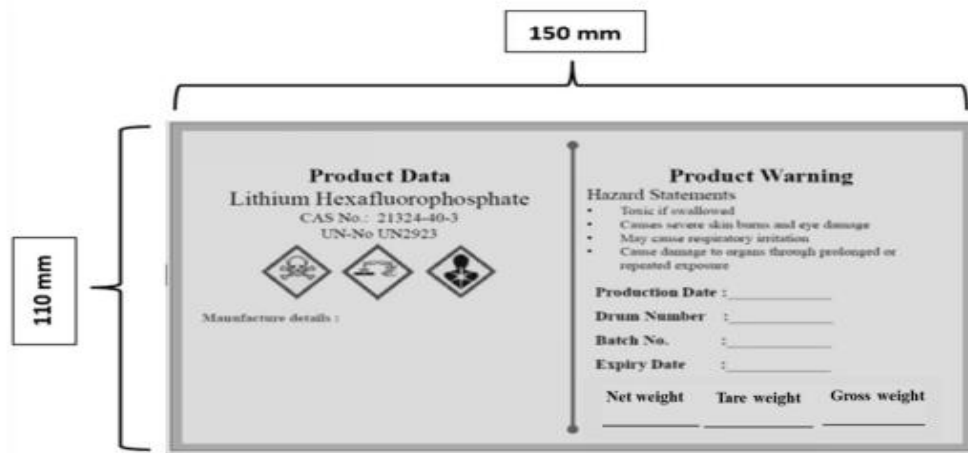


FIG 1 LABEL OF LITHIUM HEXAFLUOROPHOSPHATE



FIG 2 DANGEROUS GOODS SIGNS

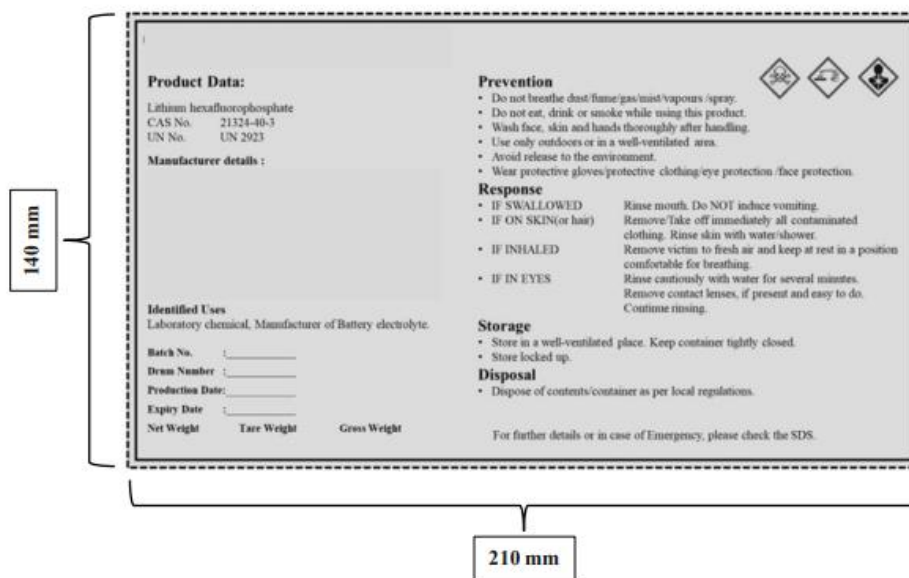


FIG. 3 SAFETY DATA SHEET LABEL

Table 1 Requirements for Lithium Hexafluorophosphate

(Clause 4.1.2)

SI No.	Characteristic	Category	Requirements	Methods of Test (Ref to Annex)
(1)	(2)	(3)	(4)	(5)
(i)	Purity, percent, <i>Min</i>		99.90	A
(ii)	Acid (as HF), ppm, <i>Max</i>		90	B
(iii)	Insoluble, ppm, <i>Max</i>		300	C
(iv)	Sulphate (as SO ₄ ²⁻), ppm, <i>Max</i>		5	D
(v)	Chloride (as Cl), ppm, <i>Max</i>		2	D
(vi)	Moisture, ppm, <i>Max</i>		20	E
(vii)	Aluminium (as Al), ppm, <i>Max</i>		2	F
(viii)	Cadmium (as Cd), ppm, <i>Max</i>	• Digital Devices	1	F
(ix)	Calcium (as Ca), ppm, <i>Max</i>	• Energy Storage System	2	F
(x)	Chromium (as Cr), ppm, <i>Max</i>	• EV Batteries	1	F
(xi)	Copper (as Cu), ppm, <i>Max</i>		1	F
(xii)	Iron (as Fe), ppm, <i>Max</i>		3	F
(xiii)	Lead (as Pb), ppm, <i>Max</i>		1	F
(xiv)	Magnesium (as Mg), ppm, <i>Max</i>		1	F
(xv)	Nickel (as Ni), ppm, <i>Max</i>		1	F
(xvi)	Potassium (as K), ppm, <i>Max</i>		2	F
(xvii)	Sodium (as Na), ppm, <i>Max</i>		2	F

Table 2 Packaging Requirements

(Clause 5.1.1)

SI No.	Parameter	Specification
(1)	(2)	(3)
i)	Nominal Volume, L	112 ± 1
ii)	Cap type	Airtight
iii)	Color	Silver
iv)	Design type	Round
v)	Material of Construction Body	Stainless Steel Grade SS304
vi)	Material of Construction Lid	Stainless Steel Grade SS304
vii)	Material of Construction Gasket	Silicone
viii)	Finish	Electro Polished

ix)	Overall Height (Body & Lid), mm	1 008 ± 5
x)	Body Height, mm	1 115 ± 5
xi)	Maximum Body Diameter, mm	504 ± 3
xii)	Lid Diameter, mm	200 ± 2
xiii)	Nominal Weight (body, lid, clamp and gasket), kg	54 - 55

ANNEX A
DETERMINATION OF PURITY

(Table 1)

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in test.

NOTE – ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 OUTLINE OF THE METHOD

The purity of the material is determined by eliminating all forms of impurities such as acid value, insoluble content, impurities in form of sulphates and chlorides, moisture content along with metal impurities that may be present in lithium hexafluorophosphate.

A-2.1 Procedure

Subtract all measured impurity contents from 100 to obtain the purity in percentage.

A-2.2 Calculation

$$\text{Purity percent} = 100 - \{I_1 + I_2 + I_3 + I_4 + I_5\} \dots\dots\dots \text{(formula 1)}$$

where

I_1 = HF impurity (in percent);

I_2 = Insoluble content (in percent);

I_3 = sulphate and chloride impurities (in percent);

I_4 = moisture content (in percent); and

I_5 = sum of metallic impurities (Al, Cd, Ca, Cr, Cu, Fe, Pb, Mg, Ni, K, Na in percent).

ANNEX B
DETERMINATION OF ACID (AS HF)

(Table 1)

B-1 OUTLINE OF THE METHOD

Use a micro-burette. Use bromothymol blue as an indicator. Use standard titration solution of sodium hydroxide, to titrate the acid in the specimen.

B-1.1 Apparatus

B-1.1.1 Beaker — 250 ml Polyethylene or other corrosion-resistant material

B-1.1.2 Analytical balance — 0.1 mg readability

B-1.1.3 Magnetic stirrer

B-1.1.4 Glove box — filled with inert gas

B-1.1.5 Ice cube maker

B-1.1.6 Micro burette — with least count of 0.01 ml or 0.02 ml

B-1.2 Reagents

B-1.2.1 Sodium Hydroxide Standard Titration Solution — 0.01 N

B-1.2.2 Ice Water

Add an appropriate amount of ice cubes to the DI water. Use it when the water temperature is ≤ 4 °C. The DI water and water used for making ice should be of Type 1 as per IS 1070.

B-1.2.3 Bromothymol Blue Indicator Solution

Weigh 0.1 gm of the indicator into a 250 ml glass beaker. Add 4 ml of 0.05 N NaOH and 5 ml of methanol (AR grade) and mix properly. Warm if necessary to dissolve. After complete dissolution add 50 ml methanol and make up the volume up to 250 ml volumetric flask with DI water.

B-1.3 Procedure

B-1.3.1 Take the sample under the protection of nitrogen (or argon) in glove box. Weigh accurately about 10 - 15g of the sample, quickly pour the sample into a polyethylene beaker containing about 150g of ice water. The water temperature should be in between 0 °C to 5 °C. Add 3 to 5 drops of bromothymol blue indicator solution and titrate with 0.01N sodium hydroxide with continuous stirring at high speed until it turns blue. At the same time carry out a blank test. The type and amount of reagents added into the blank test solution is the same as that of the test solution, except that no specimen is added.

B-1.4 Calculation

The acid content is calculated according to formula (2):

$$\text{HF (ppm)} = \left[\frac{M(V - V_0) \times 20.01 \times 100}{W \times 1000} \right] \times 10\,000 \dots \text{formula (2)}$$

where

V = the value of the volume of the standard titration solution of sodium hydroxide, which is consumed by the sample titration test solution, in millilitres (mL);

V_0 = the value of the volume of the standard titration solution of sodium hydroxide, which is consumed by titrating the blank test solution, in millilitres (mL);

20.01 = equivalent weight of HF;

M = the concentration of sodium hydroxide (N); and

W = weight of the sample(g).

ANNEX C**DETERMINATION OF INSOLUBLES**

(Table 1)

C-1 OUTLINE OF THE METHOD

This test method describes the procedure for analysis of Insolubles in Lithium Hexafluorophosphate.

C-1.1 Apparatus

C-1.1.1 Analytical Balance — 0.1 mg readability

C-1.1.2 Poly Propylene (PP)/Perfluoro Alkoxy (PFA) Beaker — 250 ml

C-1.1.3 Magnetic Agitator

C-1.1.4 Vacuum Pump-Connected Suction Filter Device

C-1.1.5 Micro Porous Hydrophobic Poly Tetra Fluoro Ethylene (PTFE) 0.45µm Filter

C-1.1.6 Glove Box — filled with inert gas

C-1.1.7 Desiccator

C-1.1.8 Aluminium Dish, Ultrasonic Bath, Hot Air Oven

C-1.2 Reagents

C-1.2.1 Dimethyl carbonate (DMC) with moisture content < 20 ppm

C-1.3 Procedure

C-1.3.1 Take the micro porous Hydrophobic PTFE 0.45 µm filter paper, which has been pre-dried 12 h at 110 °C. Weigh 10 g sample, accurate to 0.0002 g, in 250 ml PP/PFA beaker inside the Glove Box. Slowly add 125 gm dimethyl carbonate

Take above sample on magnetic stirrer and stir up to 10 min at constant 500 rpm to dissolve. Use stainless steel tweezers to place the weighed micro porous Hydrophobic PTFE 0.45µm filter paper which has been pre-dried at 110 °C to constant weight (m_1), in a vacuum pump-connected suction filter device.

C-1.3.2 Pour the stirred solution into the suction filter device, start the vacuum pump until the solution is completely filtered. Then use a total of 25 ml dimethyl carbonate (DMC), to wash the beaker 3 times; wash the filter device 3 times.

C-1.3.3 Remove the micro porous hydrophobic PTFE 0.45µm and dry in oven at 110 °C for 2 h. Then take it out and place it in a desiccator, cool to room temperature and then weigh again (m_2).

C-1.4 Calculation

The determination of insolubles is obtained as a result of the following calculation according to formula (3):

$$\text{Insoluble substance (ppm)} = \left[\frac{m_2 - m_1}{m} \right] \times 10^6 \dots \dots \text{formula (3)}$$

where

m_2 = weight of PTFE 0.45µ filter paper after filtration and drying (in gm);

m_1 = weight of PTFE 0.45µ filter paper before filtration (in gm); and

m = weight of sample in gm.

ANNEX D

DETERMINATION OF SULFATE AND CHLORIDE CONTENT

(Table 1)

D-1 OUTLINE OF THE METHOD

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

D-1.1 APPARATUS

D-1.1.1 Ion Chromatograph — an analytical system to separate and detect ions in solution

D-1.1.2 Anion Exchange Column — a resin based chromatographic column that can separate anions

D-1.1.3 Conductivity Detector — a detector that measures ionic conductivity of solutions

D-1.2 Reagents

D-1.2.1 Water- Deionized Water — Type 1, prepared according to IS 1070

D-1.2.2 Chloride Standard Solution

D-1.2.3 Sulfate Standard Solution

D-1.2.4 Sodium Carbonate (AR Grade)

D-1.2.5 Sodium Bicarbonate (AR Grade)

D-1.2.6 Acetonitrile (AR Grade)

D-1.2.7 Regeneration Solution — as per ion chromatograph supplier's manual

D-1.3 Procedure

D-1.3.1 Mobile Phase Preparation

D-1.3.1.1 Eluent — 3.2 mmol/L Na₂CO₃, 1.0 mmol/L NaHCO₃ and 25 percent v/v acetonitrile (AR grade) + water mixture.

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

D-1.3.1.2 Diluent — DI Water-Type 1 (IS 1070)

D-1.3.1.3 Blank — DI Water-Type 1 (IS 1070)

D-1.3.1.4 Rinsing Solution — Deionized water Type 1 (Rinsing Solution)

D-1.3.3 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

Table 3

(Clause D-1.3.3)

SI No.	Standard Cl (ppm)	Standard SO ₄ ²⁻ (ppm)
(1)	(2)	(3)
i)	0.1	0.5
ii)	0.5	1.0
iii)	1.0	2.0
iv)	2.0	5.0
v)	5.0	10.0

D-1.3.4 Sample Preparation

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

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

Note: The sample is weighed inside a glove box.

D-1.4 Determination

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 clearly shown on the instrument's display for easy reference and analysis.

ANNEX E**DETERMINATION OF MOISTURE**

(Table 1)

E-1 OUTLINE OF THE METHOD

Moisture is the presence of water in the chemical; tested using Karl Fischer Titration

E-1.1 Apparatus

E-1.1.1 Analytical Balance — Readability 0.1 mg

E-1.1.2 KF Coulometer

- a) Electrode 1 — Generator Electrode without diaphragm
- b) Electrode 2 — Double -Pt. Wire Electrode for coulometer

E-1.1.3 Glove Box — filled with inert gas

E-1.2 Reagents

E-1.2.1 KF Reagent — Hydranal Coulomat-AK

E-1.3 Procedure

E-1.3.1 The analytical procedure shall be carried out in a glove box. Transfer about 100 mL of KF reagent into glass assembly of KF coulometer, ensuring that both the generator and indicator electrodes are immersed in the KF reagent. Activate the instrument and condition the device.

E-1.3.2 Weigh about 1 gm of the specimen, accurate to 0.001 g. Put the specimen into the measuring KF cell in stirring solution. Input the exact weight of the sample into the instrument software. Stir evenly. Once the conditioning is complete, initiate the titration, which will be indicated by a 10 second timer.

E-1.3.3 Upon completion of the titration, the result will appear on screen. Record the result.

ANNEX F

DETERMINATION OF ELEMENTAL IMPURITIES

(Table 1)

F-1 General

Two methods are prescribed for determining aluminium, calcium, cadmium, chromium, copper, iron, lead, magnesium, nickel, potassium and sodium, namely, ICP-OES method as prescribed at F-2 and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

F-2 Determination of Aluminium, Calcium, Cadmium, Chromium, Copper, Iron, Lead, Magnesium, Nickel, Potassium and Sodium by Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) Method

F-2.1 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.

F-2.2 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 4. 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 4 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 4 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(Clauses F-2.2 and F-2.4)

SI No.	Element	Wavelength (nm)	Approximately Achievable Limits		Interfering Elements
			Radial Viewing (µg)	Axial Viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr
ii)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0,4	25	V, Zr
		422.673	-	-	V, Mo, Zr
iii)	Cd	214.441	1	0,9	As, Cr, Fe, Sc, Sb
		226.502	4	0,2	As, Co, Fe, Ni
		228.802	2	0,5	As, Co, Sc
iv)	Cr	205.559	1	5	Be, Fe, Mo, Ni, Ti
		267.719	4	2	Mn, P, V
		283.563	(10)	(2)	Fe, Mo, V, W
		284.324	(10)	-	Fe
v)	Cu	324.754	9	2	Cr, Fe, Mo, Ti
		327.396	4	3	Co, Ti
vi)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	9	-	-
vii)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)	(20)	Cr, Fe
viii)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr
ix)	Ni	221.648	10	2	Si
		231.604	15	2	Co, Sb
x)	K	766.490	66	20	Ar, Ba, Mg
		769.896	-	(230)	Ba
xi)	Na	330.237	(20)	300	Zn
		588.995	20	200	Ar, V
		589.592	93	20	Ba

F-2.3 Reagents and Solutions**F-2.3.1. Nitric Acid (65 percent) Suprapure****F-2.3.2 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 µg/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.

F-2.3.3 *Standard Solution*

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/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.

F-2.3.4 *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.

F-2.3.5 *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.

F-2.4 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 4.

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.

F-2.5 Procedure

F-2.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**F-2.3.5**). 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.

F-2.5.2 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.

F-2.6 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).