
पैकेजबंद प्राकृतिक मिनरल जल —
विशिष्टि

(तीसरा पुनरीक्षण)

Packaged Natural Mineral Water —
Specification

(Third Revision)

ICS 13.060.20

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FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Drinking Water and Carbonated Beverages Sectional Committee had been approved by the Food and Agriculture Division Council.

This standard was originally published in 1992 and in the preparation of this standard assistance was derived from the EEC Directive, 80/778/EEC 'Council directive relating to the quality of water intended for human consumption'.

This standard was subsequently revised in 1998 and 2005. The first revision of the standard was undertaken to delete provisions of fortified mineral water which were covered in the earlier version, to align with the revised Codex Standard for natural mineral water and to include hygienic practices in line with Codex (CAC/RCP 33-1985) 'Code of hygienic practice for collecting, processing and marketing of natural mineral waters'. The second revision was undertaken to incorporate five amendments along with the technological developments, check list for hygienic requirements during manufacturing packaged natural mineral water and consumer requirements.

This revision has been undertaken to update the test methods standards prescribed particularly with respect to incorporation/addition of advanced instrumental test methods for determination of various parameters such as inductively coupled plasma mass spectrometry (ICP-MS), liquid chromatography of ions and inductively coupled plasma optical emission spectrometry (ICP-OES), apart from incorporation of 11 amendments issued to the standard.

Separate standards have been formulated for packaged drinking water (other than packaged natural mineral water) (*see* IS 14543) and drinking water (*see* IS 10500).

The composition of the Committee responsible for the formulation of this standard is given in [Annex P](#).

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

*Indian Standard***PACKAGED NATURAL MINERAL WATER — SPECIFICATION***(Third Revision)***1 SCOPE**

This standard prescribes the requirements, methods of sampling and test for natural mineral waters offered for sale in packaged form for human consumption.

NOTE — It does not apply to natural mineral water sold or used for other purposes.

2 REFERENCES

The standards listed in [Annex A](#) 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 edition of these standards.

3 DEFINITIONS

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

3.1 Natural Mineral Water — Water clearly distinguishable from ordinary drinking water because:

- a) it is obtained directly from natural or drilled sources from underground water-bearing strata for which all possible precautions should be taken within the protected perimeters to avoid any pollution of, or external influence on, the chemical and physical qualities;
- b) it is characterized by its content of certain mineral salts and their relative proportions and the presence of trace elements or of other constituents;
- c) of the constancy of its composition and the stability of its discharge and its temperature, due account being taken of the cycles of minor natural fluctuations;
- d) it is collected under conditions which

guarantee the original microbiological purity and chemical composition of essential components;

- e) it is packaged close to the point of emergence of the source with particular hygienic precautions; and
- f) it is not subjected to any treatment other than those permitted by this standard.

3.1.1 Naturally Carbonated Natural Mineral Water — Natural mineral water which, after possible treatment in accordance with [4.1](#) and re-incorporation of gas from the same source and after packaging taking into consideration usual technical tolerance, has the same content of carbon dioxide spontaneously and visibly given off under normal conditions of temperature and pressure.

3.1.2 Non-carbonated Natural Mineral Water — Natural mineral water which, by nature and after possible treatment in accordance with [4.1](#) and after packaging taking into consideration usual technical tolerance, does not contain free carbon dioxide in excess of the amount necessary to keep the hydrogen carbonate salts present in the water dissolved.

3.1.3 Decarbonated Natural Mineral Water — Natural mineral water which, after possible treatment in accordance with [4.1](#) and after packaging, has less carbon dioxide content than that at emergence and does not visibly and spontaneously give off carbon dioxide under normal conditions of temperature and pressure.

3.1.4 Natural Mineral Water Fortified with Carbon Dioxide from the Source — Natural mineral water which, after possible treatment in accordance with [4.1](#) and after packaging, has more carbon dioxide content than that at emergence.

3.1.5 Carbonated Natural Mineral Water — Natural mineral water which, after possible treatment in accordance with [4.1](#) and after packaging, has been made effervescent by the addition of carbon dioxide

from another origin.

NOTE — Mineral water means natural mineral water as defined in [3.1](#).

3.1.6 Natural Spring Water — Natural spring water is natural mineral water which is derived from an underground formation from which water flows naturally to the surface of the earth at an identified location. Spring water shall be collected only at the spring or through a borehole tapping the underground formation feeding the spring. There shall be a natural force causing the water to flow to the surface through an orifice.

3.2 Packaged Natural Mineral Water — Natural mineral water filled into hermetically sealed containers of various compositions, forms and capacities that is, suitable for direct consumption without further treatment.

4 TREATMENT AND HANDLING

4.1 Treatments permitted include separation from unstable constituents, such as compounds containing iron, manganese, sulphur or arsenic, by decantation and/or simple filtration up to 0.5 microns, if necessary, accelerated by previous aeration.

4.2 The treatments provided in [3.1.1](#) to [3.1.5](#) and [4.1](#) above may only be carried out on condition that the mineral content of the water is not modified in its essential constituents, which give the water its properties.

4.3 The transport of natural mineral waters in bulk containers for packaging or for any other process before packaging is prohibited.

5 HYGIENIC CONDITIONS

Natural mineral water shall be collected, processed, handled, packaged and marketed in accordance with the hygienic practices given in [Annex B](#). A checklist for good hygienic practices and food safety system for packaged natural mineral water processing units given at the end of [Annex B](#).

6 REQUIREMENTS

6.1 Microbiological Requirements

6.1.1 *Escherichia coli* or thermotolerant bacteria, shall be absent in any 250 ml sample when tested in accordance with the method given in IS 15185.

6.1.2 *Coliform*, bacteria shall be absent in any

250 ml sample when tested in accordance with the method given in IS 5401 (Part 1) or IS 15185*.

6.1.3 *Faecal streptococci* and *Staphylococcus aureus*, shall be absent in any 250 ml sample when tested in accordance with the method given in IS 5887 (Part 2). *Streptococci* (*Enterococci*) may also be tested by the method specified in IS 15186*.

6.1.4 *Sulphite Reducing Anaerobe*, shall be absent in 50 ml sample when tested in accordance with the method given in [Annex C](#).

6.1.5 *Pseudomonas aeruginosa*, shall be absent in 250 ml sample when tested in accordance with the method given in Annex D* or ISO 16266-2.

6.1.6 *Yeast and Mould*, shall be absent in 250 ml sample when tested in accordance with the method given in 16069 (Part 1) .

6.1.7 *Salmonella* and *Shigella*, shall be absent in any 250 ml sample when tested in accordance with the method given in IS 15187 and IS 5887 (Part 7) respectively.

6.1.8 *Vibrio cholera* and *V. parahaemolyticus*, shall be absent in 250 ml sample when tested in accordance with the method given in IS 5887 (Part 5/Sec 1).

6.1.9 The membrane filtration technique outlined in IS 15188 may be used to pass the sample of water to be tested through membrane before the microbiological tests specified from [6.1.1](#) to [6.1.8](#) are carried out.

NOTE — In case of dispute, the method indicated by '*' in [6.1.2](#) to [6.1.3](#) shall be the referee method.

6.2 Natural mineral water shall also comply with the requirements given in [Table 1](#), [Table 2](#), [Table 3](#) and [Table 4](#).

6.3 Residues of pesticides for pesticides as given in Annex N shall be below the detectable limits. The analysis of pesticide shall be conducted by a recognized laboratory using internationally established test methods as given in [Annex N](#).

6.4 Natural spring water (*see* [3.1.6](#)) shall comply with the requirements given in this standard as specified in [6.1](#), [6.2](#) and [6.3](#), except total dissolved solids (TDS) content. TDS of natural spring water shall be not more than 750 mg/l.

7 PACKING

7.1 Natural mineral water shall be packed in clean, hygienic and tamperproof containers/bottles/pouches made of food grade plastic material or other food grade packaging materials, namely paper and paper board materials; glass (*see* IS 11984); and metal and metal alloys which may or may not contain plastic as component, compatible with the water to be packaged.

7.1.1 Plastic containers shall conform to IS 15410 and polyethylene flexible pouches shall conform

to IS 15609. Guidelines for handling of polyethylene flexible pouches are given in [Annex E](#).

7.1.2 Paper based multilayer laminated/extruded composite cartons shall conform to IS 17753 and aluminum cans shall conform to IS 18285.

7.1.3 All packaging materials of plastic origin shall pass the overall migration, colour migration and specific migration limits for toxic substances as laid down in IS 15410.

Table 1 Organoleptic and Physical Parameters

(Clause 6.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Colour, true colour unit, <i>Max</i>	2	IS 3025 (Part 4)
ii)	Odour	Agreeable	IS 3025 (Part 5)
iii)	Taste	Agreeable [Action tendency scale (a) or (b) or (c)]	IS 3025 (Part 8)
iv)	Turbidity, NTU, <i>Max</i>	2	IS 3025 (Part 10)
v)	Total dissolved solids, mg/l	150 to 700	IS 3025 (Part 16)
vi)	pH value	6.5 to 8.5	IS 3025 (Part 11)

Table 2 General Parameters Concerning Substances Undesirable in Excessive Amounts

(Clause 6.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to IS	
			Annex of this Standard	Other Standards
(1)	(2)	(3)	(4)	(5)
i)	Nitrate (as NO ₃), mg/l, <i>Max</i>	50	-	IS 3025 (Part 34/sec 1) or IS 3025 (Part 75)*
ii)	Nitrite (as NO ₂), mg/l, <i>Max</i>	0.02	-	IS 3025 (Part 34/sec 1) or IS 3025 (Part 75)*
iii)	Sulphide (as H ₂ S), mg/l, <i>Max</i>	0.05	-	IS 3025 (Part 29)
iv)	Manganese (as Mn), mg/l, <i>Max</i>	2.0	-	IS 3025 (Part 59) or IS 3025 (Part 2) or IS 3025 (Part 65)*
v)	Copper (as Cu), mg/l, <i>Max</i>	1.0	-	IS 3025 (Part 42) or IS 3025 (Part 2) or IS 3025 (Part 65)*

Table 2 (Concluded)

Sl No.	Characteristic	Requirement	Method of Test, Ref to IS	
			Annex of this Standard	Other Standards
(1)	(2)	(3)	(4)	(5)
vi)	Fluoride (as F), mg/l, <i>Max</i>	1.0	-	IS 3025 (Part 60/Sec 1) or (Part 60/Sec 2) or (Part 60/ Sec 3) or IS 3025 (Part 75)*
vii)	Barium, (as Ba), mg/l, <i>Max</i>	1.0	<u>G</u>	IS 3025 (Part 65)* or IS 15302 or IS 3025 (Part 2)
viii)	Antimony (as Sb), mg/l, <i>Max</i>	0.005	<u>H</u>	IS 3025 (Part 2) or IS 3025 (part 65)* or IS 15303
ix)	Borate (as B), mg/l, <i>Max</i>	5	<u>J</u>	IS 3025 (Part 65)* or IS 3025 (Part 2)
x)	Silver (as Ag), mg/l, <i>Max</i>	0.01	<u>K</u>	IS 3025 (Part 65)* or IS 3025 (Part 2) or IS 3025 (Part 79)
xi)	Chloride (as Cl), mg/l, <i>Max</i>	200	-	IS 3025 (Part 32) or IS 3025 (Part 75)*
xii)	Sulphate (as SO ₄), mg/l, <i>Max</i>	200	-	IS 3025 (Part 24/Sec 1) or (Part 24/Sec 2) IS 3025 (Part 75)
xiii)	Magnesium (as Mg), mg/l, <i>Max</i>	50	-	IS 3025 (Part 46) or IS 3025 (Part 2) or IS 3025 (Part 65)*
xiv)	Calcium (as Ca), mg/l, <i>Max</i>	100	-	IS 3025 (Part 40) or IS 3025 (Part 2) or IS 3025 (Part 65)*
xv)	Sodium (as Na) m mg/l, <i>Max</i>	150	-	IS 3025 (Part 45)* or IS 3025 (Part 2)
xvi)	Alkalinity (as HCO ₃), mg/l	75 to 400	-	IS 3025 (Part 23)
xvii)	Selenium (as Se), mg/l, <i>Max</i>	0.05	-	IS 3025 (Part 2) or IS 3025 (Part 56) IS 15303 or IS 3025 (Part 65)*
xviii)	Mineral oil, mg/l, <i>Max</i>	1.0	-	IS 3025 (Part 39)
xix)	Phenolic compounds (as C ₆ H ₅ OH) mg/l, <i>Max</i>	0.001	-	IS 3025 (Part 43/Sec 1)
xx)	Anionic surface active agents	Not detectable	-	IS 3025 (Part 68)* or IS 3025 (Part 78)

NOTES

1 Approved and validated international test methods from ISO/APHA/ASTM/AOAC/EPA/EN may also be followed.

2 In case of dispute, method indicated by ‘*’ shall be the reference method.

Table 3 Parameters Concerning Toxic Substances(Clause [6.2](#))

Sl No.	Characteristic	Requirement	Method of Test, Ref to IS	
			Annex of this Standard	Other Standards
(1)	(2)	(3)	(4)	(5)
i)	Arsenic (as As), mg/l, <i>Max</i>	0.01	-	IS 3025 (Part 2) or IS 3025 (Part 37) or IS 3025 (Part 65)*
ii)	Cadmium (as Cd), mg/l, <i>Max</i>	0.003	-	IS 3025 (Part 2) or IS 3025 (Part 41) or IS 3025 (Part 65)*
iii)	Cyanide (as CN), mg/l, <i>Max</i>	Absent	-	IS 3025 (Part 27/ Sec 1)* or IS 3025 (Part 27/Sec 2) or IS 3025 (Part 27/Sec 3)
iv)	Chromium(as Cr), mg/l, <i>Max</i>	0.05	K	IS 3025 (Part 2) or IS 3025 (Part 65)*
v)	Mercury(as Hg), mg/l, <i>Max</i>	0.001	-	IS 3025 (Part 48) or IS 3025 (Part 65)*
vi)	Lead(as Pb), mg/l, <i>Max</i>	0.01	-	IS 3025 (Part 2) or IS 3025 (Part 47) or IS 3025 (Part 65)*
vii)	Nickel (as Ni), mg/l, <i>Max</i>	0.02	L	IS 3025 (Part 2) or IS 302 (Part 65)*
viii)	Polychlorinated biphenyle (PCB)	Not detectable	M	-
ix)	Polynuclear aromatic hydrocarbons	Not detectable	-	-
x)	Uranium, mg/l, <i>Max</i>	0.03	-	IS 3025 (Part 65)* or IS 14194 (Part 3)

NOTES

1 Approved and validated international test methods from ISO/APHA/ASTM/AOAC/EPA/EN may also be followed.

2 In case of dispute, method indicated by ‘*’ shall be the reference method.

Table 4 Parameters Concerning Radio Active Residues(Clause [6.2](#))

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Alpha emitters, Bq/l, <i>Max</i>	0.1	IS 14194 (Part 2)
ii)	Beta emitters, Bq/l, <i>Max</i>	1	IS 14194 (Part 1)

NOTE — In case of non-conformity of radioactive residues, the source of water shall be abandoned and water shall be recalled immediately.

8 MARKING

8.1 The following particulars shall be marked legibly and indelibly on the label of the bottle/container:

- a) Name of the product (that is, natural mineral water);
- b) Supplementary designations, if any;
- c) Name and address of the processor;
- d) Brand name, if any;
- e) Batch or code number;
- f) Date of manufacture or packaging and 'Expiry/Use by';
- g) Best before date (optional);
- h) Net quantity;
- j) Location and name of the source of natural mineral water;
- k) Direction for storage; and
- m) Any other information required under the *Legal Metrology (Packaged Commodities) Rules, 2011* and *The Food Safety and Standards (Labelling and Display) Regulations, 2020*.

NOTE — The manner of declaration of date of manufacture or packaging/expiry/use by/best before, shall follow the DD/MM/YY format for products with shelf life upto three months. For products with shelf life more than three months, either DD/MM/YY format shall be used or the month and the year shall be declared in un-coded numerical sequence except that the month shall be indicated by capital letters and abbreviations (at least first three letters of the month) may be used.

8.2 Labelling Prohibitions

8.2.1 No claims concerning medicinal (preventative, alleviative or curative) effects shall be made in respect of the properties of the product covered by the standard. Claims of other beneficial effects

related to the health of the consumer shall not be made.

8.2.2 The name of the locality, hamlet or specified place may not form part of the brand name unless it refers to packaged natural mineral water collected/processed at the place designated by that brand name.

8.2.3 The use of any statement or of any pictorial device which may create confusion in the mind of the public or in any way mislead the public about the nature, origin, composition and properties of natural mineral waters put on sale is prohibited.

8.3 BIS Certification Marking

8.3.1 The product may also be marked with the Standard Mark.

8.3.2 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.

9 SAMPLING

Representative samples of natural mineral water shall be drawn and the criteria for conformity to this standard shall be established, according to the method given in [Annex F](#).

10 QUALITY OF REAGENTS

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

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(Clause 2)

LIST OF REFERRED STANDARDS

<i>IS No./Other Standards</i>	<i>Title</i>	<i>IS No./Other Standards</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)	(Part 29) : 2022 (Part 32) : 1988	Sulphide (<i>second revision</i>) Chloride (<i>first revision</i>)
IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater:	(Part 34/sec 1) : 2023	Nitrogen, Section 1 Determination of various types of nitrogen like ammoniacal, nitrate, nitrite and organic nitrogen (<i>second revision</i>)
(Part 2) : 2019/ ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (<i>first revision</i>)	(Part 37) : 2022 (Part 39) : 2021	Arsenic (<i>second revision</i>) Oil and grease (<i>second revision</i>)
(Part 4) : 2021	Colour (<i>second revision</i>)	(Part 40) : 1991	Calcium (<i>first revision</i>)
(Part 5) : 2018	Odour (<i>second revision</i>)	(Part 41) : 2023	Cadmium (<i>second revision</i>)
(Part 8) : 2023	Taste rating (<i>second revision</i>)	(Part 42) : 1992	Copper (<i>first revision</i>)
(Part 10) : 2023	Turbidity (<i>second revision</i>)	(Part 43/Sec 1) : 2022	Phenol, Section 1 4-Aminoantipyrine method with and without chloroform extraction method (<i>second revision</i>)
(Part 11) : 2022/ ISO 10523 : 2008	pH value (<i>second revision</i>)	(Part 45) : 1993	Sodium and potassium (<i>first revision</i>)
(Part 16) : 2023	Filterable residue (total dissolved solids) at 180 °C (<i>second revision</i>)	(Part 46) : 2023	Magnesium (<i>second revision</i>)
(Part 23) : 2023	Alkalinity (<i>second revision</i>)	(Part 47) : 1994	Lead (<i>first revision</i>)
(Part 24)	Sulphates,	(Part 48) : 1994	Mercury (<i>first revision</i>)
(Sec 1) : 2022	Gravimetric and turbidity methods (<i>second revision</i>)	(Part 56) : 2003	Selenium (<i>first revision</i>)
(Sec 2) : 2021/ ISO 22743 : 2006	Method by continuous flow analysis	(Part 59) : 2006 (Part 60)	Manganese (<i>first revision</i>) Fluoride,
(Part 27)	Cyanide,	(Sec 1) : 2023	Ion-selective electrode, SPANDS and liquid chromatography methods (<i>second revision</i>)
(Sec 1) : 2021	Titrimetric, colorimetric and ion-selective methods (<i>second revision</i>)	(Sec 2) : 2022/ ISO/TS 17951- 1 : 2016	Method using flow injection analysis (FIA) and spectrometric detection after off-line distillation
(Sec 2) : 2022/ ISO 14403-1 : 2012	Method using flow injection analysis		
(Sec 3) : 2021/ ISO 14403-2 : 2012	Method using continuous flow analysis (CFA)		

IS 13428 : 2024

<i>IS No./Other Standards</i>	<i>Title</i>	<i>IS No./Other Standards</i>	<i>Title</i>
(Sec 3) : 2022/ ISO/TS 17951-2 : 2016	Method using continuous flow analysis (CFA) with automated in-line distillation	(Part 7) : 1999	General guidance on methods for isolation and identification of <i>Shigella</i>
(Part 65) : 2022/ ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (<i>first revision</i>)	IS 10500 : 2012	Drinking Water — Specification (<i>second revision</i>)
(Part 68) : 2019	Anionic surfactants	IS 11984 : 1986	Specification for glass bottles for free flowing liquids
(Part 75) : 2022/ ISO 10304-1 : 2007	Determination of dissolved anions by liquid chromatography of ions — Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate	IS 14194	Radionuclides in environmental samples — Methods of estimation:
(Part 78) : 2021/ ISO 16265 : 2009	Determination of the methylene blue active substances (MBAS) index — Method using continuous flow analysis (CFA)	(Part 1) : 2020	Gross beta activity measurement (<i>second revision</i>)
(Part 79) : 2023	Silver	(Part 2) : 2022	Gross alpha activity measurement (<i>second revision</i>)
IS 4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)	(Part 3) : 2021	Uranium (<i>first revision</i>)
IS 5401 (Part 1) : 2012/ISO 4832 : 2006	Microbiology of food and animal feeding stuffs — Horizontal method for the detection and enumeration of coliforms: Part 1 Colony-count technique (<i>second revision</i>)	IS 15185 : 2016/ ISO 9308-1 : 2014	Water quality — Detection and enumeration of <i>Escherichia coli</i> and <i>Coliform</i> bacteria — Membrane filtration method for water with low bacterial background flora (<i>first revision</i>)
IS 5887	Methods for detection of bacteria responsible for food poisoning:	IS 15186 : 2002/ ISO 7899-2 : 2000	Water quality — Detection and enumeration of intestinal Enterococci — Membrane filtration method
(Part 2) : 1976	Isolation, identification and enumeration of <i>Staphylococcus aureus</i> and faecal streptococci (<i>first revision</i>)	IS 15187 : 2016/ ISO 19250 : 2010	Water quality — Detection of <i>Salmonella</i> species (<i>first revision</i>)
(Part 5/Sec 1) : 2023/ISO 21872-1 : 2017	Horizontal method for the demterination of <i>Vibrio</i> spp., Section 1 Detection of potentially enteropathogenic <i>Vibrio parahaemolyticus</i> , <i>Vibrio cholerae</i> and <i>Vibrio vulnificus</i> (<i>second revision</i>)	IS 15188 : 2022/ ISO 8199 : 2018	Water quality — General requirements and guidance for microbiological examinations by culture (<i>second revision</i>)
		IS 15302 : 2003	Determination of aluminium and barium in water by direct nitrous oxide-acetylene flame atomic absorption spectrometry
		IS 15303 : 2003	Determination of antimony, iron and selenium in water by electrothermal atomic

<i>IS No./Other Standards</i>	<i>Title</i>	<i>IS No./Other Standards</i>	<i>Title</i>
	absorption spectrometric method	IS 17753 : 2021	Paper based multilayer laminated/extruded composite cartons (aseptic and non-aseptic) for processed liquid food products and beverages — Specification
IS 15410 : 2003	Containers for packaging of natural mineral water and packaged drinking water — Specification		
IS 15609 : 2005	Polyethylene flexible pouches for the packing of natural mineral water and packaged drinking water — Specification	IS 18285 : 2023	Aluminium cans for packaged natural mineral water and packaged drinking water — Specification
IS 15669 : 2006/ ISO 10695 : 2000	Water quality — Determination of selected organic nitrogen and phosphorus compounds — Gas chromatographic method	ISO 16266-2 : 2018	Water quality — Detection and enumeration of <i>Pseudomonas aeruginosa</i> — Part 2: Most probable number method
IS 16069 (Part 1) : 2013/ISO 21527-1 : 2008	Microbiology of food and animal feeding stuffs — Horizontal method for the enumeration of yeasts and moulds: Part 1 Colony count technique in products with water activity greater than 0.95		

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ANNEX B

(Clause 5)

HYGIENIC PRACTICES

B-1 FIELD OF APPLICATION

The hygienic practices cover appropriate general techniques for collecting natural mineral water, its treatment, bottling, packaging, storage, transport, distribution and sale for direct consumption, so as to guarantee a safe, healthy and wholesome product.

B-2 PRESCRIPTIONS OF THE RESOURCES OF NATURAL MINERAL WATER**B-2.1 Protection of Alimentary Reservoirs and Aquifers****B-2.1.1 Authorization**

Any spring, well or drilling intended for the collection of natural mineral water should be approved by the local health authority or any other agency having jurisdiction for the region.

B-2.1.2 Determination of the Genesis of Natural Mineral Water

As far as it is methodologically possible in each case, a precise analysis should be carried out on the origin of natural mineral waters, the period of their residence in the ground before being collected and their chemical, physical and radiological qualities.

B-2.1.3 Perimeter of Protection

If possible areas where in natural mineral water might be polluted or its chemical and physical qualities otherwise deteriorated should be determined by a hydrologist. Where indicated by hydro geological conditions and considering the risks of pollution and physical, chemical and biochemical reactions, several perimeters with separate dimensions may be provided for.

B-2.1.4 Protective Measures

All possible precautions should be taken within the protected perimeters to avoid any pollution of, or external influence on, the chemical and physical qualities of natural mineral water.

It is recommended that regulations be established for the disposal of liquid, solid or gaseous waste, the use of substances that might deteriorate natural mineral water as well as any possibility of accidental deterioration of natural mineral water by natural

occurrences such as a change in the hydrogeological conditions. Particular consideration should be given to the following potential pollutants such as bacteria, viruses, fertilizers, hydrocarbons, detergents, pesticides, phenolic compounds, toxic metals, radioactive substances and other soluble organic or inorganic substances. Even where nature provides apparently sufficient protection against surface, pollution, potential hazards arising out of mining, hydraulic and engineering facilities etc, should be taken into consideration.

B-2.2 Hygiene Prescriptions for Collection of Natural Mineral Water**B-2.2.1 Extraction**

The withdrawal of natural mineral water shall be performed in conformity with the hydrogeological conditions in such a manner as to prevent any water other than the natural mineral water from entering or, should there be pumping facilities, prevent any extraneous water from entering. The natural mineral water thus collected or pumped should be protected in such a way that it is not polluted (whether caused by natural occurrence or actions or neglect or ill will).

B-2.2.2 Materials

The pipes, pumps or other possible devices coming into contact with natural mineral water and used for its collection should be made of such material as to guarantee that original quality of natural mineral water is not changed.

B-2.2.3 Protection of the Extraction Area

In the immediate surroundings of springs or wells, precautionary measures should be taken to guarantee that no pollutant whatsoever can enter the extraction area. The extraction area should be inaccessible to non-authorized people by providing adequate devices (for example, enclosure). Any use not aiming at the collection of natural mineral water should be forbidden in this area.

B-2.2.4 Exploitation of Natural Mineral Water

The condition of the extraction facilities, areas of extraction and perimeter protection as well as the quality of the natural mineral water should

periodically be checked. To control the stability of the chemical and physical particulars of the natural mineral water derived, besides the natural variations, automatic measurements of the typical characteristics of water should be carried out and recorded (for example, electrical conductance, temperature, content of carbon dioxide) or frequent partial analysis should be done.

B-2.3 Maintenance of Extraction Facilities

B-2.3.1 Technical Aspects

Methods and procedures for maintaining the extraction facilities should be hygienic and not be a potential hazard to human health or a source of contamination to natural mineral water. From the hygiene standpoint, servicing of the extraction installations should meet the same requirements as those required for the bottling or for treatment.

B-2.3.2 Equipment and Reservoirs

Equipment and reservoirs used for extraction of natural mineral water should be constructed and maintained in order to minimize all hazards to human health and to avoid contamination.

B-2.3.3 Storage at the Point of Extraction

The quantity of natural mineral water stored at the point of extraction should be as low as possible. The storing should furthermore guarantee protection against contamination or deterioration.

B-2.4 Transport of Natural Mineral Water

B-2.4.1 Means of Transport, Piping and Reservoirs

Any vehicle, piping or reservoir used in the processing of natural mineral water from its source to the bottling facilities, should comply with the necessary requirements and be, made of inert material such as ceramic and stainless steel which prevents any deterioration, be it by water, handling, servicing or by disinfection; it should allow easy cleaning.

B-2.4.2 Maintenance of Vehicles and Reservoirs

Any vehicle or reservoir should be properly cleaned and if necessary, disinfected and kept in good repair so as not to present any danger of contamination to natural mineral water and of deterioration of the essential qualities of natural mineral water.

B-3 ESTABLISHMENT FOR PROCESSING NATURAL MINERAL WATERS — DESIGN AND FACILITIES

B-3.1 Location

Establishments should be located in areas which are free from objectionable odours, smoke, dust or other contaminants and are not subject to flooding.

B-3.2 Roadways and Areas Used by Wheeled Traffic

Such roadways and areas serving the establishment which are within its boundaries or, in its immediate vicinity should have a hard paved surface suitable for wheeled traffic. There should be adequate drainage and provision should be made for protection of the extraction area in accordance with [B-2.2](#) where appropriate. Adequate road signals may be provided to call the attention of road users to the existence of natural mineral water extraction area.

B-3.3 Buildings and Facilities

B-3.3.1 Type of Construction

Buildings and facilities should be of sound construction in accordance with the provisions of [B-2.2](#) and maintained in good repair.

B-3.3.2 Disposition of Holding Facilities

Rooms for recreation, for storing or packaging of raw material and areas for cleaning of containers to be reused should be away from the bottling areas to prevent the end product from being contaminated. Raw materials and packaging materials which come into contact with natural mineral water should be stored apart from other material.

B-3.3.3 Adequate working space should be provided to allow for satisfactory performance of all operations.

B-3.3.4 The design should be such as to permit easy and adequate cleaning and to facilitate proper supervision of hygiene of natural mineral water.

B-3.3.5 The buildings and facilities should be designed to provide separation by partition, location or other effective means between those operations which may cause cross contamination.

B-3.3.6 Buildings and facilities should be designed

to facilitate hygienic operations by means of a regulated flow in the process from the arrival of the natural mineral water at the premises to the finished product, and should provide for appropriate temperature conditions for the process and the product.

B-3.3.7 *Natural Mineral Water Handling, Storing and Bottling Areas*

B-3.3.7.1 *Floors*

Where appropriate, floors should be of water-proof, non-absorbent, washable, non-slip and non-toxic materials, without crevices, and should be easy to clean and disinfect. Where appropriate, floors should have sufficient slope for liquids to drain to trapped outlets.

B-3.3.7.2 *Walls*

Where appropriate, should be of water-proof, non-absorbent, washable and non-toxic material and should be light coloured. Up to a height appropriate for the operation they should be smooth and without crevices, and should be easy to clean and disinfect. Where appropriate, angles between walls, between walls and floors, and between walls and ceilings should be sealed and smoothen to facilitate cleaning.

B-3.3.7.3 *Ceilings*

Ceilings should be so designed, constructed and finished as to prevent the accumulation of dirt and minimize condensation, mould growth and flaking, and should be easy to clean.

B-3.3.7.4 *Windows*

Windows and other openings should be so constructed as to avoid accumulation of dirt and those which open should be fitted with screens. Screens should be easily movable for cleaning and kept in good condition. Internal window sills, if present, should be sloped to prevent use as shelves.

B-3.3.7.5 *Doors*

Doors should have smooth, non-absorbent surfaces and, where appropriate, be self-closing and close fitting type.

B-3.3.7.6 *Stairs, lift cages and auxiliary structure*

Platforms, ladders, chutes, should be so situated and constructed as not to cause contamination. Chutes should be constructed with provision of inspection and cleaning hatches.

B-3.3.7.7 *Piping*

Piping for natural mineral water lines should be independent of potable and non-potable water.

B-3.3.8 In natural mineral water handling area all over head structures and fittings should be installed in such a manner as to avoid contamination directly or indirectly of natural mineral water and raw materials by condensation and drip, and should not hamper cleaning operations. They should be insulated where appropriate and be so designed and finished as to prevent the accumulation of dirt and to minimize condensation, mould development and flaking. They should be easy to clean.

B-3.3.9 Living quarters, toilets and areas where animals are kept should be completely separated from and should not open directly on to natural mineral water handling areas.

B-3.3.10 Where appropriate, establishments should be so designed that access can be controlled.

B-3.3.11 The use of material which cannot be adequately cleaned and disinfected, such as wood, should be avoided unless its use would not be a source of contamination.

B-3.3.12 *Canalization, Drainage Lines*

Canalization, drainage and used water lines as well as any possible waste storage area within the protected perimeter should be built and maintained in such a manner as not to present any risk whatsoever of polluting aquifers and springs.

B-3.3.13 *Fuel Storage Area*

Any storage area or tank for the storing of fuels such as coal or hydrocarbons should be designed, protected, controlled and maintained in such a manner as not to present a risk of aquifer and springs being polluted during the storage and manipulation of these fuels.

B-3.4 *Hygienic Facilities*

B-3.4.1 *Water Supply*

B-3.4.1.1 Ample supply of potable water under adequate pressure and of suitable temperature should be available with adequate facilities for its storage, where necessary, and distribution with adequate protection against contamination. The potable water should conform to the standard for drinking water (*see* IS 10500).

B-3.4.1.2 Natural mineral water, potable water, non-potable water for steam production or for refrigeration or any other use should be carried in separate lines with no cross connection between them and without any chance of back siphonage. It would be desirable that these lines be identified by different colours. Steam used in direct contact with natural mineral water and also natural mineral water contact surfaces should contain no substances which may be hazardous to health or may cause contamination.

B-3.4.2 *Effluent and Waste Disposal*

Establishments should have an efficient effluent and waste disposal system which should at all times be maintained in good order and repair. All effluent lines (including sewer systems) should be large enough to carry full loads and should be so constructed as to avoid contamination of potable water supplies.

B-3.4.3 *Changing Facilities and Toilets*

Adequate, suitable and conveniently located changing facilities and toilets should be provided in all establishments. Toilets should be so designed as to ensure hygienic removal of waste matter. These areas should be well lighted, ventilated and should not open directly on to natural mineral water handling areas. Hand washing facilities with warm or hot and cold water, a suitable hand-cleaning preparation, and with suitable hygienic means of drying hands, should be provided adjacent to toilets and in such a position that the employee will have to use them when returning to the processing area. Where hot and cold water are available mixing taps should be provided. Where paper towels are used, a sufficient number of dispensers and receptacles should be provided near each washing facility. Care should be taken that these receptacles for used paper towels are regularly emptied. Taps of a non-hand operable type are desirable. Notices should be posted directing personnel to wash their hands after using the toilet.

B-3.4.4 *Hand Washing Facilities in Natural Mineral Water Processing Areas*

Adequate and conveniently located facilities for hand washing and drying should be provided wherever the process demands. Where appropriate, facilities for hand disinfection should also be provided. Warm or hot and cold water should be available and taps for mixing the two should be provided. There should be suitable hygienic means of drying hands. Where paper towels are used, a sufficient number of dispensers and receptacles

should be provided adjacent to each washing facility. Taps of a non-hand operable type are desirable. The facilities should be furnished with properly trapped waste pipes leading to drains.

B-3.4.5 *Disinfection Facilities*

Where appropriate, adequate facilities for cleaning and disinfection of working implements and equipment should be provided. These facilities should be constructed of corrosion resistant materials, capable of being easily cleaned, and should be fitted with suitable means of supplying hot and cold water in sufficient quantities.

B-3.4.6 *Lighting*

Adequate natural or artificial lighting should be provided throughout the establishment. Where appropriate, the lighting should not alter colours and the intensity should not be less than:

- a) 540 lux (50 foot candles) at all inspection points;
- b) 220 lux (20 foot candles) in work rooms; and
- c) 110 lux (10 foot candles) in other areas.

Light bulbs and fixtures suspended over natural mineral water in any stage of production should be of a safer type and protected to prevent contamination of natural mineral water in case of breakage.

B-3.4.7 *Ventilation*

Adequate ventilation should be provided to prevent excessive heat, steam condensation and dust and to remove contaminated air. The direction of the air flow should never be from a dirty area to a clean area. Ventilation openings should be provided with a screen or other protecting enclosure of non-corrodible material. Screens should be easily removable for cleaning.

B-3.4.8 *Facilities for Storage of Waste and Inedible Material*

Facilities should be provided for the storage of waste and inedible material prior to removal from the establishment. These facilities should be designed to prevent access to waste or inedible material by pests and to avoid contamination of natural mineral water, potable water, equipment, buildings or roadways on the premises.

B-3.5 *Equipment and Utensils*

B-3.5.1 *Material* All equipment and utensils used in natural mineral water handling are a sand which may

contact the natural mineral water should be made of material which does not transmit toxic substances, odour or taste, is non-absorbent, is resistant to corrosion and is capable of withstanding repeated cleaning and disinfection. Surfaces should be smooth and free from pits and crevices. The use of wood and other materials which cannot be adequately cleaned and disinfected should be avoided except when their use would not be a source of contamination. The use of different materials is exercised in such a way that contact corrosion that can occur, should be avoided.

B-3.5.2 Hygienic Design, Construction and Installation

All equipment and utensils should be so designed and constructed as to prevent hazards and permit easy and thorough cleaning and disinfection.

B-4 ESTABLISHMENT — HYGIENE REQUIREMENTS

B-4.1 Maintenance

The buildings, equipment, utensils and all other physical facilities of the establishment, including drains, should be maintained in good repair and in an orderly condition. As far as practicable, rooms should be kept protected from steam, vapour and surplus water.

B-4.2 Cleaning and Disinfection

B-4.2.1 Cleaning and disinfection should meet the requirements of this standard.

B-4.2.2 To prevent contamination of natural mineral water, all equipment and utensils should be cleaned as frequently as necessary and disinfected, whenever circumstances demand.

B-4.2.3 Adequate precautions should be taken to prevent natural mineral water from being contaminated during cleaning or disinfection of rooms, equipment or utensils, by water and detergents or by disinfectants and their solutions. Detergents and disinfectants should be suitable for the purpose intended and should be acceptable to the official agency having jurisdiction. Any, residues of these agents on a surface which may come in contact with natural mineral water should be removed by thorough rinsing with water, before the area or equipment is again used for handling natural mineral water.

B-4.2.4 Either immediately after cessation of work for the day or at such other times as may be appropriate, floors, including drains, auxiliary structures and walls of natural mineral water

handling areas should be thoroughly cleaned.

B-4.2.5 Changing facilities and toilets should be kept clean at all times.

B-4.2.6 Roadways and yards in the immediate vicinity of and serving the premises should be kept clean.

B-4.3 Hygiene Control Programme

A permanent cleaning and disinfection schedule should be drawn up for each establishment to ensure that all areas are appropriately cleaned and that critical areas, equipment and material are designated for special attention. An individual, who should preferably be a permanent member of the staff of the establishment and whose duties should be independent of production, should be appointed to be responsible for the cleanliness of the establishment. He should have a thorough understanding of the significance of contamination and the hazards involved. All cleaning personnel should be well-trained in cleaning techniques.

B-4.4 Storage and Disposal of Waste

Waste material should be handled in such a manner as to avoid contamination of natural mineral water or potable water. Care should be taken to prevent access to waste by pests. Waste should be removed from the natural mineral water handling and other working areas as often as necessary and at least daily. Immediately after disposal of the waste, receptacles used for storage and any equipment which has come into contact with the waste should be cleaned and disinfected. The waste storage area should also be cleaned and disinfected.

B-4.5 Exclusion of Animals

Animals that are uncontrolled or that could be a hazard to health should be excluded from establishments.

B-4.6 Pest Control

B-4.6.1 There should be an effective and continuous programme for the control of pests. Establishments and surrounding areas should be regularly examined for evidence of infestation.

B-4.6.2 Should pests gain entrance to the establishment, eradication measures should be instituted. Control measures involving treatment with chemical, physical or biological agents should be undertaken by or under direct supervision of personnel who have a thorough understanding of the potential hazards to health resulting from the use of

these agents, including those hazards which may arise from residues retained in the natural mineral water, such measures should only be carried out in accordance with the recommendations of the official agency having jurisdiction.

B-4.6.3 Pesticides should only be used, if other precautionary measures cannot be used effectively. Before pesticides are applied, care should be taken to safeguard natural mineral water, equipment and utensils from contamination. After application, contaminated equipment and utensils should be thoroughly cleaned to remove residues prior to be in a used again.

B-4.7 Storage Hazardous Substances

B-4.7.1 Pesticides or other substances which may present. Hazard to health should be suitably labelled with a warning about their toxicity and use. They should be stored in locked rooms or cabinets, used only for that purpose and dispersed and handled only by authorized and properly trained personnel or by persons under strict supervision of trained personnel, extreme care should be taken to avoid contamination of natural mineral water.

B-4.7.2 Except when necessary for hygienic or processing purposes, no substance which could contaminate natural mineral water should be used or stored in natural mineral water handling areas.

B-4.8 Personal Effects and Clothing

Personal effects and clothing should not be deposited in natural mineral water handling areas.

B-5 PERSONNEL HYGIENE AND HEALTH REQUIREMENTS

B-5.1 Hygiene Training

Managers of establishments should arrange for adequate and continuing training of all natural mineral water handlers in hygienic handling of natural mineral water and in personal hygiene so that they understand the precautions necessary to prevent contamination of natural mineral water.

B-5.2 Medical Examination

Persons who come into contact with natural mineral water in the course of their work should have a medical examination prior to employment, if the official agency having jurisdiction, acting on medical advice, considers that this is necessary, whether because of epidemiological considerations or the medical history of the prospective natural mineral water handler. Medical examination of natural mineral water handlers should be carried out

periodically and when clinically or epidemiologically indicated.

B-5.3 Communicable Diseases

The management should take care to ensure that no person, whether known or suspected to be suffering from, or to be a carrier of a disease likely to be transmitted or afflicted with infected wounds, skin infections, sores or diarrhea, is permitted to work in any natural mineral water handling area in any capacity in which there is any likelihood of such a person directly or indirectly contaminating natural mineral water with pathogenic micro-organisms. Any person so affected should immediately report to the management.

B-5.4 Injuries

Any person who has a cut or wound should not continue to handle natural mineral water or natural mineral water contact surfaces until the injury is completely protected with waterproof covering which is firmly secured, and which is conspicuous in colour. Adequate first-aid facilities should be provided for this purpose.

B-5.5 Washing of Hands

Every person, while on duty in a natural mineral water handling area, should wash hands frequently and thoroughly with a suitable hand cleaning preparation under running warm water. Hands should always be washed before commencing work, immediately after using the toilet, after handling contaminated material and whenever else necessary. After handling any material which might be capable of transmitting disease, hands should be washed and disinfected immediately. Notices requiring hand-washing should be displayed. There should be adequate supervision to ensure compliance with this requirement.

B-5.6 Personal Cleanliness

Every person engaged in a natural mineral water handling area should maintain a high degree of personal cleanliness while on duty, and should, at all times while so engaged, wear suitable protective clothing including head covering and footwear, all of which should be cleanable, unless designed to be disposed of and should be maintained in a clean condition consistent with the nature of the work in which the person is engaged. Aprons and similar items should not be washed on the floor. When natural mineral water is manipulated by hand, any jewellery that cannot be adequately disinfected should be removed from the hands. Personnel

should not wear any insecure jewellery when engaged in handling of natural mineral water.

B-5.7 Personal Behaviour

Any behavior which could result in contamination of natural mineral water, such as eating, use of tobacco, chewing (for example, gum, sticks, betel nuts, etc) or unhygienic practices such as spitting, should be prohibited in natural mineral water handling areas.

B-5.8 Visitors

Precautions should be taken to prevent as far as possible visit on from visiting natural mineral water handling areas. If unavoidable, visitors should observe the provisions recommended in [B-4.8](#), [B-5.3](#), [B-5.4](#) and [B-5.7](#).

B-5.9 Supervision

Responsible for ensuring compliance by all personnel with all requirements of [B-5.1](#) to [B-5.8](#) and the responsibility should be specifically allocated to competent supervisory personnel.

B-6 ESTABLISHMENT — HYGIENIC PROCESSING REQUIREMENTS

B-6.1 Raw Material Requirements

To guarantee a good and stable quality of natural mineral water, certain criteria should be monitored regularly, namely:

- a) Spring discharge, temperature of the natural mineral water;
- b) Appearance of the natural mineral water;
- c) Odour and taste of the natural mineral water;
- d) The conductance of natural mineral water or any other adequate parameter; and
- e) The microbiological flora.

B-6.2 Should there be a perceptible lack in meeting the requirements, the necessary corrective measures are immediately to be taken.

B-6.3 Treatment

The treatment may include decantation, filtration, airing and where necessary application of off take of carbon dioxide.

B-6.3.1 Processing should be supervised by technically competent personnel.

B-6.3.2 All steps in the production process, including packaging, should be performed without unnecessary delay and under conditions which will

prevent the possibility of contamination, deterioration or development of pathogenic and, spoilage micro-organisms.

B-6.3.3 Rough treatment of containers should be avoided to prevent the possibility of contamination of the processed product.

B-6.3.4 Treatments are necessary controls and should be such as to protect against contamination or development of a public health hazard. It also protects against deterioration within the limits of good commercial practice.

B-6.4 Packaging Material and Containers

B-6.4.1 All packaging material should be stored in a clean and hygienic manner. The material should be appropriate for the product to be packed and for the expected conditions of storage and should not transmit to the product objectionable substances beyond the limits specified. The packaging material should be sound and should provide appropriate protection from contamination. Only packaging material required for immediate use should be kept in the packing or filling area.

B-6.4.2 Product containers should not have been used for any purpose that may lead to contamination of the product. In case of new containers, if there is a possibility that they have been contaminated should be cleaned and disinfected. When chemicals are used for these purposes, the container should be rinsed as, prescribed under [B-4.2.3](#). Containers should be well drained after rinsing. Used and, when necessary, unused containers should be inspected immediately before filling.

B-6.5 Filling and Sealing of Containers

B-6.5.1 Packaging should be done under conditions that preclude the introduction of contaminants into the product.

B-6.5.1 The methods, equipment and material used for sealing should guarantee a tight and impervious sealing and should not damage the containers, nor deteriorate the physical, chemical, microbiological and organoleptic qualities of natural mineral water.

B-6.6 Packaging of Containers

The packaging of containers should protect the latter from contamination and damage and allow appropriate handling and storing.

B-6.7 Lot Identification

Each container shall be permanently marked with code to identify the producing factory and the lot.

A lot is quantity of natural mineral water produced under identical conditions, all packages of which should bear a lot number that identifies the production during a particular time, interval, and usually from a particular processing line or other critical processing unit.

B-6.8 Processing and Production Records

Permanent, legible and dated records of pertinent processing and production details should be kept concerning each lot. These records should be retained for a period that exceeds the shelf life of the product or longer if required. Records should also be kept for the initial distribution by lot.

B-6.9 Product Durability

Product durability shall be declared on the container as per [see [8.1 \(g\)](#)]. It shall be based on in house shelf

life study and proper checks and records be maintained for the conformity of the declared product durability.

B-6.10 Storage and Transport of the End-Product

The end-product should be stored and transported under such conditions as will preclude contamination with and/or proliferation of micro-organisms, and protect against deterioration of the product or damage to the container. During storage, periodic inspection of the end-product should take place to ensure that only natural mineral water which is fit for human consumption is dispatched and that end-product specifications are complied with.

CHECKLIST FOR GOOD HYGIENE PRACTICES AND FOOD SAFETY SYSTEMS FOR PACKAGED NATURAL MINERAL WATER PROCESSING UNITS

(Clause 5)

<i>Sl No.</i>	<i>Requirements</i>	<i>Answers</i>		<i>Remarks</i>
		Applied	Not Applied	
(1)	(2)	(3)	(4)	(5)
A	Building, Facilities and Locations			
i)	Is the facility location area free from objectionable odour, smoke, dust or other contaminants and not subject to flooding?			
ii)	Are the areas immediately surrounding the buildings, roads, parking places, suitably paved, grassed and kept clean?			
iii)	Is adequate facility for drainage of surroundings which is designed to handle peak load?			
iv)	Is the facility used for processing water free from domestic animals?			
v)	Is the facility surroundings free from refuse, waste materials, rubbish, over grown weeds and grasses?			
vi)	Are there adequate facilities for the disposal of effluents and wastes?			
vii)	Are the buildings and facilities of sound construction and maintained in good repair?			
viii)	Are the buildings and facilities designed and maintained to prevent entrance and harboring of pests and entry of contaminants?			
ix)	Are building and facilities designed to facilitate hygienic operations?			

Sl No.	Requirements	Answers		Remarks
		Applied	Not Applied	
(1)	(2)	(3)	(4)	(5)
B	Plant and Physical Facilities			
i)	Is adequate lighting provided at working station, hand washing area, and storage areas?			
ii)	Is the lighting intensity adequate: a) 540 lux in all inspection area, and b) 220 lux in work areas and walls.			
iii)	Are light fixtures safety type and protected to prevent contamination in the event of breakage in the processing and packing area?			
iv)	Is adequate ventilation provided in process in a areas to minimize odours, noxious fumes and condensates?			
v)	Are the barriers/traps provided at drains to prevent the entry of rodents from the drains into the facility?			
vi)	Is effective screening provided against entry of birds, animals, insects, rodents, etc?			
vii)	Are doors, hatches and other openings to the building are constructed to render opening pest proof? NOTE — Installation of one or more of the following which effectively prevents pest entry will meet tills requirement a) doors self closing type; b) have air curtains; and c) have strip curtains.			
viii)	Are floors, walls, ceilings, windows and doors so designed and constructed as to prevent accumulation of dust, dirt and render them washable?			
ix)	Is product in process and storage area adequately protected from any leakage from external surfaces and other sources of contamination?			
x)	Are immediate surroundings of extraction or collection protected from entry of unauthorized persons?			
C	Raw Water Processing			
i)	In case of extraction/collection for processing are the sources free from contaminations/impurities?			
ii)	Are water storage tanks, pipe lines utilized for handling water constructed and so designed as to facilitate cleaning and inspection?			

Table (Continued)

Sl No.	Requirements	Answers		Remarks
		Applied	Not Applied	
(1)	(2)	(3)	(4)	(5)
iii)	Are inspections of containers/carriers/pipe lines of raw water supply performed for the material of construction and cleanliness?			
iv)	Are possible chances of contamination from incoming water assessed?			
v)	Are water storage tanks effectively cleaned to prevent entry of pests and potential contaminator?			
vi)	Are the storage tanks periodically cleaned and records maintained?			
vii)	Are the processed water contact surfaces regularly cleaned and sanitized?			
viii)	Is all equipment utensils so designed and constructed as to prevent hygiene hazards and prevent easy cleaning and sanitation?			
D	Post Processing Handling			
i)	Are cleaning operations of bottles containers so done as to preclude contamination of product and product contact services with residues?			
ii)	Has absence of residual cleaning chemicals been ensured?			
iii)	Is preventive maintenance in place for all processing machinery and equipment?			
iv)	Are the primary packing material and containers of food grade conforming to relevant Indian Standards?			
v)	Are packing and sealing, where required, monitored?			
vi)	Are containers visually electronically inspected for their soundness?			
vii)	Are physical hazards prevented from entering into processed water?			
viii)	Are glassware excluded from production area?			
E	Packaging Material and Finished Goods Storage			
i)	Are the primary packing material and containers of food grade conforming to relevant Indian Standards?			
ii)	Are packaging material inspected to ensure their suitability?			
iii)	Are the packing materials especially primary packing material properly stored and properly handled to preclude contamination?			

Sl No.	Requirements	Answers		Remarks
		Applied	Not Applied	
(1)	(2)	(3)	(4)	(5)
iv)	Are packaging material purchased, stored and handled in sanitary manner?			
F	Finished Product Storage and Distribution			
i)	Is first-in-first out (FIFO) of stored product maintained?			
ii)	Is storage properly sanitized and disinfected periodically?			
iii)	Are stores protected from pest infestations?			
iv)	Are coding and tracking clear and in place?			
v)	Are the instruction clear and in place?			
vi)	Are hold/release procedure in place and product identified?			
vii)	Are the records maintained for batch number, date of and volume of production?			
viii)	Are transport containers/vehicles maintained in clean condition?			
G	Customer Handling of Products			
i)	Are the storage instructions provided on containers?			
ii)	Is the shelf life period best before mentioned on containers in accordance with PFA requirements?			
iii)	Are instructions provided for handling defective damaged products?			
H	Sanitary Facilities and Control			
i)	Are toilet provided in sufficient numbers and are they provided with: a) doors of self closing type? b) opening directly into processing areas? c) hand washing signs provided in appropriate language? d) proper lighting and ventilation? e) proper maintenance to keep in clean and tidy manner?			
ii)	Are hand washing facilities provided adequately and conveniently to wash hands, foot, elbow or sensor operated taps?			
iii)	Are germicidal soaps/soap solution and hand drying facility provided?			
iv)	Are notice/instructions prominently pasted in toilet directing employees to wash their hands on entry			

Sl No.	Requirements	Answers		Remarks
		Applied	Not Applied	
(1)	(2)	(3)	(4)	(5)
	and re-entry in to the packaged natural mineral water handling areas?			
v)	Are the refuse receptacles self closing type maintained in a manner to protect from contaminations?			
J	Personnel Hygiene and Habits			
i)	Is any individual assigned to supervise overall sanitation of plant and personnel?			
ii)	Is there any person responsible for day-to-day monitoring of health and hygiene?			
iii)	Have the employees in processing, packing and maintenance been medically examined?			
iv)	Are the personnel with infectious diseases, skin infection and open lesion or any other source of microbial contamination excluded from working in process/packing areas?			
v)	Are personnel hygiene practices regularly maintained and monitored? a) clean outer garments-protective clothing? b) personal cleanliness-finger nails? c) head cover-hair restraints, caps, head bands, beard cover? d) no tobacco in any form-smoking chewing? e) no eating at workstations?			
vi)	Are protective clothing stored on the premises and not allowed to be used for outside wear?			
vii)	Are there clear legible notices defining limits of no smoking areas such as 'NO SMOKING BEYOND THIS POINT' displayed?			
viii)	Are personnel imparted regular training or hygienic food handling, processing food and personal hygiene?			
ix)	Are unsecured jewellery and other objects such as wrist watches, cufflinks, ear rings, glass bangles, stick BINDIS removed at work?			

ANNEX C

(Clause [6.1.4](#))**DETECTION AND ENUMERATION OF THE SPORES OF SULPHITE-REDUCING ANAEROBES (CLOSTRIDIA)****C-1 PRINCIPLE**

The spores of sulphite-reducing anaerobes (clostridia) are widespread in the environment. They are present in human and animal faecal matter, in waste water and, in soil. Unlike *Escherichia coli* and other coliform organism, the spores survive in water for long periods as they are more resistant than vegetative forms to the action of chemical and physical factors. They may thus give an indication of remote or intermittent pollution. They may even be resistant to chlorination at levels which are normally used for the treatment of water.

C-2 APPARATUS AND GLASSWARE

C-2.1 Screw-cap bottles or vials and stoppers of boron silicate glass of capacities 200 ml, 100 ml and 25 ml.

C-2.2 Volumetric Pipettes — of capacities 10 ml and 1 ml

C-2.3 Water Baths — thermostatically controlled

C-2.4 Test Tubes — 150 mm × 13 mm

C-2.5 Iron Wire

C-2.6 Incubator — capable of being maintained at 37 °C ± 1 °C.

C-3 ENRICHMENT CULTURE

Detection and enumeration of spores of sulphite reducing anaerobes by inoculating volumes of the sample into liquid enrichment media followed by incubation at 37 °C ± 1 °C for 44 h ± 4 h in anaerobic conditions.

C-4 CULTURE MEDIA AND REAGENTS**C-4.1 Basic Materials**

In order to improve the reproducibility of the results, it is recommended that, for the preparation of the diluents and culture media, dehydrated basic components or complete dehydrated media be used. Similarly, commercially prepared reagents may also be used. The manufacturer's instructions shall be followed. The chemical products used for the preparation of the culture media and the reagents shall be of recognized analytical quality. The water

used shall be distilled or deionized water, free from substances that might inhibit the growth of micro-organisms under the test conditions. Measurement of pH shall be made using a pH meter, measurements being referred to a temperature of 25 °C. If the prepared culture media are not used immediately, they shall unless otherwise stated, be stored in the dark at approximately 4 °C, for no longer than 1 month.

C-4.2 Differential Reinforced Clostridial Medium (DRCM)**C-4.2.1 Single Strength Basal Medium***Composition*

Peptonetryptic digest of meat	10 g
Meat extract	10 g
Yeast extract	1.5 g
Starch	1 g
Hydrated sodium acetate	5 g
Glucose	1 g
L-Cysteine-hydrochloride	0.5 g
Water	1 000 ml

- a) Mix the peptone, meat extract, sodium acetate and yeast extract with 800 ml of water;
- b) With the remaining 200 ml of distilled water, prepare a starch solution by mixing the starch in a little cold water to form a paste. Heat the rest of the water to boiling point and slowly add it to the paste with constant stirring;
- c) Then add this starch solution to the first mixture and heat to boiling point until it dissolves;
- d) Finally, add the glucose and and L-cysteine-hydrochloride dissolve;
- e) Adjust the pH between 7.1 and 7.2 with sodium hydroxide; and
- f) Transfer 25 ml aliquots of the medium into screw capped bottles of capacity 25 ml. Sterilize in the autoclave at 121 °C ± 1 °C for 15 min.

C-4.2.2 Double Strength Basal Medium

Prepare the double strength medium as in [C-4.2.1](#) but reduce the volume of water by half. Transfer

10 ml and 50 ml aliquots of the medium into screw-capped bottles of capacities 25 ml and 100 ml respectively.

C-4.3 Sodium Sulphite (Na₂SO₃), 4 Percent (m/m) Solution

Dissolve 4 g of anhydrous sodium sulphite in 100 ml of water. Sterilize by filtration. Store at between 2 °C and 5 °C.

It is advisable to prepare a fresh solution every 14 days.

C-4.4 Iron (III) Citrate (C₆H₅O₇ Fe), 7 Percent (m/m) Solution

Dissolve 7 g of iron (III) citrate in 100 ml of water. Sterilize by filtration. Store at between 2 °C and 5 °C.

It is advisable to prepare a fresh solution every 14 days.

C-4.5 Complete Medium

C-4.5.1 On the day of analysis, mix equal volumes of the solutions of sodium sulphite (*see* [C-4.3](#)) and iron (III) citrate (*see* [C-4.4](#)).

C-4.5.2 Add 0.5 ml of the mixture (*see* [C-4.5.1](#)) to each bottle of single strength medium (*see* [C-4.2.1](#)) which has been freshly heated and cooled.

C-4.5.3 Add 0.4 ml of the mixture (*see* [C-4.5.1](#)) to each 10 ml, and 2 ml to each 50 ml of double strength medium (*see* [C-4.2.2](#)) similarly treated.

C-4.6 Selection of Spores (Technique)

Before the test, the sample of water should be heated in a water bath at 75 °C ± 5 °C for 15 min from the time it reaches that temperature. A similar bottle containing the same volume of water as the test sample should be used periodically as a control in order to check the heating time required. The temperature of the water in the control bottle can be constantly recorded by thermometer.

C-4.7 Inoculation and Incubation

- a) Add 50 ml of sample (*see* [C-4.6](#)) to a 100 ml screw cap bottle containing 50 ml of the double strength complete medium (*see* [C-4.5.3](#));
- b) Add 10 ml of sample (*see* [C-4.6](#)) to a series of five 25 ml screw cap bottles containing 10 ml of double strength complete medium (*see* [C-4.5.3](#));
- c) Add 1 ml of sample (*see* [C-4.6](#)) to a series of five 25 ml screw cap bottles containing 25 ml of single strength complete medium (*see* [C-4.5.2](#));
- d) If necessary, add 1 ml of a 1 to 10 dilution of the sample (*see* [C-4.6](#)) to a series of five 25 ml screw cap bottles containing 25 ml of single strength complete medium (*see* [C-4.5.2](#));
- e) In order to carry out a qualitative examination of 100 ml of mineral water or packaged water without making an MPN count, use a 200 ml vial filled with a mixture of 100 ml of double strength complete medium (*see* [C-4.5.3](#)) and 100 ml of sample (*see* [C-4.6](#));
- f) If necessary, top up all the bottles with the single strength complete medium (*see* [C-4.5.2](#)) to bring the volume of liquid level with the neck and to ensure that only a very small volume of air remains, then seal the bottles hermetically, or incubate under anaerobic conditions;
- g) Incubate the inoculated bottles at 37 °C ± 1 °C for 44 h ± 4 h;
- h) Large volumes of culture in hermetically sealed glass bottles may explode due to gas production. The addition of iron wire, heated to redness and placed into the medium before inoculation, may aid anaerobiosis; and
- j) Bottles in which blackening is observed, as a result of the reduction of sulphite and the precipitation of iron (II) sulphide, shall be regarded as positive.

ANNEX D

(Clause [6.1.5](#))DETECTION AND ENUMERATION OF *Pseudomonas aeruginosa*D-1 *Pseudomonas aeruginosa*

Micro-organisms capable of growth and producing a water soluble, fluorescent pigment in media containing asparagines and ethanol. They also produce characteristic colonies when grown on agar medium containing milk at 42 °C. Some strains are non-pigmented.

D-2 PRINCIPLE

Measured volumes of the water sample or a dilution of the sample, are added to a selective medium in containers and incubated under the conditions given for the medium. Examination of the containers either for the presence of a water-soluble fluorescing pigment under ultraviolet irradiation or for growth is done. Sub-cultures are made from each container showing growth or fluorescence onto plates of milk agar medium. After incubation, the plates are examined for typical colonies of *Pseudomonas aeruginosa*, sub-cultures are made from each container onto the surface of a solid agar plate and incubated. Pure cultures are obtained by further subculture onto plates of the same agar medium as required. Each pure culture is finally tested for certain bio-chemical characteristics (see [D-11](#)).

D-3 DILUENT, CULTURE MEDIA AND REAGENTS

Use reagents of analytical reagent quality in the preparation of culture media and diluents, unless otherwise specified. Prepare media using reagent grade water.

D-4 CULTURE MEDIA

It is essential that the culture medium used be suitable for the type of water to be analysed and the purpose of the analysis. Use the following medium for the determination of presumed *Pseudomonas aeruginosa*:

Sl No. (1)	Constituents (2)	Single Strength (3)	Concentrated (4)
i)	DL asparagine	2 g	3.2 g
ii)	L Proline	1 g	1.6 g

Table (Concluded)

Sl No. (1)	Constituents (2)	Single Strength (3)	Concentrated (4)
iii)	Anhydrous dipotassium hydrogen phosphate	1 g	1.6 g
iv)	Magnesium sulphate heptahydrate	0.5 g	0.8 g
v)	Anhydrous potassium sulphate	10 g	16 g
vi)	Ethanol	25 ml	40 ml
vii)	Water	1 000 ml	1 000 ml

D-4.1 Preparation

Dissolve all the constituents in the water and proceed in either of the following ways:

- Add the ethanol and distribute in sterile screw capped bottles. Tighten the caps on the bottles to the point where the seal in the lid just begins to engage with the lip of the bottle, autoclave at 121 °C ± 1°C for 15 min. Tighten the caps on each bottle, immediately after removal from the autoclave, to prevent loss of ethanol by evaporation. Do not use polypropylene caps without seals; and
- Alternatively, sterilize the ethanol by filtration through a cellulose acetate or nitrate membrane of average pore size 0.22 µm and then add it aseptically to the medium after autoclaving and cooling. Adjust the pH to 7.2 ± 0.2. Store in screw capped bottles in the dark at room temperature for up to a maximum of 3 months.

D-5 CONFIRMATORY MEDIUM

Skim milk powder	100 g
Yeast extract broth (see 5)	250 ml
Agar	15 g
Hexadecyltrimethylammonium bromide (cetrimide)	0.3 g
Water	750 ml

Yeast extract broth:

Bacteriological yeast extract	3 g
Bacteriological peptone	10 g
Sodium chloride	5 g
Water	1 000 ml

D-5.1 Preparation on Medium

Prepare the yeast extract broth by dissolving all the constituents in the distilled water by steaming. Adjust the pH between 7.2 and 7.4. Sterilize by autoclaving at $121\text{ °C} \pm 1\text{ °C}$ for 20 min.

Mix the sterile yeast extract broth, cetrimide and agar, and steam this mixture until the agar has dissolved. In a separate glass container, add the skim milk powder to the distilled water and mix, preferably with a magnetic stirrer, until the powder has completely dissolved. Autoclave both solutions separately at $121\text{ °C} \pm 1\text{ °C}$ for 5 min. To prevent caramelization of the milk, take care to follow these instructions. Cool the solutions to 50 °C , aseptically add the milk solution to the agar medium and mix well.

D-6 APPARATUS AND GLASSWARE

- All glassware shall be sterilized at $170\text{ °C} \pm 5\text{ °C}$ for 1h in a dry oven or at $121\text{ °C} \pm 1\text{ °C}$ for 15 min in an autoclave before use.
- Use sterile petri dishes with a diameter of either 90 mm or 100 mm.
- Incubators, capable of being maintained, at $37\text{ °C} \pm 1\text{ °C}$ and $42\text{ °C} \pm 0.5\text{ °C}$.
- Ultraviolet lamp emitting light of wavelength $360\text{ nm} \pm 20\text{ nm}$.

NOTE — Sterile square plastics Repli dishes may be used as an alternative to glass bottles or tubes when the volume of sample or dilution of the sample under examination is 1 ml or less.

Plastics Repli dishes are square dishes divided into 25 identical compartments which can hold 1 ml of medium together with 1 ml of sample or sample dilution. The use of these dishes allow 5 replicates from each of five serial dilutions of the sample to be tested simultaneously. The dishes can be obtained pre-sterilized.

D-7 INOCULATION AND INCUBATION

Pass 250 ml or as appropriate quantity of water sample through a 0.45 micron membrane filter; the membrane is inoculated into concentrated asparine proline broth medium (see [D-4](#)).

Incubate the medium at $37\text{ °C} \pm 1\text{ °C}$ for 48 h. Examine for growth and fluorescence under an ultraviolet lamp in either a darkened room or apparatus designed to exclude visible light.

NOTE — The membrane filtration technique outlined in IS 15188 may be used to pass the sample of water to be tested through membrane before the test.

D-8 CONFIRMATION

D-8.1 Milk Agar

Sub-culture a loopful of culture medium from each container showing either fluorescence or growth onto a milk agar plate (see [D-5](#)), incubate the milk agar plates at $42\text{ °C} \pm 0.5\text{ °C}$ for 24 h. Examine the plates for growth, pigment production, and casein hydrolysis (clearing of the milk medium around the colonies) and record the reactions as shown in [Table 5](#).

D-9 DETECTION

All containers of the culture medium exhibiting either growth or fluorescence, which yield colonies (after subculture on milk agar plates) that produce either reaction col (3) or col (4) (see [Table 5](#)) shall be regarded as positive for the presence of *Pseudomonas aeruginosa*.

NOTE — Others identified as non-pigmented or atypical *Pseudomonas aeruginosa* by the procedure in [D-10](#) may be included also.

D-10 NON-PIGMENTED STRAINS

As a further step, it is possible to obtain confirmation of non-pigmented strains. If required, a suitable method is to take a loopful of culture medium and transfer it to a milk agar plate. The plate is incubated at temperature of $37\text{ °C} \pm 1\text{ °C}$ for 24 h. A well-isolated colony is selected and final confirmation is obtained by testing for certain biochemical characteristics (see [D-11](#)). Commercially available identification kits may be used.

D-11 FURTHER INFORMATION ABOUT PSEUDOMONAS AERUGINOSA

Pseudomonas aeruginosa is the type specie of the genus *Pseudomonas*. It is a gram negative, non-sporing rod which is oxidase and catalase positive. It is capable of growth at 42 °C but not at 4 °C ; it usually produces a water soluble fluorescent pigment (98 percent of strains) and exhibits oxidative metabolism as indicated by the Hugh and Leifson test. It generally reduces nitrate beyond the stage of nitrite and produces ammonia from the breakdown of acetamide.

Gelatin is liquefied, casein is hydrolysed, but starch is not hydrolyzed. The pigment pyocyanine

(blue-green) is produced by more than 90 percent of strains

Table 5 *Pseudomonas aeruginosa* Reactions

(Clauses [D-8.1](#) and [D-9](#))

Sl No.	Reaction Mode	Reaction		
		Typical	Atypical ¹⁾	
(1)	(2)	(3)	(4)	(5)
i)	Casein hydrolysis	+	+	+
ii)	Growth at 42 °C ± 0.5 °C	+	+	+
iii)	Fluorescence (under UV irradiation only)	+	+	-
iv)	Pyocyanine (blue-green) pigment	+	-	-

+ - positive reaction
- - negative reaction

¹⁾ Other bacteria can sometimes live atypical reactions (4) and (5). In such instances the procedure described in [D-9](#) should be followed.

ANNEX E

(Clause 7.1)

GUIDELINES FOR HANDLING OF POLYETHYLENE FLEXIBLE FILM MEANT FOR PACKING OF PACKAGED DRINKING WATER IN POUCHES

E-1 Polyethylene flexible film meant for packing of packaged drinking water in pouches should have suitable sturdy and dust proof outer packing to prevent contamination during transport, storage and handling. The supplier must be instructed to apply such packing immediately after the film manufacture. Such outer packaging must remain intact till the final loading of the film on the pouch filling machine. Care should however be taken to clean such outer packaging and render the same dust-free before the same is carried into the filling room.

E-2 Printing of the film must be done in such a way that the printing material does not interfere with the final product.

E-3 Such film must be stored in dry, cool and dust-free environment away from strong smelling substances, chemicals, cleaning material etc. It will be ideal to have separate store rooms exclusive for packaging material.

E-4 While handling the film the personnel should adhere to the following basic hygiene precautions:

- a) Finger nails of personnel should be trimmed close and well so that no unhygienic substances are found below the nails;
- b) Hands should be cleaned with disinfecting soap and dried, preferably gloved; and
- c) Personnel should wear head cover and mask while handling the film.

E-5 The pouch filling machine must have suitable means to sterilize the film prior to forming the pouch. UV sterilization may be considered taking into account the following aspects:

- a) The length and intensity of the lamps must be suitable for sterilizing the film on the active surface, that is, the surface that will be in contact with the product and the speed of the machine. The equipment supplier's certificate to that effect must be maintained for record;
- b) The UV lamp supplier must certify as to the expected life in number of hours. The filling

machine should have a mechanism to monitor the number of hours of usage, suitably interlocked with the rest of the equipment so that a reliable method to record the actual usage is available;

- c) Partly used and unused film must be stored with all precautions in accordance with **E-3** above; and
- d) Guide-rods, etc, that may direct the film in the formation of the flexible packaging and other contact parts must be suitably sanitized with hydrogen peroxide before start of every filling operation and records of the same maintained.

E-6 Fumigation of the filling room with suitable agent is recommended.

E-7 Size of the secondary packaging must take into account that at the retail point the flexible packaged drinking water is often refrigerated and so the secondary packaging should be of appropriate size to facilitate refrigeration in the secondary packaging itself. This would shield the pouches from contaminations.

E-8 The following storage instructions must be issued to retailer/whole seller and all concerned in the supply chain:

- a) Packaged Drinking Water in flexible packaging must be handled with care;
- b) It should be stored away from sunlight and in a cool place;
- c) It should be hygienically stored in a place away from chemicals, paints, pesticides and similar substances that can affect the product;
- d) It should also be stored away from strong-smelling substances;
- e) Chilling the product with commercially produced ice must be discouraged as it would expose the consumer to product with possible contamination from ice produced with unsafe water;
- f) To check for any leakage, etc, before opening;

- g) Instruments used for opening/cutting sachets should be kept exclusive for these pouches in a suitable place to avoid any contamination and should not be used for cutting or opening any other non-food product;
- h) Consumer must be advised to use clean scissors to open sachet; and
- j) Product should not be consumed if any foreign material is found.

ANNEX F

(Clause 9)

SAMPLING PLAN FOR PACKAGE NATURAL MINERAL WATER

F-1 GENERAL REQUIREMENTS OF SAMPLING FOR CONTAINERS UP TO AND INCLUDING 2 LITRES

F-1.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed as far as possible:

- a) Sample shall be drawn in original sealed bottle/container and kept in protected place not exposed to damp air, dust or soot; and
- b) Each bottle/container in original shall be sealed and marked with full details of sampling.

F-1.2 Scale of Sampling**F-1.2.1 Lot**

The quantity of packaged natural mineral water of the same type belonging to the same batch of manufacture and packed in a day, shall constitute a lot.

F-1.2.2 For ascertaining the conformity of the material to the requirements of the specification, samples shall be tested from each lot separately.

F-1.2.3 The number of bottles to be selected from a lot shall depend on the size of the lot and shall be according to [Table 6](#).

Separate bottle(s) shall be drawn for testing for the microbiological requirements. Additional bottles may be drawn if required for carrying out complete testing or when the samples are required to be sent in more than one laboratory.

F-1.2.3.1 The bottles shall be chosen at random from the lot. In order to ensure the randomness of

selection, procedure given in IS 4905 shall be followed.

Table 6 Scale of Sampling(Clauses [F-1.2.3](#), [F-1.2.4](#) and [F-1.3.1](#))

Sl No.	Number of Bottles in the Lot	Number of Samples
(1)	(2)	(3)
i)	Up to 5 000	3
ii)	5 001 to 10 000	5
iii)	10 001 to 15 000	7
iv)	15 001 and above	9

F-1.2.4 Initially the number of cartons equal to the number of bottles to be taken from the lot [according to col (3) of [Table 6](#)], shall be chosen at random. These cartons thus selected shall be opened and the bottles in these cartons examined visually for the condition of packing, external appearance and the fill. The lot shall be considered satisfactory for inspection of other characteristics given in the specification, if all the bottles in the cartons opened are found satisfactory for these characteristics.

F-1.2.5 In case any defective bottle is found according to [F-1.2.4](#), twice the number of cartons shall be opened and the bottles examined for these characteristics. If no defective bottle is found; the lot shall be considered satisfactory for inspection of other characteristic given in the specification.

F-1.3 Preparation of Test Samples

F-1.3.1 From each of the cartons opened according to [F-1.2.4](#), three bottles shall be taken from its different layers so as to obtain three times the required number of bottles in the sample [see col (3) of [Table 6](#)].

F-1.3.2 In case the number of cartons to be opened is according to [F-1.2.4](#), the number of cartons equal to the number of bottles in the sample shall be taken at random from these cartons and then the required number of bottles picked up according to [F-1.3.1](#).

F-1.3.3 The sample bottles selected as in [F-1.3.1](#) or [F-1.3.2](#) shall be divided at random into three equal sets and labelled with all the particulars of sampling. One of these sets of sample bottles shall be for the purchaser, another for vendor and the third for referee.

F-1.3.4 Referee Sample

Referee sample shall consist of a set of sample bottles 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 so as to be used in a case of a dispute between the two.

F-1.4 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the specification, if all the requirements are complied with.

F-2 GENERAL REQUIREMENTS OF SAMPLING FOR ABOVE 2 LITRES CONTAINERS

F-2.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed as far as possible:

- a) Sample shall be drawn in original sealed container and kept in protected place not exposed to damp air, dust or soot; and
- b) Each container in original shall be sealed and marked with full details of sampling.

F-2.2 Scale or Sampling

F-2.2.1 Lot

The quantity of packaged natural mineral water of the same type belonging to the same batch of manufacture and packed in a day, shall constitute a lot.

F-2.2.2 For ascertaining the conformity of the material to the requirements of the specification, samples shall be tested from each lot separately.

F-2.2.3 The number of containers to be selected from a lot shall depend on the size of the lot and shall be drawn at random, according to [Table 7](#). Separate container(s) shall be drawn for testing for the microbiological requirements. In order to ensure the randomness of selection, procedure given in IS 4905 shall be followed.

Table 7 Scale of Sampling

(Clause [F-2.2.3](#))

Sl No. (1)	Number of container in the Lot (2)	Number of Samples (3)
i)	0 to 500	5
ii)	501 to 1 200	8
iii)	1 201 to 3 200	13
iv)	3 201 and above	20

F-2.2.4 These containers shall first be examined visually for the condition of packing, external appearance and the fill. The lot shall be considered satisfactory for inspection of other characteristics given in the specification, if all the containers are found satisfactory for these characteristics.

F-2.2.5 In case any defective container is found according to [F-2.2.4](#), twice the number of containers shall be examined for these characteristic(s). If no defective container is found, the lot shall be considered satisfactory for inspection of other characteristics given in the specification.

F-2.3 Preparation of Test Samples

F-2.3.1 Out of the containers selected according to [F-2.2.3](#), any three containers shall be selected at random and stored separately.

F-2.3.2 Each of the sample containers selected as in [F-2.3.1](#) shall be divided at random into three equal sets and labelled with all the particulars of sampling. One of these sets of sample containers shall be for the purchaser, another for vendor and the third for referee.

F-2.3.3 Referee Sample

Referee sample shall consist of a set of sample containers 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 so as to be used in case of dispute between the two.

F-2.4 Criteria for Conformity

The lot shall be declared as conforming to the

requirements of the specification, if all the requirements are complied with.

ANNEX G

[Table 2, Sl No. (vii)]

DETERMINATION OF BARIUM CONTENT

G-1 GENERAL

It is not necessary to look for barium if sulphate is present in appreciable amount unless sample contains large amount of bicarbonate or chloride which may hold in solution small amounts of both sulphate and barium (as Ba).

G-2 REAGENTS

G-2.1 Ammonium Dichromate Solution

Dissolve 100 g of sulphate free ammonium dichromate in water and dilute to 1 000 ml.

G-2.2 Ammonium Acetate Solution

Dissolve 300 g of ammonium acetate in water, neutralize with ammonium hydroxide and dilute to 1 000 ml.

G-2.3 Dilute Ammonium Acetate Wash Solution

Dilute 20 ml of the ammonium acetate solution (see G-2.2) to 1 000 ml.

G-2.4 Potassium Iodide Solution — 10 percent

G-2.5 Standard Sodium Thiosulphate Solution — 0.1 N

G-2.6 Hydrochloric Acid — 1 : 1 (v/v)

NOTE — Reaction of acetate solutions should be alkaline rather than acidic.

G-3 PROCEDURE

Acidify 1.5 litre portion of sample with hydrochloric acid and concentrate to about 200 ml (if precipitate forms, filter off and test for barium). Add about 0.5 g of ammonium chloride and precipitate iron and aluminium with ammonium hydroxide. Boil, filter and wash. To filtrate, add excess (10 ml) ammonium acetate solution (see G-2.2) keeping total volume to about 200 ml. Heat to boiling and add with stirring about 5 ml ammonium dichromate solution. Allow to cool slowly and wash the precipitate free of chromate with dilute ammonium acetate, wash solution by decantation and filtration. Dissolve the precipitate in about 10 ml hydrochloric acid 1 : 1 (v/v) and hot water.

Wash the filter and dilute the solution to about 400 ml and add about 50 ml freshly prepared 10 percent potassium iodide solution. Mix carefully and titrate liberated iodine (I₂) after 3 min or 4 min with 0.1 N sodium thiosulphate.

1 ml 0.1 N sodium thiosulphate (Na₂S₂O₃) = 4.578 mg barium (Ba).

ANNEX H

[Table 2, Sl No. (viii)]

DETERMINATION OF ANTIMONY BY SPECTROPHOTOMETRIC METHOD

H-1 PRINCIPLE

Pentavalent antimony in aqueous hydrochloric acid solution reacts with rhodamine B to form coloured complex extractable with organic solvents. Intensity of extracted colour is measured spectrometrically at 565 nm wavelength.

H-2 APPARATUS**H-2.1 Suitable Spectrophotometer****H-2.2 Erlenmeyer Flask****H-3 REAGENTS****H-3.1 Hydrochloric Acid Solution — 6 N****H-3.2 Dilute Phosphoric Acid — 3 N**

Dilute 70 ml phosphoric acid (85 percent) to 1 litre with distilled water.

H-3.3 Rhodamine B Solution — 0.02 percent (in distilled water)**H-3.4 Antimony Standard Solutions**a) *Stock Solution* — 100 µg antimony/ml

Dissolve 0.100 g pure Antimony in 25 ml sulphuric acid with gradual heating. Cool and then cautiously dilute to 1 litre with distilled water.

b) *Working Solution* — 1 µg/ml

Dilute 2.0 ml stock solution to 200 ml with distilled water.

Cool reagents [H-3.1](#), [H-3.2](#), [H-3.3](#) and approximately 100 ml benzene, and eight 125 ml separators with teflon stopcocks in refrigerator before use. Maintain temperature at 5 °C to 10 °C during extraction and colour development.

H-3.5 Sulphuric Acid**H-3.6 Perchloric Acid****H-3.7 Benzene****H-4 PROCEDURE**

CAUTION — Safety practices should be followed for handling perchloric acid and sulphuric acid. Contact of perchloric acid with oxidizable or combustible materials or dehydrating or reducing agents may result in fire or explosion. Use goggles, barrier shields and other devices as necessary for personal protection.

Transfer aliquot to 125 ml Erlenmeyer flask, add 5 ml of sulphuric acid, and evaporate to fumes of SO₃. Cool the flask, add 10 drops of 70 percent perchloric acid (HClO₄) and again evaporate to white fumes. Cool the digest in ice-bath for 30 min, and then slowly add 5 ml pre-cooled 6 N hydrochloric acid with the help of pipette. Let it stand in ice-bath for 15 min, then add 8 ml pre-cooled 3 N phosphoric acid. (Until colour is extracted into benzene, perform subsequent operations as quickly as possible. Colour is stable in benzene for several hours). Immediately add 5 ml pre-cooled rhodamine B solution, stopper, and shake vigorously. Transfer to pre-cooled 125 ml separator. Pipette 10 ml pre-cooled benzene into separator, shake vigorously for 1min, and discard aqueous layer. Transfer benzene layer (red if antimony is present) into test tube and let water settle. Rinse 1 cm cell with extract, fill the cell, and read absorption at 565 nm against benzene blank taken through entire determination.

H-5 PREPARATION OF STANDARD CURVE

Pipette 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml antimony working standard solutions into 125 ml

Erlenmeyer flask; add 5 ml sulphuric acid to each, and proceed as [H-4](#). Plot absorption against µg of antimony.

H-6 CALCULATION

Calculate the µg (or mg) of antimony from the graph corresponding to the observed absorption value.

ANNEX J

[Table 2, *Sl No. (ix)*]

DETERMINATION OF BORATE

J-1 PRINCIPLE

Reaction of azomethine-H, which is the condensation product of H-acid (8-amino-naphth-1-ol-3, 6-disulfonic acid) and salicylaldehyde, with dissolved forms of borate at a pH of about 6. Formation of a yellow complex that is, measured spectrometrically at the absorption maximum in the range of 410 nm to 420 nm.

J-2 REAGENTS**J-2.1 Azomethine-H, Solution**

Dissolve 1.0 g of azomethine-H sodium salt [8-N-(2-hydroxybenzylidene)-amino-naphth-1-ol-3, 6- disulphonic acid] ($C_{17}H_{12}NNaO_8S_2$) and 3.0 g of L \pm ascorbic acid ($C_6H_8O_6$) in water and dilute to 100 ml in a volumetric flask.

The solution is stable for up to one week when stored in a polyethylene bottle at a temperature of between 4 °C and 6 °C.

J-2.2 Buffer Solution (pH 5.9)

Mix 250 g of ammonium acetate (CH_3COONH_4), 250 ml of water, 80 ml of sulphuric acid (H_2SO_4) (ρ -1.21 g/ml), 5 ml of phosphoric acid (H_2PO_4) (ρ - 1.71 g/ml), 1.0g of citric acid ($C_6H_8O_7H_2O$) and 1.0 g of disodiummethylenediamine-tetra acetic acid dihydrate ($C_{10}H_{14}N_2Na_2O_8.2H_2O$) by stirring and gentle heating.

J-2.3 Reagent Solution

Mix equal volumes of reagents [J-2.1](#) and [J-2.2](#). Prepare this solution on the day of use and store in a polyethylene bottle.

J-2.4 Borate, stock solution corresponding to 1.0 g of B per litre. Dissolve 5.719 g of boric acid (H_3BO_3) in 1 000 ml of water. Store in a polyethylene bottle. 1 ml of this stock solution contains 1.0 mg of borate, expressed as B.

J-2.5 Boron, standard solution 1 corresponding to 10.0 mg of B per litre. Dilute 10 ml of borate stock solution (see [J-2.4](#)) to 1 000 ml with water. 1 ml of this solution contains 10.0 μ g of borate, expressed as B.

J-2.6 Boron, standard solution 2 corresponding to 1.0 mg of B per litre. Dilute 10 ml of borate

solution (see [J-2.5](#)) to 100 ml with water. 1 ml of this standard solution contains 1.0 μ g of borate, expressed as B.

J-2.7 Calcium Hydroxide [$Ca(OH)_2$]**J-3 APPARATUS**

J-3.1 Ordinary laboratory apparatus made of polypropylene, polyethylene or polytetrafluoroethylene, where applicable.

J-3.2 Spectrometer, for use in the wavelength range of 410 nm to 420 nm, with cells of an optical path length between 10 mm and 50 mm.

J-4 PROCEDURE**J-4.1 Determination**

Transfer 25.0 ml of the sample, or a smaller amount of the sample diluted to 25 ml with distilled water, into a 100 ml polyethylene flask. Add 10 ml of azomethine-H (see [J-2.1](#)). Mix and allow to stand in the dark for 2 h at 20 °C \pm 1°C, then measure the absorbance at the absorption maximum in the range of 410 nm to 420 nm against distilled water in a cell of optical path length 10 mm, using the spectrometer set up according to the manufacturer's instructions and after setting the zero with distilled water in the cell. Alternatively use a cell of 50 mm optical path length for low boron concentrations of up to about 0.2 mg of boron per litre. Check the wavelength of the absorption maximum whenever a new batch of this reagent is used.

NOTE — The reaction time may be shortened by keeping the treated sample at a temperature of 30 °C. In this case the sample, the blank and the calibration samples should be treated accordingly, because the intensity of colour is temperature dependent.

J-4.2 Blank Test

Carry out a blank test by treating 25 ml of water as described in [J-4.1](#). Ensure that the blank value is in the range of 0.1 absorption units to 0.17 absorption units per 10 mm; if the absorption is higher, then check the reagents and the distilled water for their borate content.

NOTE — The following procedure maybe used to check the quality of reagents and the distilled water.

Measure into three separate borate-free beakers (preferably polytetrafluorethylene) 25 ml, 100 ml and 250 ml aliquots of the distilled water. Make each slightly alkaline by the addition of the same small (for example, 200 mg) amount of calcium hydroxide (see J-2.7) to each. Evaporate the 100 ml and 250 ml aliquots to a volume of just less than 25 ml and adjust their volumes to precisely 25 ml by the addition of a little extra distilled water, as necessary. Carry out the procedure given in J-4.1 on these aliquots.

Carry out a blank determination with each of the aliquots. If borate is present in the distilled water, the borate found increases in proportion to the volume of the aliquot taken. Erratic results indicate external borate contamination. Relatively high but constant results indicate impure reagents.

J-4.3 Prevention of Contamination

As borate is widespread in the environment, significant contamination may occur during trace determinations. The following sources of contamination, and remedies, should be considered.

Laboratory glassware is usually made from borosilicate glass. Special borate-free thermally resistant glass is obtainable, but for routine purposes, old borosilicate glass, well rinsed in hydrochloric acid, may be used for acidic solutions, but should never be used for neutral or alkaline solutions, or for prolonged storage at any pH value. Borosilicate glassware previously used with alkaline solutions shall not be used without very thorough acid rinsing. Polyethylene flasks and plastics pipettes are preferable.

Detergents and soaps used for glassware and lab coats should be borate free, and the use of towels and tissues, for drying shall be avoided.

Toiletries, talcum powder and cosmetics used by technicians often contain borate and should be avoided or removed, especially prior to undertaking accurate low-level determinations.

Water and reagents may contain borate and blanks should be carried out at least in duplicate and should agree.

J-4.4 Calibration

J-4.4.1 Zero mg/l to 0.20 mg/l of Boron Calibration Graph

To a series of six 25 ml one mark plastics flasks add

respectively 0 ml, 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of boron standard solution 2 (see J-2.6), dilute to the mark with distilled water and mix. This gives concentrations of 0 mg, 0.04 mg, 0.08 mg, 0.12 mg, 0.16 mg and 0.20 mg of boron per litre respectively. Analyze each standard solution as described in J-4.1, measuring the absorbance values in a 50 mm optical path length cell compared against distilled water. Prepare a calibration graph by plotting the absorbance values against the known concentrations in milligrams of boron per litre for each standard.

J-4.4.2 Zero mg/l to 1.00 mg/l of Boron Calibration Graph

Repeat the above calibration, using 0 ml, 5 ml, 10 ml, 15 ml, 20 ml and 25 ml of boron standard solution 2 (see J-2.6) respectively to give concentrations of 0 mg, 0.2 mg, 0.4 mg, 0.6 mg, 0.8 mg and 1.0 mg of boron per litre respectively. Analyze each standard solution as described in J-4.1, but this time measuring the absorbance values using a 10 mm optical path length cell compared against distilled water. Prepare a separate calibration graph.

J-4.4.3 Calculation of Factor f

It is essential that a linear calibration graph be achieved in both cases; if not then check the solutions and repeat the calibration. Calculate the reciprocal value for the slope, factor f , for each graph.

J-5 CALCULATION

Calculate the borate content, in milligrams of boron per litre, from the formula:

$$\frac{(A_1 - A_0)f V_{1Max}}{V_1}$$

where

- A_1 = absorbance of the sample;
- A_0 = absorbance of the blank;
- f = calibration factor, determined from the appropriate calibration curve (reciprocal value of the slope, in milligrams of boron per litre;
- V_{1Max} = maximum volume, in millilitres, of the sample; and
- V_1 = volume, in millilitres, of the sample; .

ANNEX K

[[Table 2, Sl No. \(x\)](#) and [Table 3, Sl No. \(iv\)](#)]

DETERMINATION OF SILVER AND CHROMIUM CONTENTS

K-1 PRINCIPLE

Metals in solution are determined directly by atomic absorption spectrophotometry. Suspended metals are separated by membrane filtration or suspension is dissolved and analyzed.

K-2 APPARATUS**K-2.1 Atomic Absorption Spectrophotometer**

Spectrophotometer capable of operating at conditions are given in [Table 8](#).

K-3 REAGENTS**K-3.1 De-ionized Distilled Water**

Distilled, ammonia free. Pass through ion exchange column of mixed strongly acidic cation and strongly basic anion exchange resins. Regenerate resins according to manufacturer's instructions.

K-3.2 Nitric Acid

Dilute 500 ml redistilled nitric acid to 1 000 ml with water.

NOTE — Perform distillation in hood with protective ash in place.

K-3.3 Hydrochloric Acid

Dilute 500 ml hydrochloric acid to 1 000 ml with water and distill in all pyrex or equivalent glass apparatus.

K-3.4 Metal Solutions**K-3.4.1 Stock Solutions**

Accurately weigh amount of metal specified in [Table 9](#) into a beaker and add dissolving medium. When metal is completely dissolved, transfer quantitatively into 1 000 ml volumetric flask and dilute to volume with water.

K-3.4.2 Working Solutions

Prepare daily. Dilute aliquots of stock solutions with water to make more than or equal to 4 standard solutions of each element within the range of detection as given in [Table 9](#). Add 1.5 ml nitric acid per litre to all working standard solutions before diluting to volume. Add 1ml lanthanum chloride for every 10 ml making the working standard solution.

K-3.5 Lanthanum Stock Solution

Slowly add 250 ml hydrochloric acid to 58.65 g lanthanum oxide (La_2O_3 , purity 99.9 percent by mass), dissolve and dilute to 1 000 ml.

K-3.6 Ammonium Pyrrolidine Dithiocarbamate Solution

Dissolve 1 g ammonium pyrrolidine dithiocarbamate in 100 ml water. Prepare fresh daily.

K-4 PREPARATION OF SAMPLE**K-4.1 Dissolved Metals**

As soon as practicable after collection, filter known volume of sample through 0.45 μm membrane. Use firstly 50 ml to 100 ml to rinse the flask and discard. Collect the filtrate and preserve the solution by adding 3 ml nitric acid 1 : 1 (v/v) per litre.

K-4.2 Suspended Metals

Transfer the residue and membrane from [K-4.1](#) to a 250 ml beaker and add 3 ml nitric acid. Cover with watch glass and heat gently to dissolve the membrane. Increase heating and evaporate to dryness. Cool and add 3 ml nitric acid and heat until digestion is complete, generally indicated by light coloured residue. Add 2 ml hydrochloric acid (1 : 1, v/v) and heat gently to dissolve the residue. Wash the watch glass and beaker with water and filter. Wash the filter and discard. Dilute the filtrate with water to such a concentration that it is within the range of the instrument.

K-4.3 Total Metal

Transfer an aliquot of well mixed sample to the beaker and add 3 ml nitric acid. Heat and evaporate to dryness (do not boil). Cool and add 3 ml nitric acid and heat until digestion is complete, generally indicated by light coloured residue. Add 2 ml hydrochloric acid (1 : 1, v/v) and heat gently to dissolve the residue. Wash the watch glass and beaker with water and filter. Wash the filter and discard. Dilute the filtrate with water to such a concentration that it is within the range of the instrument.

K-5 DETERMINATION

Transfer an aliquot of the sample to a 250 ml beaker and dilute to 100 ml with water. Prepare blank and standard solution in the same manner. Adjust the pH of the sample and standard solutions to 2.5 with hydrochloric acid using a pH meter. Transfer quantitatively to a 200 ml volumetric flask, add 2.5 ml of ammonium pyrrolidine dithiocarbamate solution and mix. Add 10 ml methyl isobutyl ketone and shake vigorously for 1 min. Let the layers be separated and then add water until the ketone layer

is in the neck of the flask. Centrifuge, if necessary. Aspirate the ketone layer and record readings of standards and samples against blank. The fuel-to-air ratio should be adjusted to as blue a flame as possible since organic solvents add to fuel supply. Prepare the calibration curve from the average of each standard and read the sample concentration.

K-6 CALCULATION

Metal content, mg/l = Metal, in mg, in the aliquot/l.

Table 8 Operating Parameter(Clause [K-2.1](#))

SI No.	Metal	Wavelength	Flame	Optimum Range
(1)	(2)	(3)	(4)	(5)
i)	Silver	328.1	Oxidizing air-acetylene	0.1 to 20
ii)	Chromium	357.9	Reducing air-acetylene	1 to 200

Table 9 Preparation of Metal Standard Solutions(Clauses [K-3.4.1](#) and [K-3.4.2](#))

SI No.	Metal	Weight	Compound	Dissolving Medium (1 litre Total)
(1)	(2)	(3)	(4)	(5)
i)	Silver	1.575	Silver nitrate (AgNO ₃)	Water + 10 ml redistilled nitric acid
ii)	Chromium	1.923	Chromium oxide (Cr ₂ O ₃)	Water + 10 ml redistilled nitric acid

ANNEX L

[Table 3, SI No. (vii)]

DETERMINATION OF NICKEL BY FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD**L-1 PRINCIPLE**

Formation of a complex between the metals being determined and ammonium l-pyrrolidinedithiocarbamate (APDC) and extraction at pH 2.5 with methyl-isobutyl ketone (MIBK).

Determination of the metals in this organic phase by flame atomic absorption spectrometry.

L-2 APPARATUS

L-2.1 Atomic absorption spectrometer fitted with

hollow cathode lamps for appropriate metals or electrodeless discharge lamps, and with a suitable device for allowing for the correction of the nonspecific absorbance and with a nebulizer-burner with an acetylene-air flame.

L-3 REAGENTS

L-3.1 Concentrated Nitric Acid (ρ -1.4 g/ml)

L-3.2 Nitric Acid, 1.5 mol/l

Add 100 ml of nitric acid to 600 ml of water and dilute to 1 000 ml.

L-3.3 Nickel Standard Solution Corresponding to 1 000 g/l

Weigh 1.000 g of pure metal and dissolve it in concentrated nitric acid, heating to effect complete dissolution. Allow to cool and transfer each solution quantitatively to a 1000 ml volumetric flask, dilute to the mark with water and mix. For preparing standard solution, it is also permissible to use metal salts of accurately known composition. One ml of the standard solution contains 1.00 mg of nickel.

L-3.4 Sodium Hydroxide — 2.5 mol/l

Dissolve 100g of sodium hydroxide in water and dilute to 1 litre.

L-3.5 Hydrochloric Acid — 0.3 mol/l

Mix 25 ml of concentrated hydrochloric acid (ρ -1.19 g/ml) with water and dilute to 1 litre.

L-3.6 Methyl isobutyl ketone (MIBK)
(that is, 4-methyl-2-pentanone).

L-3.7 Ammonium 1-Pyrrolidinedithiocarbamate (that is, Ammonium - Pyrrolidinocarbodithioate) (APDC) Solution — 20 g/l.

Dissolve 2.0 g of APDC in water. Make up the volume to 100 ml with water and mix. Filter the solution if a precipitate is present. If the solution is coloured, purify it by repeated extraction with MIBK until the solution is colourless. Prepare this solution freshly for each batch of samples.

L-3.8 Bromophenol Blue Indicator Solution —

One gram of bromophenol blue per litre of 50 percent (v/v) ethanol solution.

L-4 PRE-TREATMENT OF SAMPLE

L-4.1 For majority of samples, mineralization using hydrochloric acid or nitric acid will be satisfactory.

The alternative procedures of mineralization which follow are given as examples.

L-4.2 Add 5 ml of hydrochloric acid (ρ -1.19 g/ml) for each 100 ml of test portion. Heat in a steam-bath until the volume has been reduced to between 15 ml and 20 ml, making sure that the sample does not boil.

Cool and filter to remove insoluble materials that could clog the nebulizer. Collect the filtrate in a 100 ml volumetric flask. Wash the filter several times with water.

L-4.3 Add 4 ml of 15 mol/l nitric acid to 100 ml of the sample and heat until the volume is reduced to 50 ml.

Place the treated sample in a boiling flask. Add 12 ml of hydrochloric acid (ρ -1.19 g/ml). Connect the boiling flask to a condenser and reflux the solution for 2.5 h.

Cool and filter to remove insoluble materials that could clog the nebulizer. Collect the filtrate in a 100 ml volumetric flask.

Wash the condenser and filter several times with water, add this to the contents of the volumetric flask and make up to the mark with water.

L-5 PROCEDURE

L-5.1 Test Portion

Place in a 100 ml one-mark volumetric flask a test portion of the acidified sample containing 5 μ g to 20 μ g of nickel being determined. Make up to the mark with water.

L-5.2 Chelation and Extraction

Place the test portion and 100 ml of each of the calibration solutions into a series of 250 ml separating funnels fitted with polytetrafluoroethylene (PTFE) taps.

Add to each funnel 2 drops to 3 drops of bromophenol blue indicator and sodium hydroxide until a blue colour persists.

While stirring, add drop-wise hydrochloric acid until the blue colour just disappears. Then add 2 ml of hydrochloric acid in excess. The pH value shall then be 2.3 to 2.5 (see [Note 1](#)).

Add 5 ml of APDC, mix, then add 10.0 ml of MIBK. Shake vigorously for 2 min. The pH shall be approximately 2.8.

Allow the mixture to settle for at least 1 h away from light or heat in the stoppered funnel. The settling time shall be strictly the same for all the solutions. Collect the organic layer taking care to avoid any trace of the aqueous phase (centrifuge if necessary, see [Note 2](#)).

NOTES

- 1 A pH-meter may be used in place of indicator.
- 2 The settling period may be prolonged without disadvantage if it takes place in the dark at a temperature of about 5 °C. In this case it may not be necessary to centrifuge the organic phase.

L-5.3 Blank Test

Carry out a blank test in parallel with the determination, by the same procedure using the same quantities of all the reagents as in the sampling

and chelation and extraction, but replacing the test portion by water.

L-5.4 Preparation of the Sets of Calibration Solutions

Dilute with water immediately before use, the nickel standard solution in order to obtain diluted solutions containing 10 mg of nickel per litre.

In a 500 ml volumetric flask, place:

- a) 20 ml of nickel solution that contains 10 mg/l of nickel; and
- b) 0.5 ml of nitric acid.

Make up to the mark with water. This is solution S. Prepare at least four calibration solutions by diluting solution S with water so as to cover the ranges of concentrations of nickel from 0 µg/l to 200 µg/l.

Acidify each calibration solution by adding the same nitric acid which has been added to preserve the samples. The volume added shall be such that the concentrations of nitric acid are the same in the sample and in the calibration solutions.

L-5.5 Calibration and Determination

Before carrying out the spectrometric measurements, set up the spectrometer according to the manufacturer's instructions by aspirating the organic extract of a calibration solution of nickel being determined and using wave length 232 nm and oxidizing acetylene-air flame. Optimize the aspiration and flame conditions. Adjust the response of the instrument to zero absorbance with MIBK.

Aspirate the organic extracts of the calibration solutions. Plot a graph having the nickel content, in micrograms per litre of the calibration solutions as abscissae and the corresponding values of absorbance as ordinates. It is advisable that the calibration graph be checked, for example by measuring the absorbance of a calibration solution every 5 samples. Aspirate the organic extract of the test portion. Measure the absorbance and after each measurement aspirate MIBK in order to rinse the nebulizer system.

L-5.6 Calculation

By reference to the calibration graph, determine the concentrations corresponding to the absorbance of the test portion and of the blank.

The concentration, expressed in micrograms per litre, of the sample is given by the formula:

$$(Q_t - Q_b) \times \frac{100}{V}$$

where

- Q_t = nickel concentration, in microgram per litre, corresponding to the absorbance of the test portion;
- Q_b = nickel concentration, in microgram per litre, corresponding to the absorbance of the blank; and
- V = volume, in millilitre, of the acidified sample taken for the analysis.

ANNEX M

[Table 3, Sl No. (viii)]

TEST FOR POLYCHLOROBIPHENYLES (PCB)

M-1 PRINCIPLE

The procedure comprises separation of PCB from the organochlorine pesticides residues by means of silica gel adsorption column and determination of its presence by gas-liquid chromatography (GLC).

M-2 APPARATUS

M-2.1 Glass Chromatographic Column — 300 mm long, 8 mm ID with a ground-glass socket at the upper end and a stopcock at the lower end.

M-2.2 Gas Chromatograph

Equipped with electron capture detector and coupled with printer-plotter-cum-integrator.

- a) Column 1 — 1.8 m long, 3 mm ID; apiezon L grease + 0.15 percent epikote resin 1 001 on chromosorb G (acid-washed, DMCS treated, 60 to 80 mesh);
- b) Column 2 — 1.8 m long, 3 mm ID; 1.3 percent silicone gum GE - SE-52

+ 0.15 percent epikote resin 1 001, on the same support material;

Temperature:

Column oven	200 °C
Detector	2 000 °C
Injection port	200 °C

M-2.3 Electron Capture Detector

M-2.4 Kuderna-Danish Type, Evaporator — with interchangeable 10 ml graduated collection tubes

M-2.5 Snyder Columns — two-bubble micro-columns, with ground glass cones to fit the Kuderna-Danish type, 10 ml collection tubes.

M-2.6 Syringe — 5 µl capacity

M-3 REAGENTS

M-3.1 Silica Gel — 60 to 100 mesh.

Heat the gel at 260 °C for 4 h and, when it is cooled to 65 °C, place it in a desiccator. When it has cooled finally to room temperature, weigh the required amount into a glass-stoppered flask and quickly add 2.5 ml distilled water to each 100g. Immediately stopper the flask and shake it for 1.5 h on a shaker. The silica gel is ready for use.

M-3.2 N-hexane — redistilled from potassium hydroxide pellets. When concentrated 100-fold, 5 µl portion should give no significant GLC peaks.

M-3.3 Sodium Hydroxide Solution — 5 N

M-3.4 Diethyl Ether — chromatography grade

M-3.5 Cotton Wool — extracted with hexane and diethyl ether

M-3.6 Acetic Acid, glacial — redistilled

M-3.7 Chromium Trioxide — re-crystallised

M-4 SEPARATION OF PCB FROM THE MORE POLAR PESTICIDE RESIDUES

Weigh 5.0 g of the prepared gel and rapidly transfer it, by using small amounts of hexane, to the chromatographic column, in which a plug of cotton wool has been placed just above the stopcock. The stopcock may be moistened with solvent but must not be lubricated with grease. Allow the gel to settle in the column and remove and trapped air bubbles by stirring with a glass rod. Drain the surplus hexane from the column until its meniscus just touches the surface of the gel. Introduce the cleaned-up sample

extract as a solution in 1 ml of hexane, and allow the hexane to drain until the meniscus again just touches the surface of the gel. Wash the vessel that contained the extract with two 1 ml portions of hexane, adding each washing separately to the column and allowing it to run just into the gel. Place a receiver, preferably a Kuderna- Danish evaporator, beneath the column and pass 42 ml of hexane through the column at a rate not exceeding 0.7 ml/min. Collect the eluate, stopping the elution when the meniscus reaches the top of the gel, and label this fraction 1. It should contain all the PCB. Hexachlorobenzene, aldrin, *o,p'*-DDT and *pp'*-DDE, if present, are also eluted in this fraction. Change the receiver and pass 50 ml of a 10 percent solution of diethyl ether in hexane through the column. Collect the eluate and label it fraction 2. This contains the remainder of the organochlorine pesticide residues, including usually a small amount of *pp'*-DDE, and can be used for the determination of these. Concentrate fraction 1 ml to 5 ml in the Kuderna-Danish evaporator and examine it by GLC with electron capture detection, on at least two stationary phases of different polarities. If further concentration is necessary, reduce the volume of the solution with a gentle stream of dry air or nitrogen at room temperature. Compare the chromatograms from fraction 1 with those given by solutions of commercial PCB preparations when injected on the same columns as well as the correspondence between peak retention times which gives indication of the presence of PCB in the sample.

M-5 OXIDATION OF *pp'*-DDE IN THE PCB FRACTION

M-5.1 Adjust the volume of fraction 1 ml to 2 ml, concentrating, if necessary, as described above. Add 2 ml of acetic acid and, after replacing the micro Snyder column, heat the tube cautiously in the steam bath until all of the hexane has evaporated, as judged by the reduction in volume. Introduce 100 mg of chromium trioxide and place the tube in boiling water for 15 min to 20 min. Cool the mixture and shake it vigorously with 2.0 ml of hexane (accurately measured) in the stoppered tube. Neutralize the acid with 6 ml to 7 ml of 5 N sodium hydroxide, solution. Shake the tube again and then set it aside until the two layers separate. Keep the tube well stoppered to prevent loss of hexane.

M-6 DETECTION OF PCB

Inject 5 µl of the upper hexane layer of the oxidized mixture on to at least two GLC columns with different liquid phases, one of which should be Apiezon L. Compare the retention times of the peaks

so obtained with those given by the PCB reference material. Agreement between the retention times of the peaks so obtained now indicates the presence of PCB compounds in the sample with a greater degree of certainty. Any *pp'*-DDE, which was present in fraction 1, will have been converted into 4, 4'-dichlorobenzophenone. Under the GLC conditions quoted, this compound has a retention time similar to that of heptachlor epoxide and so gives a peak on the chromatogram before most of the PCB isomers encountered in practice.

A comparison of the traces before and after oxidation of fraction 1 will show how much *pp'*-DDE was originally present. Adjust the volume of the hexane solution so that the individual heights of the PCB peaks are within the linear response range of the electron capture detector. Under the prescribed GLC conditions for the Apiezon L. column, the last of the PCB isomers normally found in wildlife tissues emerges in about 120 min after injection.

ANNEX N

(Clause 6.3)

STANDARDS ON METHODS OF RESIDUE ANALYSIS

Sl No.	Name of Pesticide	Method of Test, Ref to	
		USEPA	AOAC/ISO/IS
(1)	(2)	(3)	(4)
i)	DDT(<i>o, p</i> & <i>p, p</i> -isomers of DDT, DDE and, DDD)	508	AOAC 990.06
ii)	γ -HCH (lindane)	508	AOAC 990.06
iii)	α, β & δ - HCH	508	AOAC 990.06
iv)	Endosulfan (α, β and sulphate)	508	AOAC 990.06
v)	Monocrotophos	8141A	-
vi)	Ethion	1657A	-
vii)	Chlorpyrifos	525.2, 8141A	-
viii)	Phorate (Phorate and its oxygen analogue that is, phorate sulphoxide and phorate sulphone)	8141A	-
ix)	2,4-D	515.1	-
x)	Butachlor	525.2, 8141A	-
xi)	Isoproturon	532	-
xii)	Alachlor	525.2, 507	-
xiii)	Atrazine	525.2, 8141A	-
xiv)	Methyl parathion(methyl parathion and its oxygen analogue that is, methyl-paraoxon)	8141A	IS 15669
xv)	Malathion (malathion and its oxygen analogue that is, malaaxon)	8141A	-
xvi)	Aldrin and dieldrin	525.2	AOAC 990.06

NOTE — Test methods are for guidance and reference for testing laboratory. In case of two methods, USEPA method shall be the referee method.

ANNEX P

(Foreword)

COMMITTEE COMPOSITION

Drinking Water and Carbonated Beverages Sectional Committee, FAD 14

<i>Organization</i>	<i>Representative(s)</i>
CSIR - Central Food Technological Research Institute, Mysuru	DR SRIDEVI ANNAPURNA SINGH (Chairperson)
All India Association of Natural Mineral Water Industry, Ahmedabad	SHRI BEHRAM MEHTA
All India Food Processors Association, New Delhi	SHRI K. GANESH SHRI VENKATESH SOSLE (<i>Alternate I</i>) SHRI MOHIT CHAUDHARY (<i>Alternate II</i>)
All India Network Project on Pesticide Residues, New Delhi	DR VANDANA TRIPATHY
Bhabha Atomic Research Centre, Food Technology Division (BARC), Mumbai	DR SANJAY KUMAR JHA DR SUNIL KUMAR SAHOO (<i>Alternate</i>)
Bhavan's Research Center (Microbiology), Mumbai	DR SANDHYA SHRIVASTAVA DR NISHITH DESAI (<i>Alternate</i>)
Central Ground Water Board (CGWB), Faridabad	DR NEELAM NIGAM DR S. K. SHRIVASTAVA (<i>Alternate</i>)
Central Pollution Control Board (CPCB), New Delhi	SHRI P. K. MISHRA
Centre for Science and Environment (CSE), New Delhi	MS SUNITA NARAIN SHRI AMIT KHURANA (<i>Alternate</i>)
Confederation of Indian Industry, New Delhi	MS NEHA AGGARWAL MS MAMTA ARORA BUDHIRAJA (<i>Alternate</i>)
Confederation of Indian Food Trade & Industry (CIFTI)-FICCI, New Delhi	SHRI DEEPAK JASYAL MS VARSHA YADAV (<i>Alternate</i>)
Consumer Education and Research Centre, Ahmedabad	SHRI H. S. TRIPATHI DR DOLLY A. JANI (<i>Alternate</i>)
Consumer Research, Education, Action, Training and Empowerment (CREATE), Paramakudi	DR P. DURAISINGAM
CSIR - Central Food Technological Research Institute (CSIR - CFTRI), Mysuru	DR MUKESH KAPOOR MS VANAJAKSHI (<i>Alternate</i>)
CSIR - Indian Institute of Toxicology Research, Lucknow	DIRECTOR DR KAUSAR M. ANSARI (<i>Alternate</i>)
CSIR - National Environment Engineering Research Institute, Nagpur	DR P. K. LABHASETWAR DR NOOR AFSHAN KHAN (<i>Alternate</i>)
Delhi Jal Board, New Delhi	DR SANJAY SHARMA DR ANIL KUMAR MISHRA (<i>Alternate</i>)
Envirocare Laboratories Private Limited, Thane	DR NILESH AMRITKAR DR PRITI AMRITKAR (<i>Alternate</i>)

<i>Organization</i>	<i>Representative(s)</i>
Food Research and Analysis Centre, New Delhi	SHRI ANIL KUMAR MS KAVITA RANA (<i>Alternate</i>)
Food Safety and Standards Authority of India, New Delhi	SHRI BRIJESH KUMAR
Federation of All India Packaged Drinking Water Manufacturers Association (FIPMA), Mumbai	SHRI APURVA NARENDRA DOSHI SHRI NAVEEN GOEL (<i>Alternate</i>)
ICMR - National Institute of Nutrition, Hyderabad	DIRECTOR DR A. LAXMAIAH (<i>Alternate</i>)
ICMR - National Institute of Cholera and Enteric Diseases, Kolkata	DR SHANTA DUTTA DR RANJAN KUMAR NANDY (<i>Alternate</i>)
Indian Beverage Association, New Delhi	SHRI J. P. MEENA SHRI RAJENDRA MOHAN DOBRIYAL (<i>Alternate</i>)
Bureau of Indian Standard, New Delhi	MS SHUBHANJALI UMRAO
Ministry of Jal Shakti, Department of Drinking Water and Sanitation, New Delhi	DR D. RAJASHEKHAR
Mohan Meakins Limited, Ghaziabad	DR CHAUDHARY SHALU SINGH SHRI SUDEEP TYAGI (<i>Alternate</i>)
Safe Water Network, New Delhi	SHRI RAVINDRA SEWAK MS POONAM SEWAK (<i>Alternate</i>)
The Greater Chennai Packaged Drinking Water Manufacturers Association, Chennai	SHRI J. AANTHNARAYANAN SHRI V. MURALI (<i>Alternate</i>)
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IS 13428 : 2024

Panel for Reviewing Comments on Draft Revisions IS 13428 and IS 14543, FAD 14/Panel 08

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This Indian Standard has been developed from Doc No.: FAD 14 (18332).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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