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भारतीय मानक मसौदा

वायु प्रदुषण को मापने के तरीके

भाग 30 परिवेशी वायु में पार्टिकुलेट मैटर में धातुएँ

Draft Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

Part 30 Metals in Particulate Matter in Ambient Air

(ICS. 13.040.20)

Air Quality Sectional Committee, CHD 35	Last Date for Comments: 12th November 2024

Air Quality Sectional Committee, CHD 35

FOREWORD

(*Formal clause shall be added later*)

Air pollution is a challenging environmental problem faced by modern society. Knowledge of the concentration of potentially toxic substances, like heavy metals, in the airborne particulate is essential for assessing the pollution levels and providing important information for evaluating the level of long-term exposure to the population. The effect of chronic exposure from inhaled contaminated air may be associated with detrimental health effects such as hyperpigmentation, keratosis, various types of cancer and vascular diseases.

However, heavy metals are a widely recognized collective term which covers many other elements which occur naturally in the Earth's crust. The anthropogenic releases of many of these can give rise to higher concentrations relative to the normal background values. These can attach to very small particles in the air, stay in the air for many days and travel long distances.

So far individual Indian Standard is only available for measurement of lead and nickel in ambient air. This method provides the measurement of multiple metals (concentration of metals Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Zn in ambient air) including appropriate information on sampling in various fractions of particulate matter.

In the preparation of this standard considerable assistance has been derived from (i) NAAQS by CPCB (ii) Guidelines for the measurement of Ambient Air Pollutants Volume-I by CPCB (iii) USEPA Compendium method IO-3.2 (iv) USEPA Compendium method IO-3.4 and (v) USEPA Compendium method IO-3.5.

This Indian Standard is published in several parts. The other parts in this series are:

Part 1 dust fall (First Revision)

Part 2 Sulphur Dioxide

  Sec 1 Tetrachloromercurate Pararosaniline method

  Sec 2 ultraviolet fluorescence method

Part 3 Radioactivity (particulate in air)

Part 4 suspended Particulate matter (*First Revision*)

Part 5 Sampling of Gaseous Pollutants (*First Revision*)

Part 6 oxides of nitrogen (*First Revision*)

    Sec 2 chemiluminescence method

Part 7 Hydrogen Sulphide (*First Revision*)

Part 8 sulphation rate

Part 9 oxidants

Part 10 carbon monoxide (*First Revision*)

Part 11 benzene, toluene and xylene (BTX) (*Second Revision*)

Part 12 polynuclear aromatic hydrocarbons (PAHs) in air particulate matter (*First Revision*)

Part 13 total fluorides in ambient air

Part 14 guidelines for planning the sampling of atmosphere (*Second Revision*)

Part 15 mass concentration of particulate matter in the atmosphere

  Sec 2 Beta-ray absorption method

Part 16 recommended practice for collection by filtration and determination of mass, number and optical sizing of atmospheric particulates

Part 17 C1 to C2 hydrocarbons in air by gas chromatography

Part 18 continuous analysis and automatic recording of the oxidant content of the atmosphere

Part 19 Chlorine (*first revision*)

Part 20 carbon disulphide

Part 21 non methane hydrocarbons in air by gas chromatography

Part 22 lead

Part 23 respirable suspended particulate maiter (PM 10), cyclonic flow technique

Part 24 Fine Particulate Matter (PM 2.5)

Part 25 ammonia

Part 26 Nickel

Part 27 Vapour-Phase Organic Chemicals Vinyl Chloride to nc22 Hydrocarbons in air and Gaseous Emissions by Diffusive (Passive) Sampling onto Sorbent Tubes or Cartridges Followed by Thermal Desorption (TD) and Capillary gas Chromatography (GC) Analysis.

Part 28 Vapour-Phase Organic Chemicals C3 to nC30 Hydrocarbons in Air and Gaseous Emissions – Sampling by Pumped Sorbent Tubes Followed by Thermal Desorption (TD) and Capillary gas Chromatography (GC) Analysis(Under Preparation)

Part 29 Vapor Phase Mercury in Ambient Air

Sec 1 Cold-Vapor Atomic Fluorescence Spectrometer method by Amalgamation Principle

Sec 2 Cold-Vapor Atomic Absorption or Fluorescence Spectroscopy CVAFS Method Using Acidified solution of   KMnO4

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2:2022 'Rules for rounding off numerical values (second revision)'

Draft Indian Standard

Methods For Measurement of Air Pollution
Part 29 Metals in Particulate Matter in Ambient Air

1 SCOPE

This standard (Part 30) prescribes method for the measurement of concentration of metals Aluminium (Al), Arsenic (As), Barium (Ba), Cadmium (Cd), Cobalt (Co), Chromium (Cr), Copper (Cu), Iron (Fe), Manganese (Mn), Molybdenum (Mo), Nickel (Ni), Lead (Pb), Zinc (Zn) in particulate matter in ambient air by Atomic Absorption Spectrometry (AAS), Inductively Coupled Plasma Emission Spectroscopy (ICP-OES) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS).

NOTE- Arsenic has chemical and physical properties intermediate between a metal and a non-metal, and is often referred to as a metalloid or semi-metal.

2 REFERENCE

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

IS No.	*Title*
IS 1070 : 1992	Reagent Grade Water Specification (*Fourth Revision*)
IS 2303 (Part 1/Sec 1) : 2021/ISO 719 : 2020	Grading glass for alkalinity: Part 1 hydrolytic resistance of glass grains, Section 1 Determination and classification of hydrolytic resistance at 98c (*third revision*)
IS 4167 : 2020	Glossary of terms relating to air pollution (*second revision*)
IS 5182	Methods for measurement of air pollution
Part 4 : 1999	Suspended Particulate matter (*First Revision*)
Part 14 : 2000	Guidelines for planning the sampling of atmosphere (*Second Revision*)
Part 23 : 2006	Respirable suspended particulate maiter (PM 10), cyclonic flow technique
Part 24 : 2019	Fine Particulate Matter (PM2.5)

3 TERMINOLOGY

For this standard, the definitions given in IS 4167 shall apply.

4 PRINCIPLE

This method provides for measurement of concentration of metals in particulate matter in fractions as PM, PM 10, PM 2.5 using 20.3 cm × 25.4 cm filter paper and or using 4.7 cm filter paper. The collected sample on filters may be extracted by either hot plate procedure or by microwave extraction. The method further provides analysis of extracted sample by AAS, ICP-OES and ICP-MS

5 RANGE AND SENSITIVITY

The rounded off method detection limit are suggested for guidance in Table-1 and Table-2. In Table-1 Detection limits expressed as ng/m^3 are based upon sampling rate of 1.13 m^3/min for 24 h assuming a total sample volume of 1600 m^3 for Filter Size 20.32 cm x 25.4 cm using a strip of 2.54 cm x 20.32 cm for digestion (Taking ratio of total area of exposed filter in cm^2 to area of filter taken for digestion in cm^2 as 10) and total volume of extraction in mL as 100 for metals in PM or PM10 fraction. In Table-2 Detection limits expressed as ng/m^3 are based upon sampling rate of 1 m^3/hour for 24-hr assuming a total sample volume of 24 m^3 for Filter Size 4.7 cm using whole filter for digestion (Taking ratio of total area of exposed filter in cm^2 to area of filter taken for digestion in cm^2 as 1) and total volume of extraction in mL as 100 for metals in PM10 or PM2.5 fraction. The suggested wavelengths and instrument detection levels are as prevalent in literature and may change with instrument and for other wavelengths.

Table 1 Suggested Method Detection Limit (PM or PM10 Fraction) Filter size 20.32 cm x 25.4 cm

Sl. No	Metals	Suggested Wavelength AAS Flame (nm)	Suggested Method Detection Limit AAS Flame ng/m³	Suggested Wavelength Electrothermal AAS (Graphite Furnace) (nm)	Suggested Method Detection Limit Electrothermal AAS (Graphite Furnace)	Suggested Method Detection Limit Hydride Generation ng/m³	Suggested Wavelength ICP-OES (nm)	Suggested Method Detection Limit ICP-OES ng/m³	Suggested Method Detection Limit ICP-MS ng/m³

					ng/m³				
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
i)	As	—	—	—	—	0.5	193.70	40	0.3
ii)	Al	309.3	8	309.3	2	—	308.22	25	0.02
iii)	Ba	553.6	20	553.6	1.5	—	455.40	1.5	0.100
iv)	Cd	228.8	1.5	228.8	0.5	—	226.50	3	0.02
v)	Co	240.7	20	240.7	0.5	—	228.62	5	0.01
vi)	Cr	357.9	14	357.9	0.1	—	267.72	5	0.02
vii)	Cu	324.7	8	324.7	0.5	—	324.75	4	0.01
viii)	Fe	248.3	14	248.3	0.8	—	259.94	25	—
ix)	Mn	279.5	8	279.5	0.2	—	257.61	1.5	0.02
x)	Mo	313.3	62	313.3	0.8	—	202.03	5	0.02
xi)	Ni	232.0	14	232.0	0.5	—	231.60	10	0.02
xii)	Pb	283.3	32	283.3	0.5	—	220.35	25	0.01
xiii)	Zn	213.9	4	—	—	—	213.86	1.5	0.04

Table 2 Suggested Method Detection Limit (PM10 or PM2.5 Fraction) Filter size 4.7cm

Sl. No	Metals	Suggested Wavelength AAS Flame (nm)	Suggested Method Detection Limit AAS Flame ng/m³	Suggested Wavelength Electrothermal AAS (Graphite Furnace) (nm)	Suggested Method Detection Limit Electrothermal AAS (Graphite Furnace) ng/m³	Suggested Method Detection Limit Hydride Generation ng/m³	Suggested Wavelength ICP-OES (nm)	Suggested Method Detection Limit ICP-OES ng/m³	Suggested Method Detection Limit ICP-MS ng/m³
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
i)	As	—	—	—	—	2	193.70	210	0.5
ii)	Al	309.3	40	309.3	14	—	308.22	180	0.15
iii)	Ba	553.6	125	553.6	8.5	—	455.40	10	0.03
iv)	Cd	228.8	8	228.8	0.2	—	226.50	20	0.03
v)	Co	240.7	125	240.7	2.5	—	228.62	30	0.01
vi)	Cr	357.9	85	357.9	0.5	—	267.72	30	0.15
vii)	Cu	324.7	40	324.7	3	—	324.75	25	0.02
viii)	Fe	248.3	85	248.3	5	—	259.94	180	—
ix)	Mn	279.5	40	279.5	1	—	257.61	10	0.01
x)	Mo	313.3	420	313.3	5	—	202.03	35	0.01
xi)	Ni	232.0	85	232.0	2.5	—	231.60	65	0.02
xii)	Pb	283.3	210	283.3	2.92	—	220.35	180	0.02
xiii)	Zn	213.9	20	—	—	—	213.86	10	0.10

6 INTERFERENCES

6.1 Atomic absorption spectrometry

6.1.1 In flame atomic absorption analysis, the type and temperature of the flame used is critical; with improper conditions, chemical and ionization interferences can occur. In furnace atomic absorption analysis, the advantages of enhanced sensitivity may be offset by the fact that interference is also more of a problem. High concentrations of silicon in the sample can cause an interference for most of

the elements in this method and may cause aspiration problems. No matter what elements are being measured, if large amounts of silica are extracted from the samples, the samples should be allowed to stand for several hours and centrifuged or filtered to remove the silica. If chemical interferences (when species present in the sample cause variations in the degree to which atoms are formed in the flame, or when different valence states of a single element have different absorption characteristics) are suspected in Al (SO_4, Vanadium can interfere), Cr (Iron and Nickel can interfere), Fe (High Nickel interfere), Pb (SO_4 can interfere) the method of standard additions may be used to evaluate them; and if deemed desirable, the method may be used to make an accurate determination of metal concentration in the presence of an interference. In Al, Ba, Ionization interferences are controlled by bringing all solutions to 1000 mg/L caesium (samples and standards), 1000-mg/L solution of lanthanum as a releasing agent to all samples and standards. For Ba and Pb in the presence of very large calcium concentrations (greater than 0.1 percent), molecular absorption from CaOH may be observed. This interference may be overcome by using background corrections.

6.1.2 Background or nonspecific absorption can occur from particles produced in the flame that can scatter light and produce an apparent absorption signal. Light scattering may be encountered when solutions of high salt content are being analyzed. They are most severe when measurements are made at shorter wavelengths (for example, below about 250 nm). Background absorption may also occur as the result of the formation of various molecular species that can absorb light. The background absorption can be accounted for by using atomic absorption spectrometers with automatic background correction devices or otherwise using a deuterium continuum lamp. One may further refer to manufactures manual.

6.1.3 To reduce spectral interferences (interferences that result when an atom different from the one being measured absorbs a portion of the radiation) the use of multi-element hollow cathode lamps is discouraged.

6.1.4 Physical interferences may result if the physical properties of the samples vary significantly. Changes in viscosity and surface tension can affect the sample aspiration rate and, thus, cause erroneous results. Sample dilution, the method of standard additions, or both, are used to correct such interferences.

6.1.5 Matching the matrix of the samples to the matrix of the standards minimizes interferences.

6.2 Inductively Coupled Plasma

Spectral interferences result when spectrally pure solutions of one element produce a finite output on channels assigned to other elements. Refer to manufactures manual and use recommended wavelengths to monitor selected metals using ICP in order to minimize spectral interferences.

Matrix interferences depend on the types and quantities of acids used; element emission lines may be enhanced or depressed.

These interferences may be circumvented by careful matrix matching of standards, QC solutions, and samples

6.3 Inductively Coupled Plasma Mass Spectrometry

Refer manufacturers manual for Isobaric Elemental Interferences, Abundance Sensitivity and Isobaric Polyatomic Ion Interferences

Physical interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer due to viscosity effects, at the point of aerosol formation and transport to the plasma due to surface tension, or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material reducing the effective diameter of the orifices and therefore ion transmission. Internal standardization may be effectively used to compensate for many physical interference effects.

Memory interferences result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. These can be reduced by using suitable rinse times.

7 APPARATUS

7.1 Sampler to draw concentration of metals in different fractions of particulate matter shall consist of the following:

7.1.1 Sampler as laid down in IS 5182 (Part 4) for metals in PM fraction using using 20.3×25.4 cm filter paper

7.1.2 Sampler as laid down in IS 5182 (Part 23) for metals in PM10 fraction using using 20.3×25.4 cm filter paper

7.1.3 Sampler as laid down in IS 5182 (Part 24) for metals in PM10 or PM2.5 fraction using using 4.7 cm filter paper

7.1.4 Sampler based on High Volume PM 2.5 Technology developed by CSIR-NPL for metals in PM 2.5 fraction using 20.3×25.4 cm filter paper

7.1.5 One can select suitable sampler to meet objectives of the sampling.

7.2 Hot plate or Microwave digestive system (With programmable power settings up to 600 watts).

7.3 Atomic Absorption Spectrophotometer, Inductively Coupled Plasma Emission Spectrometer, Inductively Coupled Plasma- Mass Spectrometer- Because of the diversity of instrument hardware some minimal specific details are provided for guidance. Laboratory

must discuss their requirements for Limit of Detection (LOD) and Limit of Quantification (LOQ) with reference to Ambient Air Quality Standards and select suitable instrument configuration to meet the objectives.

7.3.1 Flame Atomic Absorption Spectrophotometer (FAAS) or GFAAS (Graphite Furnace Atomic Absorption Spectrophotometer) Equipped with air/acetylene and nitrous oxide/acetylene burner heads or graphite furnace as relevant with applicable lamps, gas cylinders and regulators and all other accessories.

7.3.2 For Inductively Coupled Plasma Emission Spectrometer, the ICP source consists of a radio frequency (RF) generator capable of generating at least 1.1 kW of power, torch, tesla coil, load coil, impedance matching network, nebulizer, spray chamber, and drain. High-quality flow regulators are required for both the nebulizer argon and the plasma support gas flow. A peristaltic pump is recommended to regulate sample flow to the nebulizer. The spectrometer may be of the simultaneous (polychromator) or sequential (monochromator) type with air-path, inert gas purged, or vacuum optics. A spectral bandpass of 0.05 nm or less is required. The instrument must permit examination of the spectral background surrounding the emission lines used for metals determination. lt is necessary to be able to measure and correct for spectral background at one or more positions on either side of the analytical lines.

7.3.3 Inductively Coupled Plasma- Mass Spectrometer capable of scanning the mass 5 to 250 amu with a minimum resolution capability of 1 amu peak width at 5 percent peak height. Instrument may be fitted with a conventional or extended dynamic range detection system and all other. A Variable Speed Peristaltic Pump is suggested for solution delivery to the nebulizer. A Mass-Flow Controller is required on the nebulizer gas supply. A water-cooled spray chamber may reduce some types of interferences.

7.4 Glassware - Borosilicate and low actinic (amber colour) glassware or plasticware of equivalent quality as applicable. All reusable labware glassware, should be cleaned prior to use by soaking overnight and thoroughly washed with detergent and water, rinsed with water, and soaked for 4 h in a mixture of dilute nitric and hydrochloric acid (1+2+9). It should then be rinsed with Reagent Water Garde-I as per IS 1070 and dried. Do not use chromic acid to clean glassware.

7.5 Balance An analytical balance with readability of 0.1 mg or better. The installation of balance shall be as per manufacturer's instruction on a vibration dampening surface.

7.6 Forceps with Broad, unserrated tips to prevent damage to filters.

7.7 A clean work area in laboratory for trace element sample handling should be available. Sample containers can introduce positive and negative errors in the determination of trace elements by contributing contaminants through surface desorption or leaching and depleting element concentrations through adsorption processes.

8 REAGENTS

Quality of all reagents shall be analytical reagent grade, unless specified otherwise.

8.1 Reagent Grade Water

Reagent grade water of Grade 1 minimum as per IS 1070 shall be used.

8.2 Standards

Certified Reference Materials for Metals for Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Zn provided by Reference material producers fulfilling the requirements of ISO 17034 with stated metrological traceability to the SI

Working standards are prepared by appropriate single or multiple dilutions of the standard solutions. Mixed standards should be prepared with any chemical incompatibilities in mind. For those metals that indicate chemical or ionization interferences, the final dilution shall contain 2 percent (*v/v*) of the 50 mg/ml Cesium and lanthanum solutions. The element concentrations in the standards should be sufficiently high to produce good measurement precision and to accurately define the slope of the response curve. Concentrations of 200 μg/L are suggested.

8.2.1 *Cesium Solution (50 mg/ml)*

Dissolve 73.40 g of Cesium nitrate ($CsNO_3$) in reagent grade water. When stored in a polyethylene bottle, this solution is stable for at least 1 yr.

8.2.2 *Lanthanum Solution (50 mg/ml)*

Dissolve 156.32 g of lanthanum nitrate (La $(NO_3)_3 \cdot 6H_2O$) in a 2 percent (*v/v*) HNO_3. Dilute to volume in a l l flask with 2 percent (*v/v*) HNO_3. When stored in a polyethylene bottle, this solution is stable for at least 1 yr.

8.3 Filter Paper: Glass fibre Filter papers 20.3 × 25.4 cm or 4.7 cm polytetrafluoroethylene (PTFE) filter meeting following requirements or commercially available filter papers as EPM2000 or equivalent.

  a)   Filters are manufactured from 100 percent pure borosilicate glass.

b) High quality filters with reproducible properties must be used in sampling for metals in ambient air. They enable detailed chemical analysis of trace pollutants with minimal interference. Analyze 5 percent of the total number of filters for the presence of specific metals, prior to sample collection, to verify reproducibility and low background metal concentrations. Maximum concentration shall be Pb- 4.8; Hg- 4.4; Cd- 10.8; As- 2.8; Cu- 2.8; Fe- 5.8; Mn- 2.2; Ni- 3.0 and Cr- 2.2 ng/cm^2.

c) Particle retention: 99.99 percent efficiency in air for 0.3 μm particles at 5 cm/sec face velocity.

8.4 Hydrochloric Acid (HCl) — Concentrated.

8.5 Nitric Acid (HNO₃) — Concentrated, which have sufficiently low metal concentrations.

8.6 Sulphuric Acid (H₂SO₄) — Concentrated.

9 SAMPLING

This method lays down sampling procedure for both using 20.3 × 25.4 cm Filter paper for PM or PM10 and or using 47 mm Filter Paper for PM10 or PM2.5. The sampling for metals is specified in Table 3. Table-1 provides for current provisions as under National Ambient Air Quality Standards 2009. One can select other combinations of duration, period and frequency to meet objectives of the sampling for various metals.

Table 3 Sampling for Metals in Ambient Air

Sl No.	Pollutant	Time Weighted Average	Concentration in Ambient Air	
			Industrial, Residential, Rural and other Areas	Ecologically Sensitive Area (Notified by Central (Government)
(1)	(2)	(3)	(4)	(5)
i)	Lead (Pb), μg/m^3	Annual [1] 24 h [2]	0.50 1.0	0.50 1.0
ii)	Nickel (Ni), ng/m^3	Annual	20	20
iii)	Arsenic (As), ng / m^3	Annual	06	06

NOTE

1 Annual arithmetic mean of minimum 104 measurements in a year at a particular site taken twice a week 24 hourly at uniform intervals. (Sampler may have to be run for longer durations to collect suitable quantity in PM2.5 or PM10 fraction using a 4.7 cm filter paper)

2 Since practically sometimes it may not be possible to target sampling of 24 h in a day or 104 measurements in a year due to power failure or other site-specific problems, the values monitored for 75 percent of the time and more can be considered as representative values in the ambient air.

9.1 Location of Sampling Station/Sampler

For planning of network of sampling stations including number of stations and location of sampling station or sampler refer IS 5182 (Part 14). Record topographical features, sources of pollution, any obstructions to sampling station including any deviations specific to site and with relevance to objectives of monitoring on the sampling sheet.

9.2 Sampling procedure

Install the filter paper as per manufacturer's instructions. For automatically flow-controlled units, record the designated flow rate on the data sheet. Record the reading of the elapsed time meter. The specified length of sampling is 24 hours. During this period, take multiple readings of flow rate at regular intervals. After the required time of sampling, record the flow meter reading and take out the filter media from the sampler and put in a container or envelope and ship to laboratory. Store the envelopes in laboratory up to 30°C until analysis. The maximum sample holding times is usually 180 days. Analyse the samples within 180 days.

10 EXTRACTION OF SAMPLES

Metals can be extracted from filter paper either by either hot plate procedure or by microwave extraction.

10.1 Microwave extraction

Cut 2.54 cm × 20.3 cm strip or half the filter from the 20.3 cmx 25.4 cm filter using a stainless-steel pizza cutter or use the whole filter paper for 4.7 cm filter sample and place on its edge in a labelled centrifuge tube using vinyl gloves or plastic forceps. Using the plastic forceps, crush the filter strip down into the lower portion of the centrifuge tube to ensure acid volume will cover entire filter. Add 10.0 ml of the extraction solution to each of the centrifuge tubes (3 percent conc. HNO_3 and 8 percent conc. HCl). Place the centrifuge tubes in a Teflon vessel containing 31 ml of deionized water. Place the vessel caps with the pressure release valves on the vessels hand-tight and tighten using the capping station to a constant torque of 12 ft-lb. Place the vessels in the microwave carousel. Connect each sample vessel to the overflow vessel using the Teflon connecting tubes. Place the carousel containing the 12 vessels on to the turntable of the microwave unit. Irradiate the sample vessels at 486 W (power output) for 23 min. Allow the pressure to dissipate, then remove the carousel containing the vessels and cool in tap water for 10 min. using the caping station uncap the microwave vessels, remove the labelled centrifuge tube containing samples. Add 10ml of deionized water to each centrifuge tube. Cap the centrifuge tube tightly and mix the contents thoroughly for 2 min to 3 min to complete extraction. The final extraction volume is 20 ml based upon the above procedure. Filter the extracted fluid with Whatman No. 41 and make up the final volume to 100 ml, the filtered sample is now ready for analysis.

10.2 Hot plate procedure

Cut a 2.54 cm × 20.3 cm strip or half the filter from the 20.3 cm x 25.4 cm filter using a stainless-steel pizza cutter or use the whole filter paper for 4.7 cm filter sample. Place the filter in a beaker using vinyl gloves or plastic forceps. Cover the filter with the extraction solution (3 percent HNO & 8 percent HCl). Place beaker on the hot-plate (Temperature below 80 °C), contained in a fume hood, and reflux gently while covered with a watch glass for 30 min. Do not allow sample to dry. Remove the beakers from the hot-plate and allow to cool. Rinse the beaker walls and wash with distilled water. Add approximately 10 mL reagent grade water to the remaining filter material in the beaker and allow to stand for at least 30 min. Transfer the extraction fluid in the beaker to a 100 mL volumetric flask or another graduated vessel. Rinse the beaker and any remaining solid material with reagent grade water and add the rinses to the flask. Dilute to the mark with reagent grade water and shake. The final extraction solution concentration is 3 Percent HNO / 8 percent HCl. The filtered sample is now ready for analysis

11 PROCEDURE

The trace element concentrations in each sample can be determined by Atomic Absorption Spectrophotometer, Inductively Coupled Plasma Emission Spectrometer, Inductively Coupled Plasma- Mass Spectrometer. Choose appropriate measuring equipment to meet the objectives. Run the measuring equipment as per manufacturers instructions.

Use the rinse blank to flush the system between samples. Allow sufficient time to remove traces of the previous sample or a minimum of 1 min.

Samples having concentrations higher than the established linear dynamic range should be diluted into range and reanalyzed.

To evaluate the contribution to the absorbance from the filters and reagents used, blank samples must be analyzed. Subject the blank to the entire analysis procedure. The absorbance obtained from the aspiration of the blank solution is subtracted from the sample absorbance.

Use of following blanks is suggested. A calibration blank establishes the analytical calibration curve. Calibration blank consists of 1 percent (*v/v*) nitric acid in Reagent grade water. If the direct addition procedure is being used, add internal standards. The laboratory reagent blank assesses possible contamination from the sample preparation procedure and spectral background. Laboratory reagent blank (LRB) must contain all the reagents in the same volumes as used in processing the samples. The LRB must be carried through the entire sample digestion and preparation scheme. If the direct addition procedure is being used, add internal standards to the solution after preparation is complete.
The rinse blank flushes the instrument between samples to reduce memory interferences. Rinse blank consists of 2 percent (*v/v*) nitric acid in Reagent grade water.

11.1 Calibration

Prepare standard solutions from the stock solutions. Select at least three standards to cover linear range as recommended by method. Aspirate the standards into the flame or inject the standards into the furnace and record the absorbance. Prepare the calibration graph by plotting absorbance and concentration in μg / ml.

11.2 Metal Concentration

$$C = \frac{(M_S - M_b) \times V_S \times F_a}{V \times F_t}$$

where,

C is concentration, μg metal/m^3

Ms is metal concentration μg/mL

Mb is blank concentration μg/mL

Vs is total volume of extraction in mL

Fa is total area of exposed filter in cm^2

V is Volume of air sampled in m^3

Ft is Area of filter taken for digestion in cm^2

13 QUALITY ASSURANCE/QUALITY CONTROL MEASURE

13.1 Calibration

Periodic calibration of the AAS, ICP-OES, ICP-MS shall be performed as per the calibration plan prepared by laboratory at such intervals as determined based on use and subject to performance of intermediate checks at regular intervals.

As applicable correct data values for instrument drift or sample matrix induced interferences by applying internal standardization. Corrections for characterized spectral interferences should be applied to the data. Chloride interference corrections should be made on all samples, because of the addition of hydrochloric acid during filter extraction, as the chloride ion is a common constituent of environmental samples.

13.2 Proficiency Testing

Laboratories shall participate in proficiency testing programs as per the Proficiency Testing plan prepared by laboratory.