

SAMPLING AND ANALYSIS OF HEAVY METALS (Pb, Ni & As) IN AMBIENT AIR

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NATIONAL AMBIENT AIR QUALITY STANDARDS

S. No.	Pollutant	Time Weighted Average	Concentration in Ambient Air		
			Industrial, Residential, Rural and other areas	Ecologically Sensitive Area	Methods of measurement
1.	Lead (Pb), $\mu\text{g}/\text{m}^3$	Annual *	0.5	0.5	AAS/ICP method after sampling on EPM2000 or eq. filter paper
		24 hours **	1.0	1.0	
2.	Nickel (Ni) ng/m^3	Annual *	20	20	
3.	Arsenic (As), ng/m^3	Annual *	06	06	ED-XRF using Teflon filter

HEALTH IMPACTS

Pollutant	Source	Health effects	Environmental effects
Lead (Pb)	Combustion of solid waste, coal, oils, emissions from iron and steel production, lead smelters, tobacco-smoke	Blood, kidneys, nervous, cardiovascular and reproductive systems	Harms plants & wildlife, accumulates in soil, both terrestrial and aquatic systems is affected
Arsenic (As)	Microorganisms release volatile methylarsines and as a by-product of refining the ores of copper & lead	Causes liver, kidney, & brain damage and neurological and developmental damage.	Deposits into water bodies where it accumulates in fish, resulting in exposure to humans and wildlife.
Nickel (Ni)	Emission from metallurgical, chemical and food processing industries, wind-blown dust, combustion of coal, fuel, oil, incineration of waste.	Cancer, dizziness, Respiratory failure, Birth defects,	Adsorb to soil particles, and into groundwater.

Principle of Method

Active sampling in accordance with the guidelines of NAMP using PM10 High Volume Sampler, Extraction of samples and analysis using atomic spectrometry techniques.

SAMPLE COLLECTION & TRANSPORTATION

- After collecting the samples, transport the filters/samples to the laboratory in a shipping envelope.
- Store the samples in protective envelope up to 30⁰C till analysis
- The maximum holding time is usually 180 days. Analyse the samples within these prescribed time.

DIGESTION

Digestion: It is a process of dissolving a sample in an acid matrix, usually accompanied by heating.

Why?: To reduce interference by organic matter and to convert metals associated with inorganics & particulates to a form (usually the free metals) that can be determined by AAS or ICP.

Nitric acid will digest most samples adequately.

Nitrate is an acceptable matrix for both flame and electrothermal AA and preferred matrix for ICP-MS.

Cleaning Protocol

- Wash all labwares with laboratory detergent or Ultrasonication for 30 mins.
- Rinse
- Soak for minimum of 4 hours in 20% HNO₃.
- Rinse 3 times with DW
- Dry in dust free environment

Hot Plate Procedure

Cut 1" X 8" strip or half the filter from the 8" X 10" using pizza cutter by placing the edge of sample filter and roll it in such a way that it get into the Beaker.

Add extraction solution (3% HNO₃ and 8% HCL) of sufficient quantity to cover the entire filter sample.

Reflux gently while covering with a watch glass for 30 minute using hot plate in a Fume Hood.

Do not allow sample to dry.

Allow to cool.

Rinse the beaker walls and wash with DW.

Add 10 ml DW to the sample and allow to stand for 30 minutes.

Filter the extracted fluid with Whatman no.41.

Rinse the beaker with DW, add the rinses to the flask and make up the final volume to 100 ml.

Microwave aided Digestion

Cut 1" X 8" strip or half the filter from the 8" X 10" using a template and pizza cutter by placing the edge of sample filter and roll it in such a way that it get into the centrifuge tube.

Using vinyl gloves or plastic forceps slide down the sample into the bottom of the tube.

Add 10ml of extraction solution (3% HNO₃ and 8% HCL) and ensure that it covers the entire filter sample.

Micro oven the sample vessels at 486 W for 23 minutes.

Allow the pressure to dissipate, and cool in tap water for 10 minutes.

Add 10 ml DW to sample, cap tightly and mix the contents thoroughly for 2-3 minutes to complete extraction.

Filter the extracted fluid with Whatman no.41 and make up the final volume to 100 ml.

Thank you