

TEQIP SUMMER INTERNSHIP REPORT

Research Topic:- Understanding the occurrence and detection of mercury in natural waters.

Principle:- Determination of mercury (Hg) in filtered and unfiltered water by oxidation, purge and trap, desorption, and cold-vapor atomic absorption spectrometry (CVAAS). A carrier gas (Ar) is used for purging and Gold amalgamation is used for trapping mercury in gold traps.

- This Method is for determination of Hg in the range of 0.5–100 ng/L.

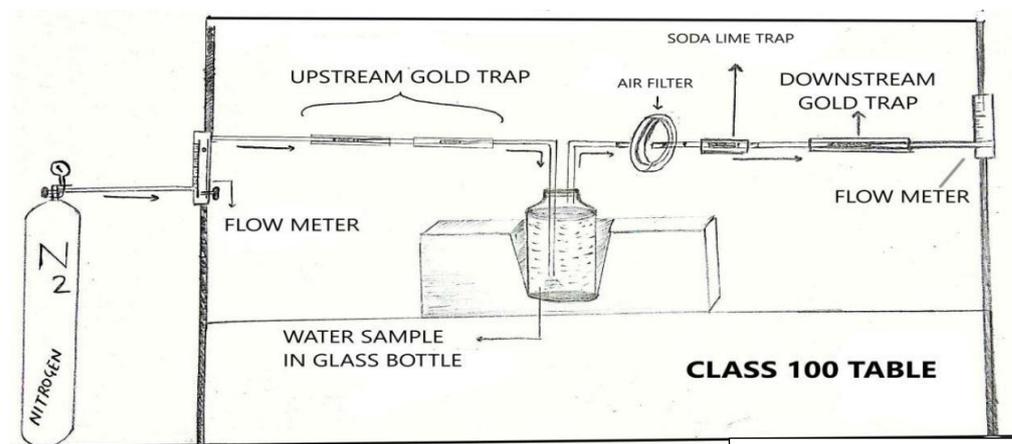
Apparatus :- Watt-man filter paper (0.45 micro meter), Tarson inline filter holder, Brooksrand Gold Traps (gold coated glass beads), Brooksrand soda lime, Poly Tetra Fluoro Ethylene (Teflon) seal Tape and tubes

Bubbler, Argon Gas Cylinder, GARDIS-5 (Mercury Vapor Analyzer), Dosing Unit, Heating Coil, Laptop or PC for operating GARDIS-5

Reagents:- Bromine Chloride (BrCl), Hydroxylamine Hydrochloride (NH₂OHHCl), Stannous Chloride (SnCl₂)

Reagent grade water (TYPE-1), Soda Lime.

Fig:01 shows the experimental setup of purge and trap method and Fig:02 shows the analytical setup for check trapped mercury.



PURGE AND TRAP SETUP

Fig :01 SETUP PURGE & TRAP METHOD



Fig:02 GARDIS-5

Summary of method:-

A 100- to 2000-mL sample is collected directly into a cleaned, pretested, fluoropolymer or glass bottle using sample handling techniques designed for collection of mercury at trace levels. For **dissolved Hg**, the sample is filtered through a 0.45-micrometre capsule. The sample is preserved by adding bromine monochloride (BrCl) solution.

Prior to analysis, all Hg in a 100-mL sample aliquot is oxidized to Hg(II) with BrCl. After oxidation, the sample is sequentially reduced with NH_2OHHCl to destroy the free halogens, then reduced with stannous chloride (SnCl_2) to convert Hg(II) to volatile Hg(0). The Hg(0) is separated from solution either by purging with nitrogen (125ml/min) by vapor/liquid separation. The Hg(0) is collected onto a gold trap. The Hg is thermally desorbed from the gold trap into an inert gas stream. Gas carries the released Hg(0) to a second gold (analytical) trap. The Hg is desorbed from the analytical trap into a gas stream that carries the Hg into the cell of a cold-vapor atomic fluorescence spectrometer (CVAFS) for detection. The **GARDIS-5** (atomic mercury vapor analyzer) is used to analyze trapped mercury in the gold trap. Quality is assured through calibration and testing of the oxidation, purging, and detection systems.

Working Tips:- Detecting low level concentration of mercury (pg) in water column. For getting mercury from water in vapor form we need to add some chemicals (BrCl for oxidation, NH_2OHHCl Used for neutralization of sample, SnCl_2 for mercury reduction). Gardis-5 is used to analyse mercury in vapor form. Dosing unit is used for checking trapping capacity of Gold Traps.

Gold Trap analysis reading data sheet

Sample	Mass (Pg)	Volume (m^3)	concentration
GOLD TRAP			
	40.6	0.367	110.35
	41.1	0.368	111.666

	41.3	0.369	111.748
GOLD TRAP			
	35.4	0.367	96.45
	37.1	0.368	100.81
	37.0	0.369	100.27

Conclusion

This intern was focused on “**understanding the occurrence and detection of mercury in natural waters**”. Briefly, 100ml of samples will be oxidized with BrCl and NH₂OH HCl pre-reduction, SnCl₂ reduction, Hg free air purging. Hg(0) collected on gold traps(gold trap pre-concentration) was quantified by Atomic Absorption Mercury Vapor Analyzer (GARDIS-5). thermal desorption, and atomic absorption quantification. The analyzer offers the detection limit close to the best atomic fluorescence systems currently available.

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