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# THE APPLICATION OF ATOMIC ABSORPTION SPECTROMETRY (AAS) AND INDUCTIVELY COUPLED PLASMA SPECTROMETRY (ICP) TO THE SOME MICROELEMENTAL (Hg, Se) ANALYSES OF WATER SAMPLES

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## Summary

Atomic absorption spectrometry (AAS) and Inductively coupled plasma spectrometry arecommonly used instrumental techniques of analysis for the quantitative determination of metals and metalloids particularly in water samples, including those from wastewater, sludges and wastes. The main advantages of AAS are its high specificity and selectivity, the sensitivity being variable over broad ranges depending on the type of atomization selected (flame, graphite, cold vapor or hydride technique).

The analysis of aqueous samples is one of the main applications for AAS and ICP-OES. This report demonstrates that the determination of mercury and selenium using SHIMADZU-AA6501F atomic absorption spectrometer with HVG and MUV, SHIMADZU-ICP 7500S plasma spectrometer with HVG in water shows all required analytical capabilities to analyze water with high precision and accuracy and excellent sensitivity and rapid analysis.

## *Mercury*

Inorganic mercury compounds (ionogenic) and organic compounds such as methyl mercury, ethyl mercury and so on may occur in water. The latter may be concentrated to a certain extent in organisms (e.g. fish). Although the absolute quantity involved is mostly only very small, in view of their toxicity these compounds must be taken into account owing to their accumulation in the food chain. It is therefore of interest in analysis to differentiate between inorganic and organically bonded mercury. The method of quantitative determination which has gained widest acceptance is the variant of AAS using the cold vapour technique with and without decomposition.

### *Mercury AAS with cold vapour method*

*Principle of the method:* Mercury ions are reduced to metallic mercury with tin(II) chloride. The metallic mercury is transferred into a quartz cuvette with the aid of a current of inert gas and the absorption of the atoms is measured in the beam of an atomic-absorption spectrometer.

### *Reagents*

*Stabilizing reagent:* Treat 500 ml of nitric acid (1.40 g/ml) with 5 g potassium dichromate ($K_2Cr_2O_7$)and make up to 1000 ml with water.

*Reduction solution:* Treat 10 g tin (II) chloride ($SnCl_2 \cdot 2\ H_2O$) with 10 ml hydrochloric acid (1.17 g/ml) and make up to 100 ml with water, shaking constantly.

### ***Selenium***

In natural waters selenium occurs only in very low concentrations. Sea water contains an average of about 0.005 mg/L Higher concentrations are rarely found in surface waters. Regionally limited ground waters form an exception: if they occur in an area with rocks and soils containing large amounts of selenium, they may contain between 0.05 and 0.1 mg/1 or in exceptional cases even more.

If selenium concentrations which greatly exceed these values occur insurface waters it must be assumed that seleniferous waste water is gaining access. Above all, this may stem from the processing (roasting) of sulphidic ores, the manufacture of sulphuric acid, or from the chemical industry.

## *Principle*

Using sodium borohydride, selenium ions are reduced to selenium hydrid transferred to a heated quartz cuvette with the aid of a current of inert gas, decomposed thermally, and the absorption of the atoms is measured the beam of an atomic-absorption spectrometer. In the hydride technique the element which is to be determined is volatilized as a gaseous hydrid and separated off from the matrix. Interferences may occur if there is considerable excess of elements such as antimony, arsenic, tin, bismuth mercury, or tellurium which may also be volatilized using this technique

Above all, heavy metals such as copper and nickel have a disturbing effect during the hydride formation itself. These interferences may be diminish by adding 300 mg of solid 2-pyridine aldoxime to the solution for measurment.

Since the hydride technique only permits quantitative detection of selenium (IV), selenium (VI) must be converted to selenium (IV) by prereduct

(boiling in a strongly hydrochloric -solution at the reflux).

*Reagents:*

Sulphuric acid (1.84 g/ml)

Hydrogen peroxide (30 %)

## Conclusion

For water analysis the Shimadzu provides excellent detection limits at low /ppb/ levels.

1. AAS method is applicable to mercury contents between 1-15 ppb in water sample using Hg 235.7 nm AA6501F by mercury cold vapor
2. AAS method is applicable to selenium contents between 8-50 ppb in water sample using Se 196.0 nm AA6501F by HVG
3. ICP method is applicable to selenium contents up 5 ppb in water sample using Se 196.0 nm ICP7500S by HVG.

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# ЦАХИЛГААН-ИСЭЛДЭЛТИЙН ДАРААХ АЛТНЫ ХҮДРИЙН УУСМАЛААС АЛТ БОЛОН БУСАД МЕТАЛЛЫГ ЯЛГАХ БОЛОМЖ

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## Товч агуулга

Алт олборлолтын дараах үлдэгдэл хүдэр (хар шлих) болон боловсруулахад хүндрэлтэй алтны үндсэн ордын хүдрийг хүчиллэг орчинд цахилгаан-исэлдэлтийн аргаар уусган, усан орчинд шилжсэн алтыг ялган авах боломжийг тогтоох судалгаа хийгдсэн. Судалгаанд Төв аймгийн Заамарын Баянголын алтны үйлдвэрийн алт олборлолтын дараах үлдэгдэл хүдэр (хар шлих)-ыг ашигласан. Цахилгаан-исэлдэлтийн дараах алтны хүдрийн уусмалаас алтыг гидразинаар ангижруулахад, NaCl-ын 10%-ын усан уусмалын электролизын дунд үүссэн хүчтэй исэлдүүлэгчид ($ClO, ClO_3^-, Cl_{2(aq)}$) болон уусмалд шилжсэн хүдрийн үндсэн элементүүд гидразинтай урвалд орж, урвалжийн хэт зарцуулалт үүсгэн алтны ангижралтыг эрс бууруулж байна. Уусмалд шилжсэн хүдрийн үндсэн элементийг (Fe, Ti зэрэг) уусмалын орчныг шүлтлэгжүүлэх замаар (PH=4-11, Eh=1.0-0.1 V) бүрэн ялгах боломжтой байгаа ч алт тэдгээр элементүүдийн хамт тунадасжин ялгарч байна. Цахилгаан-