SGS	LAKEFIELD – INDUSTRIES & ENVIRONMENT MULTI-ELEMENT DETERMINATION OF RECOVERABLE METALS IN SOIL, SEDIMENT AND SLUDGE SAMPLES BY HYDOXYLAMINE HCL EXTRACTION AND ICP/MS FINISH	Doc. No.	ME-CA-[ENV]SPE-LAK-AN-22
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1. SCOPE

This method is used to analyse soil, sediment and sludge samples for hydroxylamine HCL extractable metals and ICP/MS analysis.

2. DERIVATION

This method is derived from EPA 3050B, US EPA 6020A, and the USGS leaching protocol method 29, cold hydroxylamine HCl leach.

3. METHOD MODIFICATIONS

Method modifications from EPA SW-846 6020A:

- Internal standards used for analysis are Sc45, Ga69, In115, Tb157, and Tm169 not the recommended Li6, Sc45, Y89, Rh103, In115, Tb159, Ho165, Bi209.
- The laboratory uses the kinetic energy discrimination cell found in both the Agilent 7700 and Agilent 7900 to reduce or eliminate isobaric interferences.
- Calibration mixtures are different.

4. PRINCIPAL OF THE METHOD

2 g of sample is leached with 20 mL of 0.25 M hydroxylamine hydrochloride and agitated for 2 hours. The sample is then centrifuged, decanted, and the supernatant preserved to pH < 2 with nitric acid and submitted to ICP-MS for metals determination.

The samples are analyzed against 2% nitric acid calibration materials. Dilutions may be required to bring the analysis into the instrument linear range. The samples and calibration materials are aspirated into the plasma via nebulization, where they are desolvated, vaporized, dissociated and ionized. The ions are then transported through the interface of the instrument (sampler and skimmer cones), where the ions are focused, and mass filtered by the quadruple. The mass-separated ions are then detected. Refer to The Handbook of Inductively Coupled Plasma by Thompson and Walsh for a more detailed description of the theory of ICP-MS.

4.1. *Elements of Interest*

The table below lists some of the most common operational components for each element of interest. This table is to be used as a guideline only as some* of these components may change slightly. For additional and complete validation information refer to the validation files on the SGS network.

Element	LOQ (µg/g)	Reporting Limit (µg/g)	Element	LOQ (µg/g)	Reporting Limit (µg/g)
Silver	0.0007	0.01	**Phosphorus**		3
***Aluminum**	0.02	1	**Potassium**		4
Arsenic	0.05	0.5	**Lead**	0.0004	0.1
***Boron**	0.01	1	**Silicon**	1	3
***Barium**	0.008	0.01	**Sodium**	.77	1
***Beryllium**	0.001	0.02	**Antimony**	0.004	0.8
Bismuth	0.0008	0.09	***Selenium**	0.08	0.7
Cadmium	0.001	0.02	***Tin**	0.003	6
Calcium		3	***Strontium**	0.002	0.02
Cobalt	0.0005	0.01	**Tellurium**	0.003	0.1
***Chromium**	0.007	0.5	***Titanium**	0.003	0.1
Copper	0.004	0.1	***Thallium**	0.001	0.02
Iron	0.5	1	**Uranium**	0.0002	0.002
Mercury	0.01	0.1	***Vanadium**	0.09	1
***Lithium**	0.07	2	***Tungsten**	0.004	0.04
Magnesium	1	1	***Yttrium**	0.0004	0.004
Manganese	0.002	0.1	**Zinc**	0.007	0.7
Molybdenum	0.005	0.1	**Zirconium**	0.0009	0.03
Nickel	0.001	0.1			

**Digestion may not be complete for certain mineral forms.*

5. INTERFERENCES

5.1. *Spectral Interferences*

There are two types of spectral interferences in ICP-MS:

- The first are isobaric overlaps, which occur when there are two or more isotopes of different elements occurring at the same mass.
- The second are polyatomic/molecular overlaps, which occur when there is the formation of molecular ions interfering with the analyte isotope of interest. These could be oxides, argides, nitrides, or M^{2+} species. To correct for these interferences an alternative mass or a mass correction can be used.

5.2. *Physical Interferences*

Physical interferences that inhibit nebulization efficiency and aerosol transport efficiency are caused by high dissolved solids or concentrated acid solutions. These interferences must be reduced by either dilution of the sample, acid matrix matching or by using an appropriate internal standard element.

All samples analyzed by ICP-MS should ideally contain less than 0.2% dissolved solids, with an absolute maximum of 0.5%. An internal standard is also used for all elements to minimize the effects caused by physical interferences.

5.3. *Chemical Interferences*

Chemical interferences occur from molecular compound formation associated from the aerosol and previous samples. Memory effects occur when a sample of sufficiently high concentration has overloaded the sample introduction system. Residual concentrations of the analyte remain in the system, thus adding to the concentration of the following sample or result in molecular formation. Memory effects must be monitored and minimized by flushing the system with a slightly acidic rinse between samples.

5.4. *Instrument Interferences*

The quadrupole ICP-MS instruments are subject to interferences. An interference free mass is the best choice for analysis when possible. The potential interferences are obtained from internal studies and published isotope abundance tables. It is possible to analyze more than one isotope of an element. To correct for these interferences the Agilent Octopole Reaction System (ORS) may be used to reduce or eliminate the interferences.

5.5. *Adverse Conditions*

An adverse condition that applies to this method is the presence of high dissolved solids. This method is suitable only for samples that are less than 5% in dissolved solids after digestion. Unacceptable samples can be diluted or reconfigured to ICP-OES.

6. HEALTH & SAFETY & ENVIRONMENTAL CONDITIONS

- The chemical and biological content of samples is unknown. Therefore, samples must be handled with care and standard laboratory safety equipment (i.e., lab coat, gloves, eye protection and safety shoes) must be worn.
- All acids used in this procedure are extremely dangerous and all protective equipment must be worn (i.e., lab coat, face shield, gloves, eye protection and safety shoes).
- The samples to be analyzed have a slightly acidic matrix and should be treated with the same precautions as concentrated acids. Refer to WI-CA-[ENV]GEN-LAK-AN-002.
- All hot block digestions must be done in a properly functioning fume hood.
- Consult all safety data sheets (SDS), found on the SGS network, regarding the reagents and chemicals involved in this procedure before beginning preparation of samples.

7. SAMPLE REQUIREMENTS

Sampling information and concerns:	Representative sample must be taken.
Containers to be used:	High Density Polyethylene (HDPE) or glass
Sample size:	Optimum sample weight for dry samples 2g; Minimum sample weight for dry samples 0.5g;
Preservation required:	none
Temperature requirements for storage:	Ambient
pH requirements:	Sample should be acidic after digestion
Pre-treatment:	Sludge and sediment samples are dried and pulverized. Soil samples are screened to <2mm and a subsample is ground to pass 355µm sieve.
Holding time (from time of collection to start of analysis):	180 days (Reg. 153 protocol) Indefinite after drying
Disposal of samples:	Refer to WI-CA-[ENV]AD-LAK-AD-014

8. REFERENCES

Agilent 7700 Series ICP-MS, Masshunter Workstation User Guide, Agilent technologies, 2011.

Agilent 7700 Series ICP-MS, Hardware Maintenance Manual, Agilent Technologies, 2009-2011.

Environmental Protection Agency 1998. Inductively Coupled Plasma Mass Spectrometry. U.S.EPA Method 6020A Rev. 1

Varian 810/820 ICP-MS Customer Training Manual and Varian ICP-MS Expert On-Line Software / Hardware Guide.

Quantum ICP-MS software v.3.1, 2.11-2013

USGS Method 29, Cold Hydroxylamine HCl leach.

9. HISTORY OF REVISIONS

DATE	REV No	Section	Change
24 Jul 2023	1.0		Initial issue

10. APPROVAL

Authorization	Position	Name	Date
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