

METHOD STATEMENT



Determinand:

As, Cd, Cr, Cu, Hg, Mo, Ni, Pb, Se, Zn

Matrix:

Sample Types: soils and sludge.

Principle of Method:

Metals are determined by ICP-MS after dissolution by a boiling aqua regia digestion. The digestion is used to bring as much of the sample into solution as possible, prior to analysis.

The method is used for solid samples such as soils and for sludge samples where the solids present require an aggressive digestion to ensure dissolution.

Acidified samples are nebulised and the aerosol that is produced is transported to the plasma torch where excitation of the metal atoms occurs. Excitation is due to the high temperatures (up to 6,000K) produced by the radio frequency inductively coupled plasma.

The metal ions thus produced pass through an interface region into the mass spectrometer.

There the ions are separated by a quadrupole where only ions having a specific mass to charge ratio are passed through at any moment in time. The detector then detects these ions and the resulting electrical signals are processed into digital information that is used to indicate ion intensity and subsequently elemental concentration. Internal standardisation is used to correct for transport and matrix effects.

Sampling and Sample Preparation:

Samples are normally received in sludge or soil pots, or sealable plastic bags.

Ground soil samples are stored at room temperature, sludge and wet soil samples are refrigerated at $3 \pm 2^{\circ}\text{C}$.

Soil samples are air-dried and ground according to method WSC15 prior to analysis. Sludge samples are usually analysed on an 'as received' basis and are mixed to obtain as near a homogeneous sample as possible.

Samples are stable for 180 days (BS ISO 18512: 2007) from sampling.

Interferences

The interferences for a number of elements are well documented and understood. Within the limitations of the method, these interferences are adequately compensated for by careful choice of elemental isotopes, interference equations and the use of the helium gas.

Performance of Method:

Range of Application and Limit of Detection:

Element	Mass	Soil LOD (mg/kg)	Soil MRV (mg/kg)	Cake LOD (mg/kg)	Liquid Sludge LOD (mg/kg)	Sludge MRV (mg/kg)	Top Standard $\mu\text{g/l}$
Cr	52	0.618	2.5	3.02	1.28	5	500
Ni	60	0.319	2.5	1.52	0.984	5	500
Cu	63	0.468	2.5	8.74	6.66	10	500
Zn	66	1.16	10	36.4	22.9	50	1000
As	75	0.143	1.5	0.361	0.241	1	200
Se	82	0.0419	0.40	0.183	0.0789	0.40	20
Mo	95	0.237	1.20	0.631	0.255	0.75	50
Cd	111	0.0203	0.05	0.110	0.0949	0.15	20

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Hg	202	0.0218	0.10	0.193	0.139	0.20	2.5
Pb	208	0.304	2.5	2.90	3.38	5	500

Uncertainty of measurement:

Cr	8.993%
Ni	8.312%
Cu	9.312%
Zn	8.938%
As	9.167%
Se	8.847%
Mo	8.385%
Cd	6.92%
Hg	11.183%
Pb	8.744%

References:

- Agilent ICP-MS: ISIS 3 (Integrated Sample Introduction System).
- Agilent ICP-MS: MassHunter Workstation User Guide.
- Agilent ICP-MS: Familiarization Guide
- Agilent 7800/7900 ICPMS: Hardware Maintenance Manual
- Agilent SPS 4 Autosampler: User's Guide
- BS ISO 18512: 2007- Soil quality - Guidance on long and short term storage of soil samples.