METHOD STATEMENT

Determinand:
Determination of Mercury

Matrix:
Sample Type: dried soil, sludge and other materials

Principle of Method:
Metals are determined by ICP-MS after dissolution by a boiling aqua regia digestion. The digestion procedure 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.

ICP-MS (Inductively Coupled Plasma Mass Spectrometry) is a technique for multi – element determination of trace metals. The basis of the technique is the measurement of ions produced by an Inductively Coupled Plasma and detected using a mass spectrometer.

Acidified samples are nebulised and the aerosol that is produced is transported to the plasma torch where excitation and ionisation of the metal atoms occur. Ionisation is caused by the high temperatures (6,000 – 7,000K) inductively coupled plasma removing electrons from their orbitals. 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 to the detector at any moment in time. A dynamic reaction cell using oxygen as the reaction gas is used to reduce interference by interrupting the sequence of reactions that would otherwise cause interference. The dual mode 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.

Interferences:
Tungsten oxide interference can occur as it has the same isotopic mass as Mercury, however the oxygen DRC gas oxidises WO+ ions to higher oxide ions.

Performance of Method:
Range of Application:
LOD to 2.5µg/l
MRV: 0.10mg/kg

Limit of Detection
LOD: 0.0096µg/l

Recoveries of Compounds:

			Average Result (mg/kg)	% Recovery	% RSD	Confidence Interval (mg/kg)	Prediction Interval (mg/kg)
Standards		Low Standard	0.506	101.27	1.89		
		High Standard	2.002	100.12	2.64		
CRM	**Compost**	Agromat	0.157		7.79	0.126 - 0.158	
	Sludge	LGC6181	5.075		4.10	4.6 - 5.2	
		CRM018	6.094		2.90	3.87 - 5.68	0.57 - 8.99
		CRM031	14.12		5.41	11.4 - 15.1	
	Loamy Sand	CRM024	0.933		6.88	0.650 - 0.760	0.480 - 0.930
	Silt Clay	CRM0145	0.870		7.29	0.713 - 0.877	
	Loam	ERM-CC 140	0.128		7.06	0.066 - 0.100	0.053 - 0.113

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References:

Perkin Elmer Elan DRC-e Hardware manual.

Mercury in waters, effluent, soils and sediments etc (additional methods) 1985. (ISBN 011 7519073).

Methods for the determination of metals in soils, sediements, and sewage sludge, and plants by hydrochloric-nitric digestion, with a note on the determination of insoluble metal contents 1986. (ISBN 011 7519081).

BS ISO 18512: 2007- Soil quality – Guidance on long and short term storage of soil samples.