METHOD STATEMENT

Determinand:
Determination of V, Cr, Co, Ni, Cu, Zn, As, Se, Mo, Cd, Sn, Tl & Pb

Matrix:
Sample Type: soils, sludge and other materials requiring a hot concentrated acid digest to bring the elements into solution

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 quadropole where only ions having a specific mass to charge ratio are passed through at any moment in time. 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. Internal standardisation is used to correct for transport and matrix effects.
A table of the isotopes measured and the internal standards used is given below.

Elan ICP-MS			
Element	**Mass**	**Internal Standard used**	**Mass**
V	51	Ge	72
Cr	52	Ge	72
Co	59	Ge	72
Ni	60	Ge	72
Cu	65	Ge	72
Zn	66	Ge	72
As*	75	Ge	72
Se	82	Ge	72
Mo	98	In	115
Cd	111	In	115
Sn	118	In	115
Tl	205	Bi	209
Pb**	208	Bi	209

* Due to As at mass 75 being susceptible to chloride interference the result obtained for As with no interference equation applied may give a false high result. As int has an interference equation applied that takes into account any chloride and selenium in the sample as well as krypton contamination in the Argon. As int 2 has an interference equation taking into account Se levels only. Results should be reported routinely from As int.
** Note that Pb 208 is a summation of masses Pb 206, Pb 207 & Pb 208 due to its isotopic ratio varying from source to source.

Interferences:
Careful choice of isotopes, the use of reaction gas, interference equations and optimum plasma conditions are all used to minimise any potential interferences.

Performance of Method:

Range of Application:

V - LOD to 200 µg/l
Cr - LOD to 500 µg/l
Co - LOD to 50 µg/l
Ni - LOD to 500 µg/l
Cu - LOD to 500 µg/l
Zn - LOD to 1000 µg/l
As - LOD to 200 µg/l
Se - LOD to 20 µg/l
Mo - LOD to 50 µg/l
Cd - LOD to 20 µg/l
Sn - LOD to 50 µg/l
Tl - LOD to 50 µg/l
Pb - LOD to 500 µg/l

All analytical ranges may be extended by sample dilution.

Limit of Detection and Recoveries of Compounds:

			Low Standard	High Standard
	LOD mg/kg	**MRV mg/kg**	**Recovery %**	**Recovery %**
V	1.87	2.0	95.68	102.91
Cr	1.49	2.5	97.66	105.35
Co	0.021	0.100	102.86	102.89
Ni	0.75	2.5	102.52	102.76
Cu	0.37	2.5	102.68	102.56
Zn	4.39	10.0	104.27	103.37
As	1.41	1.5	104.55	103.73
Se	0.29	0.40	103.41	101.92
Mo	1.19	1.20	101.94	100.69
Cd	0.008	0.04	102.34	101.17
Sn	1.198	2.3	105.01	100.65
Tl	0.157	0.25	99.60	99.68
Pb	0.689	2.5	95.01	100.66

	Clay Soil	Loam Soil	Light Sandy Soil	Knostrop HL Sludge	Knostrop LL Sludge
	Recovery %	**Recovery %**	**Recovery %**	**Recovery %**	**Recovery %**
V	97.24	96.55	108.06	102.55	103.35
Cr	100.14	100.36	106.02	105.04	105.51
Co	97.78	97.95	102.61	101.76	103.33
Ni	95.56	95.82	96.52	101.11	102.57
Cu	93.05	92.63	87.83	98.71	100.32
Zn	94.56	94.05	77.21	99.58	102.21
As	96.69	97.78	106.69	103.22	104.88
Se	90.87	91.69	92.99	99.61	100.77
Mo	95.18	93.62	102.12	99.46	100.26
Cd	99.49	98.54	91.22	99.15	100.84
Sn	94.32	93.28	104.45	105.7	106.21
Tl	100.51	100.39	109.13	98.87	99.48
Pb	100.96	99.65	104.49	97.75	100.78

References:

Perkin Elmer SCIEX Elan DRC-e Hardware Guide.
BS ISO 18512: 2007- Soil quality – Guidance on long and short term storage of soil samples.