METHOD STATEMENT

Determinand:
Determination of Metals: Cd, Cr, Cu, Mn, Ni, Pb, Sn, V, Zn, Al, Ca , Fe , K, Mg , Na , P, S

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-OES (inductively coupled plasma optical emission spectroscopy) 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.
The acidified samples are then analysed by the ICP-OES instrument to determine the concentration of metals present. Excitation of the sample within the 6,000°C plasma causes ionisation of atoms, which in turn causes the emission of electromagnetic radiation at specific wavelengths for each element. The intensity of the emission is measured and quantified by comparison against standards with known concentrations of elements.

Sampling and Sample Preparation:
Samples are normally received in sludge or soil pots.
Ground soil samples are stored at room temperature, sludge and wet soil samples are refrigerated at 3 ± 2°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
Spectral Interference may occur from the presence of other elements. The spectral lines have been chosen so that overlap is minimal. Elements within standards have been chosen to minimise chemical interference.

Performance of Method:

Range of Application:

Compound	mg/L
Cd	LOD – 0.4
Cr	LOD - 10
Cu	LOD - 20
Li	LOD - 2
Mn	LOD - 100
Ni	LOD - 10
Pb	LOD - 20
Sn	LOD - 1
V	LOD - 4
Zn	LOD - 40
Al	LOD - 400
Ca	LOD - 1000
Fe	LOD - 1000

Compound	mg/L
K	LOD - 300
Mg	LOD - 200
Na	LOD - 500
P	LOD - 400
S	LOD - 1000

All analytical ranges may be extended by sample dilution.

Limit of Detection and Recoveries of Compounds:

	LOD mg/l	Soil LOD mg/kg	MRV mg/kg	Cake LOD* (2g/25% DS)	Sludge LOD* (5g/3% DS)	Low Standard	High Standard	Clay Soil	Loam Soil	Sandy Soil	Knostrop sludge
						Recovery %	Recovery %	Recovery %	Recovery %	Recovery %	Recovery %
Al	0.1604	8.02	74	16.04	53.47	102.66	97.81	102.32	107.29	104.25	100.30
Fe	1.2428	62.14	218	124.28	414.27	101.6	107.45	107.48	109.30	109.47	108.03
K	0.3314	16.57	87	33.14	110.47	99.32	98.88	99.64	102.42	102.11	102.11
Mn	0.0826	4.13	32.7	8.26	27.53	102.16	102.41	95.63	96.04	100.30	99.41
Na	0.7436	37.18	150	74.36	247.87	100.34	99.27	102.07	101.68	103.50	102.18
Zn	0.0254	1.27	12.3	2.54	8.47	102.59	100.66	104.51	104.40	108.02	103.91
Ca	0.7846	39.32	222	78.64	262.13	101.99	98.98	100.56	101.33	101.64	100.29
Li	0.0016	0.08	4.15	0.16	0.53	105.63	100.33	104.15	102.48	104.11	102.94
Cr	0.0068	0.34	4.30	0.68	2.27	103.25	101.04	102.37	101.86	104.85	102.88
Cu	0.0116	0.58	7.8	1.16	3.87	99.78	99.17	102.89	101.73	103.29	102.14
Ni	0.0086	0.43	5.10	0.86	2.87	103.90	101.26	108.21	108.04	112.72	109.48
P	0.5374	26.87	58.4	53.74	179.13	100.93	98.04	106.27	105.90	109.97	105.69
Pb	0.013	0.65	6.2	1.30	4.33	103.38	100.24	92.54	95.08	96.75	93.75
S	0.58	29.0	178	58.00	193.33	101.79	100.95	95.04	101.35	100.39	103.02
Sn	0.0118	0.59	1.8	1.18	3.93	97.1	98.33	110.36	109.68	115.57	113.58
V	0.004	0.2	1.54	0.40	1.33	102.79	101.0	104.29	103.51	105.75	103.44
Cd	0.00092	0.046	0.11	0.09	0.31	102.04	101.38	106.23	105.31	108.59	106.01
Mg	0.0742	3.71	38	7.42	24.73	101.03	101.16	100.85	97.32	103.79	103.14

References:

Perkin Elmer User Training course.

Perkin Elmer Optima 7100, 7200 and 7300 series Hardware guide manual.