

## METHOD STATEMENT

### **Determinand:**

Metals

(Arsenic, cadmium, chromium, copper, lead, nickel, zinc, iron, boron, mercury, selenium, antimony, barium and molybdenum).

### **Matrix:**

Waste acceptance criteria (WAC) leachates.

### **Principle of Method:**

The WAC leachate is prepared in accordance with method BSEN 12457.3 and a portion of the sample is diluted 20 fold with a blank standard prior to analysis. The concentration of the metals are determined by the technique of inductively coupled plasma mass spectroscopy (ICPMS).

An ICP source consists of a flowing stream of argon gas ionised by an applied radio frequency field. This field is inductively coupled to ionised gas by a coil surrounding a quartz 'torch' that supports and confines the plasma. A sample aerosol is generated in a nebuliser and spray chamber and is carried into the plasma through an injector tube. The sample aerosol is injected directly into the ICP subjecting the constituent atoms to temperatures of between 6000 and 8000 K. Because this results in almost complete dissociation of molecules a significant reduction in chemical interferences is achieved.

### **Sampling and Sample Preparation:**

The WAC leachate is prepared in accordance with method BSEN 12457.3 and a portion of the sample  $0.5 \pm 0.02$  ml is diluted to  $10 \pm 0.02$ ml using a blank standard.

### **Interferences:**

The instrument has a collision cell, which allows Helium gas to be used to reduce the effect of polyatomic interferences.

Correction factors have been applied for those elements that have significant interferences that cannot be eliminated by using Helium collision gas.

The correction on Mercury mass 202 is to account for an interference from Tungsten Oxide(WO). A tungsten interference correction solution is included on every run so that the correction equation can be checked and adjusted as necessary.

Selenium mass 78 in collision mode is used to check for bromide interference on Selenium mass 82 in standard mode

The technique of ICPMS analysis reduces the effects of chemical interference on the final analytical measurements to insignificant levels for the elements determined in the acid matrix.



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## Performance of Method:

Determinand	Range of Application (mg/l)	LOD (mg/l)	Routine Reporting Limit (mg/l)
Arsenic	0.005 - 2.0	0.0011	0.005
Cadmium	0.0001 - 1.0	0.00008	0.0001
Chromium	0.0025 - 2.0	0.0018	0.0025
Copper	0.010 - 20	0.0025	0.010
Lead	0.010 - 10	0.0006	0.01
Nickel	0.020 - 2.0	0.0026	0.02
Zinc	0.025 - 20	0.021	0.025
Iron	0.300 - 40	0.087	0.30
Boron	1.000 - 20	0.29	0.80
Mercury	0.0005 - 0.20	0.0004	0.0005
Selenium	0.01 - 0.40	0.0055	0.01
Antimony	0.006 - 0.40	0.0021	0.006
Barium	0.060 - 20	0.053	0.06
Molybdenum	0.002 - 2.0	0.0014	0.002

	% RSD	% Recovery	% Uncert.
Boron	3.12	101.78	10.67
Chromium	2.73	104.14	11.07
Iron	2.22	102.26	8.03
Nickel	2.13	103.79	9.79
Copper	2.15	101.59	7.15
Zinc	2.88	103.01	9.72
Arsenic	1.81	104.75	11.09
Selenium	2.37	98.32	7.86
Molybdenum	1.97	102.55	8.41
Cadmium	1.91	101.09	6.99
Antimony	2.35	98.53	7.02
Barium	3.57	93.66	15.09
Mercury	5.60	96.96	10.02
Lead	1.98	103.48	8.97

Date of testing: - November 2016

## References:

Digestion for metals 3030E HNO<sub>3</sub> digestion, Standard Methods 19<sup>th</sup> Ed. 1995. APHA.

