

METHOD STATEMENT

Determinand:

Metals:-

(Aluminium, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Manganese, Molybdenum, Nickel, Selenium, Strontium, Tellurium, Thallium, Tin, Titanium, Uranium, Vanadium, Zinc, Phosphorus).

Matrix:

Treated sewage, Untreated sewage, Trade effluents, Surface waters, Groundwaters, Land leachates and prepared leachates

Principle of Method:

Metals are determined by ICP-MS after heated dissolution in the presence of nitric acid. The digestion pre-treatment ensures that any suspended or colloidal forms are converted to soluble forms. Filtered (otherwise known as dissolved or soluble) metals may also be determined, by filtration through a 0.45µm membrane filter prior to analysis.

Sampling and Sample Preparation:

Samples undergo a hot nitric acid digest in accordance with Method WAS 011. The prepared samples are stored until required for analysis.

Samples are stable for times stated below, (In-House Data) from sampling.

| | |
|----|-------------------------|
| Al | 14 Days (In-House Data) |
| As | 29 Days (In-House Data) |
| Ba | 9 Days (In-House Data) |
| Be | 17 Days (In-House Data) |
| B | 7 Days (In-House Data) |
| Cd | 17 Days (In-House Data) |
| Cr | 17 Days (In-House Data) |
| Co | 17 Days (In-House Data) |
| Cu | 14 Days (In-House Data) |
| Fe | 17 Days (In-House Data) |
| Pb | 16 Days (In-House Data) |
| Mn | 17 Days (In-House Data) |
| Mo | 15 Days (In-House Data) |
| Ni | 17 Days (In-House Data) |
| P | 17 Days (In-House Data) |
| Sb | 29 Days (In-House Data) |
| Se | 29 Days (In-House Data) |
| Sr | 17 Days (In-House Data) |
| Te | 11 Days (In-House Data) |
| Tl | 17 Days (In-House Data) |
| Sn | 7 Days (In-House Data) |
| Ti | 14 Days (In-House Data) |
| U | 29 Days (In-House Data) |
| V | 17 Days (In-House Data) |
| Zn | 17 Days (In-House Data) |



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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 and by using interference equations. Appendix IV has details of the interference equations used.

Additional isotopes for Cu, Zn, Fe, Sn, Se, Mo and Te have been included in the method in order to investigate suspected interference on individual samples.

Performance of Method:

Total Metals

Perkin Elmer Elan DRC ICPMS

| Determinand | MCERTS Accreditation | Range of Application (µg/l) | LOD (µg/l) | Routine Reporting Level (µg/l) |
|-------------|----------------------|-----------------------------|------------|--------------------------------|
| Aluminium | ✓ | 15 - 1000 | 14.68 | 15 |
| Antimony | ✓ | 1.2 - 20 | 0.3617 | 1.2 |
| Arsenic | ✓ | 1.0 - 500 | 0.9512 | 1.0 |
| Barium | | 2.8 - 1000 | 1.0823 | 2.8 |
| Beryllium | | 0.1 - 100 | 0.0934 | 0.1 |
| Boron | | 19 - 1000 | 8.2749 | 19 |
| Cadmium | ✓ | 0.07 - 100 | 0.0632 | 0.07 |
| Chromium | ✓ | 0.8- 100 | 0.4361 | 0.8 |
| Cobalt | ✓ | 1.6 - 100 | 0.4797 | 1.6 |
| Copper | ✓ | 1.9 - 100 | 1.8830 | 1.9 |
| Iron | ✓ | 120 - 10000 | 115.6165 | 120 |
| Lead | ✓ | 0.5 - 100 | 0.3373 | 0.5 |
| Manganese | | 5.6 - 1000 | 5.594 | 5.6 |
| Molybdenum | | 2 - 100 | 1.9959 | 2 |
| Nickel | ✓ | 1.7 - 100 | 1.6837 | 1.7 |
| Phosphorus | | 26 - 1000 | 18.2665 | 26 |
| Selenium | | 0.8 - 150 | 0.7121 | 0.8 |
| Strontium | | 20 - 1000 | 19.0494 | 20 |
| Tellurium | | 0.38 - 20 | 0.1242 | 0.38 |
| Thallium | | 0.8 - 100 | 0.7554 | 0.8 |
| Tin | ✓ | 3.2 - 100 | 3.1194 | 3.4 |
| Titanium | | 2.6 - 100 | 0.5476 | 2.6 |
| Uranium | | 0.31 - 30 | 0.3056 | 0.31 |
| Vanadium | | 0.9 - 100 | 0.3056 | 0.9 |
| Zinc | ✓ | 14 -1000 | 13.0881 | 14 |

| Determinand | Low standard | | | High standard | | |
|-------------|--------------|-------|--------|---------------|-------|--------|
| | Conc. µg/l | RSD % | Bias % | Conc. µg/l | RSD % | Bias % |
| Aluminium | 200 | 4.89 | +5.33 | 800 | 5.24 | -0.86 |
| Antimony | 4 | 5.07 | +1.58 | 16 | 3.68 | -1.15 |
| Arsenic | 100 | 3.14 | +0.10 | 400 | 1.98 | +0.59 |
| Barium | 200 | 8.99 | +7.71 | 800 | 2.45 | +4.99 |
| Beryllium | 20 | 8.44 | +0.95 | 80 | 8.95 | -4.61 |
| Boron | 200 | 9.93 | +1.84 | 800 | 9.04 | -0.72 |



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| Determinand | Low standard | | | High standard | | |
|-------------|--------------|-------|--------|---------------|-------|--------|
| | Conc. µg/l | RSD % | Bias % | Conc. µg/l | RSD % | Bias % |
| Elan | | | | | | |
| Cadmium | 20 | 3.01 | -1.24 | 80 | 2.57 | -3.32 |
| Chromium | 20 | 4.16 | +4.89 | 80 | 2.56 | +4.67 |
| Cobalt | 20 | 3.64 | -3.77 | 80 | 2.69 | -3.45 |
| Copper | 20 | 5.24 | -1.50 | 80 | 3.45 | -6.81 |
| Iron | 2000 | 2.98 | -0.86 | 8000 | 3.70 | -1.62 |
| Lead | 20 | 3.97 | -0.32 | 80 | 2.09 | -3.82 |
| Manganese | 200 | 2.60 | -2.51 | 800 | 1.71 | -0.60 |
| Molybdenum | 20 | 6.06 | +3.86 | 80 | 4.29 | 2.39 |
| Nickel | 20 | 3.82 | +1.12 | 80 | 5.53 | -0.04 |
| Phosphorus | 200 | 6.12 | +5.65 | 800 | 4.53 | 2.89 |
| Selenium | 30 | 3.35 | +0.33 | 120 | 3.10 | -0.66 |
| Strontium | 200 | 7.83 | +0.54 | 800 | 4.43 | -3.06 |
| Tellurium | 4 | 3.49 | +0.99 | 16 | 3.06 | -5.04 |
| Thallium | 20 | 2.68 | -0.97 | 80 | 2.18 | -2.92 |
| Tin | 20 | 4.41 | +2.76 | 80 | 2.12 | +6.92 |
| Titanium | 20 | 4.06 | +6.08 | 80 | 4.38 | +1.52 |
| Uranium | 20 | 2.81 | -0.11 | 80 | 4.02 | +1.47 |
| Vanadium | 20 | 3.76 | +0.12 | 80 | 2.70 | +2.23 |
| Zinc | 200 | 1.52 | +3.69 | 800 | 1.57 | +1.71 |

| Determinand | Spike Level | Treated Sewage (Finham) | | Trade effluent (Trimpley) | | Trade effluent (Walkers) | | Untreated sewage (Finham/Alfreton*) | |
|-------------|-------------|-------------------------|--------|---------------------------|---------|--------------------------|--------|-------------------------------------|--------|
| | | RSD % | Rec. % | RSD % | Rec. % | RSD % | Rec. % | RSD % | Rec. % |
| Elan | | | | | | | | | |
| Aluminium | 20% | 5.16 | 102.14 | 4.69 | 103.05 | - | - | 5.43 | 99.84 |
| | 80% | 5.07 | 95.84 | 5.66 | 101.66 | 5.10 | 97.10 | 4.87 | 96.77 |
| Antimony | 20% | 3.94 | 97.24 | 3.12 | 99.75 | 4.77 | 99.38 | 3.32 | 96.21 |
| | 80% | 4.51 | 98.98 | 4.20 | 98.15 | 4.92 | 97.05 | 3.28* | 96.40* |
| Arsenic | 20% | 2.57 | 104.27 | 1.94 | 100.86 | - | - | 2.12 | 102.33 |
| | 80% | 2.35 | 103.34 | 2.25 | 101.40 | 3.88 | 113.07 | 2.19 | 102.92 |
| Barium | 80% | 4.48 | 105.32 | - | - | 4.69 | 107.54 | 2.63 | 106.42 |
| Beryllium | 80% | 7.82 | 93.00 | - | - | 8.36 | 96.15 | 9.42 | 93.49 |
| Boron | 80% | 6.98 | 97.12 | - | - | 6.63 | 100.34 | 6.84 | 101.94 |
| Cadmium | 20% | 2.35 | 97.15 | 2.17 | 98.41 | 3.29 | 98.25 | 2.62 | 98.71 |
| | 80% | 4.07 | 94.03 | 2.58 | 97.56 | 2.88 | 93.60 | 2.06 | 95.28 |
| Chromium | 20% | 3.18 | 105.35 | 2.99 | 104.84 | 4.52 | 106.37 | 3.13 | 106.57 |
| | 80% | 2.97 | 106.14 | 3.37 | 106.78 | 4.42 | 106.47 | 2.96 | 105.89 |
| Cobalt | 20% | 3.28 | 98.48 | 2.93 | 97.88 | 2.74 | 99.74 | 3.14 | 99.73 |
| | 80% | 2.95 | 97.52 | 2.93 | 101.28 | 3.95 | 101.23 | 2.58 | 101.29 |
| Copper | 20% | 4.93 | 95.10 | 3.63 | 91.75 | - | - | 3.47* | 91.51* |
| | 80% | 2.32 | 90.38 | 2.65 | 93.18 | 3.94 | 88.49 | 2.20* | 93.26* |
| Iron | 20% | 4.18 | 99.86 | 3.61 | 100.8 | - | - | 4.42 | 102.03 |
| | 80% | 2.91 | 98.54 | 3.48 | 102.140 | 3.25 | 94.74 | 3.48 | 98.87 |
| Lead | 20% | 2.36 | 97.03 | 2.59 | 96.81 | 2.04 | 93.57 | 1.94 | 98.03 |
| | 80% | 1.76 | 95.46 | 2.73 | 97.99 | 3.82 | 92.25 | 2.49 | 95.84 |
| Manganese | 20% | 2.50 | 93.48 | 2.35 | 96.30 | 3.81 | 103.49 | 2.77* | 93.34* |



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| Determinand | Spike Level | Treated Sewage (Finham) | | Trade effluent (Trimpley) | | Trade effluent (Walkers) | | Untreated sewage (Finham/Alfreton*) | |
|-------------|-------------|-------------------------|--------|---------------------------|--------|--------------------------|--------|-------------------------------------|--------|
| | | RSD % | Rec. % | RSD % | Rec. % | RSD % | Rec. % | RSD %. | Rec. % |
| Elan | 80% | 2.10 | 96.30 | 2.08 | 96.74 | 2.66 | 99.80 | 2.03* | 95.61* |
| Molybdenum | 80% | 3.80 | 103.08 | - | - | 5.19 | 101.81 | 3.62 | 102.69 |
| Nickel | 20% | 3.72 | 98.95 | 3.54 | 102.54 | 4.23 | 99.37 | 3.59 | 102.78 |
| | 80% | 3.56 | 98.37 | 3.16 | 103.97 | 3.90 | 99.11 | 4.53 | 98.75 |
| Phosphorus | 20% | 4.82 | 105.47 | 4.46 | 98.56 | - | - | 4.95 | 107.15 |
| | 80% | 4.47 | 103.87 | 4.34 | 98.35 | 4.05 | 103.02 | 4.91 | 101.27 |
| Selenium | 80% | 2.98 | 98.63 | - | - | 4.64 | 104.31 | 1.82* | 96.16* |
| Strontium | 80% | 3.29 | 94.61 | - | - | 3.39 | 97.54 | 3.17 | 96.15 |
| Tellurium | 80% | 2.44 | 91.34 | - | - | 3.04 | 93.94 | 2.99 | 90.81 |
| Thallium | 80% | 1.66 | 96.61 | - | - | 3.66 | 94.28 | 2.48 | 96.94 |
| Tin | 20% | 3.14 | 104.6 | 4.34 | 101.93 | - | - | 5.73 | 95.78 |
| | 80% | 2.14 | 105.29 | 3.10 | 105.99 | - | - | 3.01 | 102.03 |
| Titanium | 80% | 5.00 | 100.20 | 3.43 | 104.14 | 9.15 | 84.82 | 7.05 | 94.26 |
| Uranium | 80% | 4.66 | 102.62 | 3.19 | 101.48 | 7.58 | 82.89 | 3.74 | 103.45 |
| Vanadium | 80% | 3.18 | 105.66 | - | - | 3.93 | 107.64 | 2.93 | 105.66 |
| Zinc | 20% | 2.61 | 95.57 | 3.57 | 98.84 | - | - | 2.74* | 93.50* |
| | 80% | 1.78 | 95.13 | 1.97 | 97.47 | - | - | 2.40* | 93.87* |

| Determinand | Groundwater | | Landfill leachate | | Prepared leachate | | Surface water | |
|-------------|-------------|--------|-------------------|--------|-------------------|--------|---------------|--------|
| | RSD % | Rec. % | RSD % | Rec. % | RSD %. | Rec. % | RSD %. | Rec. % |
| Elan | 4.54 | 97.60 | 6.27 | 95.46 | 5.12 | 99.01 | 4.19 | 98.05 |
| Aluminium | 4.54 | 97.60 | 6.27 | 95.46 | 5.12 | 99.01 | 4.19 | 98.05 |
| Antimony | 3.82 | 97.99 | 3.71 | 98.42 | 3.20 | 98.04 | 4.58 | 97.80 |
| Arsenic | 1.39 | 101.40 | 2.73 | 104.19 | 2.46 | 102.00 | 3.51 | 102.88 |
| Barium | 3.15 | 104.99 | 2.54 | 105.65 | 2.39 | 105.94 | 4.36 | 105.86 |
| Beryllium | 4.90 | 97.02 | 6.83 | 93.19 | 8.73 | 93.24 | 9.28 | 91.72 |
| Boron | 5.28 | 99.71 | 7.34 | 97.50 | 9.12 | 97.84 | 6.16 | 96.98 |
| Cadmium | 2.01 | 96.45 | 2.88 | 92.33 | 2.03 | 97.56 | 2.56 | 95.65 |
| Chromium | 1.62 | 105.82 | 3.12 | 105.95 | 2.66 | 105.92 | 2.83 | 105.46 |
| Cobalt | 1.68 | 96.78 | 3.52 | 95.97 | 2.65 | 97.98 | 3.46 | 97.26 |
| Copper | 1.60 | 90.65 | 2.26 | 92.67 | 8.76 | 95.21 | 7.73 | 92.34 |
| Iron | 3.04 | 98.97 | 4.13 | 98.77 | 3.57 | 99.20 | 4.31 | 98.99 |
| Lead | 1.63 | 96.54 | 2.12 | 95.13 | 2.31 | 97.47 | 2.96 | 96.02 |
| Manganese | 2.60 | 98.66 | 3.45 | 100.11 | 4.06 | 99.18 | 3.22 | 99.91 |
| Molybdenum | 4.17 | 102.75 | 4.44 | 102.55 | 3.27 | 102.25 | 5.40 | 102.20 |
| Nickel | 2.53 | 99.08 | 3.75 | 95.06 | 5.50 | 100.84 | 3.38 | 99.38 |
| Phosphorus | 6.44 | 101.91 | 6.68 | 98.98 | 5.49 | 102.11 | 5.37 | 101.12 |
| Selenium | 2.84 | 99.44 | 3.61 | 104.18 | 2.67 | 99.76 | 3.36 | 98.65 |
| Strontium | 5.08 | 96.69 | 3.04 | 97.57 | 4.00 | 97.73 | 3.72 | 95.93 |
| Tellurium | 2.28 | 91.35 | 2.44 | 93.78 | 1.36 | 94.37 | 2.59 | 92.78 |
| Thallium | 1.87 | 98.02 | 2.18 | 97.44 | 7.57 | 96.81 | 2.51 | 97.48 |
| Tin | 2.57 | 105.98 | 1.87 | 103.42 | 1.83 | 106.31 | 2.16 | 105.56 |
| Titanium | 4.13 | 100.74 | 3.73 | 97.46 | 6.81 | 101.10 | 4.04 | 101.79 |
| Uranium | 2.75 | 102.67 | 3.40 | 103.77 | 7.76 | 100.57 | 4.67 | 102.20 |
| Vanadium | 1.72 | 104.29 | 3.75 | 105.58 | 7.87 | 102.47 | 3.49 | 103.79 |
| Zinc | 1.42 | 96.16 | 3.48 | 90.48 | 2.00 | 99.82 | 1.70 | 97.02 |



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Uncertainty of Measurement

Total Metals

The reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2, which gives a level of confidence of approximately 95%

Perkin Elmer Elan DRC ICPMS

| Determinand (total) | Uncertainty of Measurement (%) |
|---------------------|--------------------------------|
| Aluminium | 11.16 |
| Arsenic | 7.16 |
| Boron | 19.73 |
| Barium | 14.95 |
| Beryllium | 22.76 |
| Cadmium | 10.46 |
| Cobalt | 9.08 |
| Chromium | 14.37 |
| Copper | 17.73 |
| Iron | 9.89 |
| Manganese | 11.35 |
| Molybdenum | 9.62 |
| Nickel | 11.48 |
| Phosphorus | 11.26 |
| Lead | 9.88 |
| Antimony | 11.57 |
| Selenium | 7.72 |
| Tin | 12.44 |
| Strontium | 11.21 |
| Tellurium | 16.29 |
| Titanium | 17.83 |
| Thallium | 9.07 |
| Uranium | 10.19 |
| Vanadium | 12.16 |
| Zinc | 11.18 |

Filtered Metals

Perkin Elmer Elan DRC ICPMS

| Determinand | Range of Application (µg/l) | LOD (µg/l) | Routine Reporting Level (µg/l) |
|-------------|-----------------------------|------------|--------------------------------|
| Aluminium | 15 - 1000 | 11.1462 | 15 |
| Antimony | 1.2 - 20 | 0.4522 | 1.2 |
| Arsenic | 1.0 - 500 | 0.9513 | 1.0 |
| Barium | 2.8 - 1000 | 0.6621 | 2.8 |
| Beryllium | 0.1 - 100 | 0.0762 | 0.1 |
| Boron | 19 - 1000 | 18.4982 | 19 |
| Cadmium | 0.07 - 100 | 0.0179 | 0.07 |
| Chromium | 4.7 - 100 | 0.7934 | 4.7 |
| Cobalt | 1.6 - 100 | 0.0579 | 1.6 |
| Copper | 1.9 - 100 | 1.8125 | 1.9 |



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| Determinand | Range of Application (µg/l) | LOD (µg/l) | Routine Reporting Level (µg/l) |
|-------------|-----------------------------|------------|--------------------------------|
| Iron | 120 - 10000 | 51.5151 | 120 |
| Lead | 0.5 - 100 | 0.4687 | 0.5 |
| Nickel | 1.7 - 100 | 1.5312 | 1.7 |
| Phosphorus | 26 - 1000 | 10.3066 | 26 |
| Selenium | 0.8 - 150 | 0.4602 | 0.8 |
| Tellurium | 0.38 - 20 | 0.3738 | 0.38 |
| Uranium | 0.31 - 30 | 0.2005 | 0.31 |
| Vanadium | 0.9 - 100 | 0.6333 | 0.9 |
| Zinc | 14 - 100 | 5.5766 | 14 |

| Determinand | Ground water | | | Land Leachate | | |
|-------------|--------------|-------|--------|---------------|-------|--------|
| | Conc. µg/l | RSD % | Rec. % | Conc. µg/l | RSD % | Rec. % |
| Elan | | | | | | |
| Aluminium | 12.80 | 2.70 | 97.57 | 3.85 | 3.84 | 94.81 |
| Antimony | 0.32 | 4.14 | 102.77 | 0.72 | 3.55 | 102.47 |
| Arsenic | 9.01 | 2.38 | 101.91 | 1.33 | 2.82 | 104.34 |
| Barium | 60.0 | 3.54 | 101.25 | 5.76 | 2.84 | 104.18 |
| Beryllium | 0.01 | 3.52 | 106.22 | 0.01 | 4.11 | 106.74 |
| Boron | 86.2 | 2.52 | 101.34 | 929 | 3.83 | 102.65 |
| Cadmium | 0.01 | 1.92 | 98.88 | 0.01 | 2.54 | 98.00 |
| Chromium | 0.26 | 2.42 | 103.24 | 0.72 | 2.70 | 101.7 |
| Cobalt | 0.07 | 2.44 | 99.79 | 2.86 | 3.27 | 98.77 |
| Copper | 1.04 | 2.26 | 93.22 | 7.87 | 3.13 | 90.23 |
| Iron | 25.5 | 2.50 | 100.18 | 509 | 3.22 | 98.76 |
| Lead | <0.5 | 3.05 | 97.32 | <0.5 | 2.45 | 96.73 |
| Nickel | 0.20 | 2.52 | 97.38 | 4.27 | 2.81 | 95.56 |
| Phosphorus | 0.65 | 2.08 | 101.01 | <19 | 3.09 | 101.20 |
| Selenium | 0.33 | 2.47 | 100.87 | 3.14 | 3.04 | 106.39 |
| Tellurium | 0.12 | 2.27 | 96.45 | 0.13 | 2.97 | 101.33 |
| Uranium | 1.04 | 3.58 | 103.71 | 5.63 | 5.13 | 107.15 |
| Vanadium | 1.53 | 2.41 | 98.30 | <0.9 | 2.99 | 98.65 |
| Zinc | 6.20 | 2.28 | 96.05 | 4.15 | 2.78 | 90.22 |

Uncertainty of Measurement

The reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2, which gives a level of confidence of approximately 95%.

| Determinand | Uncertainty of Measurement (%) | |
|-------------|--------------------------------|-----------------|
| | Total Metals | Filtered Metals |
| Aluminium | 11.16 | 11.22 |
| Arsenic | 7.16 | 8.17 |
| Boron | 19.73 | 9.44 |
| Barium | 14.95 | 8.25 |
| Beryllium | 22.76 | 15.41 |
| Cadmium | 10.46 | 5.29 |



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| Determinand | Uncertainty of Measurement (%) | |
|-------------|--------------------------------|-----------------|
| | Total Metals | Filtered Metals |
| Cobalt | 9.08 | 5.62 |
| Chromium | 14.37 | 7.44 |
| Copper | 17.73 | 17.89 |
| Iron | 9.89 | 6.07 |
| Manganese | 11.35 | - |
| Molybdenum | 9.62 | - |
| Nickel | 11.48 | 9.00 |
| Phosphorus | 11.26 | 5.26 |
| Lead | 9.88 | 8.06 |
| Antimony | 11.57 | 8.10 |
| Selenium | 7.72 | 10.43 |
| Tin | 12.44 | - |
| Strontium | 11.21 | - |
| Tellurium | 16.29 | - |
| Titanium | 17.83 | - |
| Thallium | 9.07 | 7.21 |
| Uranium | 10.19 | 13.90 |
| Vanadium | 12.16 | 6.32 |
| Zinc | 11.18 | 16.08 |

Total Metals

Perkin Elmer NexION 300D ICP-MS

| Determinand | MCERTS Accreditation | Range of Application (µg/l) | LOD (µg/l) | Routine Reporting Level (µg/l) |
|-------------|----------------------|-----------------------------|------------|--------------------------------|
| Aluminium | Y | 15 - 1000 | 14.5311 | 15 |
| Antimony | Y | 0.5 - 20 | 0.4370 | 1.2 |
| Arsenic | Y | 1.0 - 500 | 0.2955 | 1.0 |
| Barium | | 2.8 - 1000 | 0.3504 | 2.8 |
| Beryllium | | 0.1 - 100 | 0.0652 | 0.1 |
| Boron | | 19 - 1000 | 5.3353 | 19 |
| Cadmium | Y | 0.07 - 100 | 0.0198 | 0.07 |
| Chromium | Y | 0.8 - 100 | 0.1378 | 0.8 |
| Cobalt | Y | 1.6 - 100 | 0.1205 | 1.6 |
| Copper | Y | 1.9 - 100 | 1.2159 | 1.9 |
| Iron | Y | 120 - 10000 | 45.8063 | 120 |
| Lead | Y | 0.5 - 100 | 0.1208 | 0.5 |
| Lithium | | 0.2 - 100 | 0.1029 | 0.2 |
| Manganese | | 5.6 - 1000 | 0.3244 | 5.6 |
| Molybdenum | | 2 - 100 | 0.3803 | 2 |
| Nickel | | 1.7 - 100 | 0.6512 | 1.7 |
| Phosphorus | Y | 26 - 1000 | 21.9145 | 26 |
| Selenium | | 0.8 - 150 | 0.4492 | 0.8 |
| Strontium | | 20 - 1000 | 0.3454 | 20 |
| Tellurium | | 0.38 - 20 | 0.0305 | 0.38 |



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| Determinand | MCERTS Accreditation | Range of Application (µg/l) | LOD (µg/l) | Routine Reporting Level (µg/l) |
|-------------|----------------------|-----------------------------|------------|--------------------------------|
| Thallium | | 0.8 - 100 | 0.0513 | 0.8 |
| Titanium | | 2.6 - 100 | 0.2013 | 2.6 |
| Uranium | | 0.31 - 30 | 0.0388 | 0.31 |
| Vanadium | | 0.9 - 100 | 0.1435 | 0.9 |
| Zinc | Y | 14 -1000 | 2.7773 | 14 |

| Determinand | Low standard | | | High standard | | |
|-------------|--------------|-------|--------|---------------|-------|--------|
| | Conc. µg/l | RSD % | Bias % | Conc. µg/l | RSD % | Bias % |
| Aluminium | 200 | 3.54 | 6.09 | 800 | 2.13 | 4.91 |
| Antimony | 4 | 7.37 | 5.93 | 16 | 5.64 | -7.09 |
| Arsenic | 100 | 3.57 | 4.66 | 400 | 2.84 | 4.49 |
| Barium | 200 | 8.08 | 9.79 | 800 | 7.37 | 5.63 |
| Beryllium | 20 | 9.13 | 0.47 | 80 | 9.17 | -0.82 |
| Boron | 200 | 6.00 | 5.03 | 800 | 5.51 | 6.15 |
| Cadmium | 20 | 3.87 | 3.85 | 80 | 2.83 | 4.09 |
| Chromium | 20 | 4.37 | 4.50 | 80 | 3.58 | 3.80 |
| Cobalt | 20 | 4.64 | 6.05 | 80 | 3.92 | 5.38 |
| Copper | 20 | 3.66 | -0.04 | 80 | 2.86 | 0.19 |
| Iron | 2000 | 4.77 | 6.30 | 8000 | 3.57 | 5.72 |
| Lead | 20 | 4.10 | 9.43 | 80 | 2.84 | 5.29 |
| Lithium | 20 | 9.49 | 4.02 | 80 | 9.54 | 0.60 |
| Manganese | 200 | 9.37 | -3.53 | 800 | 8.24 | -3.94 |
| Molybdenum | 20 | 3.90 | 4.38 | 80 | 5.37 | 3.15 |
| Nickel | 20 | 9.14 | -2.07 | 80 | 7.81 | -1.88 |
| Phosphorus | 200 | 4.3 | 6.02 | 800 | 2.36 | 5.04 |
| Selenium | 30 | 6.70 | 1.32 | 120 | 5.67 | 0.47 |
| Strontium | 200 | 7.18 | 6.60 | 800 | 7.47 | 4.45 |
| Tellurium | 4 | 6.28 | 4.83 | 16 | 7.99 | -3.04 |
| Thallium | 20 | 6.27 | -2.35 | 80 | 5.95 | 8.79 |
| Titanium | 20 | 9.29 | -0.18 | 80 | 9.64 | -3.22 |
| Uranium | 20 | 6.57 | 9.78 | 80 | 5.24 | 1.61 |
| Vanadium | 20 | 9.09 | -3.08 | 80 | 8.68 | -3.36 |
| Zinc | 200 | 3.52 | 7.22 | 800 | 2.93 | 7.38 |

| Determinand | Spike Level | Treated sewage (Finham) | | Trade effluent (to controlled waters) (Trimpley) | | Trade effluent (to sewer) (Walkers) | | Untreated sewage (Finham) | |
|-------------|-------------|-------------------------|--------|--|--------|-------------------------------------|--------|---------------------------|--------|
| | | RSD % | Rec. % | RSD % | Rec. % | RSD % | Rec. % | RSD % | Rec. % |
| Aluminium | 80% | 3.71 | 101.21 | 3.94 | 106.19 | 4.16 | 107.00 | 3.71 | 101.21 |
| Antimony | 80% | 3.55 | 109.73 | 3.34 | 111.12 | 5.02 | 103.40 | 7.45 | 99.80 |
| Arsenic | 80% | 3.09 | 109.26 | 2.59 | 109.68 | 4.34 | 108.29 | 3.09 | 109.26 |
| Barium | 80% | 5.97 | 104.16 | 6.35 | 106.32 | 8.29 | 103.36 | 7.34 | 109.76 |
| Beryllium | 80% | 9.98 | 100.93 | 9.10 | 98.96 | 9.23 | 102.31 | 9.40 | 101.94 |
| Boron | 80% | 5.97 | 107.96 | 6.21 | 109.16 | 6.24 | 109.30 | 5.72 | 104.94 |
| Cadmium | 80% | 3.38 | 103.60 | 3.01 | 106.47 | 4.61 | 103.48 | 3.18 | 103.69 |
| Chromium | 80% | 3.67 | 105.48 | 4.16 | 105.84 | 4.76 | 105.23 | 4.34 | 105.89 |



METHOD STATEMENT



| Determinand | Spike Level | Treated sewage (Finham) | | Trade effluent (to controlled waters) (Trimpley) | | Trade effluent (to sewer) (Walkers) | | Untreated sewage (Finham) | |
|-------------|-------------|-------------------------|--------|--|--------|-------------------------------------|--------|---------------------------|--------|
| | | | | | | | | | |
| Cobalt | 80% | 3.66 | 104.97 | 4.39 | 106.64 | 4.41 | 104.66 | 4.70 | 105.68 |
| Copper | 80% | 3.43 | 92.90 | 4.13 | 97.27 | 3.98 | 94.46 | 3.72 | 95.73 |
| Iron | 80% | 3.27 | 106.73 | 3.79 | 105.58 | 4.88 | 100.94 | 3.93 | 102.09 |
| Lead | 80% | 3.45 | 99.18 | 3.38 | 104.87 | 4.98 | 95.83 | 3.44 | 98.48 |
| Lithium | 80% | 8.97 | 98.05 | 9.09 | 99.45 | 6.87 | 94.47 | 8.03 | 99.02 |
| Manganese | 80% | 9.74 | 97.12 | 9.96 | 97.22 | 7.29 | 99.58 | 7.36 | 97.96 |
| Molybdenum | 80% | 3.89 | 111.66 | 3.78 | 106.48 | 5.46 | 102.56 | 5.84 | 106.94 |
| Nickel | 80% | 7.56 | 96.64 | 8.81 | 94.06 | 7.74 | 96.96 | 6.12 | 96.64 |
| Phosphorus | 80% | 4.03 | 109.44 | 3.58 | 109.84 | 3.83 | 103.10 | 2.59 | 103.37 |
| Selenium | 80% | 6.78 | 101.93 | 6.39 | 99.71 | 7.60 | 107.43 | 4.29 | 102.65 |
| Strontium | 80% | 9.20 | 104.57 | 9.09 | 105.34 | 6.31 | 109.51 | 6.98 | 108.09 |
| Tellurium | 80% | 7.74 | 90.64 | 7.26 | 94.24 | 6.76 | 90.99 | 7.25 | 95.78 |
| Thallium | 80% | 8.01 | 99.81 | 8.61 | 107.32 | 5.03 | 98.97 | 9.69 | 98.73 |
| Titanium | 80% | 9.33 | 103.61 | 9.44 | 96.92 | 6.81 | 103.34 | 9.15 | 96.79 |
| Uranium | 80% | 7.04 | 106.41 | 5.99 | 106.54 | 5.53 | 94.63 | 7.50 | 101.07 |
| Vanadium | 80% | 9.20 | 104.36 | 9.70 | 98.23 | 6.97 | 106.43 | 7.78 | 103.47 |
| Zinc | 80% | 3.49 | 102.93 | 3.25 | 107.90 | 3.54 | 104.63 | 3.49 | 101.91 |

| Determinand | Ground water | | Land leachate | | Prepared leachate | | Surface water | |
|-------------|--------------|--------|---------------|--------|-------------------|--------|---------------|--------|
| | RSD % | Rec. % | RSD % | Rec. % | RSD %. | Rec. % | RSD %. | Rec. % |
| NexION | | | | | | | | |
| Aluminium | 7.60 | 102.24 | 8.83 | 93.86 | 9.02 | 98.48 | 9.73 | 101.83 |
| Antimony | 7.07 | 92.64 | 5.18 | 98.99 | 6.32 | 97.66 | 5.63 | 95.38 |
| Arsenic | 8.35 | 96.58 | 8.07 | 101.04 | 8.43 | 98.33 | 8.69 | 100.63 |
| Barium | 5.25 | 104.69 | 5.97 | 100.81 | 6.88 | 107.32 | 6.32 | 105.21 |
| Beryllium | 8.90 | 99.12 | 9.67 | 102.58 | 7.72 | 99.59 | 9.22 | 101.38 |
| Boron | 6.52 | 104.36 | 5.19 | 108.88 | 6.63 | 105.58 | 5.55 | 107.36 |
| Cadmium | 4.25 | 100.56 | 4.40 | 99.53 | 3.94 | 101.00 | 4.33 | 99.98 |
| Chromium | 6.65 | 99.04 | 8.60 | 100.04 | 7.61 | 99.74 | 9.36 | 99.11 |
| Cobalt | 5.97 | 96.44 | 7.63 | 96.79 | 6.87 | 98.56 | 7.94 | 96.42 |
| Copper | 5.99 | 91.35 | 7.70 | 96.22 | 6.07 | 90.79 | 6.24 | 94.37 |
| Iron | 8.19 | 99.37 | 7.65 | 97.15 | 6.98 | 98.06 | 7.84 | 97.06 |
| Lead | 4.98 | 98.64 | 4.49 | 91.94 | 5.80 | 101.85 | 5.56 | 93.11 |
| Lithium | 7.77 | 97.83 | 8.58 | 100.69 | 7.93 | 98.91 | 6.97 | 98.61 |
| Manganese | 7.44 | 96.71 | 9.87 | 93.89 | 7.79 | 97.52 | 9.69 | 95.82 |
| Molybdenum | 4.97 | 95.30 | 4.40 | 107.63 | 4.35 | 105.78 | 4.27 | 108.28 |
| Nickel | 5.83 | 94.37 | 8.07 | 94.18 | 6.98 | 98.08 | 8.63 | 94.06 |
| Phosphorus | 4.52 | 106.44 | 4.07 | 109.43 | 4.19 | 105.76 | 3.64 | 110.58 |
| Selenium | 5.73 | 95.24 | 6.35 | 101.06 | 5.29 | 101.47 | 105.46 | 102.67 |
| Strontium | 6.25 | 103.94 | 8.19 | 100.78 | 6.13 | 107.12 | 9.11 | 103.99 |
| Tellurium | 8.46 | 90.68 | 9.14 | 90.64 | 8.50 | 94.63 | 9.75 | 90.99 |
| Thallium | 5.94 | 106.20 | 8.16 | 99.35 | 4.09 | 109.11 | 9.67 | 101.38 |
| Titanium | 8.86 | 98.40 | 9.26 | 101.28 | 8.43 | 99.48 | 9.72 | 99.33 |
| Uranium | 5.18 | 104.51 | 5.27 | 102.07 | 5.72 | 104.30 | 6.09 | 103.43 |
| Vanadium | 6.78 | 99.58 | 8.78 | 103.29 | 7.44 | 98.67 | 9.18 | 100.55 |
| Zinc | 5.59 | 98.28 | 8.38 | 94.90 | 7.42 | 100.99 | 8.01 | 95.56 |



METHOD STATEMENT



Uncertainty of Measurement

Perkin Elmer NexION 300D ICP-MS

The reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2, which gives a level of confidence of approximately 95%.

| Determinand (total) | Uncertainty of Measurement (%) |
|---------------------|--------------------------------|
| Aluminium | 15.88 |
| Arsenic | 19.59 |
| Boron | 17.40 |
| Barium | 20.69 |
| Beryllium | 21.96 |
| Boron | 17.59 |
| Cadmium | 11.05 |
| Cobalt | 13.17 |
| Chromium | 13.79 |
| Copper | 13.46 |
| Iron | 12.49 |
| Manganese | 22.11 |
| Molybdenum | 20.83 |
| Nickel | 19.21 |
| Phosphorus | 18.32 |
| Lead | 11.63 |
| Lithium | 18.37 |
| Antimony | 18.43 |
| Selenium | 16.69 |
| Strontium | 21.27 |
| Tellurium | 13.40 |
| Titanium | 22.87 |
| Thallium | 19.09 |
| Uranium | 16.33 |
| Vanadium | 21.11 |
| Zinc | 12.41 |

| Determinand | Range of Application (µg/l) | LOD (µg/l) | Routine Reporting Level (µg/l) |
|-------------|-----------------------------|------------|--------------------------------|
| Aluminium | 15 - 1000 | 6.2427 | 15 |
| Antimony | 1.2 - 20 | 0.1588 | 1.2 |
| Arsenic | 1.0 - 500 | 0.1531 | 1.0 |
| Barium | 2.8 - 1000 | 0.1187 | 2.8 |
| Beryllium | 0.1 - 100 | 0.0225 | 0.1 |
| Boron | 19 - 1000 | 2.5740 | 19 |
| Cadmium | 0.07 - 100 | 0.0094 | 0.07 |
| Chromium | 0.8 - 100 | 0.0326 | 0.8 |
| Cobalt | 1.6 - 100 | 0.0239 | 1.6 |
| Copper | 1.9 - 100 | 0.5029 | 1.9 |
| Iron | 120 - 10000 | 21.7249 | 120 |
| Lead | 0.5 - 100 | 0.0366 | 0.5 |
| Nickel | 1.7 - 100 | 0.1386 | 1.7 |
| Lithium | 0.2 - 100 | 0.0275 | 0.2 |



METHOD STATEMENT



| Determinand | Range of Application (µg/l) | LOD (µg/l) | Routine Reporting Level (µg/l) |
|-------------|-----------------------------|------------|--------------------------------|
| Phosphorus | 26 – 1000 | 23.2069 | 26 |
| Selenium | 0.8 - 150 | 0.0773 | 0.8 |
| Tellurium | 0.38 - 20 | 0.0438 | 0.38 |
| Uranium | 0.31 – 30 | 0.0792 | 0.31 |
| Vanadium | 0.9 - 100 | 0.0200 | 0.9 |
| Zinc | 14 - 1000 | 1.6319 | 14 |

Filtered Metals

| Determinand | Ground water | | Land leachate | |
|-------------|--------------|--------|---------------|--------|
| | RSD % | Rec. % | RSD % | Rec. % |
| Elan | | | | |
| Aluminium | 5.14 | 97.43 | 5.69 | 93.74 |
| Antimony | 2.8 | 99.61 | 1.90 | 103.01 |
| Arsenic | 3.37 | 97.71 | 4.91 | 98.95 |
| Barium | 3.47 | 99.97 | 2.58 | 101.44 |
| Beryllium | 4.84 | 99.61 | 4.61 | 99.33 |
| Boron | 5.15 | 100.37 | 4.78 | 100.56 |
| Cadmium | 2.09 | 100.18 | 1.59 | 101.68 |
| Chromium | 4.15 | 96.55 | 3.24 | 96.29 |
| Cobalt | 3.73 | 94.02 | 3.72 | 96.03 |
| Copper | 3.18 | 98.84 | 4.05 | 103.58 |
| Iron | 6.22 | 96.64 | 3.26 | 95.90 |
| Lead | 2.52 | 102.28 | 2.07 | 103.69 |
| Lithium | 4.84 | 97.43 | 4.62 | 97.70 |
| Nickel | 3.53 | 92.30 | 3.60 | 95.23 |
| Phosphorus | 4.22 | 101.02 | 4.50 | 100.48 |
| Selenium | 2.87 | 97.67 | 2.76 | 101.44 |
| Tellurium | 2.60 | 90.30 | 2.10 | 97.31 |
| Uranium | 2.92 | 104.52 | 2.81 | 103.57 |
| Vanadium | 4.03 | 99.48 | 2.87 | 99.24 |
| Zinc | 7.04 | 103.02 | 7.26 | 107.19 |

Uncertainty of Measurement

| Determinand (Filtered) | Uncertainty of Measurement (%) |
|------------------------|--------------------------------|
| Aluminium | 18.09 |
| Arsenic | 10.66 |
| Boron | 15.85 |
| Barium | 9.29 |
| Beryllium | 14.31 |
| Cadmium | 6.81 |
| Cobalt | 16.14 |
| Chromium | 11.83 |
| Copper | 12.35 |
| Iron | 13.47 |
| Nickel | 20.18 |
| Phosphorus | 18.86 |
| Lead | 9.48 |



METHOD STATEMENT



| Determinand (Filtered) | Uncertainty of Measurement (%) |
|------------------------|--------------------------------|
| Lithium | 14.10 |
| Antimony | 6.82 |
| Selenium | 10.47 |
| Tellurium | 17.01 |
| Uranium | 11.72 |
| Vanadium | 10.14 |
| Zinc | 21.58 |

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