# METHOD STATEMENT

**Determinand:**
Chloridazon, Metamitron, Simazine, Bentazone, Metribuzin, Atrazine, Isoproturon, Diuron, Quinoxyfen, Dichlorprop-p, Mecoprop, Pentachlorophenol, PFOS (Heptadecafluorooctanesulfonic acid), PFOA (Perfluoroctanoic acid) and DEHP (Diethylhexyl phthalate), Metsulfuron-methyl and Sulfosulfuron (screen only)

**Matrix:**
Surface, Ground and Saline waters

**Principle of Method:**
The compounds of interest are extracted from an aqueous matrix via online SPE, utilising Thermo Scientific's EQuan LC system, equipped with a Hypersil Gold aQ pre-concentration column. The compounds are then backflushed from the pre-concentration column via a gradient run and are quantified by high resolution, accurate mass (HRAM) liquid chromatography mass spectrometry (LC-MS).

**Sampling and Sample Preparation:**
Samples should be taken in an STL101 bottle. No preservative is required.
Samples are stored between 5 ± 3°C prior to analysis.

Samples are stable for times stated below, from sampling

| | |
|---|---|
| Quinoxyfen | 11 Days (in-house data) |
| DEHP | 14 Days (in-house data) |
| PFOA, PFOS, Sulfosulfuron and Pentachlorophenol | 26 Days (in-house data) |
| Chloridazon, Metamitron, Simazine, Bentazone, Metribuzin, Atrazine, Isoproturon, Diuron, Dichlorprop-p, Mecoprop and Metsulfuron-methyl | 49 Days (in-house data) |

**Interferences:**
The LC-MS system operates at a mass spectral resolution of 70,000 FWHM and therefore the technique is extremely selective, however in theory any substance with an equivalent LC retention time, and which generates ions within 5ppm of the analytes' monoisotopic mass, may interfere.

**Performance of Method:**

## Precision, Bias and Limit of Detection

| Determinand | LOD µg/l | LOQ µg/l | MRL µg/l | Low Std | | High Std | |
|---|---|---|---|---|---|---|---|
| | | | | %RSD | %Bias | %RSD | %Bias |
| Atrazine | 0.000451 | 0.0009 | 0.02 | 6.13 | -2.51 | 3.44 | 1.37 |
| Dichlorprop-p | 0.00296 | 0.0059 | 0.02 | 3.45 | -2.82 | 2.71 | 1.32 |
| Diuron | 0.000621 | 0.0012 | 0.004 | 6.88 | -3.56 | 3.03 | -0.61 |
| Isoproturon | 0.000665 | 0.0013 | 0.004 | 4.45 | -1.40 | 3.58 | 1.85 |
| Perfluorooctanoic acid | 0.0000516 | 0.00010 | 0.00013 | 4.77 | -1.68 | 4.92 | -0.15 |
| Perfluorooctanesulfonic acid | 0.0000632 | 0.00013 | 0.00013 | 9.61 | -3.16 | 4.77 | 1.38 |
| Quinoxyfen | 0.000759 | 0.0015 | 0.002 | 6.26 | -2.01 | 4.32 | 0.69 |
| Simazine | 0.000548 | 0.0011 | 0.002 | 8.15 | -4.85 | 4.15 | 1.16 |
| Metamitron | 0.00311 | 0.0062 | 0.02 | 11.40 | -3.06 | 6.69 | -0.27 |
| DEHP | 0.0305 | 0.061 | 0.2 | 5.71 | -2.27 | 4.63 | 1.75 |
| Bentazone | 0.00630 | 0.013 | 0.02 | 4.49 | -3.48 | 3.08 | -0.53 |
| Pentachlorophenol | 0.000790 | 0.0016 | 0.02 | 3.34 | -3.56 | 2.02 | -0.27 |
| Chloridazon | 0.00126 | 0.0025 | 0.004 | 4.31 | -1.49 | 3.59 | 1.35 |
| Metribuzin | 0.000912 | 0.0018 | 0.004 | 3.09 | -0.87 | 3.38 | 0.21 |
| Mecoprop-p | 0.00272 | 0.0054 | 0.02 | 4.46 | -3.04 | 2.87 | -0.30 |
| Sulfosulfuron | 0.00451 | 0.0090 | 0.02 | 7.83 | -3.27 | 11.11 | 4.15 |
| Metsulfuron-methyl | 0.00801 | 0.0160 | 0.02 | 9.12 | -0.05 | 7.13 | 1.70 |

## Matrix Spike Recoveries

| Determinand | Saline Water - Aberdaron | | Surface Water – Pocklington River, Upstream | | Ground Water – Huncote BH | |
|---|---|---|---|---|---|---|
| | %RSD | %Rec. | %RSD | %Rec. | %RSD | %Rec. |
| Atrazine | 4.95 | 102.21 | 7.32 | 111.94 | 5.48 | 100.45 |
| Dichlorprop-p | 7.50 | 95.62 | 3.23 | 104.63 | 2.47 | 101.76 |
| Diuron | 3.47 | 100.34 | 4.73 | 101.74 | 2.89 | 99.61 |
| Isoproturon | 3.69 | 101.34 | 5.03 | 103.5 | 3.34 | 101.84 |
| Perfluorooctanoic acid | 5.26 | 103.59 | 5.49 | 106.22 | 4.02 | 101.87 |
| Perfluorooctanesulfonic acid | 6.99 | 111.08 | 8.96 | 105.74 | 3.76 | 100.39 |
| Quinoxyfen | 4.77 | 104.59 | 4.48 | 103.85 | 4.47 | 99.31 |
| Simazine | 7.31 | 97.91 | 5.32 | 111.31 | 5.17 | 98.17 |
| Metamitron | 12.33 | 94.39 | 6.87 | 92.88 | 8.80 | 106.64 |
| DEHP | 4.70 | 101.63 | 4.09 | 99.65 | 4.64 | 101.74 |
| Bentazone | 3.12 | 98.59 | 3.98 | 102.23 | 3.42 | 99.02 |
| Pentachlorophenol | 2.64 | 100.98 | 2.32 | 100.75 | 3.45 | 101.27 |
| Chloridazon | 3.16 | 103.21 | 5.97 | 97.95 | 3.11 | 100.74 |
| Metribuzin | 4.48 | 102.33 | 4.65 | 105.34 | 3.41 | 100.68 |
| Mecoprop-p | 8.14 | 93.34 | 3.96 | 107.11 | 3.72 | 101.25 |
| Sulfosulfuron | 17.33 | 89.47 | 11.38 | 73.07 | 10.19 | 92.74 |
| Metsulfuron-methyl | 11.64 | 102.89 | 11.76 | 107.00 | 9.68 | 106.54 |

## Uncertainty of Measurement

The Uncertainty of Measurement has been calculated following the procedure given in GOP 5.4N.

| Determinand | Uncertainty of Measurement % | Minimum UoM (µg/L) |
|---|---|---|
| Atrazine | 25.96 | 0.00050 |
| Dichlorprop-p | 10.73 | 0.0016 |
| Diuron | 11.29 | 0.0004 |
| Isoproturon | 10.67 | 0.0004 |
| Perfluorooctanoic acid | 17.19 | 0.00007 |
| Perfluorooctanesulfonic acid | 24.42 | 0.00004 |
| Quinoxyfen | 15.77 | 0.0004 |
| Simazine | 24.87 | 0.0004 |
| Metamitron | 39.76 | 0.0015 |
| DEHP | 22.10 | 0.030 |
| Bentazone | 12.30 | 0.003 |
| Pentachlorophenol | 9.67 | 0.0005 |
| Chloridazon | 22.69 | 0.0006 |
| Metribuzin | 12.28 | 0.0009 |
| Mecoprop-p | 17.84 | 0.0016 |
| Sulfosulfuron | 30.19 | 0.0029 |
| Metsulfuron-methyl | 16.92 | 0.003 |

**References:**

In-house developed method