

## METHOD STATEMENT

### Determinand:

Determination of selected phenoxyalkanoic, benzonitrile and other compounds

### Matrix:

Sample Type: Treated, Ground and Raw Waters.

### Principle of Method:

The method is a direct aqueous injection procedure. Samples are analysed by high performance liquid chromatography using a triple quadrupole mass spectrometer as a detector. The aqueous sample is injected and the organic compounds are separated and then identified and quantified with mass spectrometric detection (MSD) in multiple reaction monitoring (MRM) mode. Quantitation is based on an internal standardisation procedure.

### Interferences:

HPLC-MS/MS is an extremely selective technique and interferences should only be encountered very rarely. Any interfering compounds would have to display the identical MRM transition at the same retention time, this is extremely unlikely in potable water samples. However, any compound, which passes through the extraction procedure, and has a similar liquid chromatographic retention time and mass spectrometric properties to the compound of interest, will cause interference. Samples containing high humic or fulvic loading have been demonstrated to not cause significant ion suppression for the compounds.

### Performance of Method:

#### Range of Application:

<u>Determinand</u>	<u>Operational Calibration Range</u>
ASULAM	LOD - 0.250 µg/l
BENAZOLIN	LOD - 0.250 µg/l
BENTAZONE	LOD - 0.250 µg/l
BROMOXYNIL	LOD - 0.250 µg/l
CLOPYRALID	LOD - 0.250 µg/l
2,4-D	LOD - 0.250 µg/l
2,4-DB	LOD - 0.250 µg/l
DICAMBA	LOD - 0.250 µg/l
2,4-DP (DICHLORPROP)	LOD - 0.250 µg/l
FLUROXYPYR	LOD - 0.250 µg/l
IMAZAPYR	LOD - 0.250 µg/l
IOXYNIL	LOD - 0.250 µg/l
MCPA	LOD - 0.250 µg/l
MCPB	LOD - 0.250 µg/l
MCPP (MECOPROP)	LOD - 0.250 µg/l
PICLORAM	LOD - 0.250 µg/l
PENTACHLOROPHENOL	LOD - 0.250 µg/l
PROPAMOCARB	LOD - 0.250 µg/l
2,4,5-T	LOD - 0.250 µg/l
2,3,6-TRICHLOROBENZOIC ACID	LOD - 0.250 µg/l
2,4,5-TP (FENOPROP)	LOD - 0.250 µg/l
TRICLOPYR	LOD - 0.250 µg/l



# METHOD STATEMENT



## Limit of Detection, Uncertainty of measurement and Recoveries of Compounds

Instrument Q1.

Determinand	LOD ng/litre	Direct Standards				Elvington (Hard Hardness)	
		Low Standard		High Standard		PCV Spike	
		Mean	%RSD	Mean	%RSD	%Recovery	%RSD
2,3,6-TrichloroBenzoic Acid	3	48.9	6.0%	201.6	4.0%	99.9%	3.8%
2,4,5-T	5	50.6	8.3%	201.7	6.4%	101.6%	5.6%
2,4,5-TP (Fenoprop)	6	50.9	6.3%	201.0	6.7%	99.9%	5.5%
2,4-D	3	50.2	9.3%	202.8	4.7%	99.5%	5.6%
2,4-DB	6	49.0	8.7%	196.1	6.0%	98.7%	4.7%
2,4-DP (Dichloroprop)	5	51.3	9.0%	198.6	5.6%	101.6%	6.5%
Asulam	2	49.6	4.3%	200.7	1.9%	100.1%	2.7%
Benazolin	3	50.3	7.8%	204.8	4.4%	99.9%	6.3%
Bentazone	2	49.2	3.3%	200.6	1.3%	99.8%	2.7%
Bromoxynil	3	49.9	7.5%	206.9	4.5%	105.1%	5.9%
Clopyralid	5	49.1	7.2%	200.7	6.3%	100.4%	4.3%
Dicamba	4	49.5	5.0%	203.5	3.4%	100.2%	7.2%
Fluroxypyr	3	50.5	8.2%	202.0	4.8%	100.1%	5.2%
Imazapyr	3	49.7	5.8%	200.6	4.6%	107.8%	6.1%
Ioxynil	3	50.3	6.3%	202.0	5.4%	102.9%	4.9%
MCPA	4	49.4	6.9%	201.3	5.1%	99.5%	5.5%
MCPB	4	51.0	6.0%	199.7	5.9%	101.1%	5.7%
MCPP	3	50.2	6.8%	201.0	4.3%	100.0%	6.6%
PCP	6	49.3	9.8%	199.3	9.2%	99.6%	7.9%
Picloram	6	50.4	5.1%	205.5	3.5%	110.1%	4.0%
Propamocarb	2	48.8	4.4%	201.2	2.2%	99.2%	2.8%
Triclopyr	9	50.6	12.3%	199.8	8.6%	98.2%	8.4%

Instrument Q2

Determinand	LOD ng/litre	Direct Standards				Elvington (Hard Hardness)	
		Low Standard		High Standard		PCV Spike	
		Mean	%RSD	Mean	%RSD	%Recovery	%RSD
2,3,6-Trichlorobenzoic acid	2	49.9	4.8	200.8	4.9%	98.0%	4.0%
2,4,5-T	4	51.9	6.2	199.7	4.0%	101.9%	4.4%
2,4,5-TP (Fenoprop)	3	51.7	6.5	198.5	3.6%	101.3%	4.8%
2,4-D	4	50.8	4.5	199.3	5.2%	99.3%	4.5%
2,4-DB	4	51.9	7.1	199.9	5.0%	98.7%	6.3%
2,4-DP (Dichloroprop)	4	51.5	6.1	198.5	3.8%	101.0%	5.0%
Asulam	3	50.4	3.0	200.4	2.8%	99.7%	3.5%
Benazolin	3	51.7	4.7	201.0	5.2%	98.6%	4.7%
Bentazone	1	49.1	1.6	200.7	1.3%	98.1%	1.4%
Bromoxynil	3	50.4	4.4	200.4	5.5%	102.5%	5.7%
Clopyralid	3	51.5	4.6	200.2	4.7%	102.7%	5.0%
Dicamba	3	50.3	4.3	199.7	3.4%	99.9%	3.5%
Fluroxypyr	3	51.2	5.0	201.6	4.6%	98.6%	4.8%
Imazapyr	2	49.1	4.7	196.9	4.4%	106.3%	6.0%
Ioxynil	3	49.5	3.8	196.4	4.6%	100.5%	4.9%
MCPA	3	51.4	5.4	198.9	4.1%	100.1%	4.5%
MCPB	6	51.1	6.0	200.6	4.0%	101.7%	4.7%
MCPP (Mecoprop)	2	51.1	6.1	199.4	4.4%	101.8%	5.1%
Pentachlorophenol	5	49.8	6.8	194.2	5.3%	98.3%	5.6%
Picloram	4	50.8	4.8	199.2	4.1%	119.5%	5.4%
Propamocarb	2	50.5	4.5	201.6	1.8%	101.6%	3.2%
Triclopyr	3	50.2	7.4	199.5	5.1%	100.4%	3.6%



## METHOD STATEMENT



### References:

Agilent 1200 Series, Reference Manuals.

Agilent 6400 QQQ LC/MS Techniques and Operation, Agilent Technologies Course Number R1893A, Student Manuals Volumes 1 and 2.

Agilent 6460 Triple Quad LC/MS System, Quick Start Guide

Agilent 6400 Triple Quad LC/MS, Maintenance and Familiarization Guides.

Agilent 6400 Triple Quad LC/MS System, Concept Guide.

