

METHOD STATEMENT



Determinand:

Determination of organochlorine pesticides, synthetic pyrethroids, and selected non-polar organic compounds.

Matrix:

Sample Type: Raw and potable waters.

Principle of Method:

Approximately 200mL of sample is extracted with 5.0+0.2 mL of hexane/ethyl acetate (in a 1:1 ratio mix). The extract is then analysed by gas chromatography using triple quad mass spectrometric detection in MRM mode.

Sampling and Sample Preparation:

Samples are taken in 500mL amber or green glass bottles containing 0.9mL of sodium thiosulfate, 10g L⁻¹, as preservative and are stored at 1-5°C on receipt at the laboratory. Samples should be extracted (or solvent added) within the number of days from sampling as stated in the stability data given below:

Compound	Stability - Days
Aldrin	20
Chlorbufam	21
Chlorothalonil	20
Chlorpropham	21
Chlorpyrifos-ethyl	28
Chlorpyrifos-methyl	28
Chlorthal-dimethyl	21
Clomazone	21
Cypermethrin	20
Deltamethrin	20
Diazinon	21
Dichlobenil	20
Dieldrin	20
Diflufenican	21
Ethofumesate	28
Ethoprophos	21
Fenpropimorph	21
alpha-HCH	20
beta-HCH	20
gamma-HCH	20
Heptachlor	10
cis-Heptachlor Epoxide	20
trans-Heptachlor Epoxide	20
Hexachlorobenzene	20
lambda-Cyhalothrin	14
Methoxychlor	20
o,p'-DDE	20
o,p'-DDT	20
o,p'-TDE	20
Parathion-ethyl	21



Compound	Stability - Days
Pendimethalin	28
Pentachlorobenzene	21
cis-Permethrin	20
trans-Permethrin	20
Picolinafen	28
Pirimicarb	28
Pirimiphos-methyl	21
Propham	21
Propyzamide	21
p,p'-DDE	20
p,p'-DDT	20
p,p'-TDE	20
Tecnazene	20
Triallate	21
Trifluralin	20

If sample extracts cannot be analysed immediately after preparation, they are stored in a fridge at 5±3°C until analysis can be completed. If the samples show any signs of degradation from the AQC results or blank contamination, then the samples would be re-analysed or re-prepped where feasible.

Interferences

Any compound, which passes through the extraction procedure and that co-elutes with any of the analytes and produces a significant response to the relevant ions being monitored.

Performance of Method:

Range of Application:

The operational range is from the limit of detection to 150 ng L⁻¹. Samples producing results above this range should be diluted to bring the sample response below that of the top standard.

Limit of Quantification:

Limits of Quantification (LOQ) are given in µg L⁻¹, the highest LOQ has been applied across all instruments.

<i>Determinand</i>	<i>AG2 Limit of Quantification (µg L⁻¹)</i>	<i>AG3 Limit of Quantification (µg L⁻¹)</i>	<i>AG5 Limit of Quantification (µg L⁻¹)</i>	<i>METHOD (Standardised) Limit of Quantification (µg L⁻¹)</i>
Aldrin	0.007	0.005	0.008	0.008
Chlorbufam	0.004	0.005	0.007	0.007
Chlorothalonil	0.006	0.003	0.005	0.006
Chlorpropham	0.005	0.004	0.003	0.005
Chlorpyrifos-ethyl	0.007	0.008	0.007	0.008
Chlorpyrifos-methyl	0.006	0.004	0.008	0.008
Chlorthal-dimethyl	0.007	0.008	0.008	0.008
Clomazone	0.007	0.005	0.006	0.007
Cypermethrin	0.005	0.004	0.008	0.008

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<i>Determinand</i>	<i>AG2 Limit of Quantification (μg L^{-1})</i>	<i>AG3 Limit of Quantification (μg L^{-1})</i>	<i>AG5 Limit of Quantification (μg L^{-1})</i>	<i>METHOD (Standardised) Limit of Quantification (μg L^{-1})</i>
op-DDE	0.003	0.005	0.005	0.005
pp-DDE	0.003	0.005	0.004	0.005
op-DDT	0.004	0.004	0.004	0.004
pp-DDT	0.004	0.004	0.004	0.004
Deltamethrin	0.005	0.009	0.006	0.009
Diazinon	0.007	0.003	0.005	0.007
Dichlobenil	0.003	0.004	0.004	0.004
Dieldrin	0.008	0.008	0.009	0.009
Diflufenican	0.009	0.009	0.006	0.009
Ethofumesate	0.007	0.008	0.007	0.008
Ethoprophos	0.008	0.006	0.006	0.008
Fenpropimorph	0.010	0.008	0.007	0.010
alpha-HCH	0.005	0.004	0.006	0.006
gamma-HCH	0.005	0.004	0.003	0.005
Heptachlor	0.005	0.004	0.003	0.005
cis-Heptachlor Epoxide	0.007	0.006	0.005	0.007
trans-Heptachlor Epoxide	0.007	0.005	0.007	0.007
Hexachlorobenzene	0.005	0.005	0.005	0.005
Lambda-Cyhalothrin	0.006	0.005	0.006	0.006
Methoxychlor	0.006	0.004	0.004	0.006
Parathion	0.006	0.010	0.012	0.012
Pendimethalin	0.009	0.009	0.009	0.009
Pentachlorobenzene	0.006	0.003	0.004	0.006
cis-Permethrin	0.004	0.005	0.006	0.006
trans-Permethrin	0.004	0.005	0.004	0.005
Picolinafen	0.009	0.007	0.006	0.009
Pirimicarb	0.014	0.010	0.016	0.016
Pirimiphos-methyl	0.008	0.005	0.005	0.008
Propham	0.004	0.004	0.005	0.005
Propyzamide	0.009	0.008	0.007	0.009
op-TDE	0.004	0.004	0.005	0.005
pp-TDE	0.003	0.003	0.005	0.005
Tecnazene	0.008	0.005	0.004	0.008
Triallate	0.006	0.004	0.006	0.006
Trifluralin	0.007	0.005	0.004	0.007

Recoveries of Compounds and Uncertainty of measurement:

Instrument, WAKGCQQQ02, AG2:

Determinand	Low Standard 30ng/L Spike		High Standard 100ng/L Spike		Elvington Treated Water (Hard Water) PCV Spike		Uncertainty
	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD	
Aldrin	98.78%	4.78%	101.20%	7.37%	99.39%	3.35%	± 9.86 %
Chlorbufam	97.72%	6.79%	98.28%	4.17%	95.69%	4.13%	± 11.67 %

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Determinand	Low Standard 30ng/L Spike		High Standard 100ng/L Spike		Elvington Treated Water (Hard Water) PCV Spike		Uncertainty
	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD	
Chlorothalonil	92.46%	6.06%	99.44%	6.72%	106.90%	7.20%	± 19.08 %
Chlorpropham	97.72%	5.75%	100.69%	3.14%	99.65%	3.51%	± 7.71 %
Chlorpyrifos-ethyl	99.39%	6.79%	100.24%	5.35%	97.73%	3.17%	± 12.62 %
Chlorpyrifos-methyl	97.81%	5.30%	100.19%	3.03%	100.38%	3.82%	± 9.98 %
Chlorthal-dimethyl	97.67%	2.82%	100.36%	8.24%	98.91%	2.61%	± 9.20 %
Clomazone	96.00%	7.55%	98.47%	2.82%	95.48%	2.64%	± 12.40 %
Cypermethrin	96.18%	3.67%	100.64%	6.56%	97.55%	2.94%	± 13.29 %
Deltamethrin	97.12%	4.60%	101.00%	5.21%	98.83%	5.09%	± 10.07 %
Diazinon	97.34%	4.61%	101.18%	7.69%	99.83%	2.86%	± 8.90 %
Dichlobenil	106.86%	7.13%	102.64%	6.40%	99.61%	5.95%	± 11.41 %
Dieldrin	95.65%	3.69%	100.30%	10.61%	95.66%	4.95%	± 11.29 %
Diflufenican	96.16%	4.04%	99.82%	6.96%	98.23%	3.00%	± 10.84 %
Ethofumesate	98.30%	7.74%	99.76%	6.26%	99.78%	6.26%	± 15.67 %
Ethoprophos	98.02%	6.79%	95.40%	5.05%	91.64%	4.09%	± 17.72 %
Fenpropimorph	99.24%	6.52%	101.01%	5.11%	98.58%	4.62%	± 19.90 %
HCH-alpha	105.25%	2.68%	102.68%	4.78%	100.72%	2.06%	± 8.28 %
HCH-gamma	101.02%	2.75%	101.87%	4.56%	99.98%	2.05%	± 7.26 %
Heptachlor	93.87%	7.23%	99.75%	6.05%	100.98%	4.75%	± 9.68 %
cis-Heptachlor Epoxide	96.76%	4.34%	99.60%	9.06%	98.59%	3.83%	± 9.44 %
trans-Heptachlor Epoxide	99.69%	3.46%	100.35%	7.44%	99.27%	3.16%	± 12.41 %
Hexachlorobenzene	105.42%	3.44%	103.20%	5.23%	100.98%	3.00%	± 8.54 %
lambda-Cyhalothrin	99.19%	9.91%	102.63%	11.81%	102.04%	9.67%	± 13.73 %
Methoxychlor	98.83%	12.73%	101.10%	8.06%	93.51%	4.71%	± 22.74 %
op-DDE	99.92%	2.34%	101.37%	5.33%	99.71%	1.18%	± 6.25 %
op-DDT	96.06%	1.66%	99.94%	5.99%	98.76%	1.92%	± 9.00 %
op-TDE	99.19%	3.74%	100.56%	7.40%	95.12%	2.77%	± 13.17 %
Parathion-ethyl	98.34%	7.45%	99.59%	6.51%	98.17%	3.63%	± 10.99 %
Pendimethalin	100.16%	11.89%	98.88%	5.06%	100.38%	6.33%	± 16.08 %
Pentachlorobenzene	104.83%	5.88%	102.72%	5.71%	100.47%	4.60%	± 9.78 %
cis-Permethrin	96.89%	4.87%	100.74%	6.79%	99.14%	3.30%	± 9.73 %
trans-Permethrin	96.68%	4.74%	100.72%	6.39%	98.40%	3.32%	± 9.73 %
Picolinafen	97.88%	6.14%	99.46%	3.13%	99.29%	4.50%	± 10.54 %
Pirimicarb	99.83%	9.16%	100.04%	6.09%	100.21%	9.52%	± 21.71 %
Pirimiphos-methyl	94.46%	6.09%	97.66%	4.29%	96.15%	4.17%	± 12.89 %
pp-DDE	98.61%	1.60%	100.93%	5.86%	98.73%	4.84%	± 7.22 %
pp-DDT	94.69%	5.89%	99.19%	6.12%	99.71%	2.89%	± 10.26 %
pp-TDE	95.27%	6.22%	100.33%	7.48%	94.27%	4.53%	± 17.92 %
Propham	100.14%	4.26%	100.24%	3.19%	98.99%	2.80%	± 7.89 %
Propyzamide	96.31%	6.84%	96.98%	4.37%	93.90%	4.96%	± 15.46 %
Tecnazene	103.03%	3.62%	100.54%	6.28%	98.25%	6.42%	± 9.57 %
Triallate	95.01%	3.40%	99.53%	8.71%	97.89%	3.49%	± 10.42 %
Trifluralin	95.55%	2.73%	100.38%	6.06%	99.65%	2.34%	± 8.79 %

METHOD STATEMENT



Instrument, WAKGCQQQ03, AG3:

Determinand	Low Standard 30ng/L Spike		High Standard 100ng/L Spike		Elvington Treated Water (Hard Water) PCV Spike		Uncertainty
	% Recovery	RSD	% Recovery	RSD	% Recovery	RSD	
Aldrin	101.56%	4.57%	101.84%	7.25%	98.01%	4.65%	± 20.90 %
Chlorbufam	96.90%	8.00%	98.67%	3.43%	96.03%	3.40%	± 14.74 %
Chlorothalonil	96.87%	7.89%	98.79%	8.16%	103.92%	10.55%	± 11.18 %
Chlorpropham	96.19%	5.80%	100.18%	3.75%	100.10%	2.27%	± 9.28 %
Chlorpyrifos-ethyl	96.05%	5.99%	97.28%	5.10%	98.74%	5.00%	± 14.91 %
Chlorpyrifos-methyl	98.88%	5.23%	100.45%	4.32%	99.66%	4.24%	± 10.16 %
Chlorthal-dimethyl	101.24%	8.47%	100.10%	8.41%	100.89%	8.07%	± 12.61 %
Clomazone	98.66%	8.12%	99.06%	4.36%	96.61%	4.49%	± 12.85 %
Cypermethrin	93.46%	4.11%	98.63%	3.04%	97.77%	2.45%	± 18.33 %
Deltamethrin	89.83%	4.96%	98.73%	3.95%	97.12%	2.42%	± 20.59 %
Diazinon	95.69%	6.41%	98.71%	2.71%	99.86%	3.53%	± 15.21 %
Dichlobenil	103.60%	9.74%	101.27%	6.56%	101.11%	7.40%	± 20.61 %
Dieldrin	98.86%	4.05%	101.99%	2.73%	99.08%	3.73%	± 15.81 %
Diflufenican	95.44%	2.80%	99.15%	1.95%	99.39%	2.52%	± 14.90 %
Ethofumesate	100.27%	8.97%	99.27%	3.57%	100.31%	6.07%	± 17.94 %
Ethoprophos	93.10%	7.74%	95.36%	5.12%	93.75%	6.55%	± 18.68 %
Fenpropimorph	96.85%	9.31%	100.70%	7.48%	99.93%	5.43%	± 13.44 %
HCH-alpha	103.97%	8.36%	102.45%	7.83%	102.87%	8.16%	± 17.01 %
HCH-gamma	100.78%	1.52%	100.46%	1.38%	99.97%	1.55%	± 12.31 %
Heptachlor	99.73%	8.49%	99.75%	6.53%	102.95%	9.03%	± 24.06 %
cis-Heptachlor Epoxide	97.29%	6.06%	99.74%	6.14%	97.45%	3.49%	± 15.58 %
trans-Heptachlor Epoxide	95.57%	5.23%	99.51%	4.99%	96.22%	4.62%	± 15.71 %
Hexachlorobenzene	105.49%	6.98%	103.45%	7.59%	102.34%	6.93%	± 14.55 %
lambda-Cyhalothrin	96.22%	8.42%	99.46%	5.70%	99.40%	11.14%	± 17.70 %
Methoxychlor	96.04%	7.02%	98.60%	6.13%	95.96%	5.62%	± 21.05 %
op-DDE	102.72%	2.81%	99.98%	5.53%	99.79%	2.77%	± 13.94 %
op-DDT	98.92%	1.93%	100.47%	1.87%	99.43%	2.04%	± 13.39 %
op-TDE	103.49%	2.10%	101.51%	2.16%	98.69%	2.36%	± 13.51 %
Parathion-ethyl	94.39%	5.21%	99.71%	2.91%	99.71%	3.24%	± 14.84 %
Pendimethalin	96.17%	7.76%	98.53%	5.61%	97.79%	4.62%	± 16.79 %
Pentachlorobenzene	102.58%	8.93%	103.27%	6.87%	102.35%	7.18%	± 20.66 %
cis-Permethrin	94.52%	6.88%	98.86%	4.20%	98.68%	3.64%	± 14.72 %
trans-Permethrin	93.68%	7.66%	99.34%	3.76%	99.84%	3.84%	± 17.87 %
Picolinafen	96.83%	6.25%	99.24%	4.18%	98.92%	6.18%	± 14.92 %
Pirimicarb	103.73%	9.65%	100.68%	9.84%	100.20%	6.73%	± 17.18 %
Pirimiphos-methyl	96.78%	6.57%	99.00%	3.77%	97.49%	3.87%	± 11.61 %
pp-DDE	101.37%	2.20%	100.79%	2.32%	99.83%	2.30%	± 13.05 %
pp-DDT	94.48%	2.59%	99.09%	1.57%	100.79%	2.39%	± 15.44 %

METHOD STATEMENT



Determinand	Low Standard 30ng/L Spike		High Standard 100ng/L Spike		Elvington Treated Water (Hard Water) PCV Spike		Uncertainty
	% Recovery	RSD	% Recovery	RSD	% Recovery	RSD	
pp-TDE	100.19%	4.24%	101.17%	3.47%	97.75%	3.77%	± 16.52 %
Propham	98.29%	5.48%	100.05%	2.68%	99.05%	3.76%	± 8.84 %
Propyzamide	99.51%	8.35%	97.88%	5.32%	94.69%	6.27%	± 16.86 %
Tecnazene	99.06%	8.11%	101.36%	6.56%	103.08%	7.70%	± 17.39 %
Triallate	97.83%	3.41%	100.46%	4.55%	102.22%	4.99%	± 12.65 %
Trifluralin	93.02%	2.99%	99.21%	2.90%	99.29%	2.41%	± 17.54 %

METHOD STATEMENT



Instrument, WAKGCQQQ05, AG5:

Determinand	Low Standard 30ng/L Spike		High Standard 100ng/L Spike		Elvington Treated Water (Hard Water) PCV Spike		Uncertainty
	% Recovery	RSD	% Recovery	RSD	% Recovery	RSD	
Aldrin	97.38%	4.92%	101.61%	2.89%	98.80%	4.55%	±15.75%
Chlorbufam	94.43%	6.92%	94.91%	4.25%	95.02%	5.12%	± 15.57%
Chlorothalonil	91.89%	7.86%	99.96%	3.42%	100.99%	2.69%	±17.50%
Chlorpropham	95.99%	5.16%	98.93%	4.41%	100.39%	4.08%	± 12.25%
Chlorpyrifos-ethyl	95.54%	8.74%	98.44%	3.34%	98.28%	3.91%	± 10.68%
Chlorpyrifos-methyl	95.18%	7.17%	99.46%	3.47%	97.71%	3.67%	± 10.32%
Chlorthal-dimethyl	99.16%	4.59%	100.87%	2.74%	101.40%	2.82%	±11.15%
Clomazone	97.63%	6.50%	97.31%	4.13%	98.03%	5.24%	± 12.27%
Cypermethrin	99.21%	8.30%	101.24%	3.46%	100.75%	3.32%	±9.17%
Deltamethrin	95.45%	5.65%	98.04%	3.18%	99.19%	2.68%	±13.23%
Dichlobenil	101.50%	2.60%	101.51%	3.74%	99.44%	4.22%	±10.54%
Diazinon	96.64%	2.98%	99.67%	2.56%	100.90%	3.46%	±8.26%
Dieldrin	100.04%	3.01%	101.19%	2.23%	99.80%	2.88%	±12.11%
Diflufenican	97.30%	2.61%	100.35%	1.62%	100.33%	2.77%	±8.49%
Ethofumesate	97.45%	6.17%	97.97%	4.30%	100.05%	4.97%	± 15.35%
Ethoprophos	92.63%	7.16%	94.29%	3.85%	92.97%	5.50%	± 19.47%
Fenpropi-morph	98.45%	7.78%	101.31%	3.50%	101.17%	4.40%	± 13.11%
HCH-alpha	101.14%	3.13%	101.90%	2.21%	101.84%	3.41%	±8.45%
HCH-gamma	100.44%	2.47%	102.12%	1.90%	101.26%	3.04%	±7.93%
Heptachlor	96.82%	3.81%	101.42%	1.83%	99.41%	3.21%	±9.51%
cis-Heptachlor Epoxide	101.31%	3.57%	101.66%	4.02%	101.75%	5.38%	±13.06%
trans-Heptachlor Epoxide	99.05%	4.31%	101.50%	3.53%	99.77%	5.66%	±12.45%
Hexachlorobenzene	101.50%	3.08%	103.64%	2.31%	102.60%	2.87%	±9.22%
lambda-Cyhalothrin	100.97%	8.97%	99.87%	4.53%	98.98%	4.82%	±14.79%
Methoxychlor	99.54%	6.59%	101.59%	2.89%	101.55%	2.41%	±8.57%
op-DDE	100.54%	2.76%	101.97%	2.66%	101.71%	3.63%	±8.17%
op-DDT	99.83%	1.89%	100.63%	1.22%	99.71%	2.64%	±7.73%
op-TDE	97.04%	3.00%	100.39%	1.91%	100.36%	3.24%	±9.72%
Parathion-ethyl	95.11%	7.35%	98.04%	3.48%	98.98%	3.41%	±12.02%
Pendimethalin	99.25%	8.35%	99.34%	5.28%	98.04%	6.81%	± 18.95%
Pentachlorobenzene	102.33%	2.72%	103.88%	3.45%	103.24%	3.03%	±10.78%
cis-Permethrin	98.82%	7.24%	102.24%	4.70%	101.36%	4.38%	±12.80%
trans-Permethrin	99.08%	5.01%	101.83%	3.35%	101.10%	3.93%	±12.09%
Picolinafen	97.02%	7.33%	99.95%	3.68%	99.25%	3.85%	± 10.56%
Pirimicarb	103.97%	9.39%	101.03%	5.33%	98.90%	4.81%	± 16.59%
Pirimiphos-methyl	94.38%	8.91%	97.82%	3.81%	98.42%	3.96%	± 12.26%
pp-DDE	98.45%	3.00%	100.42%	2.30%	100.89%	3.02%	±8.44%
pp-DDT	94.34%	2.64%	98.92%	2.00%	99.15%	2.69%	±12.51%
pp-TDE	96.95%	2.15%	100.05%	2.99%	99.00%	2.33%	±11.82%
Propham	98.68%	4.26%	99.68%	3.82%	100.15%	3.48%	± 11.74%

METHOD STATEMENT



Determinand	Low Standard 30ng/L Spike		High Standard 100ng/L Spike		Elvington Treated Water (Hard Water) PCV Spike		Uncertainty
	% Recovery	RSD	% Recovery	RSD	% Recovery	RSD	
Propyzamide	97.63%	7.77%	97.67%	4.59%	97.77%	5.82%	± 14.33%
Tecnazene	97.81%	2.69%	101.15%	2.60%	100.83%	3.23%	±10.69%
Triallate	97.99%	2.61%	100.66%	2.01%	101.65%	2.73%	±8.41%
Trifluralin	100.89%	6.12%	100.85%	1.92%	101.50%	2.65%	±7.28%

References:

In house method