

METHOD STATEMENT

Determinand:

This method covers the determination of a wide range of organochlorine, organophosphorous and organonitrogen compounds.

Matrix:

Ground waters, Surface waters, Treated Sewage effluents, Trade effluents and Landfill leachates.

Principle of Method:

A wide range of organic compounds are isolated from aqueous matrix using a two stage extraction technique. The compounds are determined by Electron impact GC-MSMS in selected reaction monitoring (SRM) mode using argon as a collision gas.

Sampling and Sample Preparation:

Samples should be collected in glass bottles with PTFE lined screw caps and be stored at $5\pm 3^{\circ}\text{C}$. Samples are stable for 7 days (Standard Methods:-ISBN 0-87553-161-X) from sampling.

Interferences:

GC-MS/MS is an extremely selective technique and interferences should only be encountered very rarely, however in theory, any compound which is extracted by the procedure, which has a GC retention time similar to the compound of interest and which produces both parent and daughter ions similar to that of the compounds in question, may interfere.

Performance of Method:

Range of Method: Typically 2ng/l - 200ng/l or 20ng/l - 1000ng/l without dilution (Dependant on Compound)

Compound	LOD ng/L	Low Standard		High Standard	
		% Bias	% RSD	% Bias	% RSD
1,3,5-Trichlorobenzene	1.125	-8.1	10.6	-2.3	6.2
124-Trichlorobenzene	1.44*	-4.7	10.8	-1.4	7.0
Hexachlorobutadiene	0.657	-7.8	11.8	-0.6	8.5
123-Trichlorobenzene	0.847	-6.8	10.5	-3.1	6.0
Dichlorvos	0.97*	-2.9	6.1	-3.1	5.5
EPTC	3.803	-1.6	7.0	2.1	5.5
Dichlobenil	0.820	0.2	4.5	3.5	3.2
Mevinphos	0.819	-9.6	11.4	-6.9	15.6
Pentachlorobenzene	1.001	-7.7	8.5	-1.5	6.9
Tecnazene	3.330	-2.1	3.1	0.7	2.5
Propachlor	2.924	-2.7	3.9	0.3	4.9
Trifluralin	3.583	-3.8	5.1	1.9	3.7
24D-Methyl	3.627	-2.0	7.4	-0.2	5.6
Chlorpropham	2.901	-1.3	3.4	0.3	2.3
Phorate	1.187	-5.8	5.7	-0.6	4.1
24D-Ethyl	3.871	-3.4	7.6	-0.3	5.0
24D-Isopropyl	4.081	-3.5	9.1	0.1	5.2
alpha-HCH	1.42*	-2.3	4.1	0.5	2.8
Hexachlorobenzene	1.01*	-1.3	7.4	-0.2	5.6
Propazine	3.315	-4.0	3.9	-0.9	4.7
Diazinon	2.31*	-0.5	7.8	3.6	6.2
Propetamphos	0.72*	-2.5	5.3	4.2	6.1
Atrazine	5.64*	-3.3	4.2	0.3	3.6
Simazine	7.05*	-1.3	7.0	2.3	4.7
Dimethoate	1.376	-2.1	9.5	-0.8	7.2
Trietazine	2.861	-3.0	8.1	-1.1	4.6
Propyzamide	5.280	-5.8	6.7	-1.3	4.6
beta-HCH	0.92*	-3.7	3.1	1.3	2.9
Triallate	4.095	-2.0	3.2	0.4	2.8



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Compound	LOD ng/L	Low Standard		High Standard	
		% Bias	% RSD	% Bias	% RSD
Pirimicarb	2.094	-3.6	3.7	0.2	3.9
24D-n-Butyl	4.561	-6.3	9.8	0.8	4.9
gamma-HCH	1.49*	-2.2	3.3	1.4	2.6
24D-Isobutyl	4.289	-3.5	8.0	-0.6	5.1
Alachlor	4.647	-3.3	4.9	0.4	2.7
PCB 28	0.838	-3.6	14.5	1.6	4.0
Fenpropidin	7.973	-3.0	11.5	-0.1	6.1
Prometryn	3.312	-2.5	8.3	-0.6	4.7
Pirimiphos-Methyl	0.883	-1.6	6.6	5.1	5.9
Ametryn	3.994	-3.8	6.1	0.6	4.2
Heptachlor	0.980	-7.9	7.9	0.6	7.0
Parathion-Methyl	0.749	-6.4	4.6	-1.6	4.7
Terbutryn	3.335	-3.4	8.0	-0.8	5.4
Malathion	0.954	-5.5	3.5	-0.8	3.4
Ethofumesate	3.303	-4.4	9.1	-0.1	5.7
Fenpropimorph	6.510	-1.5	6.5	1.8	5.3
PCB 52	0.693	0.4	7.3	2.9	5.0
Fenitrothion	0.875	-7.4	5.0	-0.8	4.4
Chlorpyrifos-Ethyl	0.627	-0.9	4.0	4.9	6.1
Aldrin	1.15*	3.1	16.0	6.4	8.3
Fenthion	0.956	-5.8	5.0	-0.7	5.2
Triademefon	3.544	-1.7	8.5	1.2	5.1
Parathion-Ethyl	0.839	-3.4	5.3	1.1	3.7
Cyanazine	7.519	-2.5	8.4	0.2	7.2
Pendimethalin	4.832	-9.8	12.5	-3.0	7.1
Isodrin	1.37*	9.5	18.2	9.0	10.0
Chlorfenvinphos	1.21*	-4.2	5.1	0.1	4.6
Heptachlor Epoxide	1.349	-3.1	5.7	1.9	3.6
op-DDE	0.76*	-1.0	7.8	0.3	7.9
PCB 101	0.833	0.0	6.5	2.0	7.0
trans-Chlordane	1.189	-3.1	7.2	0.6	6.5
cis-Chlordane	1.199	-4.6	6.8	-0.2	8.2
alpha-Endosulphan	1.73*	-1.3	7.4	0.6	7.1
Flutriafol	12.316	-0.8	8.0	-3.2	14.5
pp-DDE	0.91*	6.6	12.2	4.9	9.7
OP-TDE	0.849	2.7	9.3	-0.1	6.3
Dieldrin	1.67*	-3.1	6.7	0.9	4.8
Endrin	1.34*	-2.8	4.4	0.7	7.3
PCB 118	1.243	-0.1	6.5	2.3	5.8
op-DDT	0.95*	-1.0	6.2	3.1	5.2
PP-TDE	1.14*	2.4	10.4	-1.7	7.8
PCB 153	0.737	2.2	7.5	5.2	8.5
beta-Endosulphan	1.81*	1.7	7.1	0.8	7.1
Triazophos	0.79*	0.5	7.9	5.1	8.1
Carbophenothion	1.310	-2.5	10.1	1.1	7.2
PCB 138	0.905	3.8	8.5	5.5	9.1
Propiconazole	4.965	-3.7	8.9	0.1	6.7
pp-DDT	1.16*	-3.0	9.3	7.3	5.3
Diflufenican	8.946	-2.2	5.2	-0.5	6.2
Tebuconazole	10.476	-0.7	12.7	-1.1	9.4
PCB 180	1.153	2.7	7.1	2.1	5.8
Phosalone	1.463	-3.2	5.9	-0.3	5.7
Azinphos-Methyl	0.671	-6.7	5.5	0.0	5.1
Azinphos-Ethyl	0.839	1.3	9.3	5.1	7.6
cis-Permethrin	0.94*	-4.0	5.7	0.2	4.4
trans-Permethrin	0.75*	-4.1	5.3	-0.3	4.2
Coumaphos	0.854	-2.0	6.3	1.5	6.1
Cyfluthrin	0.91*	-5.5	4.0	0.8	4.0



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Compound	LOD ng/L	Low Standard		High Standard	
		% Bias	% RSD	% Bias	% RSD
Cypermethrin	1.48*	-4.6	0.0	0.0	4.0

* - From the MCERTS validation

Compound	Groundwater		Surface Water		Landfill Leachate		Trade Effluent	
	% Rec.	% RSD	% Rec.	% RSD	% Rec.	% RSD	% Rec.	% RSD
1,3,5-Trichlorobenzene	99.7	9.2	98.0	6.3	97.6	5.4	93.9	13.4
124-Trichlorobenzene	97.8	6.2	99.1	5.1	99.0	6.7	98.3	10.2
Hexachlorobutadiene	99.0	7.5	99.2	6.8	101.1	7.9	96.3	11.9
123-Trichlorobenzene	95.8	5.9	98.0	4.6	97.7	5.5	96.8	7.9
Dichlorvos	98.3	5.4	98.9	7.8	97.4	6.4	97.4	6.1
EPTC	102.5	10.2	98.0	6.1	97.8	5.0	97.2	7.9
Dichlobenil	101.8	4.3	101.5	4.8	101.3	3.5	99.4	4.7
Mevinphos	95.8	12.8	101.0	18.5	99.0	17.6	101.3	15.8
Pentachlorobenzene	95.6	8.5	99.9	6.6	100.6	5.9	98.9	7.1
Tecnazene	98.9	2.7	100.6	4.1	100.4	3.0	98.8	3.8
Propachlor	99.6	4.8	97.9	3.4	98.8	4.9	98.6	5.0
Trifluralin	99.6	3.1	98.4	3.8	99.6	3.0	96.1	5.0
24D-Methyl	99.5	6.3	95.4	6.1	97.2	5.6	99.7	7.1
Chlorpropham	98.3	2.4	100.5	4.1	100.5	3.0	102.3	2.3
Phorate	99.3	4.5	101.1	4.8	101.2	4.5	103.1	5.4
24D-Ethyl	99.8	6.0	91.7	6.0	96.4	5.5	100.4	8.9
24D-Isopropyl	99.4	4.5	96.3	6.7	98.3	5.1	99.3	6.6
alpha-HCH	99.5	3.2	100.6	4.2	100.1	2.8	99.6	3.9
Hexachlorobenzene	97.4	7.7	96.4	8.1	99.9	6.9	97.8	6.2
Propazine	98.2	4.2	98.3	3.9	98.6	4.8	99.4	5.4
Diazinon	102.8	6.3	101.5	6.1	101.1	4.6	101.0	7.4
Propetamphos	103.6	5.0	102.4	4.3	101.5	3.5	102.4	6.9
Atrazine	98.9	2.5	99.5	2.7	98.6	2.7	98.8	3.3
Simazine	101.4	4.4	102.5	5.4	101.8	5.3	100.7	5.5
Dimethoate	96.4	5.7	97.1	7.3	98.7	5.8	97.2	5.6
Trietazine	98.1	5.8	98.3	5.2	98.2	5.3	99.3	7.8
Propyzamide	98.7	5.4	99.8	5.1	99.6	5.0	100.8	6.7
beta-HCH	99.1	2.4	100.6	4.0	99.8	4.4	98.9	5.9
Triallate	97.7	4.3	100.1	4.3	100.1	3.3	99.6	3.4
Pirimicarb	100.3	2.8	100.2	4.0	101.6	4.2	102.2	3.3
24D-n-Butyl	99.1	4.9	82.1	14.5	97.3	8.6	98.5	8.3
gamma-HCH	99.5	2.2	101.9	3.9	100.8	2.9	102.2	4.0
24D-Isobutyl	97.9	5.3	81.9	13.3	96.4	8.1	95.6	9.3
Alachlor	99.0	3.2	100.7	4.0	100.4	3.1	99.3	7.3
PCB 28	98.0	5.1	94.5	6.3	99.6	3.6	96.1	5.7
Fenpropidin	99.8	4.2	99.2	5.6	105.2	5.8	108.4	7.8
Prometryn	98.1	4.3	99.2	4.5	99.1	4.5	100.1	7.0
Pirimiphos-Methyl	104.3	5.0	103.2	5.8	101.9	4.0	101.2	8.0
Ametryn	99.8	5.2	99.7	3.9	100.2	3.9	101.1	6.3
Heptachlor	97.3	8.9	94.3	8.0	100.2	7.0	94.8	8.6
Parathion-Methyl	99.0	4.7	102.1	4.5	102.4	4.9	107.1	6.8
Terbutryn	98.6	5.9	98.2	4.8	99.2	4.5	101.8	7.2
Malathion	100.3	4.4	99.7	4.6	101.5	3.3	104.5	4.8
Ethofumesate	98.8	5.3	98.5	4.8	98.8	4.4	100.6	7.0
Fenpropimorph	99.2	6.1	95.9	6.5	98.9	7.1	101.3	8.5



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Compound	Groundwater		Surface Water		Landfill Leachate		Trade Effluent	
	% Rec.	% RSD	% Rec.	% RSD	% Rec.	% RSD	% Rec.	% RSD
PCB 52	100.0	7.8	95.2	6.3	100.9	5.4	94.8	6.6
Fenitrothion	100.5	4.6	102.0	4.0	102.4	5.3	107.4	8.0
Chlorpyrifos-Ethyl	102.7	4.5	101.6	4.5	101.7	3.4	101.3	7.1
Aldrin	103.6	8.2	96.1	10.9	103.8	8.1	98.8	8.2
Fenthion	98.1	5.4	100.4	5.0	100.0	4.0	102.4	4.0
Triademefon	100.8	6.6	100.3	5.2	100.6	4.9	101.3	6.5
Parathion-Ethyl	101.0	3.8	101.0	3.5	100.1	3.9	99.3	5.6
Cyanazine	105.1	5.8	111.1	5.6	110.8	6.0	111.8	6.6
Pendimethalin	96.2	8.7	96.5	6.6	97.6	6.2	96.6	9.1
Isodrin	107.0	8.8	102.1	10.1	107.1	9.8	101.7	9.8
Chlorfenvinphos	99.7	3.6	101.6	5.2	102.9	3.0	106.4	4.3
Heptachlor Epoxide	99.3	5.0	102.0	4.6	101.2	4.2	100.9	3.9
op-DDE	96.7	9.3	91.3	10.2	98.2	7.5	94.0	8.4
PCB 101	97.6	8.9	91.3	10.1	98.9	6.8	94.0	9.5
trans-Chlordane	95.5	9.9	92.3	8.1	97.9	6.2	94.9	10.0
cis-Chlordane	95.6	9.9	93.2	10.5	98.7	7.3	95.6	8.8
alpha-Endosulphan	96.6	9.4	102.2	6.4	101.0	6.7	101.2	6.5
Flutriafol	104.0	7.0	109.0	6.7	108.7	7.5	113.8	8.2
pp-DDE	101.0	11.7	94.4	11.5	102.3	9.6	92.4	14.0
OP-TDE	96.3	8.6	96.0	9.2	98.7	7.6	103.6	9.1
Dieldrin	99.1	5.7	99.4	4.8	99.5	4.2	98.9	4.7
Endrin	98.9	4.7	100.3	5.5	101.7	5.5	100.4	5.9
PCB 118	97.0	9.5	90.2	8.0	98.7	6.4	93.7	8.1
op-DDT	102.4	4.4	98.9	6.0	100.6	7.3	95.6	6.6
PP-TDE	96.7	9.9	97.3	10.2	97.7	9.3	103.2	9.8
PCB 153	100.4	9.7	91.0	10.6	101.3	8.3	91.6	12.4
beta-Endosulphan	94.9	9.1	98.1	6.5	99.1	5.8	98.5	6.6
Triazophos	103.3	6.3	101.1	6.0	100.7	3.5	101.4	9.2
Carbophenothion	101.6	5.9	96.7	7.5	101.4	4.9	97.2	6.2
PCB 138	101.3	10.2	92.7	10.1	101.6	8.1	93.4	9.8
Propiconazole	101.7	7.3	99.2	5.1	102.4	4.9	104.6	8.5
pp-DDT	107.4	9.9	101.7	9.0	103.3	6.6	96.8	10.7
Diflufenican	96.5	7.5	99.5	5.8	99.5	5.6	98.5	4.6
Tebuconazole	103.2	7.4	100.7	6.0	107.3	7.0	113.0	9.9
PCB 180	98.9	8.3	88.9	9.4	97.4	5.9	91.3	11.4
Phosalone	100.3	6.2	100.2	6.0	101.2	5.0	101.1	6.2
Azinphos-Methyl	99.9	4.7	112.6	6.8	105.8	4.6	108.3	6.7
Azinphos-Ethyl	104.6	7.8	104.6	6.2	103.2	5.3	104.1	7.9
cis-Permethrin	99.4	4.3	95.1	5.8	99.0	4.5	97.2	6.6
trans-Permethrin	98.6	3.6	95.5	4.9	98.5	4.3	97.2	7.1
Coumaphos	100.2	6.1	110.0	6.5	103.4	6.7	107.7	6.2
Cyfluthrin	99.7	3.7	97.2	4.5	99.9	3.6	99.6	6.6
Cypermethrin	98.9	3.4	96.6	4.0	99.5	4.0	98.1	6.3



METHOD STATEMENT



MCERTS Validation

Compound	MCERTS Accredited	LOD ng/L	Esholt STW - Final Effluent			
			Low Spike		High Spike	
			% Rec.	% RSD	% Rec.	% RSD
1,2,4-TCB	✓	1.44	90.71	7.42	95.35	5.54
Dichlorvos	✓	0.97	93.26	11.41	100.50	8.03
Aldrin	✓	1.15	95.70	6.52	98.72	5.05
Isodrin	✓	1.37	94.65	9.71	100.04	5.50
Endrin	✓	1.34	99.77	5.21	101.03	3.76
Dieldrin	✓	1.67	93.04	14.24	95.81	6.19
alpha-HCH	✓	1.42	99.75	8.07	101.44	4.08
beta-HCH	✓	0.92	99.91	6.96	101.49	5.43
gamma-HCH	✓	1.49	100.53	7.53	101.02	5.98
Atrazine	✓	5.64	96.16	5.87	102.36	5.05
Simazine	✓	7.05	94.87	11.52	102.46	9.36
Chlorfenvinphos	✓	1.21	96.32	6.13	101.10	5.77
Diazinon	✓	2.31*	98.69	8.81	99.52	4.04
cis-Permethrin	✓	0.94	93.58	4.33	98.13	3.57
trans-Permethrin	✓	0.75	93.99	5.42	98.85	4.00
Total Permethrin	✓	1.63	93.79	4.73	98.49	3.75
Cyfluthrin	✓	0.91	94.46	3.32	99.92	3.82
Cypermethrin	✓	1.48	94.73	3.10	99.77	3.67
Hexachlorobenzene	✓	1.01	98.66	8.85	99.28	5.69
Propetamphos	✓	0.72	97.50	4.73	99.22	4.28
Triazophos	✓	0.79	91.85	8.20	99.66	5.86
alpha-Endosulphan	✓	1.73	101.42	10.21	101.15	5.10
beta-Endosulphan	✓	1.81	98.02	6.60	100.06	6.33
op-DDE	✓	0.76	97.05	7.12	99.19	5.04
pp-DDE	✓	0.91	96.73	6.32	99.49	4.71
op-DDT	✓	0.95	95.53	3.73	98.33	2.78
pp-DDT	✓	1.16	92.71	7.55	96.87	6.30
pp-TDE	✓	1.14	102.83	6.06	102.40	4.44



METHOD STATEMENT



MCERTS Validation

Compound	Cooper Bridge STW - Final Effluent				Bulmer & Lumb - Trade Effluent			
	Low Spike		High Spike		Low Spike		High Spike	
	% Rec.	% RSD	% Rec.	% RSD	% Rec.	% RSD	% Rec.	% RSD
1,2,4-TCB	92.99	9.68	96.14	5.11	94.36	10.04	98.18	4.59
Dichlorvos	92.92	9.81	99.99	9.49	94.85	7.86	99.05	8.99
Aldrin	91.99	9.15	94.22	6.05	96.96	10.99	92.28	10.96
Isodrin	93.13	8.40	95.31	7.99	96.44	11.58	93.69	10.06
Endrin	100.96	7.31	100.85	5.53	101.23	10.29	97.58	9.63
Dieldrin	95.97	11.54	96.31	7.06	99.43	13.23	98.99	8.53
alpha-HCH	101.77	7.12	101.08	3.82	102.99	8.57	99.78	3.92
beta-HCH	103.85	7.41	102.36	6.08	104.67	9.01	100.61	5.96
gamma-HCH	98.51	8.32	97.24	9.69	103.63	8.96	99.69	5.69
Atrazine	106.40	7.93	101.56	4.35	98.36	5.58	99.08	5.01
Simazine	94.89	11.19	99.59	9.21	94.05	10.53	97.78	6.09
Chlorfenvinphos	89.87	8.31	93.49	5.97	93.28	8.96	99.01	5.98
Diazinon	90.57	10.25	92.42	7.78	92.85	6.11	94.46	4.85
cis-Permethrin	90.19	6.43	95.33	3.77	95.43	4.41	94.31	7.82
trans-Permethrin	91.54	5.71	96.36	3.74	99.92	6.03	97.12	5.48
Total Permethrin	90.86	5.89	95.84	3.68	97.68	5.06	95.72	6.52
Cyfluthrin	94.53	4.15	99.90	3.50	97.60	3.13	99.99	2.32
Cypermethrin	93.21	4.40	99.68	4.02	97.40	3.54	99.56	3.61
Hexachlorobenzene	96.55	8.13	97.89	6.11	100.15	10.93	97.45	6.72
Propetamphos	88.39	4.23	92.40	5.11	96.08	10.46	96.64	5.26
Triazophos	83.51	8.99	91.87	7.91	91.51	8.82	97.99	7.11
alpha-Endosulphan	98.89	8.55	99.05	6.06	100.66	11.19	98.12	6.31
beta-Endosulphan	99.76	10.31	99.43	8.43	102.76	11.84	98.75	8.63
op-DDE	93.60	10.06	92.91	6.48	98.70	10.91	92.41	11.35
pp-DDE	93.82	6.72	92.76	6.24	98.38	9.42	91.14	13.08
op-DDT	95.64	4.25	97.39	3.25	96.40	3.79	96.85	3.16
pp-DDT	92.40	6.44	96.86	2.19	96.44	5.27	97.08	8.66
pp-TDE	100.64	8.04	99.92	7.24	105.12	11.00	100.06	7.07



METHOD STATEMENT



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%.

Compound	Uncertainty of Measurement %
123-Trichlorobenzene	19.17
124-Trichlorobenzene	21.26
135-Trichlorobenzene	25.04
24-D-Ethyl	20.52
24-D-Isobutyl	28.93
24-D-Isopropyl	15.95
24-D-Methyl	17.43
24-D-n-Butyl	26.22
Alachlor	20.44
Aldrin	19.42
alpha-Endosulphan	22.91
alpha-HCH	19.81
Ametryn	13.26
Atrazine	8.38
Azinphos-Ethyl	16.41
Azinphos-Methyl	20.76
beta-Endosulphan	21.56
beta-HCH	22.16
Carbophenothion	15.69
Chlorfenvinphos	13.42
Chlorpropham	9.30
Chlorpyrifos-Ethyl	11.00
cis-Chlordane	26.90
cis-Permethrin	15.91
Coumaphos	18.76
Cyanazine	27.80
Cyfluthrin	17.26
Cypermethrin	16.63
Diazinon	11.58
Dichlobenil	12.50
Dichlorvos	18.43
Dieldrin	14.83
Diflufenican	16.77
Dimethoate	18.26
Endrin	17.15
EPTC	21.27
Ethofumesate	15.83
Fenitrothion	17.52
Fenpropidin	19.61
Fenpropimorph	18.35
Fenthion	13.69
Flutriafol	27.34
gamma-HCH	13.21
Heptachlor	22.35
Heptachlor Epoxide	13.75
Hexachlorobenzene	20.92



METHOD STATEMENT



Compound	Uncertainty of Measurement %
Hexachlorobutadiene	23.44
Isodrin	21.94
Malathion	11.88
Mevinphos	43.64
op'-DDE	25.76
op'-DDT	16.87
op'-TDE	25.28
Parathion-Ethyl	10.26
Parathion-Methyl	15.50
PCB 101	25.84
PCB 118	27.87
PCB 138	27.48
PCB 153	29.58
PCB 180	27.21
PCB 28	14.55
PCB 52	22.41
Pendimethalin	20.74
Pentachlorobenzene	21.25
Phorate	14.90
Phosalone	18.96
Pirimicarb	10.20
Pirimiphos-Methyl	12.94
pp'-DDE	33.28
pp'-DDT	19.42
pp'-TDE	31.01
Prometryn	15.08
Propachlor	13.12
Propazine	12.29
Propetamphos	10.98
Propiconazole	17.55
Propyzamide	15.45
Simazine	13.71
Tebuconazole	24.33
Tecnazene	8.84
Terbutryn	15.34
trans-Chlordane	24.91
trans-Permethrin	16.37
Triademefon	15.31
Triallate	12.15
Triazophos	11.46
Trietazine	16.97
Trifluralin	14.99

References

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