

Determination of Organophosphate Pesticides in Surface water using Gas Chromatography with mass selective detection (MSD).

1. Scope:

This section method (SM) documents the selected organophosphate pesticides analysis in surface water by all authorized section personnel. This method is not applicable for Ethoprop, Azinphos-methyl and Profenofos.

2. Principle:

The surface water sample is extracted with methylene chloride. The extract is passed through sodium sulfate to remove residual water. The anhydrous extract is evaporated to almost dryness on a rotary evaporator and diluted to a final volume of 1.0 mL with acetone. The extract is then analyzed by a gas chromatograph equipped with a mass selective detector (MSD).

3. Safety:

3.1 All general laboratory safety rules for sample preparation and analysis shall be followed.

3.2 Methylene chloride is a regulated and controlled carcinogenic hazardous substance. It must be stored and handled in accordance with California Code of Regulations, Title 8, Subchapter 7, Group 16, Article 110, Section 5202.

3.3 All solvents should be handled with care in a ventilated area.

4. Interferences:

There are matrix interferences that cause quantitative problems. Therefore the calibration standards will be made up in appropriate matrix.

5. Apparatus and Equipment:

5.1 Rotary evaporator (Büchi/Brinkman or equivalent)

5.2 Nitrogen evaporator (Meyer N-EVAP Organomation Model # 112 or equivalent)

5.3 Vortex-vibrating mixer

- 5.4 Balance (Mettler SM-L) or equivalent
- 5.5 Gas Chromatograph equipped with a mass selective detector (MSD)

6. Reagents and Supplies

- 6.1 Methylene Chloride, nanograde or equivalent pesticide grade
- 6.2 Acetone, nanograde or equivalent pesticide grade
- 6.3 Anhydrous Sodium Sulfate, granular
- 6.4 Diazinon CAS# 333-41-5
- 6.5 Disulfoton CAS# 298-04-4
- 6.6 Chlorpyrifos CAS# 2921-88-2
- 6.7 Malathion CAS# 121-75-5
- 6.8 Methidation CAS# 950-37-8
- 6.9 Fenamiphos CAS# 22224-92-6
- 6.10 Dichlorvos CAS# 62-73-7
- 6.11 Phorate CAS# 298-02-2
- 6.12 Fonofos CAS# 66767-39-3
- 6.13 Dimethoate CAS# 60-51-5
- 6.14 Parathion methyl CAS# 298-00-0
- 6.15 Tribufos (DEF) CAS# 78-48-8
- 6.16 Conical tube with glass stopper, 15-mL graduated, 0.1 mL subdivision
- 6.17 Separatory funnel, 2 L
- 6.18 Boiling flask, 500 mL
- 6.19 Funnel, long stem, 10 mm diameter
- 6.20 Disposable Pasteur pipettes, and other laboratory ware as needed
- 6.21 Recommended analytical columns:

For MSD - 1,4-bis(dimethylsiloxy)phenylene dimethyl polysiloxane (Restek Rxi-5Sil MS or equivalent) fused silica column, 30 m x 0.25 mm x 0.25 μ m film thickness.

7. Standards Preparation:

- 7.1 Dilute the 1 mg/mL Organophosphate standards obtained from the CDFCA/CAC Environmental Analysis Standards Repository with acetone to make up a series of mixed working standards (see 10.2). These standards shall be prepared to cover the linear range from 0.025 η g/ μ L to 0.5 η g/ μ L for OP screen and 0.01 η g/ μ L to 0.5 η g/ μ L for low level diazinon and chlorpyrifos.
- 7.2 The calibration standards are added to matrix blank extracts (9.1.2.1) to correct for matrix background interference.

- 7.3 Store standards according to manufacturing requirement. Keep all standards in designated refrigerator for storage.
- 7.4 The expiration date of each mixed working standard is six months from the preparation date or same as stock standards, if sooner.
- 7.5 A portion of the new standard will be vialled and set aside in the refrigerator. This will be used when doing the intermediate check and the check for a new set of standards. The intermediate check will be performed before the standard is 3 months old and be documented along with the comparison for that set of standards. There should be <20% difference between the response of the new standard or the intermediate check standard and the response of the vialled standard.

8. Sample Preservation and Storage:

All water samples and sample extracts shall be stored in the refrigerator (4 ± 3 °C).

9. Test Sample Preparation:

9.1 Sample Preparation

9.1.1 Remove samples from refrigerator and allow samples to come to room temperature before extraction.

9.1.2 Preparation of matrix blank and matrix spike:

The Department of Pesticide Regulations (DPR) provides the background water for matrix blank and spikes.

9.1.2.1 Matrix blank: Weigh out approximate 1000 g of background water and follow the test sample extraction procedure.

9.1.2.2 Matrix spike: Weigh out approximate 1000 g of background water. Spike a client requested amount of organophosphate pesticides into the background water and let it stand for 1 minute. Follow the test sample extraction procedure.

9.2 Test Sample Extraction

- 9.2.1 Record the weight of the whole bottle water sample to 0.1 g by subtracting the weight of the sample container before and after water has been transferred into a separatory funnel.
- 9.2.2 Shake with 100 ± 5 mL of methylene chloride for 2 minutes. Vent frequently to relieve pressure.
- 9.2.3 After phases have separated, drain lower methylene chloride layer through 20 ± 4 g of anhydrous sodium sulfate and glass wool, into a 500 mL boiling flask.
- 9.2.4 Repeat steps 9.2.2 & 9.2.3 two more times using 80 ± 5 mL of methylene chloride each time. Combine the extracts in the same boiling flask.
- 9.2.5 After draining the final extraction, rinse the sodium sulfate with 25 ± 5 mL of methylene chloride.
- 9.2.6 Evaporate the sample extract to 2 - 4 mL on a rotary evaporator using a water bath at 35 ± 2 °C and 15 - 20 inch Hg vacuum. Add 2 - 4 mL of acetone and rotoevaporate to 1 - 2 mL. Transfer the extract to a calibrated 15 mL graduated test tube.
- 9.2.7 Rinse flask 3 more times with 2 - 4 mL of acetone and transfer each rinse to the same test tube.
- 9.2.8 Evaporate the extract to a volume slightly less than 1 mL in a water bath at 38 ± 2 °C under a gentle stream of nitrogen. Then bring to a final volume of 1.0 mL with acetone, mix well and transfer into two autosampler vials.
- 9.2.9 Submit extract for GC/MS analysis.

10. Instrument Calibration:

- 10.1 The calibration standards are added to a matrix blank extract to correct for matrix background.
- 10.2 A calibration standard curve consists of minimum of three levels. Standard concentrations of 0.01, 0.025, 0.05, 0.1, 0.25 and 0.5 $\text{ng}/\mu\text{L}$ are recommended. Calibration is obtained using a linear or quadratic regression with the correlation coefficient (r) equal to or greater than 0.995.

11.1 Injection Scheme

Follow the sequence of Solvent, Calibration standards, Solvent, Matrix Blank, Matrix Spike, Test Samples (maximum of 10-12 samples) and Calibration standards. Injection of an old sample or matrix blank before the sequence analysis to condition the instrument is recommended.

11.2 GC Instrumentation

11.2.1 Recommended instrument (GC/MSD) parameters: Injector 250 °C; MSD transfer line heater 280 °C; oven temperature 80 °C, hold 2 min., ramp @ 20 °C/min. to 250 °C, hold 4 min.; injection volume 2 or 3 µL.

Ions Selected for SIM Acquisition:

Diazinon	137, 152, 179, 304 ,	Retention time: 11.9 min
Disulfoton	88 , 97, 142, 274,	Retention time: 12.2 min
Malathion	93, 125, 127, 173 ,	Retention time: 14.1 min
Chlorpyrifos	125, 197 , 258, 314,	Retention time: 11.2 min
Methidathion	58, 85, 93, 145 ,	Retention time: 9.88 min
Fenamiphos	154, 217, 288, 303 ,	Retention time: 9.26 min
DDVP	79, 109 , 185,	Retention time: 11.2 min
Phorate	75 , 97, 121, 260,	Retention time: 9.72 min
Dimethoate	87 , 93, 125, 126,	Retention time: 12.0 min
Fonofos	109 , 137, 246,	Retention time: 10.7 min
Me Parathion	63, 109, 125, 263 ,	Retention time: 9.94 min
DEF	169 , 202,	Retention time: 9.73 min

(Quantitation ions are in bold)

12. Quality Control:

12.1 Each set of samples shall have a matrix blank and minimum of one matrix spike sample.

12.2 The matrix blank should be free of target compounds.

12.3 The recoveries of the matrix spike shall be within the control limits.

12.3.1 When spike recoveries fall outside the control limits, the chemist must

investigate the cause. The entire extraction set of samples is re-analyzed. If the spike recoveries fall within the limit, then the results from the re-analyzed samples shall be reported.

12.3.2 If the spike recoveries still fall outside the control limits, the client will be notified. The backup samples will be re-extracted for analysis.

12.4 The retention time should be within ± 2 percent of that of the standard.

12.5 All calibration standards analyzed for a sample set will be used in the calibration curve. If the calibration curve does not meet the acceptance criteria the samples shall be re-run. If the calibration criteria are met the sample results will be reported. If the calibration criteria are still not met a method deviation will be prepared and approved by the supervisor or designee. The client will be notified of the deviation and a copy of the method deviation detailing what was changed and why it was changed will be included with the sample results and the data will be flagged to let the data user know of the deviation.

12.6 The sample must be diluted if results fall outside the linear range of the standard curve.

12.7 Bracketing standard curves should have a percent change less than 20%.

12.8 Method Detection Limits (MDL)

The method detection limit refers to the lowest concentration of analyte that a method can detect reliably. To determine the MDL, 7 replicate water samples are spiked at 0.05 ppb for OP screen and 7 replicate water samples are spiked at 10 ppt for low level diazinon and chlorpyrifos and 7 replicates were spikes at 0.02 ppb for malathion. The standard deviation from the spiked sample recoveries are used to calculate the MDL for each analyte using the follow equation:

$$\text{MDL} = tS$$

Where t is the Student t test value for the 99% confidence level with n-1 degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the n=7 replicate used to determine the MDL, t=3.143.

12.9 Reporting limit (RL):

The reporting limit (RL) refers to the level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. Per client

agreement, the RL is chosen in a range 1-5 times the MDL except in special cases. (See 15.5)

MDL data and the RL are tabulated in Appendix IA and IB.

12.10 Method Validation Recovery Data and Control Limits:

12.10.1 The method validation consisted of five sample sets. Each set included seven levels of fortification (0.01, 0.025, 0.05, 0.10, 0.25, 0.5 ppb) and a method blank. All spikes and method blank samples were processed through the entire analytical method.

12.10.2 Upper and lower warning and control limits are set at ± 2 and ± 3 standard deviations of the average % recovery, respectively.

12.10.3 The method validation consisted of five sample sets. Each set included six levels of fortification and a method blank. All spikes and method blank samples were processed through the entire analytical method.

Method validation results and control limits are tabulated in Appendix IB.

12.11 Estimated Measurement Uncertainty:

Total uncertainty for this method is 17% at 95% confidence interval.

12.12 Trend Identification

12.12.1 All matrix spike recoveries for OP analysis will be put into control charts and monitored for trends. Three trend characteristics will be evaluated at least bi-yearly by the supervisor or designee.
2 of 3 points above or below 2/3 of the UCL or LCL.
7 continuous points above or below the center line (CL)
14 points alternating above and below the CL.

12.12.2 When results indicate an out of control situation the supervisor or designee will indicate this on the control chart and take appropriate corrective action, which may include monitoring the results more closely to initiating a formal corrective action with root cause investigation.

13. Calculations:

Quantitation is based on external standard (ESTD) calculation using either the peak area or height. The software uses a linear or quadratic curve fit, with all levels weighted equally. Alternatively, at chemist discretion, concentrations may be calculated using

the response factor for the standard whose value is closest to the level in the sample.

$$\text{ppb} = \frac{(\text{sample peak ht. or area}) (\text{std. conc.}) (\text{std. vol. injected}) (\text{sample final vol., (mL)})(1000)}{(\text{std. peak ht. or area}) (\text{sample vol. injected}) (\text{sample wt., g})}$$

14. Reporting Procedure:

14.1 Identification of Analyte

For responses within calibration range, compare the retention time of the peaks with the retention time of standards. For positive results retention times shall not vary from the standards more than 2 percent.

14.2 Sample results are reported out according to the client's analytical laboratory specifications.

15. Discussion and References:

- 15.1 Sample response and quantitation vary depending on matrix background in the samples. The calibration standards were added to a matrix blank extract to correct for matrix background interference.
- 15.2 Some of the late eluting compounds were observed to suffer gradual losses in sensitivity. We recommend changing the injector liner and trimming the column when this occurs.
- 15.3 The client requested a lower reporting limit for both diazinon and chlorpyrifos. We re-validated this method using GC/MSD as the analysis instrument to achieve the lower reporting limit for those two compounds.

16. References:

- 16.1 *EPA Method 507, Pesticides, Capillary Column*. EPA Test Method for Drinking Water and Raw Source Water, 1987.
- 16.2 Hsu, J. and Hernandez J. *Determination of Organophosphate Pesticides in Surface Water using Gas Chromatography*, 1997, Environmental Monitoring Method, Center for Analytical Chemistry, CDFA.

Appendix IA

Determination of Method Detection Limit (MDL) and Reporting Limit (RL)

Spike/analyte	Diazinon			Disulfoton			Chlorpyrifos		
			Avg.			Avg.			Avg.
0.05/ ppb Spk 1	0.04709	0.04664	0.04687	0.04203	0.04528	0.04366	0.04784	0.04804	0.04794
0.05/ ppb Spk 2	0.04901	0.04975	0.04938	0.03938	0.03474	0.03706	0.04991	0.05010	0.05001
0.05/ ppb Spk 3	0.04465	0.04871	0.04668	0.04050	0.03653	0.03852	0.04580	0.04566	0.04573
0.05/ ppb Spk 4	0.04851	0.05026	0.04939	0.04640	0.04365	0.04503	0.04775	0.04768	0.04772
0.05/ ppb Spk 5	0.04405	0.04447	0.04426	0.04774	0.04583	0.04679	0.04459	0.04420	0.04440
0.05/ ppb Spk 6	0.04154	0.04181	0.04168	0.04740	0.04446	0.04593	0.04222	0.04262	0.04242
0.05/ ppb Spk 7	0.03949	0.04188	0.04069	0.03821	0.03487	0.03654	0.04093	0.04070	0.04082
		SD	0.00348			0.00441			0.00326
MDL= 3.14 * SD		MDL	0.01093			0.01384			0.01024
		RL	0.01			0.04			0.01
Spike/analyte	Malathion			Methidathion			Fenamiphos		
			Avg.			Avg.			Avg.
0.05/ ppb Spk 1	0.04549	0.04553	0.04551	0.03980	0.04117	0.04049	0.04614	0.04229	0.04422
0.05/ ppb Spk 2	0.04877	0.04895	0.04886	0.04612	0.04541	0.04577	0.04490	0.04879	0.04685
0.05/ ppb Spk 3	0.04489	0.04101	0.04295	0.03971	0.03883	0.03927	0.04202	0.04175	0.04189
0.05/ ppb Spk 4	0.04693	0.04568	0.04631	0.04224	0.04092	0.04158	0.04880	0.04839	0.04860
0.05/ ppb Spk 5	0.04169	0.04129	0.04149	0.03380	0.03328	0.03354	0.04403	0.04333	0.04368
0.05/ ppb Spk 6	0.04208	0.04177	0.04193	0.03967	0.03922	0.03945	0.04305	0.04289	0.04297
0.05/ ppb Spk 7	0.04121	0.04039	0.04080	0.04004	0.03957	0.03981	0.04196	0.03691	0.03944
		SD	0.00298			0.00362			0.00305
MDL= 3.14 * SD		MDL	0.00935			0.01136			0.00957
		RL	0.02			0.05			0.05

Determination of Method Detection Limit (MDL) and Reporting Limit (RL)

Spike/analyte	Malathion		
			Avg.
0.02/ ppb Spk 1	0.02160	0.02590	0.02375
0.02/ ppb Spk 2	0.01830	0.02260	0.02045
0.02/ ppb Spk 3	0.01690	0.02170	0.01930
0.02/ ppb Spk 4	0.01850	0.02230	0.02040
0.02/ ppb Spk 5	0.01710	0.02340	0.02025
0.02/ ppb Spk 6	0.01410	0.01960	0.01685
0.02/ ppb Spk 7	0.01830	0.02220	0.02025
Standard deviation		SD	0.00203
MDL= 3.14 * SD		MDL	0.00638
Reporting limit		RL	0.02

Appendix IB

Spike/analyte	DDVP	Avg.		Phorate	Avg.		Fonofos	Avg.	
0.05/ ppb Spk 1	0.04130	0.04339	0.04235	0.04292	0.04329	0.04311	0.04369	0.04362	0.04366
0.05/ ppb Spk 2	0.04210	0.04447	0.04329	0.04396	0.04350	0.04373	0.04652	0.04794	0.04723
0.05/ ppb Spk 3	0.04034	0.04069	0.04052	0.04084	0.04006	0.04045	0.04155	0.04126	0.04141
0.05/ ppb Spk 4	0.03780	0.04184	0.03982	0.04263	0.04252	0.04258	0.04368	0.04409	0.04389
0.05/ ppb Spk 5	0.03835	0.03789	0.03812	0.04031	0.03962	0.03997	0.04167	0.04151	0.04159
0.05/ ppb Spk 6	0.03834	0.03724	0.03779	0.03725	0.03734	0.03730	0.03935	0.03893	0.03914
0.05/ ppb Spk 7	0.03534	0.03528	0.03531	0.03577	0.03555	0.03566	0.03822	0.03774	0.03798
		SD	0.00276			0.00305			0.00343
MDL= 3.14 * SD		MDL	0.00868			0.00959			0.01076
		RL	0.05			0.05			0.04
Spike/analyte	Dimethoate	Avg.		Methyl Parathion	Avg.		DEF	Avg.	
0.05/ ppb Spk 1	0.03922	0.03874	0.03898	0.04111	0.04046	0.04079	0.04293	0.04358	0.04326
0.05/ ppb Spk 2	0.04397	0.04344	0.04371	0.04610	0.04631	0.04621	0.04628	0.04591	0.04610
0.05/ ppb Spk 3	0.03692	0.03638	0.03665	0.03906	0.04019	0.03963	0.04186	0.04259	0.04223
0.05/ ppb Spk 4	0.03869	0.03900	0.03885	0.04044	0.03966	0.04005	0.04388	0.04400	0.04394
0.05/ ppb Spk 5	0.03068	0.03089	0.03079	0.03278	0.03343	0.03311	0.03993	0.04046	0.04020
0.05/ ppb Spk 6	0.03617	0.03964	0.03791	0.03637	0.03720	0.03679	0.03932	0.03886	0.03909
0.05/ ppb Spk 7	0.03801	0.03736	0.03769	0.03748	0.03708	0.03728	0.03696	0.03786	0.03741
		SD	0.00383			0.00406			0.00301
MDL= 3.14 * SD		MDL	0.01202			0.01276			0.00946
		RL	0.04			0.03			0.05

Method Validation Data

Analyte	Spike ppb	Set 1			Set 2			Set 3				
				Avg.			Avg.			Avg.		
Diazinon	0.01	83.4	82.7	83.1	90.1	90.4	90.3	94.5	107.0	100.8	SD	6.082
	0.025	89.2	90.9	90.1	85.6	91.3	88.5	93.6	85.2	89.4	Mean	90.2
	0.05	101.0	94.6	97.8	90.0	89.8	89.9	89.3	93.4	91.4	UCL	108.5
	0.1	85.0	85.5	85.3	86.2	87.9	87.1	89.3	89.4	89.4	UWL	102.4
	0.25	92.9	93.5	93.2	80.4	81.0	80.7	88.3	86.3	87.3	LWL	78.1
	0.5	93.7	93.7	93.7	98.1	98.8	98.5	89.4	87.0	88.2	LCL	72.0
Disulfoton	0.01	84.1	83.1	83.6	112.0	105.0	108.5	114.0	114.0	114.0	SD	10.855
	0.025	73.6	72.3	73.0	80.5	78.4	79.5	85.2	83.8	84.5	Mean	85.4
	0.05	74.1	73.2	73.7	84.6	86.0	85.3	90.7	87.4	89.1	UCL	117.9
	0.1	85.3	85.7	85.5	81.2	79.2	80.2	85.0	83.1	84.1	UWL	107.1
	0.25	79.8	78.7	79.3	76.1	74.7	75.4	83.5	82.4	83.0	LWL	63.7
	0.5	79.7	78.1	78.9	95.2	94.6	94.9	85.0	84.2	84.6	LCL	52.8
Chlorpyrifos	0.01	111.0	109.0	110.0	98.5	101.0	99.8	102.0	102.0	102.0	SD	7.133
	0.025	98.1	97.6	97.9	89.4	90.4	89.9	86.1	87.6	86.9	Mean	92.9
	0.05	97.7	98.4	98.1	90.7	93.9	92.3	89.8	90.0	89.9	UCL	114.3
	0.1	88.1	88.4	88.3	87.2	87.4	87.3	87.3	86.8	87.1	UWL	107.2
	0.25	93.8	94.0	93.9	81.0	81.2	81.1	87.0	86.4	86.7	LWL	78.6
	0.5	94.8	93.8	94.3	98.9	99.6	99.3	88.0	87.1	87.6	LCL	71.5
Malathion	0.01	88.0	87.8	87.9	90.6	94.4	92.5	99.4	97.0	98.2	SD	4.642
	0.025	93.0	96.8	94.9	91.6	90.3	91.0	89.2	88.4	88.8	Mean	91.8
	0.05	99.0	98.9	99.0	91.7	92.8	92.3	90.3	89.1	89.7	UCL	105.7
	0.1	89.0	91.2	90.1	88.2	86.6	87.4	91.2	88.0	89.6	UWL	101.1
	0.25	95.7	95.7	95.7	81.8	82.3	82.1	89.8	88.2	89.0	LWL	82.5
	0.5	97.3	96.0	96.7	99.0	99.1	99.1	89.0	87.3	88.2	LCL	77.8

Method Validation Data (continued)

Methidathion	0.01	97.3	91.2	94.3	81.2	81.7	81.5	92.1	95.1	93.6	SD	8.648
	0.025	107.0	103.0	105.0	84.4	81.5	83.0	90.4	83.6	87.0	Mean	91.6
	0.05	107.0	101.0	104.0	88.7	86.2	87.5	83.3	81.9	82.6	UCL	117.6
	0.1	103.0	99.6	101.3	85.0	83.9	84.5	87.9	87.7	87.8	UWL	108.9
	0.25	106.0	104.0	105.0	80.1	80.5	80.3	93.0	92.0	92.5	LWL	74.3
	0.5	101.0	100.0	100.5	95.5	95.8	95.7	84.4	82.5	83.5	LCL	65.7

Fenamiphos	0.01	75.7	73.1	74.4	77.3	78.6	78.0	77.6	76.9	77.3	Sd	6.793
	0.025	86.5	85.6	86.1	78.3	77.0	77.7	77.4	78.4	77.9	Mean	84.4
	0.05	93.0	90.7	91.9	90.3	82.1	86.2	84.5	79.2	81.9	UCL	104.8
	0.1	93.0	91.4	92.2	83.8	82.8	83.3	81.3	83.5	82.4	UWL	98.0
	0.25	96.3	94.0	95.2	77.8	77.2	77.5	85.5	86.3	85.9	LWL	70.8
	0.5	94.8	92.8	93.8	95.1	94.9	95.0	82.7	82.2	82.5	LCL	64.0

DDVP	ppb	Set 1	Avg.		Set 2	Avg.		Set 3	Avg.			
	0.01	86.0	74.8	80.4	81.3	80.1	80.7	77.3	93.9	85.6	SD	7.765
	0.025	90.9	89.3	90.1	74.6	81.7	78.2	89.3	81.8	85.6	Mean	86.4
	0.05	84.0	85.6	84.8	81.6	81.2	81.4	81.7	82.3	82.0	UCL	109.7
	0.1	109.0	107.0	108.0	85.0	84.1	84.6	86.0	89.0	87.5	UWL	101.9
	0.25	99.2	91.7	95.5	76.6	76.5	76.6	85.2	85.4	85.3	LWL	70.8
0.5	92.4	89.0	90.7	94.4	96.4	95.4	84.1	81.0	82.6	LCL	63.1	

Fonofos	0.01	95.5	89.9	92.7	92.2	87.6	89.9	108.0	84.3	96.2	SD	4.794
	0.025	90.3	92.8	91.6	82.6	82.2	82.4	85.6	84.2	84.9	Mean	88.6
	0.05	86.4	84.9	85.7	85.8	83.4	84.6	87.0	82.8	84.9	UCL	103.0
	0.1	86.5	87.2	86.9	86.6	85.5	86.1	89.3	88.0	88.7	UWL	98.2
	0.25	92.5	91.1	91.8	80.3	85.6	83.0	87.1	86.4	86.8	LWL	79.0
	0.5	91.7	90.4	91.1	101.0	101.0	101.0	88.8	86.3	87.6	LCL	74.3

Dimethoate	0.01	97.5	87.7	92.6	72.0	74.7	73.4	106.0	97.2	101.6	SD	14.969
	0.025	137.0	136.0	136.5	84.3	82.5	83.4	79.9	83.8	81.9	Mean	90.2
	0.05	102.0	98.7	100.4	79.1	80.2	79.7	73.7	73.6	73.7	UCL	135.1
	0.1	105.0	101.0	103.0	81.4	81.6	81.5	83.9	84.3	84.1	UWL	120.1
	0.25	99.7	97.8	98.8	76.0	76.4	76.2	88.9	88.3	88.6	LWL	60.3
	0.5	93.8	92.9	93.4	94.2	92.6	93.4	82.6	80.7	81.7	LCL	45.3

Methyl Parathion	0.01	89.3	81.8	85.6	82.3	76.5	79.4	93.9	82.3	88.1	SD	12.244
	0.025	106.0	99.0	102.5	80.5	81.0	80.8	83.5	81.0	82.3	Mean	89.4
	0.05	103.0	96.9	100.0	76.7	78.7	77.7	74.4	75.3	74.9	UCL	126.1
	0.1	120.0	116.0	118.0	79.9	80.0	80.0	86.1	88.8	87.5	UWL	113.9
	0.25	108.0	105.0	106.5	75.6	75.6	75.6	90.3	91.5	90.9	LWL	64.9
	0.5	104.0	104.0	104.0	92.9	94.1	93.5	82.8	82.1	82.5	LCL	52.7

DEF	0.01	82.6	76.6	79.6	76.2	78.8	77.5	83.2	82.9	83.1	Sd	6.175
	0.025	91.5	93.5	92.5	93.8	83.5	88.7	82.9	83.6	83.3	Mean	88.0
	0.05	94.8	92.5	93.7	83.5	82.8	83.2	83.4	83.3	83.4	UCL	106.5
	0.1	91.6	91.9	91.8	88.7	87.6	88.2	87.1	87.8	87.5	UWL	100.4
	0.25	98.2	97.1	97.7	83.9	83.2	83.6	88.4	87.7	88.1	LWL	75.7
	0.5	96.5	94.9	95.7	99.4	99.9	99.7	88.5	86.6	87.6	LCL	69.5

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