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Title: Determination of Ethalfluralin, Trifluralin, Benfluralin, Prodiamine, Pendimethalin, Oxyfluorfen, and Oryzalin in Surface Water

1. Scope:

This section method (SM) provides stepwise procedure for selective Dinitroaniline compounds and Oxyfluorfen analysis in surface water. It is followed by all authorized EA personnel.

2. Principle:

The dinitroanilines and oxyfluorfen are extracted from surface water samples with methylene chloride. The extract is passed through sodium sulfate to remove residual water. The anhydrous extract is evaporated on a rotary evaporator and then a solvent exchange is performed with acetone. The extract is concentrated to a final volume of 1 mL where 0.5 mL is removed and vialed for GCMS-SIM (Gas Chromatography with Mass Spectrometer operated in the Single Ion Monitoring mode) or GCMS/MS analysis. The remaining 0.5mL is evaporated to just dryness and then brought up to a final volume of 0.5mL with methanol for analysis of oryzalin on LCMS.

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.

4. Interferences:

There were no matrix interferences that caused quantitative problems during method development and validation.

5. Apparatus and Equipment:

5.1 Rotary Evaporator (Buchi/Brinkman or equivalent)
5.2 Nitrogen Evaporator (Meyer N-EVAP Organomation Model #112 or equivalent)
5.3 Balance (Mettler PC 4400 or equivalent)
5.4 Vortex-vibrating mixer

5.5 Gas Chromatograph equipped with a mass selective detector (MSD)
5.6 Gas Chromatograph equipped with a triple stage quadropole detector (MS/MS)
5.7 Liquid Chromatograph equipped with an ion trap (LCMS)

6. **Reagents and Supplies**:

6.1	Ethalfluralin	CAS#55283-68-6
6.2	Trifluralin	CAS#1582-09-8
6.3	Benfluralin	CAS#1861-40-1
6.4	Prodiamine	CAS#29091-21-2
6.5	Pendimethalin	CAS#40487-42-1
6.6	Oxyfluorfen	CAS#42874-03-3
6.7	Oryzalin	CAS#19044-88-3

6.8 Methylene Chloride, nanograde or equivalent pesticide grade
6.9 Acetone, nanograde or equivalent pesticide grade
6.10 Water, MS grade, Burdick & Jackson or equivalent
6.11 Methanol, MS grade, Burdick & Jackson or equivalent
6.12 Formic Acid, HPLC grade
6.13 Ammonium formate, reagent grade or equivalent
6.10 Separatory funnel, 2 L
6.11 Boiling flask, 500 mL
6.12 Sodium Sulfate, ACS grade
6.13 Funnels, long stem, 60°, 10 mm diameter
6.14 Volumetric Pipette, 0.5 mL
6.15 Graduated conical tubes with glass stopper, 15 mL
6.16 Glass wool, Pyrex® fiber glass slivers 8 microns
6.17 Disposable Pasteur pipettes, and other laboratory ware as needed
6.18 Recommended analytical columns:

For MSD - 5% (Phenyl)-methylpolysiloxane (HP-5MS or equivalent) fused silica column, 30 m x 0.25 mm id x 0.25 µm film thickness.

For HPLC/MS – Waters SymmetryShieldRP_{18} 5 µm, 3.9 x 150 mm cartridge
Guard column: Waters SymmetryShieldRP_{18} 5 µm, 3.9 x 20 mm cartridge
Guard column holder: Waters Sentry guard holder universal.

7. Standards Preparation:

7.1 The individual dinitroaniline and oxyfluorfen stock standards of 1.0 mg/mL were obtained from the CDFA/CAC Standards Repository. The standards were diluted

to 10 µg/mL with acetone for identification purposes. Oryzalin was prepared in methanol at a concentration of 10 µg/mL for infusion into the LCMS.

A combination standard of 10 µg/mL was prepared from the individual mg/mL standards with acetone. The standard was also used to dilute the following concentrations: 0.025, 0.05, 0.1, 0.2, 0.5, and 1 µg/mL in acetone for GC instrument calibration. The 10 µg/mL of oryzalin in methanol was diluted to the same concentrations as above for LC instrument calibration.

7.2 Keep all standards in the designated refrigerator for storage.

7.3 The expiration date of each standard is six months from the preparation date.

8. **Sample Preservation and Storage**:

Store all samples waiting for extraction in a separate refrigerator (0 - 5 °C).

9. **Test Sample Preparation**:

9.1 Background Preparation

The Department of Pesticide Regulation (DPR) provided the surface water for background to be used in method validation and QC.

9.2 Preparation of blank and spike

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

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

9.3 Test Sample Extraction

9.3.1 Record the weight of water samples to 0.1 g by subtracting the weight of the sample container before and after water has been transferred into a separatory funnel.

9.3.2 Shake with 100 ± 5 mL of methylene chloride for 2 minutes. Vent frequently to relieve pressure.

9.3.3 After phases have separated, drain lower the methylene chloride layer through 20 ± 4 g of anhydrous sodium sulfate and glass wool, into a 500 mL boiling flask.

9.3.4 Repeat steps 9.3.1 & 9.3.2 two more times using 80 ± 5 mL of methylene chloride each time. Combine the extracts in the same boiling flask.

9.3.5 After draining the final extraction, rinse the sodium sulfate with 25 ± 5 mL of methylene chloride.

9.3.6 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.

9.3.7 Rinse flask 3 more times with 2 - 4 mL of acetone and transfer each rinse to the same test tube.

9.3.8 Evaporate the sample 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 0.5mL to two autosampler vials with inserts. Submit extract for GCMS-Triple Stage quadrapole analysis.

9.3.9 The remaining 0.5 mL sample extract is placed back in the water bath and evaporated to just dryness. Pipet 0.5 mL of methanol into the test tube and vortex well. Transfer extract to an autoampler vial to analyze on LCMS for oryzalin.

10. **Instrument Calibration**:

10.1 The calibration standard curve consists of a minimum of three levels. The lowest level must be at or below the corresponding reporting limits.

10.2 The calibration curves for the GCMS and Triple Quad were obtained using quadratic fit. The LCMS calibration curves were obtained using linear regression.

11. **Analysis:**

11.1 HPLC-MS

11.1.1 HPLC Instrument: Waters model 2695 HPLC and auto-sampler with column heater and remote control through Thermo Finnigan Xcalibur system.

Column: Waters SymmetrySheid RP_{18} 5 µm, 3.9 x 150 mm column
Column Temperature: 40 °C
Mobile Phase: Gradient
Solvent 1: 3762 mL water, 200 mL methanol, 38 mL 1M ammonium formate and 4.0 mL formic acid.
Solvent 2: 3600 mL methanol, 360 mL water, 36 mL 1.0 M ammonium formate, 4 mL formic acid.
Gradient:

Time(min)	Flow rate	Mobile Phase 1	Mobile Phase 2
0	0.75	85.0	15.0
3.0	0.75	85.0	15.0
4.0	0.75	50.0	50.0
10.0	0.75	50.0	50.0
14.0	0.75	40.0	60.0
16.0	0.75	5.0	95.0
22.0	0.75	5.0	95.0
24.5	0.75	85.0	15.0
27.0	0.75	85.0	15.0

Injection Volume:20 µL

11.1.2 Liquid Chromatograph Mass spectrometer (LC-MS) and Operating Parameters

Model:	Finnigan Model DECA ion trap MS
Ion Source Type:	Atmospheric pressure Ionization (APCI)
Source Polarity:	Positive
APCI Vaporizer Temp:	450 °C
Capillary Temperature:	220 °C
Sheath Gas:	60
Auxiliary Gas:	10
Mode of operation:	MS/MS

Compound Name	Retention Time (min.)	Molecular Weight	Mass Range	Product Ions
Oryzalin	18.96	346.36	95-400	288, 305

Note:The column conditions, temperature, mobile phase, etc. may slightly shift retention time.

11.1.3 Operating parameter

Parent Mass(m/z)	Isolation Width (m/z)	Normalized Collision Energy (%)	Activation Q	Activation Time (msec.)
347	2.0	30.0	0.250	30.0

11.2 GC-Triple Quad Instrumentation:

11.2.1 Model: Varian Triple Quad 320-MS
Column: Varian Factor Four VF-5ms x 0.25mm x0.25µm

Temperature Program: initial column temperature 80 °C, hold 1 min., ramp at 15 °C/min. to temperature of 180 °C and hold for 3 min. ramp at 15 °C/min. to final temperature of 300°C and hold for 3 min.;

Injector Temperature: 250 °C
Injection volume: 1 uL.

Compound	Retention Time (min)	Precursor ion	Product Ion	Collison Energy/-ev
Ethalfluralin	10.28	333	316	-10
Trifluralin	10.52	335	290	-15
Benfluralin	10.62	335	276	-15
Prodiamine	13.91	350	275	-10
Pendimethalin	14.86	281	252	-10
Oxyfluorfen	15.97	361	300	-15

11.3 GCMS Instrumentation:

11.3.1Model: Agilent GCMS

Column: 5% (Phenyl)-methylpolysiloxane (HP-5MS or equivalent) fused silica column, 30 m x 0.25 mm id x 0.25 µm film thickness.

Temperature Program: initial column temperature 80 °C, hold 1 min., ramp at 15 °C/min. to temperature of 180 °C and hold for 3 min. ramp at 15 °C/min. to final temperature of 300°C and hold for 3 min.;

Injector Temperature: 250 °C
Transfer line Temperature: 280 °C

Compound	Retention Time (min.)	Selected ions	Starting time (min.)
Ethalfluralin	9.41	**276**, 316, 333	6.00
Trifluralin	9.62	264, **306**, 335	9.52
Benfluralin	9.69	264, **292**, 335	9.52
Prodiamine	13.27	279, **321**, 333	12.50
Pendimethalin	14.23	**252**, 253, 281	13.85
Oxyfluorfen	15.38	**252**, 300, 361	14.85

Quantitation ions are in bold.

12. **Quality Control**:

12.1 Method Detection Limits (MDL)

Method Detection Limit (MDL) refers to the lowest concentration of the analyte that a method can detect reliably. To determine the MDL, 7 surface water samples are spiked at 0.05ppb and processed through the entire method along with a blank. The standard deviation derived from the spiked sample recoveries was used to calculate the MDL for each analyte using the following equation:

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 replicates used to determine the MDL, t=3.143.

The results for the standard deviations and MDL are in Appendix 1.

12.2 Reporting Limit (RL)

Reporting limit (RL) refers to a level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. The RL is chosen in a range 1-5 times the MDL, as per client agreement. The reporting limit for the dinitroanilines and oxyfluorfen is 0.05 ppb.

12.3 Method Validation

The method validation consisted of four sample sets. Each set included five levels of fortification and a method blank. All spikes and method blanks were processed through the entire analytical method. Spike levels and recoveries for the selective dinitroaniline and oxyfluorfen are shown in Appendix 2.

12.4 Control Charts and Limits

Control charts were generated using the data from the method validation for each analyte. The upper and lower warning and control limits are set at $\pm$ 2 and 3 standard deviations of the % recovery, respectively, shown in Appendix 2.

12.5 Acceptance Criteria

12.5.1 Each set of samples will have a matrix blank and a spiked matrix sample.
12.5.2 The retention time should be within $\pm$ 2 per cent of that of the standards.
12.5.3 The recoveries of the matrix spikes shall be within the control limits.
12.5.4 The sample shall be diluted if results fall outside of the calibration curve.

13. **Calculations:**

Quantitation is based on an external standard (ESTD) calculation using either the peak area or height. The LCMS software used a linear curve fit, with all levels weighted equally. The software for the triple quadrapole uses a quadratic curve fit, with all levels weighted 1/nx and the GCMS uses quadratic curve fit, with all levels weighted equally. Alternatively, at the chemist's discretion, sample results may be calculated using the response factor for the standard.

$$\text{ppb} = \frac{(\text{sample peak area or ht}) \times (\text{std conc}) \times (\text{std vol. Injected}) \times (\text{final vol of sample})(1000\ \mu\text{L/mL})}{(\text{std.peak area or ht}) \times (\text{sample vol injected}) \times (\text{sample wt (g)})}$$

14. **Reporting Procedure:**

Sample results are reported out according to the client's analytical laboratory specification sheets.

15. **Discussion and References:**

15.1 The triple quadrapole will used as the primary instrument for the analysis of the dinitroanalines and oxyfluorfen. The MSD will be used as a backup instrument. The LCMS is used for the analysis of oryzalin since it wasn't very sensitive on the GC.

15.2 A storage stability study was done with this project. The storage stability study consisted of a 5 ppb spike level and 3 replicates over a 28 day period. Fifteen bottles containing backgrouond water were spiked and stored in the refrigerator until analyzed on 0, 4, 7, 14, and 28 days. Along with the storage spikes a blank and method control spike were also extracted. This storage study showed no degradation for the dinitroaniline compounds or oxyfluorfen. The results are shown in Appendix 3.

15.3 We have observed gradual losses in sensitivity caused by the sample matrix. We recommend changing the injector liner and trimming the column when this occurs.

15.4 Solid phase extraction has been tried for sample preparation as part of our method development. The recoveries were low and inconsistent for some compounds.

15.5 GC-Triple Quad analysis of the samples produced a sample response and quantitation varied depending on matrix background in the samples. Therefore the calibration standards were added to a matrix blank extract to correct for matrix background interference. This is unnecessary for LCMS analysis.

15.6 References:

15.61 J.L Kish, E.M. Thruman, E.A. Scribner, and L.R. Zimmerman; *Methods of Analysis by the U.S. Geological Survey Organic Geochemistry Research Group—Determination of Selected Herbicides Metabolites and Their Degradaion Products in Water Using Solid-Phase Extraction and Gas*

Chromatography/Mass, U.S. Geological Survey Kansas Water Science Center

15.62 Hsu, J. and Feng, H.; *Determination of Organophosphate Pesticides in the surface water using Gas Chromatography*, 2004, Environmental monitoring method, Center for Analytical Chemistry, CDFA

Appendix 1

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

Results: Varian GC/TQMS

Spk\Analyte	Ethalfluralin	Trifluralin	Benfluralin	Prodiamine	Pendimethalin	Oxyfluorfen
0.05ppb spk 1	0.0366	0.0410	0.0384	0.0375	0.0358	0.0332
0.05ppb spk 2	0.0463	0.0402	0.0387	0.0384	0.0359	0.0369
0.05ppb spk 3	0.0416	0.0399	0.0451	0.0414	0.0377	0.0343
0.05ppb spk 4	0.05	0.0478	0.0465	0.0427	0.0464	0.0403
0.05ppb spk 5	0.0479	0.0398	0.0433	0.0391	0.0385	0.0375
0.05ppb spk 6	0.0461	0.0408	0.0437	0.0415	0.0394	0.0381
0.05ppb spk 7	0.0495	0.0512	0.0487	0.0493	0.0425	0.0425
SD	0.00479	0.00460	0.00382	0.00395	0.00382	0.00322
MDL	0.0150	0.0144	0.0120	0.0124	0.0120	0.0101
RL	0.05	0.05	0.05	0.05	0.05	0.05

Appendix 1: continued

Results: Agilent GC/MSD

Spk\Analyte	Ethalfluralin	Trifluralin	Benfluralin	Prodiamine	Pendimethalin	Oxyfluorfen
0.05ppb spk 1	0.044	0.040	0.038	0.051	0.045	0.052
0.05ppb spk 2	0.052	0.047	0.044	0.060	0.054	0.061
0.05ppb spk 3	0.048	0.044	0.041	0.057	0.051	0.059
0.05ppb spk 4	0.059	0.054	0.051	0.069	0.062	0.070
0.05ppb spk 5	0.047	0.044	0.041	0.054	0.048	0.052
0.05ppb spk 6	0.049	0.045	0.042	0.057	0.051	0.056
0.05ppb spk 7	0.056	0.052	0.049	0.066	0.059	0.068
SD	0.00528	0.0049	0.0047	0.0064	0.0059	0.0072
MDL	0.017	0.015	0.015	0.020	0.019	0.023
RL	0.05	0.05	0.05	0.05	0.05	0.05

Results: Finningan LCQ Deca

Spk\Analyte	Oryzalin
0.05ppb spk 1	0.057
0.05ppb spk 2	0.057
0.05ppb spk 3	0.057
0.05ppb spk 4	0.056
0.05ppb spk 5	0.055
0.05ppb spk 6	0.057
0.05ppb spk 7	0.053
SD	0.001528
MDL	0.021
RL	0.05

Appendix 2

Method Validation Data

Results: **Varian GC/TQMS**

Analyte	Spike ppb	Recovery Set 1	(%) set 2	set 3	set 4	%	%
Ethalfluralin	0.15	110	97.7	101	97.9	Mean:	98.7
	0.3	108	107	86.2	96.4	SD:	6.4
	1	94.7	94	98.9	93.9	UCL:	117.9
	2	94.7	105	108	96.7	UWL:	111.5
	5	90.0	95.9	102	95.0	LWL:	85.9
						LCL:	79.5
Trifluralin	0.15	109	91.4	103	89.8	Mean:	97.4
	0.3	108	104	88.5	92.7	SD:	6.6
	1	96.5	92	97.6	95.2	UCL:	117.2
	2	96.8	106	106	91.5	UWL:	110.6
	5	92.6	89.9	102	95.6	LWL:	84.2
						LCL:	77.6
Benfluralin	0.15	103	86	101	87.7	Mean:	96.7
	0.3	107	104	83.5	92.6	SD:	7.0
	1	98.3	92.5	101	93.6	UCL:	117.7
	2	94.9	108	104	95.1	UWL:	110.7
	5	91	90.4	102	97.3	LWL:	82.7
						LCL:	75.7
Prodiamine	0.15	120	95.1	112	99.0	Mean:	101
	0.3	117	113	77.6	97.2	SD:	11.4
	1	102	93.7	113	90.2	UCL:	135.2
	2	92.6	108	115	92.5	UWL:	123.8
	5	90.1	93.9	100.9	91.0	LWL:	78.2
						LCL:	66.8

Results: **Varian GC/TQMS**

Analyte	Spike ppb	Recovery Set 1	(%) set 2	set 3	set 4	%	%
Pendimethlin	0.15	109	94.4	109	95.3	Mean:	98.0
	0.3	112	106	85.1	98.0	SD:	8.9
	1	101	91.6	105	90.2	UCL:	124.7
	2	94.2	99.6	115	92.2	UWL:	115.8
	5	86.6	88.3	99.3	88.5	LWL:	80.2
						LCL:	71.3
Oxyfluorfen	0.15	114	96.5	112.4	95.3	Mean:	100.4
	0.3	115	113	75.2	101	SD:	12.8
	1	105	90.7	107	91.3	UCL:	138.8
	2	97.6	109	128	91.9	UWL:	126.0
	5	84.1	89.8	106	85.7	LWL:	74.8
						LCL:	62.0

Results: **Agilent GC/MSD**

Analyte	Spike ppb	Recovery Set 1	(%) set 2	set 3	set 4		%
Ethalfluralin	0.15	94.3	84.9	95.9	91.2	Mean:	99.6
	0.3	104	114	105	90.6	SD:	9.6
	1	123	107	100	106	UCL:	128.4
	2	96.3	114	103	89.2	UWL:	118.8
	5	96.9	92.9	92.7	92.6	LWL:	80.4
						LCL:	70.8
Trifluralin	0.15	91.3	82	91.3	87.3	Mean:	97.1
	0.3	101	111	102	87.7	SD:	9.4
	1	119	104	96.5	102	UCL:	125.3
	2	94.6	112	102	88.0	UWL:	115.9
	5	95.8	92.4	91.4	92.0	LWL:	78.3
						LCL:	68.9

Results: **Agilent GC/MSD**

Benfluralin	0.15	90.0	80.0	89.3	84.7	Mean:	96.0
	0.3	99.7	110	99.1	85.7	SD:	9.5
	1	118	103	96.1	101	UCL:	124.5
	2	94.0	111	101	87.5	UWL:	115.0
	5	95.8	92.0	91.4	91.6	LWL:	77.0
						LCL:	67.5
Prodiamine	0.15	116	96.7	117	108	Mean:	112
	0.3	121	135	121	102	SD:	11.0
	1	130	116	113	115	UCL:	145.0
	2	106	121	120	97.5	UWL:	134.0
	5	105	103	99.2	98.8	LWL:	90.0
						LCL:	79.0
Pendimethlin	0.15	112	89.6	106	98.1	Mean:	108
	0.3	117	126	120	95.5	SD:	10.6
	1	123	111	108	111	UCL:	139.8
	2	105	120	119	95.0	UWL:	129.2
	5	106	100	97.6	97.9	LWL:	86.8
						LCL:	76.2
Oxyfluorfen	0.15	124	87.3	111	103	Mean:	113
	0.3	125	131	134	102	SD:	12.1
	1	123	120	115	118	UCL:	149.6
	2	110	120	123	102	UWL:	137.2
	5	114	105	99.9	100	LWL:	88.8
						LCL:	76.7

Results: **Finningan LCQ Deca**

Analyte	Spike ppb	Recovery Set 1	(%) set 2	set 3	set 4		%
Oryzlin	0.15	92.7	96.0	73.3	84.0	Mean:	83.6
	0.3	86.0	91.7	100	77.7	SD:	9.3
	1	87.2	91.2	77.9	68.0	UCL:	111.5
	2	93.0	70.6	80.5	68.5	UWL:	102.2
	5	90.4	81.8	79.6	81.0	LWL:	65.0
						LCL:	55.7

Appendix 3 Storage Stability Study

		Day 0		Day 4		Day 7		Day 14		Day 28	
Analyte		ppb	%R	ppb	%R	ppb	%R	ppb	%R	ppb	%R
Ethalfluralin	blk	nd		nd		nd		nd		nd	
	spk	0.836	83.6%	0.875	87.5%	0.849	84.9%	0.796	79.6%	0.804	80.4%
	spk 1	0.865	86.5%	0.894	89.4%	0.877	87.7%	0.961	96.1%	1.00	100%
	spk 2	0.873	87.3%	0.857	85.7%	0.858	85.8%	1.03	103%	1.04	104%
	spk 3	0.831	83.1%	0.821	82.1%	0.895	89.5%	0.941	94.1%	0.872	87.2%
Trifluralin	blk	nd		nd		nd		nd		nd	
	spk	0.795	79.5%	0.851	85.1%	0.877	87.7%	0.818	81.8%	0.83	83.0%
	spk 1	0.825	82.5%	0.862	86.2%	0.828	82.8%	0.948	94.8%	0.964	96.4%
	spk 2	0.734	73.4%	0.838	83.8%	0.88	88.0%	1.06	106.0%	1.03	103%
	spk 3	0.797	79.7%	0.833	83.3%	0.913	91.3%	0.94	94.0%	0.832	83.2%
Benfluralin	blk	nd		nd		nd		nd		nd	
	spk	0.840	84.0%	0.827	82.7%	0.859	85.9%	0.806	80.6%	0.838	83.8%
	spk 1	0.875	87.5%	0.854	85.4%	0.858	85.8%	0.983	98.3%	0.962	96.2%
	spk 2	0.853	85.3%	0.874	87.4%	0.878	87.8%	1.03	103%	1.06	106%
	spk 3	0.856	85.6%	0.828	82.8%	0.879	87.9%	0.930	93.0%	0.885	88.5%
Prodiamine	blk	nd		nd		nd		nd		nd	
	spk	0.858	85.8%	0.852	85.2%	0.899	89.9%	0.832	83.2%	0.813	81.3%
	spk 1	0.906	90.6%	0.881	88.1%	0.834	83.4%	1.02	102%	0.97	97.0%
	spk 2	0.905	90.5%	0.910	91.0%	0.953	95.3%	1.09	109%	1.10	110%
	spk 3	0.899	89.9%	0.851	85.1%	0.908	90.8%	0.979	97.9%	0.907	90.7%
Pendimethlin	blk	nd		nd		nd		nd		nd	
	spk	0.82	82.0%	0.825	82.5%	0.881	88.1%	0.796	79.6%	0.802	80.2%
	spk 1	0.898	89.8%	0.836	83.6%	0.785	78.5%	0.948	94.8%	0.953	95.3%
	spk 2	0.900	90.0%	0.875	87.5%	0.871	87.1%	1.02	102%	1.04	104%
	spk 3	0.868	86.8%	0.783	78.3%	0.857	85.7%	0.906	90.6%	0.868	86.8%

Oxyfluorfen	blk	nd		nd		nd		nd		nd	
	spk	0.775	77.5%	0.824	82.4%	0.884	88.4%	0.819	81.9%	0.726	72.6%
	spk 1	0.889	88.9%	0.810	81.0%	0.788	78.8%	0.984	98.4%	0.977	97.7%
	spk 2	0.857	85.7%	0.849	84.9%	0.913	91.3%	0.991	99.1%	1.04	104%
	spk 3	0.838	83.8%	0.752	75.2%	0.869	86.9%	0.867	86.7%	0.837	83.7%
Oryzalin	blk	nd		nd		nd		nd		nd	
	spk	0.900	90.0%	0.963	96.3%	0.95	95.0%	0.960	96.0%	0.795	79.5%
	spk 1	0.963	96.3%	0.929	92.9%	0.937	93.7%	0.918	91.8%	0.881	88.1%
	spk 2	0.898	89.8%	0.824	82.4%	0.867	86.7%	1.02	102%	0.884	88.4%
	spk 3	0.999	99.9%	0.997	99.7%	0.803	80.3%	1.03	103%	0.870	87.0%

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Revision Log:

Date	What was revised? Why?