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Study 249. Statewide Urban Pesticide Use and Water Quality Monitoring

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1 INTRODUCTION

Urban pesticide use includes structural pest control, landscape maintenance, rights-of-way pest control, public health protection, and residential applications. The average annual non-residential reported pesticide use in California urban areas from 1995-2005 is ~14 million pounds of active ingredients (a.i.) (CDPR, 2006). The non-residential urban use of pesticides in 2006 in California amounted to ~500,000 lbs a.i. for permethrin, ~446,000 lbs a.i. for diuron, and ~220,000 lbs a.i. for oryzalin. The use of fipronil, which is exclusively used in urban areas in California (Gunasekara et al., 2007), has increased dramatically from 650 lbs in 2000 to ~100,000 lbs in 2006 (Ensminger, 2007). While residential use of pesticides is not reported, total annual urban use may be approximately estimated for many pesticides using pesticide sales data and reported use data. For example, the mean estimated annual urban use of pesticides during 2004 – 2005 in California was ~700,000 lbs a.i. for malathion, 200,000 lbs a.i. for carbaryl, and 740,000 lbs a.i. for pyrethroids. Based on comparison of pesticide use and sales data, pesticide applications in urban areas account for approximately 70% of pesticide sales in California in 2005 (Spurlock, 2007). Higher urban pesticide use is anticipated in condense urbanized areas such as the San Francisco Bay, Sacramento, Los Angeles, and San Diego areas.

When urban use pesticide residues transport to storm drains or urban waterways by stormwater runoff (Guo et al., 2007) or urban baseflow, they may cause toxicity to sensitive aquatic species (Gunasekara et al., 2007; He et al., 2007). For example, stormwater samples collected from Chollas Creek, a highly urbanized watershed in San Diego, California exhibited toxicity to *Strongylocentrotus* and *Ceriodaphnia*. Toxicity identification evaluations suggested that organophosphate pesticides, most likely diazinon and chlorpyrifos, were responsible for the toxicity observed in *Ceriodaphnia* (Schiff et al., 2002). Another urban runoff study conducted in

the great Los Angeles area indicated that commercial and high-density residential land uses showed the highest diazinon concentrations as opposed to other urban land uses (Schiff and Sutula, 2004). The widespread urban use of organophosphate pesticides in California resulted in several urban waterbodies being placed in the Clean Water Act Section 303(d) list (SWRCB, 2007).

As urban uses of both diazinon and chlorpyrifos were suspended in recent years, pyrethroid use has increased sharply. Recent monitoring of urban watersheds has identified potential pyrethroid impacts (Bondarenko et al., 2007; He et al., 2007; Xu et al., 2007; Yang et al., 2007). Nearly all creek sediments collected in Roseville, California - a typical suburban development in the Sacramento area caused toxicity to the amphipod *Hyalella azteca*, and about half the samples caused nearly complete mortality. The pyrethroid bifenthrin was the primary cause of the toxicity, with additional contributions from the pyrethroids cyfluthrin and cypermethrin. The dominant sources of these pyrethroids are structural pest control by professional applicators and/or homeowner use of insecticides, particularly lawn care products (Weston et al., 2005). The authors concluded that the suburbs of Roseville are probably not unique, and similar sediment quality degradation is likely in other suburban areas, particularly in dry regions where landscape irrigation can dominate seasonal flow.

Pyrethroids are just one of the pesticide groups that are widely used in urban areas in California. A recent retail store survey indicated that more than 320 pesticide products were on sale at three stores in the San Francisco Bay area, and these products contained 99 different a.i.'s (Moran, 2005). Another survey of 14 stores located in Sacramento County and the San Francisco Bay area revealed 542 different products on sale, which contained 112 different a.i.'s (Flint, 2003), representing a wide spectrum of urban insecticides and herbicides. Data concerning how and to what degree the urban use of these pesticides impacts urban watersheds is lacking. Urban monitoring data are therefore needed in order to assess the potential impacts of urban pesticide use on aquatic systems. A statewide consistent monitoring program will provide useful data on the environmental fate of urban use pesticides and for use in the development of management measures. This one-year project represents the Phase I of a multi-year monitoring program that includes urban pesticide use screening monitoring of water quality and focused investigations on potential source(s) and mitigation measures for source reduction.

2 OBJECTIVES

This project attempts to address the following:

- Which pesticides (if any) are present in California urban surface waters at levels that have the potential to impact the beneficial uses of those waters?
- What are the potential sources of pesticides in urban runoff and their relative contribution to the receiving water problem?

The overall goal of the multiyear project is to assess urban pesticide use and water quality in urban drainage and receiving water from stormwater runoff and baseflow in California's major urbanized areas. The objectives of the first-year study include:

- Determining which pesticides are present in urban runoff,
- Evaluating the magnitude of measured concentrations relative to water quality or aquatic toxicity benchmarks, and
- Assessing the effect of waterbody type (stormwater drain vs. urban creek) and season (stormwater vs. non-stormwater) on pesticide concentration and/or loading.

The proposed study will also measure other water quality constituents, including dissolved oxygen, pH, electrolytic conductivity, and turbidity, to provide essential background information and an overall picture of water quality as it relates to aquatic life criteria.

3 PERSONNEL

The study will be conducted by staff from the Environmental Monitoring Branch, Surface Water Protection Program, under the general direction of Dr. Kean S. Goh, Environmental Program Manager. Key personnel are listed below:

Project Leader: Li-Ming (Lee) He, Ph.D. and Michael Ensminger, Ph.D.

Field Coordinator: Kevin Kelley and Matthew Goehring

Senior Scientist: Frank Spurlock, Ph.D.

Laboratory Liaison: Carissa Ganapathy

Analytical Chemists: California Department of Food and Agriculture, Center for Analytical Chemistry. Staff Chemists

Questions concerning this monitoring project should be directed to Li-Ming (Lee) He at (916) 327-7479 or by email at lhe@cdpr.ca.gov or Michael Enslinger at (916) 324-4186 or by email at menslinger@cdpr.ca.gov.

4 STUDY PLAN

4.1 Sampling

Monitoring will be conducted in four large urban areas in northern and southern California, including the greater Sacramento area, the greater San Francisco Bay area, the greater Los Angeles area, and the greater San Diego area. This project will be coordinated or leveraged with existing DPR-funded investigations including those monitoring efforts already underway through UC Riverside (Gan et al., 2007; Greenberg, 2007), UC Davis (Gan et al., 2007), and UC Cooperative Extension, or established relationships with Regional Water Quality Control Boards, Southern California Coastal Water Research Project, and California stormwater management agencies/organizations.

All monitoring sites will be established in each area by cooperating with local water quality agencies using currently available information including land use data, catchment area, representativeness, historical water quality data, safety and accessibility, and site reconnaissance.

There will be four or seven monitoring sites in each area (Appendices 1 and 2). The monitoring sites are categorized to stormdrain discharges and receiving waters. Stormdrain discharges refer to stormdrain outfalls, which represent effluents that may contain pesticides. Such samples would represent worst-case conditions (i.e., highest possible pesticide concentrations). If the compounds are not detected in the discharges themselves, it is unlikely that toxicity would remain after release to and dilution within a receiving water. Receiving waters refer to any waterbody that receives runoff discharges. Sampling at stormdrains and receiving waters is intended to identify potential pesticides contributed by each category of discharges, their concentration at the point of discharge, and differences between dry- and wet-flow conditions.

Sampling will take place in both dry flow (baseflow) and wet flow (rainstorm) conditions during the year of monitoring. Three dry- and three wet-flow samplings will be conducted at each sampling site in each area. The dry-flow sampling will be conducted during the dry weather season (May through September). The wet-flow sampling will include the time of the first flush of the first storm (2008-2009 water year) and the first flushes of two other rainstorm events (2007-2008 water year).

The composite sampling method will be used during a storm event wherever feasible. Safety for equipment setup and/or limitation of appropriate field equipment availability may prevent the use of composite sampling. In this case, grab sampling will be used. Composite samples will be collected using polyethylene carboys and subsequently split into amber glass bottles. Grab samples will be collected into 1-L amber glass bottles. Samples will be collected as close to center channel as possible. Excluding receiving water samples, all other discharges will be sampled just prior to the point of release to a receiving water to avoid the effect of dilution and mixing from other inputs upstream. Samples will be stored on ice (~4 °C) and transported to the designated laboratory for chemical analysis.

Sediment samples (0 to 3 samples) will be collected into glass jars near the location of a stormdrain outfall during the dry-flow season. Effluent quality is inherently variable, and it is possible that we could fail to obtain water samples from one or more of the discharges at times of significant pesticide input to a receiving water. Since sediments serve as an integrator of long-term pesticide exposure, it is likely that any significant and persistent discharge would potentially produce elevated pesticide concentrations in nearby sediments.

4.2 Field Measurement

Physicochemical properties of water will be determined using a portable multi-parameter water quality monitoring system. Water parameters measured *in situ* at each site during each sampling event will include pH, temperature, conductivity, turbidity, and dissolved oxygen. Salinity and total dissolved solids will be estimated from conductivity. These data will be used to supplement and help interpret other data including pesticides.

To provide for interpretation of the monitoring data in the context of the hydrological conditions present during monitoring events, rainfall amount and duration will be recorded from the nearest reliable rain gauge for each monitoring event. Stormdrain discharge or stream flow rates will be measured to characterize the flow regime in effect and to estimate the total loading of target pesticides. Flow will be measured using a deployable flowmeter or a portable flow probe. Flow data may also be obtained from an existing stream gauge station where available.

All field measured data and other information will be recorded to a standardized field datasheet.

4.3 Laboratory Analysis

Pesticide analytes were determined based on current urban pesticide use or sale information (Flint, 2003; Kreidich et al., 2005; Moran, 2007), historical surface water monitoring data (CDPR, 2007), preliminary results from urban monitoring in progress (Gan et al., 2007), and aquatic toxicity (USEPA, 2007a). The current proposed list of analytes includes the following pesticide groups:

- Insecticides - Pyrethroids (in sediment only), carbamates, organophosphates, and fipronil & metabolites;
- Herbicides - Dinitroanilines, photosynthetic inhibitors (e.g., triazines), and auxin transport inhibitors (e.g., phenoxy).

The full list of pesticides, including specific analyte names, is available in Appendix 3. These pesticides will be analyzed using currently available analytical methods. All samples will be analyzed by the California Department of Food and Agriculture's Center for Analytical Chemistry, which is located in Sacramento, California. Analytical methods, analytes, method detection limits, and reporting limits for this study are also given in Appendix 3. Details of the chemical analysis methods and method detection/reporting limits for newly developed methods will be updated when available. The list of analytes may be updated or modified at any time as new information becomes available or due to changes in available resources.

We will also measure total suspended solids (TSS) in water samples and total organic carbon (TOC) in both water and sediment samples using standard methods.

5 QUALITY ASSURANCE

Quality assurance and quality control (QA/QC) will be performed in accordance with Standard Operating Procedure QAQC001.00 (Segawa, 1995), which includes criteria for QC samples. Ten percent of the total number of samples will be submitted as field blanks, blind spikes, or field duplicates. In addition, QA/QC procedures developed by US EPA (1998) and for SWAMP by SWRCB (Puckett, 2002) will be consulted where applicable.

6 DATA MANAGEMENT

All data generated by this project will be maintained in the DPR facility. DPR staff (its contractors if any) will be responsible for collection of samples and field data. The project leaders will be responsible for managing all data including field information, field measurements, laboratory analytical data using an existing database or developing a new database for the project.

7 DATA ANALYSIS AND REPORTING

Water quality will be assessed based on comparison of analytical data with water quality objectives or benchmarks indicating potential for biological effects. Water quality objectives established by US EPA or California will be used for comparison to measured concentrations whenever available. The benchmarks for pesticides developed by US EPA will be used when water quality standards are not available (USEPA, 2007c). Other aquatic effects data may also be used for comparison to measured concentrations, including LC₅₀ or EC₅₀ (e.g., Table 1) (US EPA, 2007a). The content of pyrethroids in sediment will be compared with sediment toxicity data (Table 2) developed by Amweg et al. (2005). Data collected from this project will be analyzed to determine summary statistics of concentrations and detection frequencies, and

statistical analysis to determine the effect of different factors such as runoff type (e.g. stormwater vs. baseflow), season or geographic location may be conducted. Final project report(s) will be prepared by project leader(s) in coordination with other project team members.

Table 1. LC₅₀ or EC₅₀ of pesticides for select standard aquatic organisms (USEPA, 2007b) derived from US EPA Pesticide ECOTOX Database (2007a)

Insecticides LC ₅₀ in µg/L				Herbicides EC ₅₀ in µg/L ³	
Active Ingredients	A. bahia ¹	Daphnia ²	C. dubia ²	Active Ingredients	Green Algae
Beta-Cyfluthrin	0.00242	0.17	0.14	Atrazine	4.3
Bifenthrin	0.00397	0.32	0.07	Bromacil	6.8
Carbaryl	5.7	1.25	11.6	Dicamba	61
Chlorpyrifos	0.035	0.21	0.058	Dichlobenil	2700
Cyfluthrin	0.00242	0.17	0.14	Diuron	1.3
Cypermethrin	0.0047	0.36	0.889	Glyphosate	3530
Deltamethrin	0.0017	0.037	NA	Linuron	38.8
Diazinon	4.2	0.65	0.21	Oryzalin	24
Esfenvalerate	NA	0.27	0.28	Simazine	82
Fipronil	0.14	15.6	17.5		
Imidacloprid	38	NA	NA		
Lambda-Cyhalothrin	0.0041	1.04	0.3		
Malathion	2.2	1.6	1.14		
Permethrin	0.02	1.25	0.55		
Tralomethrin	NA	0.15	0.26		

¹ *Americamysis bahia* (*Mysiopsis bahia*) with the common name mysid shrimp. 96 h in marine water.

² *Daphnia* and *Ceriodaphnia dubia*, 48 h in freshwater.

³ 96-120 h.

NA - not available.

Table 2. Sediment toxicity of *Hyaella azteca* to pyrethroids (10-d LC₅₀ in µg/g sediment organic carbon) (Amweg et al., 2005)

Bifenthrin	Cyfluthrin	Deltamethrin	Esfenvalerate	Lambda-cyhalothrin	Permethrin
0.18	1.08	0.79	0.89	0.45	4.88

8 PROJECT SCHEDULE

This one-year project represents the Phase I of a multi-year monitoring program that includes urban pesticide use screening monitoring of water quality and focused investigations on potential source(s) and mitigation measures for source reduction. The schedule of major activities associated with this project is shown in the following table (Table 3). Some of the tasks listed in the table will be accomplished as scheduled, but they can be modified and improved as more information is obtained from ongoing monitoring, for example, sampling protocol, chemical analysis, or data quality criteria may be modified as needed.

Table 3. Project Schedule

Tasks	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC	JAN
Identify sampling locations	██████████		██████████												
Develop field datasheet/contract	██████████		██████████												
Develop detailed sampling protocol			██████████												
Conduct sampling and measurement					██████████										
Perform laboratory analysis			██████████												
Data Management			██████████												
Perform data analysis and interpretation						██████████									
Final Report														██████████	

9 BUDGET

Provided below is the proposed budget for the project. Since the project will be carried out in the 2008 calendar year, the total cost has been proportioned in FY07-08 and FY08-09 as shown below.

FY07-08

Sample	Samples	Price/Sample	Cost
Stormdrain water	54	3715	200610
Receiving water	21	3715	78015
Sediment	14	800	11200
Field duplicate	8	3715	29720
Field Blank	4	3715	14860
Total	101		334,405*

*See Appendix 1 for details.

The cost for continuing QC samples is not included.

FY08-09

Sample	Samples	Price/Sample	Cost
Stormdrain water	54	3715	200610
Receiving water	21	3715	78015
Sediment	0	800	0
Field duplicate	4	3715	14860
Field Blank	0	3715	0
Total	79		293,485*

*See Appendix 2 for details.

The cost for continuing QC samples is not included.

10 REFERENCES

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Appendix 1. FY07-08 Monitoring sites, pesticides screening, and analytical cost estimate for urban pesticides use and water quality

Sample Type	#Sampling Sites				# Sampling Events		Analytes									Price/Sample	Cost
	Sacramento Area	Bay Area	LA Area	San Diego Area	Rainstorm	Baseflow	# Samples	Fipronil (1)	Pyrethroids(2)	Carbamates(3)	Organophosphates (4)	photosynthetic inhibitors (5)	Dinitroanilines (6)	Auxin transport inhibitors (7)	TSS and TOC (8)		
Stormdrain Discharges	3	5	5	5	1	2	54	\$ 500	\$ -	\$ 800	\$ 320	\$ 720	\$ 800	\$ 575	\$ -	\$ 3,715	\$ 200,610
Receiving Water	1	2	2	2	1	2	21	\$ 500	\$ -	\$ 800	\$ 320	\$ 720	\$ 800	\$ 575	\$ -	\$ 3,715	\$ 78,015
Sediment (same sites as stormdrain discharges) (9)	2	4	4	4	0	1	14	\$ -	\$ 800	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ 800	\$ 11,200
Field Duplicate (water only)	1	1	1	1	1	1	8	\$ 500	\$ -	\$ 800	\$ 320	\$ 720	\$ 800	\$ 575	\$ -	\$ 3,715	\$ 29,720
Field Blank (water only)	1	1	1	1	0	1	4	\$ 500	\$ -	\$ 800	\$ 320	\$ 720	\$ 800	\$ 575	\$ -	\$ 3,715	\$ 14,860
Total	4	7	7	7			101									\$ 15,660	\$ 334,405

(1) Analytes include degradates.

(2) Sediment sampling for 1 or 2 sites in each region.

(3) Carbaryl included.

(4) Malathion included. Only 2 sites in each region. 1/2 the price was used in estimating the cost.

(5) Atrazine, simazine, diuron, bromacil included.

(6) Oryzalin and prodiamine included.

(7) The phenoxy screen includes dicamba.

(8) To be analyzed in house or using a contract lab.

(9) Sediment sampling and analysis are limited to pyrethroids only.

Appendix 2. FY08-09 Monitoring sites, pesticides screening, and analytical cost estimate for urban pesticides use and water quality

Sample Type	# Sampling Sites				# Sampling Events		Analytes								Price/Sample	Cost	
	Sacramento Area	Bay Area	LA Area	San Diego Area	Rainstorm	Baseflow	# Samples	Fipronil (1)	Pyrethroids(2)	Carbamates(3)	Organophosphates (4)	photosynthetic inhibitors (5)	Dinitroanilines (6)	Auxin transport inhibitors (7)			TSS and TOC (8)
Stormdrain Discharges	3	5	5	5	2	1	54	\$ 500	\$ -	\$ 800	\$ 400	\$ 720	\$ 800	\$ 575	\$ -	\$ 3,795	\$ 200,610
Receiving Water	1	2	2	2	2	1	21	\$ 500	\$ -	\$ 800	\$ 400	\$ 720	\$ 800	\$ 575	\$ -	\$ 3,795	\$ 78,015
Sediment (same sites as stormdrain discharges) (9)	0	0	0	0	0	0	0	\$ -	\$ 800	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ 800	\$ -
Field Duplicate (water only)	1	1	1	1	1	0	4	\$ 500	\$ -	\$ 800	\$ 400	\$ 720	\$ 800	\$ 575	\$ -	\$ 3,795	\$ 14,860
Field Blank (water only)	1	1	1	1	0	0	0	\$ 500	\$ -	\$ 800	\$ 400	\$ 720	\$ 800	\$ 575	\$ -	\$ 3,795	\$ -
Total	4	7	7	7			79									\$ 15,980	\$ 293,485

(1) Analytes include degradates.

(2) Sediment sampling for 1 or 2 sites in each region.

(3) Carbaryl included.

(4) Malathion included. Only 2 sites in each region. 1/2 the price was used in estimating the cost.

(5) Atrazine, simazine, diuron, bromacil included.

(6) Oryzalin and prodiamine included.

(7) The phenoxy screen includes dicamba.

(8) To be analyzed in house or using a contract lab.

(9) Sediment sampling and analysis are limited to pyrethroids only.

Appendix 3. Analytes, method detection limit, and reporting limit[‡]

Matrix: Water

Analyte Group: Carbamates (CB)

Method: HPLC

Compound	Method Detection Limit	Reporting Limit
	(µg/L)	(µg/L)
Aldicarb Sulfoxide	0.011	0.05
Aldicarb Sulfone	0.02	0.05
Methomyl	0.01	0.05
3-OH Carbofuran	0.011	0.05
Aldicarb	0.01	0.05
Carbofuran	0.01	0.05
Carbaryl	0.011	0.05
Oxymyl	0.02	0.05
Methiocarb	0.016	0.05

[‡] Listed in these tables are analytes included in the long screening list of pesticides, some of which may not be used in urban areas. Some of the pesticides presented in the table may not be analyzed when a short screening list is used for chemical analysis due to higher costs for analyzing a full list of pesticides.

Matrix: Water

Analyte Group: Dinitroanilines (DN)

Method: GC/TQMS

Compound	Method Detection Limit	Reporting Limit
	($\mu\text{g/L}$)	($\mu\text{g/L}$)
Benfluralin	0.012	0.05
Ethalfuralin	0.015	0.05
Oryzalin (LCQ)	0.0048	0.05
Pendimethalin	0.012	0.05
Prodiamine	0.012	0.05
Trifluralin	0.014	0.05
Oxyfluorfen	0.01	0.05

Matrix: Water

Analyte Group: Fipronil & Metabolites (FP)

Method: GC/MSD in the SIM mode

Compound	Method Detection Limit	Reporting Limit
	($\mu\text{g/L}$)	($\mu\text{g/L}$)
Fipronil	0.004	0.05
Desulfinyl fipronil	0.003	0.05
Desulfinyl fipronil amide	0.005	0.05
Fipronil sulfide	0.003	0.05
Fipronil sulfone	0.005	0.05
Fipronil amide	0.005	0.05

Matrix: Water

Analyte Group: Organophosphates (OP)

Method: GC/FPD

Compound	Method Detection Limit	Reporting Limit
	($\mu\text{g/L}$)	($\mu\text{g/L}$)
Ethoprop	0.0098	0.05
Diazinon	0.011	0.04
Disulfoton	0.0093	0.04
Chlorpyrifos	0.0109	0.04
Malathion	0.0117	0.04
Methidathion	0.0111	0.05
Fenamiphos	0.0125	0.05
Azinphos methyl	0.0099	0.05
Dichlorvos	0.0098	0.05
Phorate	0.0083	0.05
Fonofos	0.0080	0.04
Dimethoate	0.0079	0.04
Methyl Parathion	0.0080	0.03
Tribufos	0.0142	0.05
Profenofos	0.0114	0.05
GC/MS	($\mu\text{g/L}$)	($\mu\text{g/L}$)
Diazinon	0.0012	0.01
Chlorpyrifos	0.0079	0.01
<i>*in clean American River water</i>		

Matrix: Water

Analyte Group: Auxin transport inhibitors [Phenoxy (PX)]

Method: GC/MS

Compound	Method Detection Limit	Reporting Limit
	($\mu\text{g/L}$)	($\mu\text{g/L}$)
Dicamba	0.064	0.1
2,4-D	0.064	0.1
MCPA	0.064	0.1
Triclopyr	0.064	0.1

Matrix: Sediment

Analyte Group: Pyrethroids (PY)

Method: GC/ECD

Compound	Method Detection Limit	Reporting Limit
	($\mu\text{g}/\text{kg}$)	($\mu\text{g}/\text{kg}$)
Bifenthrin	0.108	1.0
Fenopropathrin	0.109	1.0
Lambda-cyhalothrin epimer	0.117	1.0
Lambda-cyhalothrin	0.115	1.0
Permethrin cis	0.116	1.0
Permethrin trans	0.135	1.0
Cyfluthrin	0.183	1.0
Cypermethrin	0.107	1.0
Fenvalerate/esfenvalerate	0.143	1.0
Deltamethrin	0.0661	1.0
Resmethrin (GC/MSD)	0.870	1.5

Matrix: Water

Analyte Group: Photosynthetic inhibitors [Triazines (TR)]

Method: LC/MS/MS

Compound	Method Detection Limit	Reporting Limit
	($\mu\text{g/L}$)	($\mu\text{g/L}$)
Atrazine	0.020	0.05
Simazine	0.013	0.05
Diuron	0.022	0.05
Prometon	0.016	0.05
Bromacil	0.031	0.05
Prometryn	0.016	0.05
Hexazinone	0.040	0.05
Metribuzin	0.025	0.05
Norflurazon	0.019	0.05
DEA	0.01	0.05
ACET	0.03	0.05
DACT	0.016	0.05