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Mail Stop 412

June 1, 2015

To: GS-W-ALL

From: Donna N. Myers, Chief, Office of Water Quality

Subject: Approval of New USGS Method (O-2437-15) Determination of Pesticides and Pesticide Degradates by Direct Aqueous Injection (DAI) LC-MS/MS

The Office of Water Quality (OWQ) has approved a new filtered-water analytical method, O-2437-15, National Water Quality Laboratory (NWQL) laboratory schedule (LS) 2437, for the determination of 229 current use pesticides and pesticide degradates (see attached table 1).

The U.S. Geological Survey (USGS) developed this method to support studies of selected pesticide compounds (113 pesticides and 116 pesticide degradates) in a variety of filtered water-matrix types including groundwater, surface water, and drinking water. Field filtration of 10-milliliter samples for LS2437 is required as described in National Field Manual for the Collection of Water Quality Data (NFM), Chapter 5.2.2.B (Sandstrom and Wilde, 2014).

The pesticide compounds in the new method were considered high priority for method development and future monitoring studies in the USGS because of potential for occurrence and persistence in streams and groundwater, and toxicity to humans and aquatic organisms. The high-priority pesticide compounds included 78 new compounds not previously monitored in USGS analytical methods for determination of pesticides in water as well as many pesticide compounds determined infrequently or in small research studies. In addition, 96 pesticide compounds are included in commonly used USGS analytical methods that will be useful for long-term monitoring studies. A unique feature of the new method is the relatively large number of pesticide degradates included that provides the ability to more thoroughly study the fate of pesticides in the environment, as well as evaluate the toxicity and exposure effects.

All method analytes are determined without sample pre-concentration or isolation using direct injection of an aliquot of the filtered sample onto a liquid chromatograph-tandem mass spectrometer (LC-MS/MS). Pesticide compounds are determined in the samples in two groups, one by the MS/MS operated in positive electrospray ionization (ESI+) mode, the other in negative electrospray ionization (ESI-) mode. For each group an aliquot of the sample (100 μ L) is injected onto a liquid chromatographic column (C18 phase), and the analytes are separated using binary mobile phase of formic acid/ammonium formate-methanol for ESI+ mode or acetic acid-acetonitrile for ESI- mode. A dynamic multiple reaction monitoring (MRM) method is used on the MS/MS to collect specific quantification and qualifier ions for each analyte. Analyte identification is based on agreement between known standards with respect to chromatographic retention time and the ratio of the two characteristic MRMs. The concentration of each identified component is determined using the internal standard technique.

The Techniques and Method report describes the analytical method and presents characteristics of the method validation including bias and variability in different matrices, method detection levels (MDLs), and holding-time studies. The report also describes a comparison of the new DAI LC-MS/MS analytical method with two existing NWQL analytical methods (LS2033 and LS2060) for determination of pesticides. The results provided a characterization of the performance of the new method during implementation in routine production, including bias and variability of analytes in LS2437 over a range of environmental matrices and concentrations and in comparison to LS2033 and LS2060.

The MDLs were determined in reagent water using the U.S. Environmental Protection Agency (EPA) procedure (40 CFR part 142, appendix B). The MDLs range from 1 to 103 nanograms per liter (ng/L) for 183 analytes analyzed in the ESI positive mode, and from 2 to 106 ng/L for 42 analytes analyzed in the ESI negative mode. Four analytes had MDLs between 100 and 250 ng/L. After the method is implemented into routine productions the MDLs and reporting levels will be updated to reflect long-term method performance. The stability studies in reagent water demonstrated that the majority of the pesticide compounds were stable after 14 days of storage at 4 degrees Celsius, so these were selected as the practical holding time and storage temperature for routine sample processing.

Field samples are collected using USGS protocols for organic contaminants according to procedures in the NFM, Chapter 4 (U.S. Geological Survey, 2006). Samples are filtered at the field site using small disposable 25-mm-diameter syringe-tip filters (Q762FLD) (Sandstrom and Wilde, 2014) and samples are collected and stored in 20-mL vials (PEST bottle type, Q21FLD) until analysis.

Because there is a large backlog of samples requiring data analysis for LS2437, the NWQL plans to manage new requests for LS2437 by the custom proposal process during FY 2015. Please contact labhelp (labhelp@usgs.gov) to request sample analysis using LS2437 for samples in FY 2015.

This method approval process follows the technical procedures specified in OWQ Technical Memorandum 04.01 and the method is described in a USGS Techniques and Methods Report:

Suggested Citation:

Sandstrom, M.W., Kanagy, L.K., Anderson, C.E., and Kanagy, C.J., in press, Determination of pesticides and pesticide degradates in filtered water by direct aqueous injection liquid chromatography tandem mass spectrometry: U.S. Geological Survey Techniques and Methods book 5, chap. B11

The report will be made available through the USGS Publications Warehouse.

If you have any questions about the new analytical method, or would like a copy of the report, when it is available, please contact Mark Sandstrom (sandstro@usgs.gov, 303-236-3943) or Jeff McCoy (jefmccoy@usgs.gov, 303-236-3940).

If you have questions about the method approval process, please contact Donna Myers (dnmyers@usgs.gov, 703-648-5012).

References cited:

U.S. Geological Survey, 2006, Collection of water samples (ver. 2.0): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A4, September 2006, accessed [May 29, 2015], available at <http://pubs.water.usgs.gov/twri9A4/>

Sandstrom, M.W., and Wilde, F.D., 2014, Syringe-filter procedure for processing samples for analysis of organic compounds by DAI LC-MS/MS: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A5, Section 5.2.2.B., available at <http://water.usgs.gov/owq/FieldManual/chapter5/pdf/5.2.2.B.pdf> .

Table 1. Information about pesticide compounds in direct aqueous-injection liquid chromatography-tandem mass spectrometry (LC-MS/MS) method; (USGS method O-2437-15; NWQL laboratory schedule 2437.

[USGS, U.S. Geological Survey; lab code 9030, positive electrospray ionization, lab code 9031, negative electrospray ionization; CASRN, chemical abstracts registry number; pesticide group, pesticide active ingredient for degradates; -, data not available; na, not applicable; IS, Internal standard]

Pesticide compound	USGS Parameter code	Lab Code	CASRN	Analytical use	Analytical method group	Pesticide group	New USGS analyte
1H-1,2,4-Triazole	68498	9030	116421-29-5	Degradate	fungicide	Propiconazole	Yes
2-(1-hydroxyethyl)-6-methylaniline (HEMA)	68611	9030	196611-19-5	Degradate	acetanilide & amide	Acetochlor/Metolachlor	Yes
2-(2-ethyl-6-methyl- phenyl)aminopropan-1-ol	68595	9030	61520-53-4	Degradate	acetanilide & amide	Metolachlor	No
2-Amino-N-isopropylbenzamide	68503	9030	30391-89-0	Degradate	acid	Bentazone	No
2-Aminobenzimidazole	68502	9030	934-32-7	Degradate	fungicide	Benomyl	Yes
2-Chloro-2,6-diethylacetanilide	68525	9030	6967-29-9	Degradate	acetanilide & amide	Alachlor	No
2-Chloro-N-(2-ethyl-6- methylphenyl)acetamide	68521	9030	32428-71-0	Degradate	acetanilide & amide	Acetochlor/Metolachlor	No
2-Hydroxy-4-isopropylamino-6-amino- s-triazine	68659	9030	19988-24-0	Degradate	triazine	Atrazine	No
2-Hydroxy-6-ethylamino-4-amino-s- triazine	68656	9030	7313-54-4	Degradate	triazine	Atrazine	No
2-Hydroxyatrazine	68660	9030	2163-68-0	Degradate	triazine	Atrazine	No
2,3,3-trichloroprop-2-ene-1-SA	68691	9031	65600-62-6	Degradate	carbamate & thiocarbamate	Triallate	Yes
2,4-D	68500	9031	94-75-7	Pesticide	acid	2,4-D	No
3-Hydroxycarbofuran	68508	9030	16655-82-6	Degradate	carbamate & thiocarbamate	Carbofuran	No
3-Phenoxybenzoic acid	68873	9031	3739-38-6	Degradate	pyrethroid & phenylpyrazine	Permethrin	Yes
3,4-Dichlorophenylurea	68226	9030	2327-02-8	Degradate	sulfonylurea & urea	Diuron	No
4-(Hydroxymethyl)pendimethalin	68511	9031	56750-76-6	Degradate	miscellaneous	Pendimethalin	No
4-Chlorobenzylmethyl sulfoxide	68514	9030	-	Degradate	carbamate & thiocarbamate	thiobencarb	Yes
4-Hydroxy molinate	68515	9030	66747-12-4	Degradate	carbamate & thiocarbamate	Molinate	Yes
4-Hydroxy-tert-fluometuron	68619	9030	-	Degradate	sulfonylurea & urea	Fluometuron	Yes
4-Hydroxychlorothalonil	68336	9031	28343-61-5	Degradate	fungicide	Chlorothalonil	No
4-Hydroxyhexazinone A	68517	9030	72576-13-7	Degradate	triazine	Hexazinone	Yes
Acephate	68519	9030	30560-19-1	Pesticide	organophosphate	Acephate	Yes
Acetochlor	68520	9030	34256-82-1	Pesticide	acetanilide & amide	Acetochlor	No

Acetochlor OA	68522	9031	194992-44-4	Degradate	acetanilide & amide	Acetochlor	No
Acetochlor SA	68523	9031	187022-11-3	Degradate	acetanilide & amide	Acetochlor	No
Acetochlor SAA	68524	9031	-	Degradate	acetanilide & amide	Acetochlor	No
Alachlor	65064	9030	15972-60-8	Pesticide	acetanilide & amide	Alachlor	No
Alachlor OA	68526	9031	171262-17-2	Degradate	acetanilide & amide	Alachlor	No
Alachlor SA	68871	9031	142363-53-9	Degradate	acetanilide & amide	Alachlor	No
Alachlor SAA	68527	9031	494847-34-1	Degradate	acetanilide & amide	Alachlor	No
Aldicarb	68528	9030	116-06-3	Pesticide	carbamate & thiocarbamate	Aldicarb	No
Aldicarb sulfone	68529	9030	1646-88-4	Degradate	carbamate & thiocarbamate	Aldicarb	No
Aldicarb sulfoxide	68530	9030	1646-87-3	Degradate	carbamate & thiocarbamate	Aldicarb	No
Ametryn	68533	9030	834-12-8	Pesticide	triazine	Ametryn	No
Asulam	68536	9030	3337-71-1	Pesticide	carbamate & thiocarbamate	Asulam	Yes
Atrazine	65065	9030	1912-24-9	Pesticide	triazine	Atrazine	No
Azinphos-methyl	65066	9030	86-50-0	Pesticide	organophosphate	Azinphos-methyl	No
Azinphos-methyl oxon	68211	9030	961-22-8	Degradate	organophosphate	Azinphos-methyl	No
Azoxystrobin	66589	9030	131860-33-8	Pesticide	fungicide	Azoxystrobin	No
Bentazon	68538	9031	25057-89-0	Pesticide	acid	Bentazone	No
Bifenthrin	65067	9030	82657-04-3	Pesticide	pyrethroid & phenylpyrazine	Bifenthrin	No
Bromacil	68542	9030	314-40-9	Pesticide	miscellaneous	Bromacil	No
Bromoxynil	68543	9031	1689-84-5	Pesticide	acid	Bromoxynil	No
Butralin	68545	9030	33629-47-9	Pesticide	miscellaneous	Butralin	Yes
Butylate	65068	9030	2008-41-5	Pesticide	carbamate & thiocarbamate	Butylate	No
Carbaryl	65069	9030	63-25-2	Pesticide	carbamate & thiocarbamate	Carbaryl	No
Carbendazim	68548	9030	10605-21-7	Degradate	fungicide	Benomyl	Yes
Carbofuran	65070	9030	1563-66-2	Pesticide	carbamate & thiocarbamate	Carbofuran	No
Carboxy molinate	68549	9030	66747-13-5	Degradate	carbamate & thiocarbamate	Molinate	Yes
Chlorimuron-ethyl	68872	9030	90982-32-4	Pesticide	sulfonylurea & urea	Chlorimuron-ethyl	No
Chlorosulfonamide acid	68551	9031	130-45-0	Degradate	sulfonylurea & urea	Halosulfuron-methyl	Yes
Chlorpyrifos	65072	9030	2921-88-2	Pesticide	organophosphate	Chlorpyrifos	No
Chlorpyrifos oxon	68216	9030	5598-15-2	Degradate	organophosphate	Chlorpyrifos	No
Chlorsulfuron	61678	9030	64902-72-3	Pesticide	sulfonylurea & urea	Chlorsulfuron	No

cis-Cyhalothric acid	68553	9031	68127-59-3	Degradate	pyrethroid & phenylpyrazine	Bifenthrin; lambda-cyhalothrin; Tefluthrin	Yes
cis-Permethrin	68769	9030	61949-76-6	Pesticide	pyrethroid & phenylpyrazine	Permethrin	No
Cyanazine	66592	9030	21725-46-2	Pesticide	triazine	Cyanazine	No
Chlorthal-monomethyl	68560	9031	887-54-7	Degradate	acid	Dacthal	No
Dechlorofipronil	68561	9031	-	Degradate	pyrethroid & phenylpyrazine	Fipronil	Yes
Dechlorometolachlor	68562	9030	126605-22-9	Degradate	acetanilide & amide	Metolachlor	No
Deethylatrazine	68552	9030	6190-65-4	Degradate	triazine	Atrazine	No
Desiodo flubendiamide	68563	9030	-	Degradate	miscellaneous	Flubendiamide	Yes
Deisopropyl prometryn	68564	9030	4147-57-3	Degradate	triazine	Prometryn	No
Deisopropylatrazine	68550	9030	1007-28-9	Degradate	triazine	Atrazine	No
Demethyl fluometuron	68591	9030	3032-40-4	Degradate	sulfonylurea & urea	Fluometuron	No
Demethyl hexazinone B	68566	9030	56611-54-2	Degradate	triazine	Hexazinone	Yes
Demethyl norflurazon	68567	9030	23576-24-1	Degradate	miscellaneous	Norflurazon	No
Desulfinylfipronil	66607	9031	205650-65-3	Degradate	pyrethroid & phenylpyrazine	Fipronil	No
Desulfinylfipronil amide	68570	9031	-	Degradate	pyrethroid & phenylpyrazine	Fipronil	No
Diazinon	65078	9030	333-41-5	Pesticide	organophosphate	Diazinon	No
Diazinon oxon	68236	9030	962-58-3	Degradate	organophosphate	Diazinon	No
Dicamba	68571	9031	1918-00-9	Pesticide	acid	Dicamba	No
Dichlorvos	68572	9030	62-73-7	Degradate	organophosphate	Naled	No
Dicrotophos	68573	9030	141-66-2	Pesticide	organophosphate	Dicrotophos	No
Didealkylatrazine	68547	9030	3397-62-4	Degradate	triazine	Atrazine	No
Diflubenzuron	68576	9030	35367-38-5	Pesticide	sulfonylurea & urea	Diflubenzuron	Yes
Diflufenzopyr	68577	9031	109293-97-2	Pesticide	sulfonylurea & urea	Diflufenzopyr	Yes
Diketonitrile isoxaflutole	68578	9031	143701-75-1	Degradate	miscellaneous	Isoxaflutole	Yes
Dimethenamid	68580	9030	87674-68-8	Pesticide	acetanilide & amide	Dimethenamid	No
Dimethenamid OA	68581	9031	-	Degradate	acetanilide & amide	Dimethenamid	No
Dimethenamid SA	68582	9031	205939-58-8	Degradate	acetanilide & amide	Dimethenamid	No
Dimethenamid SAA	68583	9031	-	Degradate	acetanilide & amide	Dimethenamid	Yes
Dimethoate	66596	9030	60-51-5	Pesticide	organophosphate	Dimethoate	No
Dimethoate oxon	68661	9030	1113-02-6	Degradate	organophosphate	Dimethoate	Yes
Disulfoton	67595	9030	298-04-4	Pesticide	organophosphate	Disulfoton	No
Disulfoton oxon	68586	9030	126-75-0	Degradate	organophosphate	Disulfoton	Yes
Disulfoton oxon sulfone	68588	9030	2496-91-5	Degradate	organophosphate	Disulfoton	Yes
Disulfoton oxon sulfoxide	68587	9030	2496-92-6	Degradate	organophosphate	Disulfoton	Yes
Disulfoton sulfone	68589	9030	2497-06-5	Degradate	organophosphate	Disulfoton	No

Disulfoton sulfoxide	68590	9030	2497-07-6	Degradate	organophosphate	Disulfoton	No
Diuron	66598	9030	330-54-1	Pesticide	sulfonylurea & urea	Diuron	No
EPTC	65080	9030	759-94-4	Pesticide	carbamate & thiocarbamate	EPTC	No
EPTC R248722	68594	9030	-	Degradate	carbamate & thiocarbamate	EPTC	Yes
Ethoprop	68596	9030	13194-48-4	Pesticide	organophosphate	Ethoprop	No
Ettoxazole	68598	9030	153233-91-1	Pesticide	miscellaneous	Ettoxazole	Yes
Famoxadone	67609	9030	131807-57-3	Pesticide	fungicide	Famoxadone	No
Fenamiphos	68599	9030	22224-92-6	Pesticide	organophosphate	Fenamiphos	No
Fenamiphos sulfone	68600	9030	31972-44-8	Degradate	organophosphate	Fenamiphos	No
Fenamiphos sulfoxide	68601	9030	31972-43-7	Degradate	organophosphate	Fenamiphos	No
Fenbutatin oxide	68602	9030	13356-08-6	Pesticide	miscellaneous	Fenbutatin oxide	Yes
Fentin	68603	9030	668-34-8	Pesticide	fungicide	Fentin	Yes
Fipronil	66604	9031	120068-37-3	Pesticide	pyrethroid & phenylpyrazine	Fipronil	No
Fipronil amide	68604	9031	-	Degradate	pyrethroid & phenylpyrazine	Fipronil	Yes
Fipronil sulfide	66610	9031	120067-83-6	Degradate	pyrethroid & phenylpyrazine	Fipronil	No
Fipronil sulfonate	68605	9031	209248-72-6	Degradate	pyrethroid & phenylpyrazine	Fipronil	Yes
Fipronil sulfone	66613	9031	120068-36-2	Degradate	pyrethroid & phenylpyrazine	Fipronil	No
Flubendiamide	68606	9031	272451-65-7	Pesticide	miscellaneous	Flubendiamide	Yes
Flumetsulam	61679	9030	98967-40-9	Pesticide	miscellaneous	Flumetsulam	No
Fluometuron	68608	9030	2164-17-2	Pesticide	sulfonylurea & urea	Fluometuron	No
Fonofos	65084	9030	944-22-9	Pesticide	organophosphate	Fonofos	No
Halosulfuron-methyl	61680	9030	100784-20-1	Pesticide	sulfonylurea & urea	Halosulfuron-methyl	No
Hexazinone	65085	9030	51235-04-2	Pesticide	triazine	Hexazinone	No
Hexazinone TP C	68612	9030	72585-88-7	Degradate	triazine	Hexazinone	Yes
Hexazinone TP D	68613	9031	30243-77-7	Degradate	triazine	Hexazinone	Yes
Hexazinone TP E	68614	9031	72576-14-8	Degradate	triazine	Hexazinone	Yes
Hexazinone TP F	68574	9030	-	Degradate	triazine	Hexazinone	Yes
Hexazinone TP G	68713	9030	-	Degradate	triazine	Hexazinone	Yes
Hydroxy mono demethyl fluometuron	68617	9030	-	Degradate	sulfonylurea & urea	Fluometuron	Yes
Hydroxyacetochlor	68615	9030	60090-47-3	Degradate	acetanilide & amide	Acetochlor	No
Hydroxyalachlor	68616	9030	56681-55-1	Degradate	acetanilide & amide	Alachlor	No
Hydroxydiazinon	68618	9030	29820-16-4	Degradate	organophosphate	Diazinon	Yes
Hydroxyfluometuron	68620	9030	-	Degradate	sulfonylurea & urea	Fluometuron	Yes

Hydroxymetolachlor	68622	9030	131068-72-9	Degradate	acetanilide & amide	Metolachlor	No
Hydroxyphthalazinone	68623	9030	-	Degradate	sulfonylurea & urea	Di flufenzopyr	Yes
Hydroxysimazine	68624	9030	2599-11-3	Degradate	triazine	Simazine	No
Hydroxytebuthurion	68621	9030	59962-54-8	Degradate	sulfonylurea & urea	Tebuthiuron	Yes
Imazamox	68625	9030	114311-32-9	Pesticide	miscellaneous	Imazamox	Yes
Imazaquin	61682	9030	81335-37-7	Pesticide	miscellaneous	Imazaquin	No
Imazethapyr	61683	9030	81335-77-5	Pesticide	miscellaneous	Imazethapyr	No
Imidacloprid	68426	9030	138261-41-3	Pesticide	miscellaneous	Imidacloprid	No
Indoxacarb	68627	9030	173584-44-6	Pesticide	miscellaneous	Indoxacarb	Yes
Isoxaflutole	68632	9030	141112-29-0	Pesticide	miscellaneous	Isoxaflutole	No
Isoxaflutole acid RPA 203328	68633	9031	142994-06-7	Degradate	miscellaneous	Isoxaflutole	Yes
Kresoxim-methyl	67670	9030	143390-89-0	Pesticide	fungicide	Kresoxim-methyl	No
Lactofen	68638	9030	77501-63-4	Pesticide	miscellaneous	Lactofen	Yes
Linuron	68639	9030	330-55-2	Pesticide	sulfonylurea & urea	Linuron	No
Malaoxon	68240	9030	1634-78-2	Degradate	organophosphate	Malathion	No
Malathion	65087	9030	121-75-5	Pesticide	organophosphate	Malathion	No
MCPA	68641	9031	94-74-6	Pesticide	acid	MCPA	No
Metalaxyl	68437	9030	57837-19-1	Pesticide	fungicide	Metalaxyl	No
Metconazole	66620	9030	125116-23-6	Pesticide	fungicide	Metconazole	No
Methamidophos	68644	9030	10265-92-6	Pesticide	organophosphate	Methamidophos	Yes
Methidathion	65088	9030	950-37-8	Pesticide	organophosphate	Methidathion	No
Methomyl	68645	9030	16752-77-5	Pesticide	carbamate & thiocarbamate	Methomyl	No
Methomyl oxime	68646	9030	13749-94-5	Degradate	carbamate & thiocarbamate	Methomyl	No
Methoxyfenozide	68647	9030	161050-58-4	Pesticide	miscellaneous	Methoxyfenozide	Yes
Metolachlor	65090	9030	51218-45-2	Pesticide	acetanilide & amide	Metolachlor	No
Metolachlor hydroxy morpholinone	68649	9030	61520-54-5	Degradate	acetanilide & amide	Metolachlor	Yes
Metolachlor OA	68650	9031	152019-73-3	Degradate	acetanilide & amide	Metolachlor	No
Metolachlor SA	68651	9031	171118-09-5	Degradate	acetanilide & amide	Metolachlor	No
Metribuzin	68652	9030	21087-64-9	Pesticide	triazine	Metribuzin	No
Metribuzin DA	68568	9030	35045-02-4	Degradate	triazine	Metribuzin	Yes
Metribuzin DADK	68569	9031	52236-30-3	Degradate	triazine	Metribuzin	Yes
Metribuzin DK	68653	9031	56507-37-0	Degradate	triazine	Metribuzin	Yes
Molinate	65091	9030	2212-67-1	Pesticide	carbamate & thiocarbamate	Molinate	No
Myclobutanil	66632	9030	88671-89-0	Pesticide	fungicide	Myclobutanil	No
N-(3,4-Dichlorophenyl)-N-methylurea	68231	9030	3567-62-2	Degradate	sulfonylurea & urea	Diuron	No
Naled	68654	9030	300-76-5	Pesticide	organophosphate	Naled	No
Nicosulfuron	61685	9030	111991-09-4	Pesticide	sulfonylurea & urea	Nicosulfuron	No

Norflurazon	67685	9030	27314-13-2	Pesticide	miscellaneous	Norflurazon	No
Novaluron	68655	9030	116714-46-6	Pesticide	sulfonylurea & urea	Novaluron	Yes
O-Ethyl-O-methyl-S-propyl phosphorothioate	68597	9030	76960-87-7	Degradate	organophosphate	Ethoprop	No
O-Ethyl-S-methyl-S-propyl phosphorodithioate	68657	9030	76936-72-6	Degradate	organophosphate	Ethoprop	Yes
O-Ethyl-S-propyl phosphorothioate	68658	9030	31110-62-0	Degradate	organophosphate	Ethoprop	Yes
Orthosulfamuron	68662	9030	213464-77-8	Pesticide	sulfonylurea & urea	Orthosulfamuron	Yes
Oryzalin	68663	9031	19044-88-3	Pesticide	miscellaneous	Oryzalin	No
Oxamyl	68664	9030	23135-22-0	Pesticide	carbamate & thiocarbamate	Oxamyl	No
Oxamyl oxime	68665	9030	30558-43-1	Degradate	carbamate & thiocarbamate	Oxamyl	No
Oxyfluorfen	65093	9030	42874-03-3	Pesticide	miscellaneous	Oxyfluorfen	No
Paraoxon	68666	9030	311-45-5	Degradate	organophosphate	Parathion-ethyl	No
Paraoxon-methyl	68648	9030	950-35-6	Degradate	organophosphate	Parathion-methyl	No
Parathion-methyl	65089	9030	298-00-0	Pesticide	organophosphate	Parathion-methyl	No
Pendimethalin	65098	9030	40487-42-1	Pesticide	miscellaneous	Pendimethalin	No
Phorate	68668	9030	298-02-2	Pesticide	organophosphate	Phorate	No
Phorate oxon	68669	9030	2600-69-3	Degradate	organophosphate	Phorate	No
Phorate oxon sulfone	68670	9030	2588-06-9	Degradate	organophosphate	Phorate	Yes
Phorate oxon sulfoxide	68671	9030	2588-05-8	Degradate	organophosphate	Phorate	Yes
Phorate sulfone	68672	9030	2588-04-7	Degradate	organophosphate	Phorate	No
Phorate sulfoxide	68673	9030	2588-03-6	Degradate	organophosphate	Phorate	No
Phosmet	65101	9030	732-11-6	Pesticide	organophosphate	Phosmet	No
Phthalazinone	68675	9030	-	Degradate	sulfonylurea & urea	Diflufenzopyr	Yes
Piperonyl butoxide	65102	9030	51-03-6	Pesticide	pyrethroid & phenylpyrazine	Piperonyl butoxide	No
Profenofos	68676	9030	41198-08-7	Pesticide	organophosphate	Profenofos	No
Prometon	67702	9030	1610-18-0	Pesticide	triazine	Prometon	No
Prometryn	65103	9030	7287-19-6	Pesticide	triazine	Prometryn	No
Pronamide	67706	9030	23950-58-5	Pesticide	acetanilide & amide	Pronamide	No
Propanil	66641	9030	709-98-8	Pesticide	acetanilide & amide	Propanil	No
Propargite	68677	9030	2312-35-8	Pesticide	miscellaneous	Propargite	No
Propazine	68678	9030	139-40-2	Pesticide	triazine	Propazine	No
Propiconazole	66643	9030	60207-90-1	Pesticide	fungicide	Propiconazole	No
Propoxur	68679	9030	114-26-1	Pesticide	carbamate & thiocarbamate	Propoxur	No
Prosulfuron	61687	9030	94125-34-5	Pesticide	sulfonylurea & urea	Prosulfuron	No
Pymetrozine	68680	9030	123312-89-0	Pesticide	miscellaneous	Pymetrozine	Yes

Pyraclostrobin	66646	9030	175013-18-0	Pesticide	fungicide	Pyraclostrobin	No
Pyridaben	68682	9030	96489-71-3	Pesticide	miscellaneous	Pyridaben	Yes
Pyrimidinol	68505	9030	2814-20-2	Degradate	organophosphate	Diazinon	Yes
Pyriproxyfen	68683	9030	95737-68-1	Pesticide	miscellaneous	Pyriproxyfen	Yes
sec-Aceto chlor-OA	68684	9031	152019-74-4	Degradate	acetanilide & amide	Aceto chlor/Metolachlor	Yes
sec-Alachlor-OA	68685	9031	-	Degradate	acetanilide & amide	Alachlor	Yes
Siduron	68686	9030	1982-49-6	Pesticide	sulfonylurea & urea	Siduron	No
Simazine	65105	9030	122-34-9	Pesticide	triazine	Simazine	No
Sulfentrazone	68687	9031	122836-35-5	Pesticide	sulfonylurea & urea	Sulfentrazone	Yes
Sulfometuron-methyl	68688	9030	74222-97-2	Pesticide	sulfonylurea & urea	Sulfometuron-methyl	No
Sulfosulfuron	68689	9030	141776-32-1	Pesticide	sulfonylurea & urea	Sulfosulfuron	Yes
Sulfosulfuron ethyl sulfone	68690	9030	-	Degradate	sulfonylurea & urea	Sulfosulfuron	Yes
Tebuconazole	66649	9030	107534-96-3	Pesticide	fungicide	Tebuconazole	No
Tebufozenide	68692	9030	112410-23-8	Pesticide	miscellaneous	Tebufozenide	Yes
Tebupirimfos	68693	9030	96182-53-5	Pesticide	organophosphate	Tebupirimfos	No
Tebupirimfos oxon	68694	9030	-	Degradate	organophosphate	Tebupirimfos	Yes
Tebuthiuron	68695	9030	34014-18-1	Pesticide	sulfonylurea & urea	Tebuthiuron	No
Tebuthiuron TP 104	68575	9030	-	Degradate	sulfonylurea & urea	Tebuthiuron	Yes
Tebuthiuron TP 106	68714	9030	-	Degradate	sulfonylurea & urea	Tebuthiuron	Yes
Tebuthiuron TP 108	68696	9030	39222-73-6	Degradate	sulfonylurea & urea	Tebuthiuron	Yes
Tebuthiuron TP 109 (OH)	68697	9030	-	Degradate	sulfonylurea & urea	Tebuthiuron	Yes
Terbacil	68698	9030	5902-51-2	Pesticide	miscellaneous	Terbacil	No
Terbufos	68699	9030	13071-79-9	Pesticide	organophosphate	Terbufos	No
Terbufos oxon	68700	9030	56070-14-5	Degradate	organophosphate	Terbufos	Yes
Terbufos oxon sulfone	68701	9030	56070-15-6	Degradate	organophosphate	Terbufos	No
Terbufos oxon sulfoxide	68702	9030	56165-57-2	Degradate	organophosphate	Terbufos	Yes
Terbufos sulfone	68703	9030	56070-16-7	Degradate	organophosphate	Terbufos	No
Terbufos sulfoxide	68704	9030	10548-10-4	Degradate	organophosphate	Terbufos	No
Terbutylazine	66651	9030	5915-41-3	Pesticide	triazine	Terbutylazine	No
Tetraconazole	66654	9030	112281-77-3	Pesticide	fungicide	Tetraconazole	No
Thiobencarb	65107	9030	28249-77-6	Pesticide	carbamate & thiocarbamate	Thiobencarb	No
trans-Permethrin	68708	9030	61949-77-7	Pesticide	pyrethroid & phenylpyrazine	Permethrin	No
Triallate	68710	9030	2303-17-5	Pesticide	carbamate & thiocarbamate	Triallate	No
Tribufos	68711	9030	78-48-8	Pesticide	organophosphate	Tribufos	No
Triclopyr	68712	9031	55335-06-3	Pesticide	acid	Triclopyr	No
Trifloxystrobin	66660	9030	141517-21-7	Pesticide	fungicide	Trifloxystrobin	No

Surrogates:

Acetochlor-d11 (surrogate)	90517	9030	1189897-44-6	IS	acetanilide & amide	Acetochlor	na
Alachlor-d13 (surrogate)	90518	9030	1015856-63-9	IS	acetanilide & amide	Alachlor	na
Butachlor SA (surrogate)	90624	9031	187022-12-4	IS	acetanilide & amide	Butachlor SA	na
Dimethachlor SA (surrogate)	90625	9031	-	IS	acetanilide & amide	Dimethachlor SA	na
Metolachlor-d6 (surrogate)	90553	9030	-	IS	acetanilide & amide	Metolachlor	na
2,4-D-d3 (surrogate)	91986	9031	202480-67-9	IS	acid	2,4-D	na
Carbaryl-d7 (surrogate)	90519	9030	362049-56-7	IS	carbamate & thiocarbamate	Carbaryl	na
Carbofuran-d3 (surrogate)	90521	9030	1007459-98-4	IS	carbamate & thiocarbamate	Carbofuran	na
Thiobencarb-d10 (surrogate)	90556	9030	-	IS	carbamate & thiocarbamate	Thiobencarb	na
Carbendazim-d4 (surrogate)	90520	9030	291765-95-2	IS	fungicide	Carbendazim	na
Tebuconazole-d6 (surrogate)	90555	9030	-	IS	fungicide	Tebuconazole	na
Diazinon-d10 (surrogate)	90523	9030	100155-47-3	IS	organophosphate	Diazinon	na
Malathion-d10 (diethyl-d10) (surrogate)	90552	9030	347841-48-9	IS	organophosphate	Malathion	na
3-Phenoxybenzoic acid (surrogate)	90516	9031	-	IS	pyrethroid & phenylpyrazine	Permethrin	na
cis-Permethrin-13C6 (surrogate)	90558	9030	-	IS	pyrethroid & phenylpyrazine	Permethrin	na
Diflubenzuron-d4 (surrogate)	90524	9030	-	IS	sulfonylurea & urea	Diflubenzuron	na
Diuron-d6 (surrogate)	90808	9030	-	IS	sulfonylurea & urea	Diuron	na
Linuron-d6 (dimethyl-d6) (surrogate)	90529	9030	-	IS	sulfonylurea & urea	Linuron	na
Nicosulfuron-d6 (surrogate)	90554	9030	1189419-41-7	IS	sulfonylurea & urea	Nicosulfuron	na
Deethylatrazine-d6 (surrogate)	90522	9030	-	IS	triazine	Atrazine	na
Hexazinone-d6 (surrogate)	90527	9030	-	IS	triazine	Hexazinone	na
Sample volume	3406L	3406	-	-	-	-	na