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Report Date: 2019-12-04 **Final Report**

South Dakota Agricultural Laboratories has examined the sample of

Limfinite Package Id :	20191113-003
Lab Sample Id :	19PE008836
Customer Sample Id :	North Lagoon - AltEn
Sample Description :	WS C WW - water
Date Collected :	2019-11-12
Date Received :	2019-11-13

RESULTS

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	33.9	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	7070	5	LC-MS/MS	2019-11-21	2019-11-29
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	64.5	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	312	5	LC-MS/MS	2019-11-21	2019-11-22
Glufosinate	ppb	10.3	10	LC-MS/MS	2019-11-14	2019-11-22
Glyphosate	ppb	206	10	LC-MS/MS	2019-11-14	2019-11-22
Imidacloprid	ppb	40.8	5	LC-MS/MS	2019-11-21	2019-11-21
Ipconazole	ppb	181	5	LC-MS/MS	2019-11-21	2019-11-21
Isavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22

Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	15.1	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	149	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	634	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	2450	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	2400	5	LC-MS/MS	2019-11-21	2019-11-21
Trifloxystrobin	ppb	36.0	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

QUALITY ASSURANCE

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	19PE008837	96.5	ND	ND	ND
Azoxystrobin	ppb	19PE008838	95.5	ND	ND	ND
Bifenthrin	ppb	ND	111	ND	ND	ND
Brassinazole	ppb	19PE008838	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	ND	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	ND	110	ND	ND	ND
Clothianidin	ppb	19PE008837	102	ND	ND	ND
Cyfluthrin 1-4	ppb	ND	110	ND	ND	ND
Cyhalothrin 1-2	ppb	ND	110	ND	ND	ND
Cypermethrin 1-4	ppb	ND	108	ND	ND	ND
Cyproconazole	ppb	19PE008838	118	ND	ND	ND
Deltamethrin 1-2	ppb	ND	108	ND	ND	ND
Desthio-Prothioconazole	ppb	19PE008838	119	ND	ND	ND
Difenoconazole	ppb	19PE008838	116	ND	ND	ND
Dimoxystrobin	ppb	19PE008838	123	ND	ND	ND
Dinotefuron	ppb	19PE008837	95.6	ND	ND	ND
Epoxiconazole	ppb	19PE008838	110	ND	ND	ND
Fluconazole	ppb	19PE008838	124	ND	ND	ND
Fluoxastrobin	ppb	19PE008838	103	ND	ND	ND
Glufosinate	ppb	<10	112	ND	ND	ND
Glyphosate	ppb	188	82.3	ND	ND	ND
Imidacloprid	ppb	19PE008837	108	ND	ND	ND
Ipconazole	ppb	19PE008838	103	ND	ND	ND
Isavuconazole	ppb	19PE008838	118	ND	ND	ND
Itraconazole	ppb	19PE008838	130	ND	ND	ND
Metconazole	ppb	19PE008838	110	ND	ND	ND
Nitenpyram	ppb	19PE008837	105	ND	ND	ND
Orysastrobin	ppb	19PE008838	108	ND	ND	ND
Permethrin 1-2	ppb	ND	107	ND	ND	ND
Picoxystrobin	ppb	19PE008838	107	ND	ND	ND
Posaconazole	ppb	19PE008838	117	ND	ND	ND
Propiconazole	ppb	19PE008838	116	ND	ND	ND
Prothioconazole	ppb	19PE008838	122	ND	ND	ND

Pyraclostrobin	ppb	19PE008838	105	ND	ND	ND
Ravuconazole	ppb	19PE008838	122	ND	ND	ND
Tebuconazole	ppb	19PE008838	93.5	ND	ND	ND
Tetraconazole	ppb	19PE008838	116	ND	ND	ND
Thiabendazole	ppb	19PE008838	126	ND	ND	ND
Thiacloprid	ppb	19PE008837	91.4	ND	ND	ND
Thiamethoxam	ppb	19PE008837	106	ND	ND	ND
Trifloxystrobin	ppb	19PE008838	96.5	ND	ND	ND
Uniconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

Comments:

Definitions:

ppb - parts per billion
Detection Limit - Lowest concentration that can be quantitatively reported with confidence
ND - Not Detected above the limit of quantification
Duplicate - Concentration found in repeat sample analysis
Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample
Matrix Blank - A similar-matrix, blank sample is evaluated
Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure
Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

BRIEF METHOD DESCRIPTION

Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990
A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)

Ramsteiner et al. JAOAC 57(1): 192-201 (1974)
Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.
https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

Permethrins in water - Basic Principles
Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

Neonicotinoids in water - Purpose and Scope
Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Neonicotinoids in water - References
J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Neonicotinoids in water - Basic Principles
Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

Glyphosate and Glufosinate in water - Purpose and Scope
This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

Glyphosate and Glufosinate in water - References
P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS
L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

Glyphosate and Glufosinate in water - Basic Principles
Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

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Report Date: 2019-12-04 **Final Report**

South Dakota Agricultural Laboratories has examined the sample of

Limfinite Package Id :	20191113-003
Lab Sample Id :	19PE008837
Customer Sample Id :	West Lagoon - AltEn
Sample Description :	WS C WW - water
Date Collected :	2019-11-12
Date Received :	2019-11-13

RESULTS

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	111	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	<5	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	31000	5	LC-MS/MS	2019-12-03	2019-12-03
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	66.2	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	735	5	LC-MS/MS	2019-11-21	2019-11-22
Glufosinate	ppb	ND	10	LC-MS/MS	2019-11-14	2019-11-22
Glyphosate	ppb	116	10	LC-MS/MS	2019-11-14	2019-11-22
Imidacloprid	ppb	312	5	LC-MS/MS	2019-11-21	2019-11-21
Ipconazole	ppb	134	5	LC-MS/MS	2019-11-21	2019-11-21
Isavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22

Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	150	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	216	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	2160	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	24000	5	LC-MS/MS	2019-12-03	2019-12-03
Trifloxystrobin	ppb	53.3	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

QUALITY ASSURANCE

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	ND	96.5	ND	ND	ND
Azoxystrobin	ppb	19PE008838	95.5	ND	ND	ND
Bifenthrin	ppb	19PE008836	111	ND	ND	ND
Brassinazole	ppb	19PE008838	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	19PE008836	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	19PE008836	110	ND	ND	ND
Clothianidin	ppb	32400	102	ND	ND	ND
Cyfluthrin 1-4	ppb	19PE008836	110	ND	ND	ND
Cyhalothrin 1-2	ppb	19PE008836	110	ND	ND	ND
Cypermethrin 1-4	ppb	19PE008836	108	ND	ND	ND
Cyproconazole	ppb	19PE008838	118	ND	ND	ND
Deltamethrin 1-2	ppb	19PE008836	108	ND	ND	ND
Desthio-Prothioconazole	ppb	19PE008838	119	ND	ND	ND
Difenoconazole	ppb	19PE008838	116	ND	ND	ND
Dimoxystrobin	ppb	19PE008838	123	ND	ND	ND
Dinotefuron	ppb	ND	95.6	ND	ND	ND
Epoxiconazole	ppb	19PE008838	110	ND	ND	ND
Fluconazole	ppb	19PE008838	124	ND	ND	ND
Fluoxastrobin	ppb	19PE008838	103	ND	ND	ND
Glufosinate	ppb	19PE008836	112	ND	ND	ND
Glyphosate	ppb	19PE008836	82.3	ND	ND	ND
Imidacloprid	ppb	315	108	ND	ND	ND
Ipconazole	ppb	19PE008838	103	ND	ND	ND
Isavuconazole	ppb	19PE008838	118	ND	ND	ND
Itraconazole	ppb	19PE008838	130	ND	ND	ND
Metconazole	ppb	19PE008838	110	ND	ND	ND
Nitenpyram	ppb	ND	105	ND	ND	ND
Orysastrobin	ppb	19PE008838	108	ND	ND	ND
Permethrin 1-2	ppb	19PE008836	107	ND	ND	ND
Picoxystrobin	ppb	19PE008838	107	ND	ND	ND
Posaconazole	ppb	19PE008838	117	ND	ND	ND
Propiconazole	ppb	19PE008838	116	ND	ND	ND
Prothioconazole	ppb	19PE008838	122	ND	ND	ND

Pyraclostrobin	ppb	19PE008838	105	ND	ND	ND
Ravuconazole	ppb	19PE008838	122	ND	ND	ND
Tebuconazole	ppb	19PE008838	93.5	ND	ND	ND
Tetraconazole	ppb	19PE008838	116	ND	ND	ND
Thiabendazole	ppb	19PE008838	126	ND	ND	ND
Thiacloprid	ppb	ND	91.4	ND	ND	ND
Thiamethoxam	ppb	23300	123	ND	ND	ND
Trifloxystrobin	ppb	19PE008838	96.5	ND	ND	ND
Uniconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

Comments:

Definitions:

ppb - parts per billion
Detection Limit - Lowest concentration that can be quantitatively reported with confidence
ND - Not Detected above the limit of quantification
Duplicate - Concentration found in repeat sample analysis
Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample
Matrix Blank - A similar-matrix, blank sample is evaluated
Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure
Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

BRIEF METHOD DESCRIPTION

Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990
A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)

Ramsteiner et al. JAOAC 57(1): 192-201 (1974)
Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.
https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

Permethrins in water - Basic Principles

Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

Neonicotinoids in water - Purpose and Scope

Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Neonicotinoids in water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Neonicotinoids in water - Basic Principles

Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

Glyphosate and Glufosinate in water - Purpose and Scope

This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

Glyphosate and Glufosinate in water - References

P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS
L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

Glyphosate and Glufosinate in water - Basic Principles

Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

Reviewed By: Regina Wixon, Ph.D.

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Lincoln ,NE 95922
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E-Mail: mark.pomajzl@nebraska.gov

Report Date: 2019-12-04 **Final Report**

South Dakota Agricultural Laboratories has examined the sample of

Limfinite Package Id :	20191113-003
Lab Sample Id :	19PE008838
Customer Sample Id :	Field Blank (Between North Lagoon and South Lagoon Sampling) - AltEn
Sample Description :	WS C WW - water
Date Collected :	2019-11-12
Date Received :	2019-11-13

RESULTS

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Glufosinate	ppb	<10	10	LC-MS/MS	2019-11-14	2019-11-22
Glyphosate	ppb	<10	10	LC-MS/MS	2019-11-14	2019-11-22
Imidacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Ipconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Isavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22

Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Trifloxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

QUALITY ASSURANCE

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	19PE008837	96.5	ND	ND	ND
Azoxystrobin	ppb	ND	95.5	ND	ND	ND
Bifenthrin	ppb	19PE008836	111	ND	ND	ND
Brassinazole	ppb	ND	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	19PE008836	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	19PE008836	110	ND	ND	ND
Clothianidin	ppb	19PE008837	124	ND	ND	ND
Cyfluthrin 1-4	ppb	19PE008836	110	ND	ND	ND
Cyhalothrin 1-2	ppb	19PE008836	110	ND	ND	ND
Cypermethrin 1-4	ppb	19PE008836	108	ND	ND	ND
Cyproconazole	ppb	ND	118	ND	ND	ND
Deltamethrin 1-2	ppb	19PE008836	108	ND	ND	ND
Desthio-Prothioconazole	ppb	ND	119	ND	ND	ND
Difenoconazole	ppb	ND	116	ND	ND	ND
Dimoxystrobin	ppb	ND	123	ND	ND	ND
Dinotefuron	ppb	19PE008837	95.6	ND	ND	ND
Epoxiconazole	ppb	ND	110	ND	ND	ND
Fluconazole	ppb	ND	124	ND	ND	ND
Fluoxastrobin	ppb	ND	103	ND	ND	ND
Glufosinate	ppb	19PE008836	112	ND	ND	ND
Glyphosate	ppb	19PE008836	82.3	ND	ND	ND
Imidacloprid	ppb	19PE008837	108	ND	ND	ND
Ipconazole	ppb	ND	103	ND	ND	ND
Isavuconazole	ppb	ND	118	ND	ND	ND
Itraconazole	ppb	ND	130	ND	ND	ND
Metconazole	ppb	ND	110	ND	ND	ND
Nitenpyram	ppb	19PE008837	105	ND	ND	ND
Orysastrobin	ppb	ND	108	ND	ND	ND
Permethrin 1-2	ppb	19PE008836	107	ND	ND	ND
Picoxystrobin	ppb	ND	107	ND	ND	ND
Posaconazole	ppb	ND	117	ND	ND	ND
Propiconazole	ppb	ND	116	ND	ND	ND

Prothioconazole	ppb	ND	122	ND	ND	ND
Pyraclostrobin	ppb	ND	105	ND	ND	ND
Ravuconazole	ppb	ND	122	ND	ND	ND
Tebuconazole	ppb	ND	93.5	ND	ND	ND
Tetraconazole	ppb	ND	116	ND	ND	ND
Thiabendazole	ppb	ND	126	ND	ND	ND
Thiacloprid	ppb	19PE008837	91.4	ND	ND	ND
Thiamethoxam	ppb	19PE008837	106	ND	ND	ND
Trifloxystrobin	ppb	ND	96.5	ND	ND	ND
Uniconazole	ppb	ND	122	ND	ND	ND
Voriconazole	ppb	ND	97.9	ND	ND	ND

Comments:

Definitions:

ppb - parts per billion
Detection Limit - Lowest concentration that can be quantitatively reported with confidence
ND - Not Detected above the limit of quantification
Duplicate - Concentration found in repeat sample analysis
Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample
Matrix Blank - A similar-matrix, blank sample is evaluated
Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure
Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

BRIEF METHOD DESCRIPTION

Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990

A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)
Ramsteiner et al. JAOAC 57(1): 192-201 (1974)
Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.
https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

Permethrins in water - Basic Principles

Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

Neonicotinoids in water - Purpose and Scope

Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Neonicotinoids in water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Neonicotinoids in water - Basic Principles

Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

Glyphosate and Glufosinate in water - Purpose and Scope

This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

Glyphosate and Glufosinate in water - References

P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS
L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

Glyphosate and Glufosinate in water - Basic Principles

Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

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Report Date: 2019-12-04 **Final Report**

South Dakota Agricultural Laboratories has examined the sample of

Limfinite Package Id :	20191113-003
Lab Sample Id :	19PE008839
Customer Sample Id :	North Lagoon Duplicate Sample AltEn
Sample Description :	WS C WW - water
Date Collected :	2019-11-12
Date Received :	2019-11-13

RESULTS

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	32.4	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	<5	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	<5	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	5980	5	LC-MS/MS	2019-11-21	2019-11-29
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	61.7	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	312	5	LC-MS/MS	2019-11-21	2019-11-22
Glufosinate	ppb	<10	10	LC-MS/MS	2019-11-14	2019-11-22
Glyphosate	ppb	200	10	LC-MS/MS	2019-11-14	2019-11-22
Imidacloprid	ppb	40.8	5	LC-MS/MS	2019-11-21	2019-11-21
Ipconazole	ppb	166	5	LC-MS/MS	2019-11-21	2019-11-21
Isavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22

Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	17.0	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	141	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	627	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	2470	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	2360	5	LC-MS/MS	2019-11-21	2019-11-21
Trifloxystrobin	ppb	32.4	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

QUALITY ASSURANCE

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	19PE008837	96.5	ND	ND	ND
Azoxystrobin	ppb	19PE008838	95.5	ND	ND	ND
Bifenthrin	ppb	19PE008836	111	ND	ND	ND
Brassinazole	ppb	19PE008838	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	19PE008836	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	19PE008836	110	ND	ND	ND
Clothianidin	ppb	19PE008837	102	ND	ND	ND
Cyfluthrin 1-4	ppb	19PE008836	110	ND	ND	ND
Cyhalothrin 1-2	ppb	19PE008836	110	ND	ND	ND
Cypermethrin 1-4	ppb	19PE008836	108	ND	ND	ND
Cyproconazole	ppb	19PE008838	118	ND	ND	ND
Deltamethrin 1-2	ppb	19PE008836	108	ND	ND	ND
Desthio-Prothioconazole	ppb	19PE008838	119	ND	ND	ND
Difenoconazole	ppb	19PE008838	116	ND	ND	ND
Dimoxystrobin	ppb	19PE008838	123	ND	ND	ND
Dinotefuron	ppb	19PE008837	95.6	ND	ND	ND
Epoxiconazole	ppb	19PE008838	110	ND	ND	ND
Fluconazole	ppb	19PE008838	124	ND	ND	ND
Fluoxastrobin	ppb	19PE008838	103	ND	ND	ND
Glufosinate	ppb	19PE008836	112	ND	ND	ND
Glyphosate	ppb	19PE008836	82.3	ND	ND	ND
Imidacloprid	ppb	19PE008837	108	ND	ND	ND
Ipconazole	ppb	19PE008838	103	ND	ND	ND
Isavuconazole	ppb	19PE008838	118	ND	ND	ND
Itraconazole	ppb	19PE008838	130	ND	ND	ND
Metconazole	ppb	19PE008838	110	ND	ND	ND
Nitenpyram	ppb	19PE008837	105	ND	ND	ND
Orysastrobin	ppb	19PE008838	108	ND	ND	ND
Permethrin 1-2	ppb	19PE008836	107	ND	ND	ND
Picoxystrobin	ppb	19PE008838	107	ND	ND	ND
Posaconazole	ppb	19PE008838	117	ND	ND	ND
Propiconazole	ppb	19PE008838	116	ND	ND	ND
Prothioconazole	ppb	19PE008838	122	ND	ND	ND

Pyraclostrobin	ppb	19PE008838	105	ND	ND	ND
Ravuconazole	ppb	19PE008838	122	ND	ND	ND
Tebuconazole	ppb	19PE008838	93.5	ND	ND	ND
Tetraconazole	ppb	19PE008838	116	ND	ND	ND
Thiabendazole	ppb	19PE008838	126	ND	ND	ND
Thiacloprid	ppb	19PE008837	91.4	ND	ND	ND
Thiamethoxam	ppb	19PE008837	106	ND	ND	ND
Trifloxystrobin	ppb	19PE008838	96.5	ND	ND	ND
Uniconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

Comments:

Definitions:

ppb - parts per billion
Detection Limit - Lowest concentration that can be quantitatively reported with confidence
ND - Not Detected above the limit of quantification
Duplicate - Concentration found in repeat sample analysis
Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample
Matrix Blank - A similar-matrix, blank sample is evaluated
Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure
Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

BRIEF METHOD DESCRIPTION

Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990
A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)

Ramsteiner et al. JAOAC 57(1): 192-201 (1974)
Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.
https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

Permethrins in water - Basic Principles
Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

Neonicotinoids in water - Purpose and Scope
Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Neonicotinoids in water - References
J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Neonicotinoids in water - Basic Principles
Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

Glyphosate and Glufosinate in water - Purpose and Scope
This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

Glyphosate and Glufosinate in water - References
P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS
L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

Glyphosate and Glufosinate in water - Basic Principles
Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

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Report Date: 2019-12-04 **Final Report**

South Dakota Agricultural Laboratories has examined the sample of

Limfinite Package Id :	20191113-003
Lab Sample Id :	19PE009039
Customer Sample Id :	Trip Blank - AltEn
Sample Description :	Trip Blank - AltEn
Date Collected :	2019-11-12
Date Received :	2019-11-13

RESULTS

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	<5	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Glufosinate	ppb	<10	10	LC-MS/MS	2019-11-19	2019-11-22
Glyphosate	ppb	<10	10	LC-MS/MS	2019-11-19	2019-11-22
Imidacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Ipconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Isavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22

Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Trifloxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

QUALITY ASSURANCE

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	19PE008837	96.5	ND	ND	ND
Azoxystrobin	ppb	19PE008838	95.5	ND	ND	ND
Bifenthrin	ppb	19PE008836	111	ND	ND	ND
Brassinazole	ppb	19PE008838	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	19PE008836	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	19PE008836	110	ND	ND	ND
Clothianidin	ppb	19PE008837	124	ND	ND	ND
Cyfluthrin 1-4	ppb	19PE008836	110	ND	ND	ND
Cyhalothrin 1-2	ppb	19PE008836	110	ND	ND	ND
Cypermethrin 1-4	ppb	19PE008836	108	ND	ND	ND
Cyproconazole	ppb	19PE008838	118	ND	ND	ND
Deltamethrin 1-2	ppb	19PE008836	108	ND	ND	ND
Desthio-Prothioconazole	ppb	19PE008838	119	ND	ND	ND
Difenoconazole	ppb	19PE008838	116	ND	ND	ND
Dimoxystrobin	ppb	19PE008838	123	ND	ND	ND
Dinotefuron	ppb	19PE008837	95.6	ND	ND	ND
Epoxiconazole	ppb	19PE008838	110	ND	ND	ND
Fluconazole	ppb	19PE008838	124	ND	ND	ND
Fluoxastrobin	ppb	19PE008838	103	ND	ND	ND
Glufosinate	ppb	<10	73.5	ND	ND	ND
Glyphosate	ppb	<10	83.7	ND	ND	ND
Imidacloprid	ppb	19PE008837	108	ND	ND	ND
Ipconazole	ppb	19PE008838	103	ND	ND	ND
Isavuconazole	ppb	19PE008838	118	ND	ND	ND
Itraconazole	ppb	19PE008838	130	ND	ND	ND
Metconazole	ppb	19PE008838	110	ND	ND	ND
Nitenpyram	ppb	19PE008837	105	ND	ND	ND
Orysastrobin	ppb	19PE008838	108	ND	ND	ND
Permethrin 1-2	ppb	19PE008836	107	ND	ND	ND
Picoxystrobin	ppb	19PE008838	107	ND	ND	ND
Posaconazole	ppb	19PE008838	117	ND	ND	ND
Propiconazole	ppb	19PE008838	116	ND	ND	ND
Prothioconazole	ppb	19PE008838	122	ND	ND	ND

Pyraclostrobin	ppb	19PE008838	105	ND	ND	ND
Ravuconazole	ppb	19PE008838	122	ND	ND	ND
Tebuconazole	ppb	19PE008838	93.5	ND	ND	ND
Tetraconazole	ppb	19PE008838	116	ND	ND	ND
Thiabendazole	ppb	19PE008838	126	ND	ND	ND
Thiacloprid	ppb	19PE008837	91.4	ND	ND	ND
Thiamethoxam	ppb	19PE008837	106	ND	ND	ND
Trifloxystrobin	ppb	19PE008838	96.5	ND	ND	ND
Uniconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

Comments:

Definitions:

ppb - parts per billion
Detection Limit - Lowest concentration that can be quantitatively reported with confidence
ND - Not Detected above the limit of quantification
Duplicate - Concentration found in repeat sample analysis
Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample
Matrix Blank - A similar-matrix, blank sample is evaluated
Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure
Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

BRIEF METHOD DESCRIPTION

Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990
A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)

Ramsteiner et al. JAOAC 57(1): 192-201 (1974)
Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.
https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

Permethrins in water - Basic Principles

Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

Neonicotinoids in water - Purpose and Scope

Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

Neonicotinoids in water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

Neonicotinoids in water - Basic Principles

Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

Glyphosate and Glufosinate in water - Purpose and Scope

This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

Glyphosate and Glufosinate in water - References

P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS
L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

Glyphosate and Glufosinate in water - Basic Principles

Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

Reviewed By: Regina Wixon, Ph.D.

Submitted by the customer:

Chain of Custody Record

Sample Site: AltEn, LLC Wastewater Lagoons

Project Name: AltEn Wastewater Sampling 11/12/2019

Sampler Name(s) (Print): Mark Pomajzl, Hillary Stoll, and Jason Windhorst

This CoC is for the samples collected by NDEE for NDEE (Sample Set 1). Split samples were left with AltEn, LLC. These samples were collected in the same manner as the samples retained by NDEE. The samples left with AltEn, LLC, are referred to as Sample Set 2. This CoC will be sent with Sample Set 1 to the lab. A copy of this CoC will remain with NDEE.

Sampler Name(s) (Signature): [signature]

Sample ID	Sample Date	Sample Time	Sample Type*	Grab or Composite?	Media	Comments	Lab Use
~~South Lagoon -AltEn~~	11/12/2019		WS	C	WW		
North Lagoon -AltEn	11/12/2019	10:30 AM	WS	C	WW	19PE008836	
West Lagoon - AltEn	11/12/2019	11:45 AM	WS	C	WW	19PE008837	
~~Field Blank (Before Sampling) - AltEn~~	11/12/2019		FB	N/A	DI		
Field Blank (Between North Lagoon and South Lagoon Sampling) - AltEn	11/12/2019	11:00 AM	FB	N/A	DI	19PE008838	
~~Field Blank (Between South Lagoon and West Lagoon Sampling) - AltEn~~	11/12/2019		FB	N/A	DI		
North Lagoon Duplicate Sample - AltEn	11/12/2019	10:30 AM	WSQ	C	WW	Duplicate obtained from composite sample bucket for North Lagoon.	19PE008839
Trip Blank - AltEn	11/12/2019	—	TB	N/A	DI	Trip Blank- do not open.	

*If it is a normal sample, write WS. If it is a duplicate, write WSQ. If it is a trip blank, write TB. If it is a field blank, write FB.

Relinquished by:	Received by:	Date/Time:
[signature] - 11/12/19 ~ 3:30 PM Mark Pomajzl 11/12/19 ~ 3:30 pm	C. Sanepoel 2019-11-13	9 AM
Relinquished by:	Received by:	Date/Time:

Shipment Method:

1 of ____

20191113-000
19PE008836-008839

20191113-003
19PE008836-8839

South Dakota Agricultural Laboratories
Agricultural and Analytical Testing

Pesticide Residue Sample Submission Form

South Dakota Agricultural Laboratories
1335 Western Avenue
Brookings, SD. 57006
(605) 692-7325

20191113-003
19PE008836-008839

Atten Wastewater Unit (Mark Pomajzl)

Name: Nebraska Department of Environmental Energy *Sample ID: (See Attached form)

Address: Suite 400, The Atrium, Room 5E City: Lincoln State: NE

Zip: P.O.B 98922 (68509-8922) Phone: 1 (402) 471-2936 **Email: Mark.Pomajzl@nebraska.gov

**Sample ID must be marked clearly on the sample you submit. **Results will be emailed to the provided email address.*

Billing Information: X Check box if billing is the same as the customer information

Name:_____ Address:_____

City:_____ State:_____ Zip:_____

Phone: (____) ____ - ____ Email:_____

Individual tests are $162 each, unless otherwise marked. Scans are $212 and include all of the compounds in a particular category. Acceptable samples include Vegetation, Water or Soil. Call to confirm other substrates.

Thank you for choosing South Dakota Agricultural Labs! We do add analytes to our testing regiment throughout the year. If a chemical of interest is not listed, please call us:

(605) 692-7325.

How much sample should you send?

Please send 30g of vegetation or 100g of soil to run an individual test. What does this look like? For vegetation, it would be about a quart sized bag packed full. If more than one test is required, please fill a gallon sized bag. For soil samples, please send 2 cups, if more than one test is required send 4 cups.

Analyses offered

Please turn page over to view the current pesticide analyses.

If you are interested in a screen of active ingredients, please check the box next to the **bold-faced** heading. This will include all active ingredients within the PGR screen for $212.

Example: PGR Screen ☑

If you are interested in single analyses, please circle the active ingredients. The cost of each individual analyte is $162 unless otherwise marked.

Example: Mesotrione

Sample\s received at SD Ag Labs
Date 2019-11-13
Received by Christina Schepker

Azoles ☑
Brassinazole
Cyproconazole
Difenoconazole
Epoxiconazole
Fluconazole
Ipconazole
Isavuconazole
Itraconazole
Metconazole
Posaconazole
Propiconazole
Prothioconazole
Prothioconazole
Metabolite 1
Prothioconazole
Metabolite 2
Ravuconazole
Tebuconazole
Tetraconazole
Thiabendazole
Uniconazole
Voriconazole

Permethrins ☑
Bifenthrin
Cyhalothrin 1-2
Permethrin 1-2
Cyhalothrin 1-4
Cypermethrin 1-4
Deltamethrin 1-2

IMI ☐
Imazalil
Imazamethabenz
Imazamox
Imazapic
Imazapyr
Imazaquin
Imazethapyr

Neonics ☑
Acetamiprid
Clothianidin
Thiacloprid
Imidacloprid
Thiamethoxam
Nitenpyram
Dinotefuran

Strobins ☑
Dimoxystrobin
Picoxystrobin
Azoxystrobin
Pyraclostrobin
Orysastrobin
Trifloxystrobin
Fluoxastrobin

PGR ☐
2, 4-D
2,4,5-T
2,4,5-TP
2,4-DB
2,4-DP
Bentazon
Bromacil
Bromoxynil
Clopyralid
DCPA
Dicamba
Fluroxypyr
MCPA
MCPP
Picloram
Pyrasulfotole
Quinclorac
Triclopyr

Pre-Emergents ☐
Acetochlor
Alachlor
Atrazine
Dimethenamid
Metolachlor
Metribuzin
Pendimethalin
Prometon
Simazine

SU ☐
Chlorimuron
Formasulfuron
Mesosulfuron
Metsulfuron
Nicosulfuron
Primisulfuron
Prosulfuron
Rimsulfuron
Sulfometuron
Sulfosulfuron
Thifensulfuron
Triasulfuron
Tribenuron
Triflusulfuron

Sulams ☐
Cloransulam
Florasulam
Flumetsulam
Penoxsulam
Pyroxsulam

OP Scan ☑ **$262**
Glyphosate **$212**
Glufosinate **$212**

Individual Active Ingredients
Abemectin
Acetamiprid
Aciflurofen
Aldicarb
Aldicarb sulfone
Aldicarb sulfoxide
Aminocyclopyrachlor
Aminopyralid **$212**
Benzovendiflupry
Bicyclopyrone
Carbaryl
Carbofuran
Carfentrazone
Chlorantraniliprole
Chlorophacinone
Chlorpyrifos-ethyl
Chlorpyrifos-methyl
Chlorsulfuron **$212**
Clethodim
Clethodim sulfone
Clethodim sulfoxide
Clomazone
Cyanazine
Diazinon
Diflufenzopyr
Dinotefuran
Diquat **$312**
Dithiopyr
Diuron
Diuron metabolite
Ethalfuralin **$212**
Emamectin benzoate
Fenoxaprop
Fipronil
Fipronil sulfide
Fipronil sulfone
Fluazinam
Flucarbazone
Fludioxonil
Flumioxazin
Fluopyram
Fluthiacet methyl
Flutriafol
Fluxapyroxad
Fluaziflop
Fomesafen
Halosulfuron **$212**
Hexazinone
Indaziflam
Indoxacarb
Iodosulfuron **$212**
Isoxaflutole
Lactofen
Lincomycin
Linuron
Malathion
Mandipropamid
Mesotrione
Methiocarb
Methomyl
Nitenpyram
Oryzalin
Oxamyl
Oxathiapiprolin
Oxyfluorfen
Paclobutrazol
Paraquat **$312**
Pinoxaden
Prodiamine
Propachlor
Propanil
Propazine
Propoxur
Propoxycarbazone
Pydiflumetofen
Pyroxasulfone
Quizalofop
Saflufenacil
Sedaxane
Sulfameturon methyl
Sulfentrazone
Tebuthiuron
Tembotrione
Terbacil
Thiencarbazone
Thiophanate methyl
Topramezone **$212**
Trifuralin **$212**
Trimethoprim