

BEFORE THE NEBRASKA DEPARTMENT OF ENVIRONMENT AND ENERGY

IN THE MATTER OF	)	
AltEn, LLC,	)	CASE NO. 3540
	)	
FID # 84069	)	EMERGENCY COMPLAINT
	)	AND ORDER
Respondent.	)	

This Emergency Complaint and Order is issued by the Director of the Department of Environment and Energy (hereinafter referred to as "Department") pursuant to Neb. Rev. Stat. § 81-1507(4) of the Environmental Protection Act (Act), Neb. Rev. Stat. § 81-1501 et seq. (Reissue 2014, Supp. 2015) to require immediate action to protect the public health and the environment. The Department is the agency of the State of Nebraska charged with the duty pursuant to Neb. Rev. Stat. § 81-1504(1) to exercise exclusive general supervision of the administration and enforcement of the Environmental Protection Act, and all rules and regulations and orders promulgated under the Act. The Respondent is AltEn, LLC, (hereinafter "Respondent"), whose Facility is located in Mead, Saunders County, Nebraska.

The complaint below sets out the facts establishing the existence of an emergency, and the order specifies the immediate action that must be taken to abate the emergency. This Emergency Complaint and Order is effective immediately. The Respondent shall be afforded the opportunity for a hearing upon application in writing to the director of the Department, such hearing to take place as soon as possible but not later than ten days after a request is made.

**I. COMPLAINT**

1. Respondent is AltEn, LLC, owner of the ethanol plant (hereinafter "Facility") at 1344 County Road 10, Mead, Nebraska 68041. The legal description is N SW Section 12 Township N 14 Range E 08, Saunders County, Nebraska. AltEn, LLC, is a limited liability company registered to do business in Nebraska and organized in the state of Kansas.

2. The Respondent's ethanol plant uses seed corn treated with pesticides to produce ethanol. The waste products include wastewater and distiller's grain, both containing high levels of pesticide residue.

3. The Facility discharges processed wastewater to three lagoons. The Southeast and Northeast Lagoons were built first, pursuant to the Facility's Wastewater Facility (WWF) Construction Permit # 05-0158. The West Lagoon was built in 2018 pursuant to WWF Construction Permit # 2018-0060; the three lagoons are interconnected to allow water to flow from one lagoon to another.

4. On May 9, 2018, on behalf of the Respondent, Matthew D. Goeller, P.E., Settje Agri-Services & Engineering, Inc., filed an Engineering Design form #1 (ED-1) with the Department documenting and amending the maximum operating depth and freeboard above max (the distance between the surface of the wastewater and the top of retaining banks determined necessary to ensure safe operation) for the three lagoons at the Facility. The Southeast Lagoon has a maximum operating depth of 19.0 feet, and a freeboard above max of 3.0 feet. The Northeast Lagoon has a maximum operating depth of 19.0 feet, and a freeboard above max of 3.0 feet. The West Lagoon (referred to as the Northwest Lagoon in the ED-1) has a maximum operating depth of 17.9 feet, and a freeboard above max of 2.1 feet. This ED-1 amended the maximum operating depth and freeboard for the Northeast and West lagoons from the original plans and specifications because of the addition of the third lagoon and the interconnection between the lagoons. This ED-1 was approved by the Department as part of Permit # 2018-0060.

5. On February 1, 2021, employees from the Department conducted a site visit at the Facility. See Attachment A, February 1, 2021, Site Inspection Report, hereby incorporated into this Order.

6. An Environmental Engineer from the Department's Permitting and Engineering Division conducted an inspection of the three lagoons at the Facility. Based upon her visual observations and calculations (located in Attachment A), she made the following conclusions:

- A. The West Lagoon is operating at a depth of about 18.5 feet, above the maximum operating depth of 17.9 feet and within the area designed for freeboard.
- B. The Northeast Lagoon is operating at a depth of about 19 to 19.5 feet, above the maximum operating depth of 19 feet and within the area designed for freeboard.
- C. The Southeast Lagoon is operating at a depth of about 19 to 19.5 feet, above the maximum operating depth of 19 feet and within the area designed for freeboard.

7. Testing of the West Lagoon wastewater on April 8, 2019, showed the presence of Azoxystrobin, Clothianidin, Thiabendazole, and Thiamethoxam, which are fungicides and pesticides used on treated seed corn. The levels of fungicides and pesticides reported in the test results were Azoxystrobin (33.9 parts per billion (ppb)), Clothianidin (58,400 ppb), Glyphosate (124 ppb), Thiabendazole (8450 ppb), and Thiamethoxam (35,400 ppb). See Attachment B, Test Results.

8. Testing of the Northeast Lagoon wastewater on November 12, 2019, showed the presence of Azoxystrobin (99.3 ppb), Clothianidin (7070 ppb), Glyphosate (206 ppb), Thiabendazole (2450 ppb), and Thiamethoxam (2400 ppb). Testing of the West Lagoon wastewater on November 12, 2019, showed the presence of Azoxystrobin (111 ppb), Clothianidin (31,000 ppb), Glyphosate (116 ppb), Thiabendazole (2160 ppb), and Thiamethoxam (24,000 ppb). See Attachment C, Test Results.

9. The pesticide levels found in the lagoon wastewater testing from April 8, 2019, and November 12, 2019, far exceed the registered application rates for which EPA has conducted safety assessments for pesticide products and "represents a level of

contamination that has no uniformity or limit on the number and amount of pesticides present." "Some of these pesticides are known to leach and may contaminate groundwater. Some may be persistent, and runoff will contaminate aquatic ecosystems. Additionally, there are systemic pesticides in these samples and based on the high levels detected, they can be taken up into plant tissues and result in levels of residues in nectar and pollen that may harm pollinators or in leaves or other plant parts that are consumed by birds and mammals." See Attachment D, January 13, 2021, Letter from EPA, hereby incorporated into this Order.

10. On September 13, 2019, the Department issued a Notice of Violation to the Respondent citing as a violation:

"5. The wastewater in the treatment lagoons contains pesticides from the use of treated seed corn feed stock in the ethanol production process. These pollutants are not addressed by the Best Management Practices (BMP) plan to prevent short-term and long-term surface and groundwater contamination as required by Part II.B.6 of NPDES Permit NE0137634."

11. The Department required the Respondent to "Immediately, cease land application of lagoon wastewater."

12. The Department issued a Letter of Noncompliance to the Respondent on October 2, 2020, citing the failure to immediately cease land application of lagoon wastewater as a continuing violation of the September 13, 2019, Notice of Violation.

13. The October 2, 2020, Letter of Noncompliance further cited:

"A. Failure to comply with operation and maintenance requirements.

1. The northeast lagoon liner is badly damaged. This includes damage from vegetation, as well as numerous "whales" and tears. This is a violation of Title 123, Chapter 11, Sections 001, 008.05, and 008.06.
2. The northwest lagoon liner has several "whales". Moreover, the edge of the liner is visible at the ground surface in some areas on the east side of the northwest lagoon. This is supposed to be anchored two feet below the ground, per plans and specifications submitted to the Department prior to

the construction of the northwest lagoon. This is a violation of Title 123, Chapter 11, Sections 001, 008.05, and 008.06."

14. The Respondent is currently prohibited from land applying the lagoon wastewater. According to a site visit on September 11, 2020, the Facility estimated that the average wastewater flow to the lagoons is 100,000 gallons per day. If the facility continues to discharge wastewater to the lagoons without any acceptable method approved by the Department to dispose of the wastewater, the water levels in the lagoons will continue to rise and be susceptible to overflowing the dikes as precipitation increases in the spring.

15. Respondent was issued National Pollutant Discharge Elimination System (hereafter NPDES) Permit Number NE0137634, effective July 1, 2017, and modified on October 30, 2020. This permit authorizes Respondent to discharge wastewater in accordance with permit limits. This permit in part provides:

**"Part IV. D. Prohibited Discharges**

The discharge of process wastewater to Waters of the State from the AltEn, LLC facility is prohibited. Process wastewater is defined as any wastestream generated from the chemical, physical, or biological procedures utilized to produce ethanol."

**"Appendix A. Conditions Applicable to All NPDES Permits**

**5. Need to Halt or Reduce Activity not a Defense**

It shall not be a defense for a permittee in an enforcement action that it would have been necessary to halt or reduce the permitted activity in order to maintain compliance with the conditions of this permit."

**"7. The permittee shall at all times properly operate and maintain all facilities and systems of treatment and control (and related appurtenances) which are installed or used by the permittee to achieve compliance with the conditions of this permit."**

16. NPDES Permit Number NE0137634 cites the receiving water for stormwater drainage from the Facility to be an undesignated tributary to Clear Creek (Segment LB2-10120 in the Lower Platte River Basin), both of which are waters of the State.

17. Neb. Rev. Stat. § 81-1506(1)(a) makes it “unlawful for any person to cause pollution of any air, waters, or land of the state or to place or cause to be placed any wastes in a location where they are likely to cause pollution of any air, waters, or land of the state.”

18. The Respondent is a person as defined in Neb. Rev. Stat. § 81-1502(10).

19. Pursuant to the Nebraska Environmental Quality Council’s authority to adopt rules and regulations for the purpose of water pollution control, as expressed in Neb. Rev. Stat. § 81-1505, the Council adopted rules and regulations codified as Neb. Admin. Code, Title 119, Rules and Regulations Pertaining to the Issuance of Permits under the National Pollutant Discharge Elimination Section and Neb. Admin. Code, Title 123, Rules and Regulations for the Design, Operation, and Maintenance of Wastewater Works.

20. Neb. Admin. Code, Title 119, Rules and Regulations Pertaining to the Issuance of Permits under the National Pollutant Discharge Elimination Section, Chapter 14, Section 001, states in pertinent part that:

“001.01 Duty to comply. The permittee must comply with all conditions of the permit. Any permit noncompliance constitutes a violation of the Federal and State Act and is grounds for enforcement action; for permit termination, revocation and reissuance, or modification; or denial of a permit renewal application.” [and]

“001.03 Need to halt or reduce activity not a defense. It shall not be a defense for a permittee in an enforcement action that it would have been necessary to halt or reduce the permitted activity in order to maintain compliance with the conditions of this permit.”

21. Neb. Admin. Code, Title 123, Chapter 11, § 001 states: “Wastewater treatment facilities shall be maintained in proper operating condition in accordance with this chapter and shall be operated in a manner to meet all NPDES permit requirements and not result in a prohibited bypass or an unauthorized discharge.”

22. Neb. Admin. Code, Title 123, Rules and Regulations for the Design, Operation, and Maintenance of Wastewater Works, Chapter 11 states:

“008 The operation and maintenance of wastewater lagoons shall be

conducted in the following manner:

008.06 The liners of wastewater lagoons shall be maintained so that wastewater seepage does not exceed the rate approved by the Department in the construction plans and specifications. Where no record of approved plans and specifications exist, the lagoon liner shall be maintained to meet the seepage rate prescribed by the Department for the specific wastewater being treated. The owner shall perform seepage testing when requested by the Department.

008.07 Wastewater lagoons shall be operated so that the water level is not maintained in the area designed for freeboard."

23. In a Consent Order entered into between the Department and the Respondent on April 24, 2019, Respondent was ordered to make repairs to its lagoon system. However, Respondent has failed to comply with the following provisions of this order:

"B. Within sixty (60) days of the signing of this consent order, submit to the NDEQ an independent professional engineering evaluation or the repair of the north cell of the two-cell lagoon, the south cell of the two-cell lagoon, and the emergency lagoon liners along with a timeline for the repair completion."

"D. Within thirty (30) days of receiving approval of the plan for the north cell of the two-cell lagoon repairs, respondent shall make a determination if they will repair the north cell of the two-cell lagoon and/or utilize another method of water treatment or storage will be utilized."

24. The February 1, 2021, Site Visit Report (Attachment A) further notes that Northeast lagoon and West lagoon liners have not been repaired and are still badly damaged.

25. The Director specifically finds that the purpose of maintaining freeboard is to protect the lagoons against exceptional storms and associated winds, heavy rain/snowfall, and damage to the integrity of the lagoon from wave action and potential erosion. Freeboard protects the land and waters of the State from wastewater overflows. Damaged lagoon liners with whales are also susceptible to further damage from wind and high water levels in the lagoons. The Northeast and West Lagoons remain unrepairs; using the lagoons without adequate freeboard could subject the lagoons to additional damage.

26. The Director finds that unless wastewater levels in the lagoons are lowered in accordance with II. Compliance Order section of this Order, every additional wastewater

discharge from the Facility to the lagoons will increase the lagoon levels, decrease the freeboard above max available in the lagoons, and take the lagoons further above their maximum operating depths, presenting an imminent and substantial danger to the structure and integrity of the lagoons, an imminent and substantial risk of an unpermitted and uncontrolled release of wastewater, and further damage to public health and the environment.

27. The Director finds that the Respondent, by operating its Facility lagoons at or above freeboard above the max level, has and continues to be violating the Nebraska Environmental Protection Act (Act), Neb. Rev. Stat. § 81-1501 et. seq. (Reissue 2014, Cum. Supp. 2018); Title 119; Title 123; the Facility's Wastewater Facility (WWF) Construction Permits #2018-0060, #05-0158; and NPDES Permit # NE0137634.

28. The Director finds that the Respondent, by its actions cited above and by failing to operate its Facility under the terms of its permits, NPDES Permit # NE0137634, WWF Construction Permits #2018-0060, #05-0158, and in compliance with the Act, Title 119, and Title 123, has operated its Facility in a manner that presents an emergency and imminent and substantial threat to human health and the environment.

29. The Director further finds that the Respondent, by its actions, is likely to cause and may already have caused pollution to the air, waters, and land of the state in violation of state law, rules and regulations, and issued permits.

30. The Director further finds that the Facility must immediately cease discharging wastewater into its wastewater lagoons to prevent injury and exposure to the public and the environment.

## **II. COMPLIANCE ORDER**

### **IT IS HEREBY ORDERED THAT:**

31. Respondent must immediately cease discharge of industrial wastewater into its

wastewater lagoons and take whatever action is necessary to ensure that the Facility and its equipment are operating in compliance with the Act, Title 119, Title 123, NPDES Permit # NE0137634, and WWF Construction Permits #2018-0060, #05-0158.

32. Respondent must immediately provide the Department with verification it has ceased discharge of industrial wastewater into its wastewater lagoons.

33. Respondent must not remove industrial wastewater from its wastewater lagoons except in accordance with standards and conditions for disposal of industrial wastewater laced with pesticides. It must be disposed at a location and in a manner approved by the Department.

34. Within thirty (30) days, Respondent shall submit a plan to the Department detailing a protocol for disposal of the industrial wastewater in the lagoons.

35. Respondent shall not resume discharge of any industrial wastewater into its wastewater lagoons until the Respondent provides verification to the Department that the wastewater lagoons are operating below freeboard above max and an industrial wastewater disposal protocol is in place.

36. Respondent shall promptly respond to any written communication by the Department. Any delay in responding to such communication shall be construed as non-compliance with this Order.

### **III. SUBMITTALS**

37. Any information or verification required to be submitted to the Department pursuant to this Order shall refer to FID #84069 and shall be sent to:

Reuel Anderson  
Wastewater, NDPES Compliance Section  
Nebraska Department of Environmental Quality  
PO Box 98922  
245 Fallbrook Blvd.  
Lincoln, NE 68509-8922  
402-471-2186

#### **IV. NOTICE OF OPPORTUNITY TO REQUEST A HEARING**

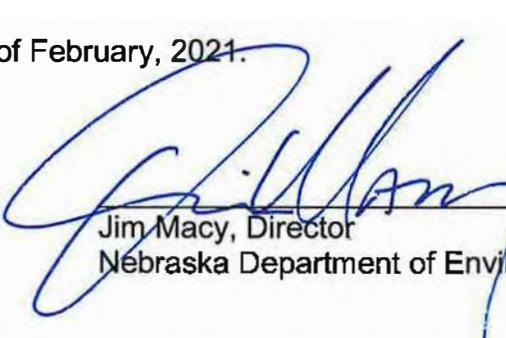
38. In accordance with Neb. Rev. Stat. §18-1507(4) this Emergency Complaint and Order shall become effective immediately. The Respondent is required to comply with the Emergency Order upon receipt.

39. The Respondent may request a hearing by submitting a written request to the Director. The request for hearing may be filed by mail to: Jim Macy, Director, State of Nebraska Department of Environmental Quality, P.O. Box 98922, Lincoln, Nebraska 68509-8922, or may be delivered to the Department's Lincoln office located at 245 Fallbrook Blvd., Lincoln, Nebraska. Such hearing shall be scheduled as soon as possible but not later than ten days after the request is made. The Emergency Complaint and Order shall remain in effect pending any hearing and shall not relieve the Respondent from immediate compliance.

40. This Emergency Complaint and Order does not preclude the Department from pursuing additional enforcement by administrative order for injunctive relief and penalties based on violations of the Act.

41. The Department further reserves the right to impose additional obligations to abate or eliminate the emergency or pollution caused by the Respondent.

Dated this 4 day of February, 2021.

  
Jim Macy, Director  
Nebraska Department of Environment and Energy

CERTIFICATE OF SERVICE

I hereby certify that a copy of the foregoing Emergency Order and Complaint, was served by certified United States mail, postage prepaid, return receipt requested this 4<sup>th</sup> day of February, 2021, upon the Respondent's Registered Agent listed below at their mailing address:

Capitol Corporate Services, Inc.  
1125 S. 103<sup>rd</sup> St.  
Suite 800  
Omaha, NE 68124

  
Lauren Triplett  
Staff Assistant, Legal Division

USPS Label # 9414811898765899429806

# Attachment A

## **Nebraska Department of Environment and Energy (NDEE)**

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### **MEMORANDUM**

**To:** File

**From:** Hillary Stoll

**Date:** February 1, 2021

**RE:** AltEn, LLC

**NDEE ID:** 84069

**Program ID:** WWF, PCS NE0137634

**Subject:** Site Visit Report (Wastewater Aspects Only)

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### **Summary of Site Visit and Information Acquired During Visit:**

Mark Pomajzl, Jason Holsten, and Hillary Stoll (myself) arrived at the AltEn, LLC facility in Mead, Nebraska at about 9:30 AM on February 1, 2021. Mr. Scott Tingelhoff, plant manager, was not at the facility, but Mr. Ken Peterson was present. We stated that we were conducting a site visit and would like to tour the facility and observe the wastewater lagoons. At no time was access denied. Mr. Peterson provided the tour. The observations included in this memorandum are only those that are related to wastewater. After the tour, we spoke with Mr. Peterson and Mr. Dean Egr for the exit summary.

### **Observations Noted:**

1. Mr. Dean Egr confirmed that the facility completed an approved wastewater construction project (NDEE Document ID 20200085932) to help reduce excessive air in the force main that transports wastewater to the Northwest (also known as “West”) lagoon cell. This involved constructing a new force main that discharges wastewater into the Northwest lagoon cell. According to Mr. Egr, this project was completed last week (Photos 4 and 6).
2. Mr. Dean Egr confirmed that a sump pump failed so they manually transported sump waste into the Northwest lagoon cell via a honeywagon (Photo 7).
3. The water levels in each of the three lagoon cells have exceeded the maximum operating depths and are operating in the area designed for freeboard. The maximum operating depths, according to the approved plans and specifications for the construction of the Northwest lagoon cell, as well as modifications to the Northeast and Southeast lagoon cells, are included in Table 1. The pipe invert elevations for the overflow pipes between the lagoon cells are included in Table 2.

4. From a visual observation of the depth marker, the Northwest cell is operating at a depth of about 18.5 feet, which is above the maximum operating depth and within the area designed for freeboard (Photos 8 and 9).
5. From a visual observation of the depth marker, the Northeast cell is operating at a depth of about 19 to 19.5 feet. It was difficult to determine the operating depth based on the depth marker due to snow (Photo 15). The invert elevation of the overflow pipe entering the Northeast lagoon cell from the Northwest lagoon cell is 116.0', per the approved plans and specifications (Table 2). The water surface in the Northeast lagoon was above this invert, suggesting that it was above the maximum operating depth and within the area designed for freeboard (Photos 10 and 11). The Northeast lagoon is designed to drain into the Southeast lagoon when it reaches its maximum operating depth. Wastewater was flowing from the Northeast lagoon to the Southeast lagoon during the site visit (Photos 16-18).
6. From a visual observation of the depth marker, the Southeast cell is operating at a depth of about 19 to 19.5 feet. It was difficult to determine the depth based on the depth marker due to snow (Photo 19). The invert elevation of the overflow pipe entering the Southeast cell from the Northeast cell is 115.3', per the approved plans and specifications (Table 2). The elevation of the water in the Southeast cell appeared to be greater than the elevation of the middle of the overflow pipe (Photos 17 and 18). Per the approved plans and specifications, the overflow pipe is fifteen inches in diameter. Since the overflow pipe is fifteen inches in diameter, this suggests that the elevation of the water in the Southeast cell was at least 115.925' (115.3' + 0.625'), which is greater than the maximum operating depth and within the area designed for freeboard.
7. The Northeast lagoon cell liner has not been repaired and is still badly damaged (Photos 12-14). The multiple "whales" in the Northwest lagoon cell liner have also not been repaired.
8. One of the fences enclosing the wastewater lagoons was left open (Photo 5).

**Table 1. Information Obtained from Document ID 20180025019 & Document ID 20180034184 (WWF Construction Permit 2018-0060)**

Lagoon Cell	Top of Berm Elevation given in Plans & Specifications	Depth at Top of Berm (ft)	Design Freeboard (ft)	Maximum Operating Depth (ft)	Maximum Operating Elevation
Northwest (Influent Cell)	118.6'	20	2.1	17.9	116.5'
Northeast (Second Cell)	118.6'	22	3.0	19	115.6'
Southeast (Final Cell)	118.6'	22	3.0	19	115.6'

**Table 2. Overflow Pipe Information Obtained from Document ID 20180025019 & Document ID 20180034184 (WWF Construction Permit 2018-0060)**

	Influent Invert Elevation	Effluent Invert Elevation
<b>Overflow Pipe from NW Cell to NE Cell</b>	116.5'	116.0'
<b>Overflow Pipe from NE Cell to SE Cell</b>	115.8'	115.3'

**Concerns:**

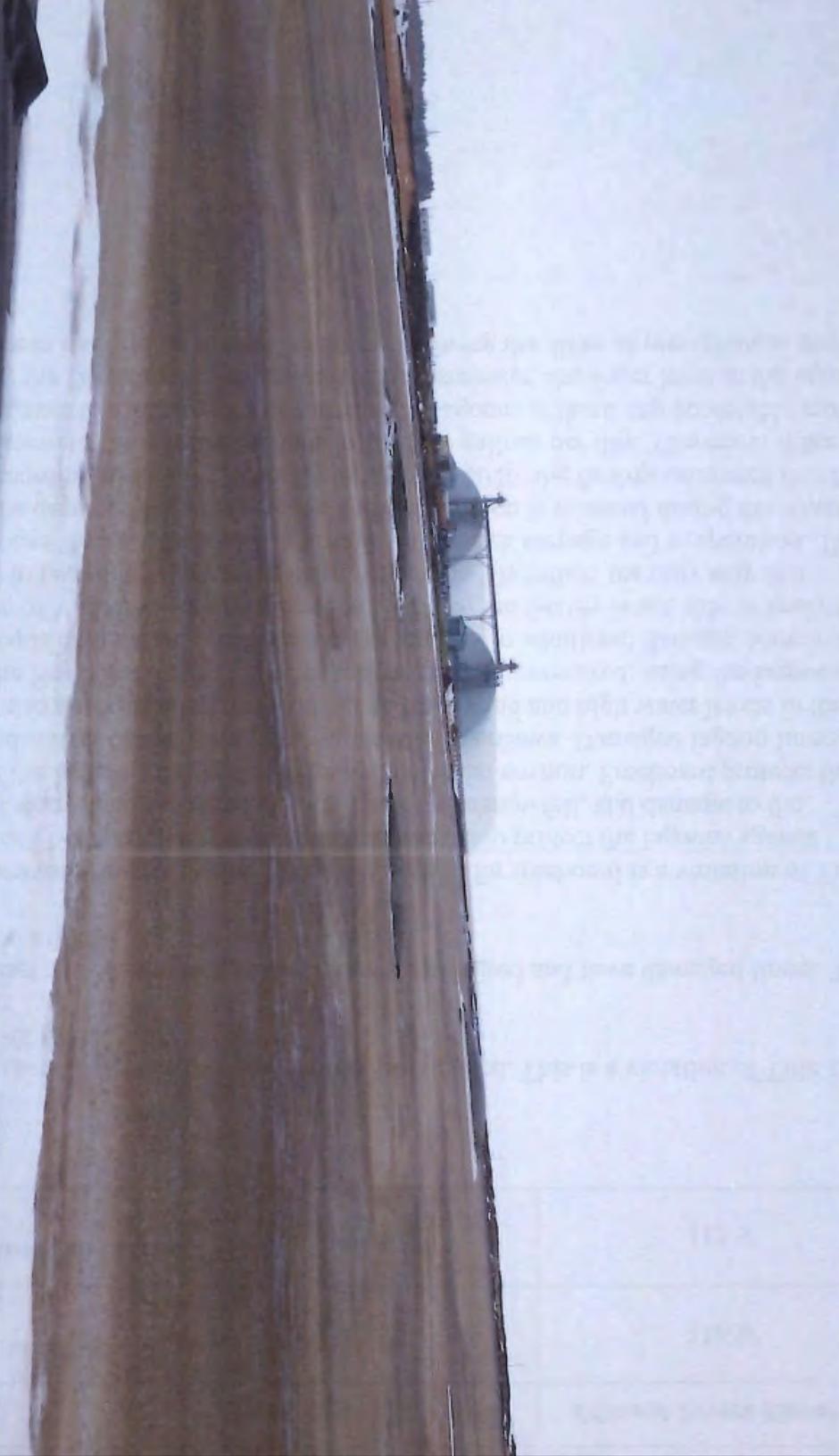
1. One of the gates to the wastewater lagoons was opened. This is a violation of Title 123, Chapter 11-008.03.
2. The Northeast and Northwest lagoons remain unrepairs and have damaged liners. This is a violation of Title 123, Chapter 11-008.06.
3. Operating a wastewater lagoon in the area designed for freeboard is a violation of Title 123, Chapter 11-008.07. The purpose of freeboard is to protect the lagoons against exceptional storms and associated winds, heavy rain/snowfall, and damage to the integrity of the lagoon from wave action and potential erosion. Freeboard protects the property and waters of the State from wastewater overflows. Damaged lagoon liners with whales are also susceptible to further damage from wind and high water levels in the lagoons. The Northeast and Northwest lagoons remain unrepairs; using the lagoons without adequate freeboard could subject the lagoons to additional damage. Moreover, per a Notice of Violation dated September 13, 2019, the facility is not able to apply the wastewater to land due to the presence of pesticides. Therefore, the only way that wastewater can “leave” the lagoons currently is through seepage and evaporation. The lagoons were designed for zero seepage and evaporation is minimal during the winter months. According to a site visit on September 11, 2020, the facility estimated that the average wastewater flow to the lagoons is 100,000 gallons per day. Therefore, if the facility continues to discharge wastewater to the lagoons without any acceptable method approved by the Department to dispose of the wastewater, the water level in the lagoons will continue to rise and be susceptible to overflowing the dikes as precipitation increases in the spring.

**Attachments**

1. Photos
2. Map

otos

e: Not all photos taken at the site are included in this memorandum.



to 1

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: A1EN, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: Southwest

Description: Overview of Northwest lagoon cell (influent cell)

n, LLC Site Visit, 2/1/2021



to 2

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Orientation Facing: Northwest

Description: Overview of Northeast lagoon cell (second cell)



to 3

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: Southwest

Description: Overview of Southeast lagoon cell (final cell)



to 4

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Project Name/Location: AltEn, LLC, Mead, NE

Project Identifier: IIS #84069, PCS NE0137634

Section Facing: N/A

Description: Newly constructed influent pipe into the Northwest lagoon cell



to 5

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Direction Facing: Southwest

Description: Open gate south of the Northwest lagoon cell



to 6

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: Southeast

Description: Influent pipe into the Northwest lagoon cell



to 7

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: N/A

Description: Tracks from a honeywagon used to transport wastewater from the sump into the Northwest lagoon cell



to 8

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Entity Name/Project Name: AltEn, LLC, Mead, NE

Entity IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: N/A

Description: Depth marker for Northwest lagoon cell



to 9

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Direction Facing: Northwest

Description: Depth marker for Northwest lagoon cell



to 10

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: South

Description: Overflow pipe from Northwest lagoon cell (right) to Northeast lagoon cell (left)



to 11

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Orientation Facing: Southwest

Description: Overflow pipe between the Northwest lagoon cell (right) and the Northeast lagoon cell (left)



to 12

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: N/A

Description: Liner tear in Northeast lagoon cell



to 13

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: Northwest

Description: Liner "whale" in Northeast lagoon cell



to 14

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Orientation Facing: Northwest

Description: Liner "whales" in Northeast lagoon cell



to 15  
e Taken: February 1, 2021  
tographer: Hillary Stoll  
ility Name/Project Name: AltEn, LLC, Mead, NE  
ility IIS number/Project Identifier: IIS #84069, PCS NE0137634  
ection Facing: Southwest  
cription: Depth marker for Northeast lagoon cell. The top line is 22 feet. The "1" for 21 feet is just visible above the snow.



Photo 16  
Date Taken: February 1, 2021  
Photographer: Hillary Stoll  
Facility Name/Project Name: AltEn, LLC, Mead, NE  
Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634  
Section Facing: Southeast  
Description: Overflow pipe from Northeast lagoon cell (left) into Southeast lagoon cell (right)



to 17

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: East

Description: Overflow pipe from Northeast lagoon cell (left) to Southeast lagoon cell (right)



to 18

Date Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Section Facing: East

Description: Overflow pipe from Northeast lagoon cell (left) entering the Southeast lagoon cell (right)



to 19

Photo Taken: February 1, 2021

Photographer: Hillary Stoll

Facility Name/Project Name: AltEn, LLC, Mead, NE

Facility IIS number/Project Identifier: IIS #84069, PCS NE0137634

Direction Facing: N/A

Description: Depth marker on Southeast lagoon cell. The top line is 23 feet (right above the snow).



All information included in this map is based on design information from Document ID 20180025019 & Document ID 20180034184.

11S: 84069  
PCS: NEO137634  
mp✓

April 29, 2019

## Attachment B

Mr. Dan Bigbee  
EA Engineering, Science, and Technology  
221 Sun Valley Blvd  
Suite D  
Lincoln, NE 68528

RE: Project: Alten LLC Digestor Sampling  
Pace Project No.: 60299511

Dear Mr. Bigbee:

Enclosed are the analytical results for sample(s) received by the laboratory on April 10, 2019. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

Some analyses have been subcontracted outside of the Pace Network. The subcontracted laboratory report has been attached.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

*Trudy Gipson*

Trudy Gipson  
trudy.gipson@pacelabs.com  
1(913)563-1405  
Project Manager

Enclosures

cc: Mr. Kent Dixon, EA Engineering, Science, and Technology



### REPORT OF LABORATORY ANALYSIS

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## CERTIFICATIONS

Project: Alten LLC Digester Sampling  
Pace Project No.: 60299511

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**Dallas Certification IDs:**

400 West Bethany Dr Suite 190, Allen, TX 75013  
Florida Certification #: E871118  
Texas T104704232-18-26  
EPA# TX00074  
Texas Certification #: T104704232-18-26

Kansas Certification #: E-10388  
Arkansas Certification #: 88-0647  
Oklahoma Certification #: 8727  
Louisiana Certification #: 30686  
Iowa Certification #: 408

## REPORT OF LABORATORY ANALYSIS

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## SAMPLE SUMMARY

Project: Alten LLC Digester Sampling  
Pace Project No.: 60299511

Lab ID	Sample ID	Matrix	Date Collected	Date Received
60299511001	WEST LAGOON	Water	04/08/19 12:35	04/10/19 09:45
60299511002	OVERFLOW LAGOON	Water	04/08/19 13:30	04/10/19 09:45
60299511003	WET CAKE	Solid	04/08/19 11:00	04/10/19 09:45
60299511004	SEED CORN	Solid	04/08/19 13:30	04/10/19 09:45

## REPORT OF LABORATORY ANALYSIS

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### SAMPLE ANALYTE COUNT

Project: Alten LLC Digestor Sampling  
 Pace Project No.: 60299511

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
60299511001	<b>WEST LAGOON</b>	EPA 8081	JL	9	PASI-D
		EPA 8151	DAT	3	PASI-D
60299511002	<b>OVERFLOW LAGOON</b>	EPA 8081	JL	9	PASI-D
		EPA 8151	DAT	3	PASI-D
60299511003	<b>WET CAKE</b>	EPA 8081	JL	9	PASI-D
		EPA 8151	DAT	3	PASI-D
60299511004	<b>SEED CORN</b>	EPA 8081	JL	9	PASI-D
		EPA 8151	DAT	3	PASI-D

### REPORT OF LABORATORY ANALYSIS

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## PROJECT NARRATIVE

Project: Alten LLC Digestor Sampling

Pace Project No.: 60299511

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**Method:** EPA 8081

**Description:** 8081 GCS Pesticides, TCLP

**Client:** EA Engineering, Science, and Technology

**Date:** April 29, 2019

### General Information:

4 samples were analyzed for EPA 8081. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

### Hold Time:

The samples were analyzed within the method required hold times with any exceptions noted below.

### Sample Preparation:

The samples were prepared in accordance with EPA 3510 with any exceptions noted below.

### Initial Calibrations (including MS Tune as applicable):

All criteria were within method requirements with any exceptions noted below.

### Continuing Calibration:

All criteria were within method requirements with any exceptions noted below.

### Surrogates:

All surrogates were within QC limits with any exceptions noted below.

QC Batch: 116453

S1: Surrogate recovery outside laboratory control limits (confirmed by re-analysis).

- WEST LAGOON (Lab ID: 60299511001)
- Tetrachloro-m-xylene (S)

### Method Blank:

All analytes were below the report limit in the method blank, where applicable, with any exceptions noted below.

### Laboratory Control Spike:

All laboratory control spike compounds were within QC limits with any exceptions noted below.

### Matrix Spikes:

All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

### Additional Comments:

#### Analyte Comments:

QC Batch: 116453

D3: Sample was diluted due to the presence of high levels of non-target analytes or other matrix interference.

- WEST LAGOON (Lab ID: 60299511001)
- Decachlorobiphenyl (S)

## REPORT OF LABORATORY ANALYSIS

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## PROJECT NARRATIVE

Project: Alten LLC Digestor Sampling  
Pace Project No.: 60299511

**Method:** EPA 8151  
**Description:** 8151 Chlorinate Herbicide TCLP  
**Client:** EA Engineering, Science, and Technology  
**Date:** April 29, 2019

**General Information:**

4 samples were analyzed for EPA 8151. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

**Hold Time:**

The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**

The samples were prepared in accordance with EPA 8151 with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**

All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**

All criteria were within method requirements with any exceptions noted below.

**Surrogates:**

All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**

All analytes were below the report limit in the method blank, where applicable, with any exceptions noted below.

**Laboratory Control Spike:**

All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**

All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

QC Batch: 116456

A matrix spike and/or matrix spike duplicate (MS/MSD) were performed on the following sample(s): 60299511002

R1: RPD value was outside control limits.

- MSD (Lab ID: 524640)
- 2,4,5-TP (Silvex)

**Additional Comments:**

This data package has been reviewed for quality and completeness and is approved for release.

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: Alten LLC Digestor Sampling  
Pace Project No.: 60299511

Sample: WEST LAGOON		Lab ID: 60299511001		Collected: 04/08/19 12:35		Received: 04/10/19 09:45		Matrix: Water		
Parameters	Results	Units	Report Limit	MDL	Reg. Limit	DF	Prepared	Analyzed	CAS No.	Qual
<b>8081 GCS Pesticides, TCLP</b>										
Analytical Method: EPA 8081 Preparation Method: EPA 3510 Leachate Method/Date: EPA 1311; 04/22/19 09:37										
gamma-BHC (Lindane)	ND	mg/L	0.0025	0.00025	.4	5	04/23/19 10:30	04/23/19 17:03	58-89-9	
Chlordane (Technical)	ND	mg/L	0.010	0.0020	.03	5	04/23/19 10:30	04/23/19 17:03	57-74-9	
Endrin	ND	mg/L	0.0010	0.00020	.02	5	04/23/19 10:30	04/23/19 17:03	72-20-8	
Heptachlor	ND	mg/L	0.00050	0.00030	.008	5	04/23/19 10:30	04/23/19 17:03	76-44-8	
Heptachlor epoxide	ND	mg/L	0.00050	0.00020	.008	5	04/23/19 10:30	04/23/19 17:03	1024-57-3	
Methoxychlor	ND	mg/L	0.0050	0.00025	10	5	04/23/19 10:30	04/23/19 17:03	72-43-5	
Toxaphene	ND	mg/L	0.015	0.010	.5	5	04/23/19 10:30	04/23/19 17:03	8001-35-2	
<b>Surrogates</b>										
Decachlorobiphenyl (S)	42	%.	12-162			5	04/23/19 10:30	04/23/19 17:03	2051-24-3	D3
Tetrachloro-m-xylene (S)	49	%.	54-127			5	04/23/19 10:30	04/23/19 17:03	877-09-8	S1
<b>8151 Chlorinate Herbicide TCLP</b>										
Analytical Method: EPA 8151 Preparation Method: EPA 8151 Leachate Method/Date: EPA 1311; 04/22/19 09:37										
2,4-D	ND	mg/L	0.010	0.0031	10	1	04/23/19 10:30	04/24/19 04:31	94-75-7	
2,4,5-TP (Silvex)	ND	mg/L	0.010	0.0023		1	04/23/19 10:30	04/24/19 04:31	93-72-1	
<b>Surrogates</b>										
2,4-DCAA (S)	32	%.	10-171			1	04/23/19 10:30	04/24/19 04:31	19719-28-9	

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: Alten LLC Digester Sampling

Pace Project No.: 60299511

Sample: OVERFLOW LAGOON		Lab ID: 60299511002		Collected: 04/08/19 13:30		Received: 04/10/19 09:45		Matrix: Water		
Parameters	Results	Units	Report Limit	MDL	Reg. Limit	DF	Prepared	Analyzed	CAS No.	Qual
<b>8081 GCS Pesticides, TCLP</b>										
Analytical Method: EPA 8081 Preparation Method: EPA 3510										
Leachate Method/Date: EPA 1311; 04/22/19 11:16										
gamma-BHC (Lindane)	ND	mg/L	0.00050	0.000050	.4	1	04/23/19 10:30	04/23/19 15:34	58-89-9	
Chlordane (Technical)	ND	mg/L	0.0020	0.00041	.03	1	04/23/19 10:30	04/23/19 15:34	57-74-9	
Endrin	ND	mg/L	0.00020	0.000040	.02	1	04/23/19 10:30	04/23/19 15:34	72-20-8	
Heptachlor	ND	mg/L	0.00010	0.000060	.008	1	04/23/19 10:30	04/23/19 15:34	76-44-8	
Heptachlor epoxide	ND	mg/L	0.00010	0.000040	.008	1	04/23/19 10:30	04/23/19 15:34	1024-57-3	
Methoxychlor	ND	mg/L	0.0010	0.000050	10	1	04/23/19 10:30	04/23/19 15:34	72-43-5	
Toxaphene	ND	mg/L	0.0030	0.0021	.5	1	04/23/19 10:30	04/23/19 15:34	8001-35-2	
<b>Surrogates</b>										
Decachlorobiphenyl (S)	67	%.	12-162			1	04/23/19 10:30	04/23/19 15:34	2051-24-3	
Tetrachloro-m-xylene (S)	72	%.	54-127			1	04/23/19 10:30	04/23/19 15:34	877-09-8	
<b>8151 Chlorinate Herbicide TCLP</b>										
Analytical Method: EPA 8151 Preparation Method: EPA 8151										
Leachate Method/Date: EPA 1311; 04/22/19 11:16										
2,4-D	ND	mg/L	0.010	0.0031	10	1	04/23/19 10:30	04/24/19 04:09	94-75-7	
2,4,5-TP (Silvex)	ND	mg/L	0.010	0.0023	1	1	04/23/19 10:30	04/24/19 04:09	93-72-1	R1
<b>Surrogates</b>										
2,4-DCAA (S)	53	%.	10-171			1	04/23/19 10:30	04/24/19 04:09	19719-28-9	

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: Alten LLC Digestor Sampling  
 Pace Project No.: 60299511

Sample: WET CAKE      Lab ID: 60299511003      Collected: 04/08/19 11:00      Received: 04/10/19 09:45      Matrix: Solid

**Results reported on a "wet-weight" basis**

Parameters	Results	Units	Report Limit	MDL	Reg. Limit	DF	Prepared	Analyzed	CAS No.	Qual
<b>8081 GCS Pesticides, TCLP</b>		Analytical Method: EPA 8081 Preparation Method: EPA 3510 Leachate Method/Date: EPA 1311; 04/22/19 11:16								
gamma-BHC (Lindane)	ND	mg/L	0.00050	0.000050	.4	1	04/23/19 10:30	04/23/19 15:48	58-89-9	
Chlordane (Technical)	ND	mg/L	0.0020	0.00041	.03	1	04/23/19 10:30	04/23/19 15:48	57-74-9	
Endrin	ND	mg/L	0.00020	0.000040	.02	1	04/23/19 10:30	04/23/19 15:48	72-20-8	
Heptachlor	ND	mg/L	0.00010	0.000060	.008	1	04/23/19 10:30	04/23/19 15:48	76-44-8	
Heptachlor epoxide	ND	mg/L	0.00010	0.000040	.008	1	04/23/19 10:30	04/23/19 15:48	1024-57-3	
Methoxychlor	ND	mg/L	0.0010	0.000050	10	1	04/23/19 10:30	04/23/19 15:48	72-43-5	
Toxaphene	ND	mg/L	0.0030	0.0021	.5	1	04/23/19 10:30	04/23/19 15:48	8001-35-2	
<b>Surrogates</b>										
Decachlorobiphenyl (S)	50	%.	12-162			1	04/23/19 10:30	04/23/19 15:48	2051-24-3	
Tetrachloro-m-xylene (S)	76	%.	54-127			1	04/23/19 10:30	04/23/19 15:48	877-09-8	
<b>8151 Chlorinate Herbicide TCLP</b>		Analytical Method: EPA 8151 Preparation Method: EPA 8151 Leachate Method/Date: EPA 1311; 04/22/19 11:16								
2,4-D	ND	mg/L	0.010	0.0031	10	1	04/23/19 10:30	04/24/19 04:52	94-75-7	
2,4,5-TP (Silvex)	ND	mg/L	0.010	0.0023	1	1	04/23/19 10:30	04/24/19 04:52	93-72-1	
<b>Surrogates</b>										
2,4-DCAA (S)	62	%.	10-171			1	04/23/19 10:30	04/24/19 04:52	19719-28-9	

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: Alten LLC Digestor Sampling

Pace Project No.: 60299511

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**Sample: SEED CORN**      Lab ID: **60299511004**      Collected: 04/08/19 13:30      Received: 04/10/19 09:45      Matrix: Solid

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*Results reported on a "wet-weight" basis*

Parameters	Results	Units	Report Limit	MDL	Reg. Limit	DF	Prepared	Analyzed	CAS No.	Qual
<b>8081 GCS Pesticides, TCLP</b> Analytical Method: EPA 8081 Preparation Method: EPA 3510										
Leachate Method/Date: EPA 1311; 04/22/19 11:16										
gamma-BHC (Lindane)	ND	mg/L	0.00050	0.000050	.4	1	04/23/19 10:30	04/23/19 16:02	58-89-9	
Chlordane (Technical)	ND	mg/L	0.0020	0.00041	.03	1	04/23/19 10:30	04/23/19 16:02	57-74-9	
Endrin	ND	mg/L	0.00020	0.000040	.02	1	04/23/19 10:30	04/23/19 16:02	72-20-8	
Heptachlor	ND	mg/L	0.00010	0.000060	.008	1	04/23/19 10:30	04/23/19 16:02	76-44-8	
Heptachlor epoxide	ND	mg/L	0.00010	0.000040	.008	1	04/23/19 10:30	04/23/19 16:02	1024-57-3	
Methoxychlor	ND	mg/L	0.0010	0.000050	10	1	04/23/19 10:30	04/23/19 16:02	72-43-5	
Toxaphene	ND	mg/L	0.0030	0.0021	.5	1	04/23/19 10:30	04/23/19 16:02	8001-35-2	
<b>Surrogates</b>										
Decachlorobiphenyl (S)	57	%.	12-162			1	04/23/19 10:30	04/23/19 16:02	2051-24-3	
Tetrachloro-m-xylene (S)	60	%.	54-127			1	04/23/19 10:30	04/23/19 16:02	877-09-8	
<b>8151 Chlorinate Herbicide TCLP</b> Analytical Method: EPA 8151 Preparation Method: EPA 8151										
Leachate Method/Date: EPA 1311; 04/22/19 11:16										
2,4-D	ND	mg/L	0.010	0.0031	10	1	04/23/19 10:30	04/24/19 05:13	94-75-7	
2,4,5-TP (Silvex)	ND	mg/L	0.010	0.0023	1	1	04/23/19 10:30	04/24/19 05:13	93-72-1	
<b>Surrogates</b>										
2,4-DCAA (S)	53	%.	10-171			1	04/23/19 10:30	04/24/19 05:13	19719-28-9	

## REPORT OF LABORATORY ANALYSIS

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**QUALITY CONTROL DATA**

Project: Alten LLC Digestor Sampling

Pace Project No.: 60299511

QC Batch: 116453

Analysis Method: EPA 8081

QC Batch Method: EPA 3510

Analysis Description: 8081 GCS TCLP Pesticides

Associated Lab Samples: 60299511001, 60299511002, 60299511003, 60299511004

METHOD BLANK: 524630

Matrix: Water

Associated Lab Samples: 60299511001, 60299511002, 60299511003, 60299511004

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Chlordane (Technical)	mg/L	ND	0.0020	0.00041	04/23/19 14:36	
Endrin	mg/L	ND	0.00020	0.000040	04/23/19 14:36	
gamma-BHC (Lindane)	mg/L	ND	0.00050	0.000050	04/23/19 14:36	
Heptachlor	mg/L	ND	0.00010	0.000060	04/23/19 14:36	
Heptachlor epoxide	mg/L	ND	0.00010	0.000040	04/23/19 14:36	
Methoxychlor	mg/L	ND	0.0010	0.000050	04/23/19 14:36	
Toxaphene	mg/L	ND	0.0030	0.0021	04/23/19 14:36	
Decachlorobiphenyl (S)	%.	69	12-162		04/23/19 14:36	
Tetrachloro-m-xylene (S)	%.	80	54-127		04/23/19 14:36	

LABORATORY CONTROL SAMPLE: 524631

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Endrin	mg/L	0.005	0.0039	77	56-152	
gamma-BHC (Lindane)	mg/L	0.005	0.0044	88	70-135	
Heptachlor	mg/L	0.005	0.0042	84	59-139	
Heptachlor epoxide	mg/L	0.005	0.0040	79	65-138	
Methoxychlor	mg/L	0.005	0.0040	80	39-160	
Decachlorobiphenyl (S)	%.			64	12-162	
Tetrachloro-m-xylene (S)	%.			73	54-127	

MATRIX SPIKE &amp; MATRIX SPIKE DUPLICATE: 524633

524634

Parameter	Units	MS		MSD		MS Result	MS % Rec	MSD Result	MSD % Rec	% Rec Limits		Max RPD	Max Qual
		60299511002	Result	Spike Conc.	MSD Conc.					Max RPD	Max Qual		
Endrin	mg/L	ND	0.005	0.005	0.0044	0.0041	88	83	39-173	6	40		
gamma-BHC (Lindane)	mg/L	ND	0.005	0.005	0.0056	0.0053	111	106	69-139	5	40		
Heptachlor	mg/L	ND	0.005	0.005	0.0046	0.0042	92	84	48-141	9	40		
Heptachlor epoxide	mg/L	ND	0.005	0.005	0.0059	0.0056	119	112	28-164	6	40		
Methoxychlor	mg/L	ND	0.005	0.005	0.0043	0.0041	86	82	20-178	5	40		
Decachlorobiphenyl (S)	%.						69	61	12-162				
Tetrachloro-m-xylene (S)	%.						69	64	54-127				

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

**REPORT OF LABORATORY ANALYSIS**

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## QUALITY CONTROL DATA

Project: Alten LLC Digester Sampling  
Pace Project No.: 60299511

QC Batch: 116456 Analysis Method: EPA 8151  
QC Batch Method: EPA 8151 Analysis Description: 8151 GCS TCLP Herbicides  
Associated Lab Samples: 60299511001, 60299511002, 60299511003, 60299511004

METHOD BLANK: 524637 Matrix: Water

Associated Lab Samples: 60299511001, 60299511002, 60299511003, 60299511004

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
2,4,5-TP (Silvex)	mg/L	ND	0.010	0.0023	04/24/19 02:44	
2,4-D	mg/L	ND	0.010	0.0031	04/24/19 02:44	
2,4-DCAA(S)	%.	61	10-171		04/24/19 02:44	

LABORATORY CONTROL SAMPLE: 524638

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
2,4,5-TP (Silvex)	mg/L	0.03	0.018	61	28-145	
2,4-D	mg/L	0.03	0.018	59	28-145	
2,4-DCAA (S)	%.			52	10-171	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 524639 524640

Parameter	Units	MS Spike		MSD Spike		MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD RPD	Max R1
		60299511002	Result	Conc.	Conc.							
2,4,5-TP (Silvex)	mg/L	ND	0.03	0.03	0.014	0.019	48	62	20-129	25	20	R1
2,4-D	mg/L	ND	0.03	0.03	0.019	0.023	59	73	12-162	20	20	
2,4-DCAA (S)	%.						48	61	10-171			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

## REPORT OF LABORATORY ANALYSIS

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## QUALIFIERS

Project: Alten LLC Digestor Sampling  
Pace Project No.: 60299511

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### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.  
ND - Not Detected at or above adjusted reporting limit.  
TNTC - Too Numerous To Count  
J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.  
MDL - Adjusted Method Detection Limit.  
PQL - Practical Quantitation Limit.  
RL - Reporting Limit - The lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.  
S - Surrogate  
1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.  
Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.  
LCS(D) - Laboratory Control Sample (Duplicate)  
MS(D) - Matrix Spike (Duplicate)  
DUP - Sample Duplicate  
RPD - Relative Percent Difference  
NC - Not Calculable.  
SG - Silica Gel - Clean-Up  
U - Indicates the compound was analyzed for, but not detected.  
N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.  
Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.  
TNI - The NELAC Institute.

### LABORATORIES

PASI-D Pace Analytical Services - Dallas

### ANALYTE QUALIFIERS

- D3 Sample was diluted due to the presence of high levels of non-target analytes or other matrix interference.
- R1 RPD value was outside control limits.
- S1 Surrogate recovery outside laboratory control limits (confirmed by re-analysis).

## REPORT OF LABORATORY ANALYSIS

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### QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: Alten LLC Digestor Sampling  
 Pace Project No.: 60299511

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
60299511001	WEST LAGOON	EPA 3510	116453	EPA 8081	116504
60299511002	OVERFLOW LAGOON	EPA 3510	116453	EPA 8081	116504
60299511003	WET CAKE	EPA 3510	116453	EPA 8081	116504
60299511004	SEED CORN	EPA 3510	116453	EPA 8081	116504
60299511001	WEST LAGOON	EPA 8151	116456	EPA 8151	116528
60299511002	OVERFLOW LAGOON	EPA 8151	116456	EPA 8151	116528
60299511003	WET CAKE	EPA 8151	116456	EPA 8151	116528
60299511004	SEED CORN	EPA 8151	116456	EPA 8151	116528

### REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
 without the written consent of Pace Analytical Services, LLC.

**Sample Condition Upon Receipt**
**WO# : 60299511**

**60299511**

 Client Name: EA Eng.

 Courier: FedEx  UPS  VIA  Clay  PEX  ECI  Pace  Xroads  Client  Other 

 Tracking #: i2 635 804 01 9576 4765 Pace Shipping Label Used? Yes  No 

 Custody Seal on Cooler/Box Present: Yes  No  Seals intact. Yes  No 

 Packing Material: Bubble Wrap  Bubble Bags  Foam  None  Other 

 Thermometer Used: T-298 Type of Ice: Ice Blue  None 

 Cooler Temperature (°C): As-read 2.2 Corr. Factor -0.1 Corrected 2.1

 Date and initials of person examining contents: 4/10/19 JJ

Temperature should be above freezing to 6°C

Chain of Custody present:	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A
Chain of Custody relinquished:	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A
Samples arrived within holding time	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A
Short Hold Time analyses (<72hr):	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A
Rush Turn Around Time requested:	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A
Sufficient volume:	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A
Correct containers used	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A
Pace containers used	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A
Containers intact:	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A
Unpreserved 5035A / TX1005/1006 soils frozen in 48hrs?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A
Filtered volume received for dissolved tests?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A
Sample labels match COC: Date / time / ID / analyses <u>all 3</u>	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A
Samples contain multiple phases? Matrix: <u>wt + SLS</u>	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A
Containers requiring pH preservation in compliance? (HNO <sub>3</sub> , H <sub>2</sub> SO <sub>4</sub> , HCl<2; NaOH>9 Sulfide, NaOH>10 Cyanide) (Exceptions: VOA, Micro, O&G, KS TPH, OK-DRO)	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A
Cyanide water sample checks:	List sample IDs, volumes, lot #'s of preservative and the date/time added.
Lead acetate strip turns dark? (Record only)	<input type="checkbox"/> Yes <input type="checkbox"/> No
Potassium iodide test strip turns blue/purple? (Preserve)	<input type="checkbox"/> Yes <input type="checkbox"/> No
Blank present	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A
Headspace in VOA vials (>6mm)	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A
Samples from USDA Regulated Area: State: <u>NE</u>	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A
Additional labels attached to 5035A / TX1005 vials in the field?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A

Client Notification/ Resolution: Copy COC to Client? Y / N Field Data Required? Y / N

Person Contacted: \_\_\_\_\_ Date/Time: \_\_\_\_\_

Comments/ Resolution: \_\_\_\_\_

 Project Manager Review: TDG Date: 4-11-19

**The Chain-of-Custody is a LEGAL DOCUMENT** All relevant fields must be completed accurately.

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Required Project Information:	
Invoicing Information:	
Project Name:	EA Eng Science and Technology
Report To:	Dan Bigbee
Attention:	Accounts Payable, Dan Bigbee
Copy To:	Kent Dixon
Company Name:	EA Eng Science and Technology
REGULATORY AGENCY	
Purchase Order No:	LSO#
Project Name:	AIEN LLC
Project Number:	AIEN LLC Digester Sampling
Reference:	Page Quote Re: 56462 (JR)
Manager:	Trudy Gipson 913-563-1405
Page Profile #:	12849 Lines 1 & 2
<b>STATE:</b>	NE

Information		Valid Matrix Codes	
MATRIX	CODE	MATRIX	CODE
DRAINING WATER	DW	WATER	WT
WASTE WATER	WW	PRODUCT	P
SOLID/FLUID	SL	SL	
ON	OL	COMPOSITE	COMPOSITE
WP	WP	START	END/GRAB
AIR	AIR		
OTHER	OT		
TISSUE	TS		

**TRIX CODE** (see valid codes to left)

**AMPLE TYPE** (G=GRAB C=COMP)

**AMPLE TEMP AT COLLECTION**

**CONTAINERS**

reserved	Preservatives
D <sub>4</sub>	
3	
H	
H <sub>2</sub> O <sub>3</sub>	
anol	
r	

**alysis Test**

P - Pest\*

P - Herb\*

Individual Chlorine (Y/N)

Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

The City of Coeur d'Alene IFCN DOCUMENT #10000000000000000000000000000000

The Charter of Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

# CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Page: 1 of 1

Information		Section B Required Project Information		Section C Invoice Information			
EA Eng Science and Technology 221 Sun Valley Blvd Suite D Lincoln, NE 68528 drgbee@east.com		Report To: Dan Bigbee Copy To: Kent Dixon Purchase Order No.: LSO #		Attention: Accounts Payable, Dan Bigbee Company Name: EA Eng Science and Technology Address: Phone: 58482 (JR) Fax: 317-7612 Project Name: AIEen LLC Project Number: AIEen LLC Digestor Sampling			
SAMPLE ID (A-Z, 0-9 / -) Multiple IDs MUST BE UNIQUE		Valid Matrix Codes MATRIX CODES Other than water CW WATER WWT WASTE WATER WW PRODUCT P SCHENKID SI Oil OI Wire WI Air AR Other OT Tissue TS		REGULATORY AGENCY NPDES GROUND WATER DRINKING WATER UST RCRA OTHER			
COLLECTED		SAMPLE TYPE (G=GRAB (see valid codes to left) G=GRAB S=STAB E=ENDOGENE)		STATE: NE			
		SAMPLE TEMP AT COLLECTION		Requested Analysis Filtered (Y/N)			
		# OF CONTAINERS		VIN			
		Unpreserved H <sub>2</sub> SO <sub>4</sub> HNO <sub>3</sub> HCl NaOH Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> Methanol Other		Analysis Test			
				Neonicotinoids Screen* Strodoilurin Screen* Triazole Screen* Pyrethroid Screen* Organophosphae Screen*			
				Residual Chlorine (Y/N)			
				Pace Project No./L			
				60294511			
ADDITIONAL COMMENTS DRAFT ANALYTICAL REQUESTS		REINQUISED BY / AFFILIATION DATE TIME		ACCEPTED BY / AFFILIATION DATE TIME		SAMPLE CONDITIONS	
Initial former Kenzigian		4/8/19 4-9-19	4:30 1630	4/8/19 4-9-19	4:30 1630	Temp in °C Received on ice (Y/N)	Liquid Liquid Cake Corn
SAMPLER NAME AND SIGNATURE D. n. c. l. L.C.R. Inc. SPC-2 DATE Signed (MM/DD/YY)						Custody sealed Coated (Y/N)	
PRINT Name of SAMPLER:							
SIG NATURE of SAMPLER:							

	Document Name: Sample Condition Upon Receipt	Document Revised: 03-14-19 Page 1 of 1
	Document No.: F-DAL-C-001-rev.9	Issuing Authority: Pace Dallas Quality Office

### Sample Condition Upon Receipt

Dallas       Ft Worth

WO# : 75106394



Client Name: Pace-t-5 Project Work order: \_\_\_\_\_

Courier: FedEx  UPS  USPS  Client  LSO  PACE  Other: \_\_\_\_\_

Tracking #: 474482452890

Custody Seal on Cooler/Box: Yes  No  Packing Material: Bubble Wrap/Bags  Foam  None  Other

Received on ice: Yes  No  Type of Ice: Wet  Blue

Thermometer Used: 17-11 Cooler Temp °C: 5.7 (Recorded) 5.7 (Correction Factor) 5.7 (Actual)

Temperature should be above freezing to 6°C

Chain of Custody relinquished	Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>
Sampler name & signature on COC	Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> (BWW)
Short HT analyses (<72 hrs)	Yes <input type="checkbox"/> No <input checked="" type="checkbox"/>
Sufficient Volume received	Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>
Correct Container used	Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>
Container Intact	Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>
Sample pH Acceptable pH Strips: _____	Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input checked="" type="checkbox"/>
Residual Chlorine Present Cl Strips: _____	Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input checked="" type="checkbox"/>
Sulfide Present Lead Acetate Strips: _____	Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input checked="" type="checkbox"/>
Are soil samples (volatiles, TPH) received in 5035A Kits	Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input checked="" type="checkbox"/>
Unpreserved 5035A soil frozen within 48 hrs	Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input checked="" type="checkbox"/>
Headspace in VOA (>6mm)	Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input checked="" type="checkbox"/>
Project sampled in USDA Regulated Area: State Sampled: <u>NE</u>	Yes <input type="checkbox"/> No <input checked="" type="checkbox"/>
Non-Conformance(s):	Yes <input type="checkbox"/> No <input checked="" type="checkbox"/>

# of Custody



Pace Analytical  
www.paceanalytical.com

Samples were sent directly to the Subcontracting Laboratory.

State Of Origin: NE  
 Cert. Needed: Yes  
 No  
 Owner Received Date: 4/10/2019 Results Requested By: 4/24/2019

on  
 atical Kansas  
 Blvd  
 66219  
 (3)563-1405

Subcontract To

Pace Analytical Dallas  
 400 West Bethany Drive  
 Suite 190  
 Allen, TX 75013  
 Phone (972)727-1123

Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	Preserved Containers		LAB USE
					Unpreserved		
AGOON	PS	4/8/2019 12:35	60299511001	Water	1	X	
LOW LAGOON	PS	4/8/2019 13:30	60299511002	Water	1	X	
KKE	PS	4/8/2019 11:00	60299511003	Solid	1	X	
ORN	PS	4/8/2019 13:30	60299511004	Solid	1	X	

Released By	Date/Time	Received By	Date/Time
		<i>Allen, TX</i>	<i>4/10/2019</i>

Comments

Temperature on Receipt °C	Custody Seal (Y) or N	Received on Ice (Y) or N	Samples Intact (Y) or N

To maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document.

Return of custody is considered complete as is since this information is available in the owner laboratory.

REPORT NUMBER

**19-109-4178**REPORT DATE  
**Apr 19, 2019**RECEIVED DATE  
**Apr 10, 2019**SEND TO  
**12352****PAGE 1/5**ISSUE DATE  
**Apr 19, 2019**

**PACE ANALYTICAL SERVICES**  
**PACE ANALYTICAL SERVICES**  
**9608 LOIRET BLVD**  
**LENEXA KS 66219-**

**REPORT OF ANALYSIS**

For: (12352) PACE ANALYTICAL SERVICES  
liquid  
60299511 Sub 9852

Analysis	Level Found As Received	Reporting Units	Reporting Limit	Method	Analyst- Date	Verified- Date
Sample ID: West Lagoon 60299511001	Lab Number: <b>8614775</b>	Date Sampled: <b>2019-04-08 1235</b>				
Aflatoxin B1	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Aflatoxin B2	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Aflatoxin G1	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Aflatoxin G2	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Aflatoxin summation	n.d.	ppb	1.00	Calculation	Auto-2019/04/17	Auto-2019/04/19
DON (Vomitoxin)	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Fumonisin B1	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Fumonisin B2	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Fumonisin B3	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Fumonisin summation	n.d.	ppm	0.10	Calculation	Auto-2019/04/17	Auto-2019/04/19
Ochratoxin	n.d.	ppb	1.0	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
T-2 toxin	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/19
Zearalenone	n.d.	ppb	50	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Sample ID: Overflow Lagoon 60299511002	Lab Number: <b>8614776</b>	Date Sampled: <b>2019-04-08 1330</b>				
Aflatoxin B1	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Aflatoxin B2	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Aflatoxin G1	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Aflatoxin G2	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17
Aflatoxin summation	n.d.	ppb	1.00	Calculation	Auto-2019/04/17	Auto-2019/04/19

The result(s) issued on this report only reflect the analysis of the sample(s) submitted.

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## REPORT NUMBER

**19-109-4178**REPORT DATE  
**Apr 19, 2019**SEND TO  
**12352**RECEIVED DATE  
**Apr 10, 2019****PAGE 2/5**ISSUE DATE  
**Apr 19, 2019**

**PACE ANALYTICAL SERVICES**  
**PACE ANALYTICAL SERVICES**  
**9608 LOIRET BLVD**  
**LENEXA KS 66219-**

**REPORT OF ANALYSIS**  
For: (12352) PACE ANALYTICAL SERVICES  
liquid  
60299511 Sub 9852

Analysis	Level Found		Reporting		Method	Analyst-Date	Verified-Date
	As Received	Units	Limit				
<b>Sample ID: Overflow Lagoon 60299511002</b>	Lab Number: <b>8614776</b> (con't)						
DON (Vomitoxin)	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
Fumonisin B1	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
Fumonisin B2	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
Fumonisin B3	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
Fumonisin summation	n.d.	ppm	0.10	Calculation	Auto-2019/04/17	Auto-2019/04/19	
Ochratoxin	n.d.	ppb	1.0	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
T-2 toxin	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/19	
Zearalenone	n.d.	ppb	50	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
<b>Sample ID: Wet Cake 6029951003</b>	Lab Number: <b>8614777</b>		Date Sampled: <b>2019-04-08 1100</b>				
Aflatoxin B1	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
Aflatoxin B2	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
Aflatoxin G1	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
Aflatoxin G2	n.d.	ppb	1.00	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/17	
Aflatoxin summation	n.d.	ppb	1.00	Calculation	Auto-2019/04/17	Auto-2019/04/19	
DON (Vomitoxin)	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/19	tjp8-2019/04/19	
Fumonisin B1	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/19	
Fumonisin B2	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/19	
Fumonisin B3	n.d.	ppm	0.1	AOAC 2008.02 (mod)	kmc4-2019/04/17	tjp8-2019/04/19	
Fumonisin summation	n.d.	ppm	0.10	Calculation	Auto-2019/04/19	Auto-2019/04/19	

The result(s) issued on this report only reflect the analysis of the sample(s) submitted.

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REPORT NUMBER

**19-109-4178**

REPORT DATE

**Apr 19, 2019**

RECEIVED DATE

**Apr 10, 2019**

SEND TO  
**12352**



**PAGE 3/5**

ISSUE DATE  
**Apr 19, 2019**

**PACE ANALYTICAL SERVICES**  
**PACE ANALYTICAL SERVICES**  
**9608 LOIRET BLVD**  
**LENEXA KS 66219-**

**REPORT OF ANALYSIS**

For: (12352) PACE ANALYTICAL SERVICES  
liquid  
60299511 Sub 9852

Analysis	Level Found	Reporting	Analyst-Date	Verified-Date
	As Received	Units	Limit	Method
Sample ID: Wet Cake 6029951003	Lab Number: 8614777 (con't)			
Ochratoxin	n.d.	ppb	1.0	AOAC 2008.02 (mod)
T-2 toxin	n.d.	ppm	0.1	AOAC 2008.02 (mod)
Zearalenone	n.d.	ppb	50	AOAC 2008.02 (mod)

All results are reported on an AS RECEIVED basis., n.d. = not detected , ppm = parts per million, ppm = mg/kg , ppb = parts per billion

cc: Account(s) 12352 PACE ANALYTICAL SERVICES

For questions please contact:

Handwritten signature of Heather Ramig  
Heather Ramig  
Account Manager  
hramig@midwestlabs.com (402)829-9891

The result(s) issued on this report only reflect the analysis of the sample(s) submitted.  
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**REPORT NUMBER****19-109-4178**REPORT DATE  
**Apr 19, 2019**RECEIVED DATE  
**Apr 10, 2019**SEND TO  
**12352****PAGE 4/5**ISSUE DATE  
**Apr 19, 2019**

**PACE ANALYTICAL SERVICES**  
**PACE ANALYTICAL SERVICES**  
**9608 LOIRET BLVD**  
**LENEXA KS 66219-**

**REPORT OF ANALYSIS**  
For: (12352) PACE ANALYTICAL SERVICES  
liquid  
60299511 Sub 9852

**Detailed Method Description(s)****Mycotoxin extraction and analysis**

Sample analysis follows MWL LCMS 020 which is based on AOAC 2008.02 (modified). Samples are ground to a homogenous consistency and placed in an extraction solution. The extract is allowed to equilibrate and then an aliquot passed through an immunoaffinity column which contains antibodies that are specific for the mycotoxins. The mycotoxins are released from the affinity column and then analyzed by either LC/MS and/or LC/MS/MS which allows identification of the mycotoxins using mass spectrometry and retention time.

**Calculation**

Analytical results are entered into applicable formulas to provide a calculated result which is reported.

The result(s) issued on this report only reflect the analysis of the sample(s) submitted.

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## CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

**PAGE 5/5**

Section A Required Client Information		Section B Required Project Information		Section C Invoice Information:		Page: 1 of 1				
Company: EA Eng Science and Technology	Report To: Dan Bigbee	Address: 221 Sun Valley Blvd Suite D	Copy To: Kent Dixon	Attention: Accounts Payable, Dan Bigbee	Company Name: EA Eng Science and Technology	REGULATORY AGENCY				
Lincoln, NE 68528		Email To: dbigbee@eaest.com	Purchase Order No.: LSO #	Address:	Pace Quote Reference: 58462 (JR)	<input type="checkbox"/> NPDES	<input type="checkbox"/> GROUND WATER	<input type="checkbox"/> DRINKING WATER		
Phone: 402-817-7612	Fax	Project Name: AltEn LLC	Project Number: AltEn LLC Digestor Sampling	Pace Project Manager: Trudy Gipson 913-563-1405	Pace Profile #: 12849 Lines 1 & 2	<input type="checkbox"/> UST	<input type="checkbox"/> RCRA	<input type="checkbox"/> OTHER		
Requested Due Date/TAT:						Site Location: NE	STATE: NE			
Requested Analysis Filtered (Y/N)										
ITEM #	Section D Required Client Information		COLLECTED		Preservatives					
	Valid Matrix Codes		COMPOSITE		Preservatives					
	MATRIX	CODE	DATE	TIME	DATE	TIME				
	DINING WATER	DW								
	WATER	WT								
	WASTE WATER	WW								
	PRODUCT	PR								
	SOIL/SOIL	SR								
	OIL	OL								
	WP	WP								
	AIR	AR								
	OTHER	OT								
	TISSUE	TS								
SAMPLE ID (A-Z, 0-9, -,.) Sample ID MUST BE UNIQUE		MATRIX CODE		SAMPLE TYPE (G=GRAB C=COMP)						
1	Midwest Laboratories		WT	4/8/19 12:38		1	1			
2	West Lagoon		WT	4/8/19 13:38		1	1			
3	Overflow Lagoon		WT	4/8/19 13:38		1	1			
4	Wat Cake		SL	4/8/19 11:00		1	1			
5										
6										
7										
8										
9										
10										
11										
12										
ADDITIONAL COMMENTS		RELINQUISHED BY / AFFILIATION		DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS	
Sub Midwest Laboratories		Mr. J. Donald / NDEQ		4/8/19	4:30	2019-04-10 16:30	4/9/19	16:30		
		Re: 8614775 TEA		4-9-19	16:30		4/10/19		5.0% AT 21.13	
SAMPLER NAME AND SIGNATURE									Temp in °C	
PRINT Name of SAMPLER: Daniel LeMaistre									Received in Lab (Y/N)	
SIGNATURE of SAMPLER:									Custody Sealed/Caller (Y/N)	
DATE Signed (MM/DD/YYYY): 4/8/19									Sample intact (Y/N)	

\*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

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F-ALL-Q-020rev.08, 12-Oct-2007

**Performed By:**

South Dakota Agricultural Laboratories  
 1335 Western Avenue  
 Brookings, South Dakota 57006  
 Phone: 605-692-7325  
 E-Mail: regina.wixon@sdaglabs.com

**Collected By:**

EA Eng Science and Technology  
 221 Sun Valley Blvd Suite D  
 Accounts Payable, Dan Bigbee  
 Lincoln, NE 68528  
 Phone: 402-817-7612  
 E-Mail: dbigbee@eaest.com

**Report Date: 2019-04-25****Final Report****South Dakota Agricultural Laboratories has examined the sample of**

Limfinite Package Id :	20190415-002
Lab Sample Id :	19PE001995
Customer Sample Id :	West Lagoon
Sample Description :	Liquid
Date Collected :	2019-04-08
Date Received :	2019-04-15

ANALYTE	RESULT
Acetamprid	ND ppb
Azoxystrobin	99.3 ppb
Bifenthrin	ND ppb
Brassinazole	ND ppb
Chlorpyrifos-ethyl	ND ppb
Chlorpyrifos-methyl	ND ppb
Clothianidin	58400 ppb
Cyfluthrin	ND ppb
Cypermethrin	ND ppb
Cyproconazole	ND ppb
Deltamethrin	<5 ppb
Dimoxystrobin	ND ppb
Epoxiconazole	ND ppb
Fluconazole	ND ppb
Glufosinate	ND ppb
Glyphosate	124 ppb
Imidacloprid	108 ppb
Isavuconazole	ND ppb
Lambda-cyhalothrin	ND ppb
Metconazole	ND ppb
Orysastrobin	ND ppb
Permethrin	ND ppb
Posaconazole	ND ppb
Propiconazole	ND ppb
Pyraclostrobin	ND ppb
Ravuconazole	ND ppb
Tebuconazole	213 ppb
Thiabendazole	8450 ppb
Thiacloprid	ND ppb
Thiamethoxam	35400 ppb

Trifloxystrobin	58.2 ppb
Uniconazole	ND ppb
Voriconazole	ND ppb

**Comments:**

ND: Not Detected

---

Page Analytical

CHAIN-OF-CUSTODY / Analytical Request Document

The Chair of Committee & the DOCUMENT All relevant fields must be completed accurately

Section A		Section B		Section C	
Required Information		Required Information		Required Information	
Company A-219484	I-A Eng Science and Technology	Request To Daren Begee	Account Name E.A Eng Science and Technology	Regulatory Agency Name REGULATORY AGENCY	
221 Sun Valley Blvd Suite D	City To Kenilworth	Phone No 1-800-542-6264	Phone No 1-800-542-6264	Address Project Name Project Manager Project Number	Address Project Name Project Manager Project Number
Fax To darenbegee@pepsci.com	ISO # 14001	Purchase Order No 402-A-77612	Project Manager ARLEN LLC	NPDES GROUND WATER DRINKING WATER	UST RCRA OTHER
Phone 402-A-77612	E-mail [REDACTED]	Project Number 14049 Lines 1 & 2	Site Location NE	STATE NE	
Requested Due Date/TAT					

ITEM #	SAMPLE ID		COLLECTED		Preservatives		ANALYST				
	Sample ID	Date Collected	Date	Time	Type	Conc.					
1	South Dakota Agricultural Laboratories										
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
ADDITIONAL COMMENTS		RElinquished By / AFFILIATION		DATE	TIME	ACCEPTED BY / AFFILIATION		DATE	TIME	SAMPLE CONDITIONS	
<i>by J. B. J. Project</i>		<i>10/15/19</i>		<i>10/15/19</i>	<i>13:30</i>	<i>10/15/19</i>		<i>10/15/19</i>	<i>13:30</i>	<i>10/15/19</i>	



**Performed By:**

South Dakota Agricultural Laboratories  
 1335 Western Avenue  
 Brookings, South Dakota 57006  
 Phone: 605-692-7325  
 E-Mail: regina.wixon@sdaglabs.com

**Collected By:**

EA Eng Science and Technology  
 221 Sun Valley Blvd Suite D  
 Accounts Payable, Dan Bigbee  
 Lincoln, NE 68528  
 Phone: 402-817-7612  
 E-Mail: dbigbee@eaest.com

**Report Date: 2019-04-29****Final Report****South Dakota Agricultural Laboratories has examined the sample of**

Limfinite Package Id :	20190415-002
Lab Sample Id :	19PE001996
Customer Sample Id :	Overflow Lagoon
Sample Description :	Liquid
Date Collected :	2019-04-08
Date Received :	2019-04-15

<b>ANALYTE</b>	<b>RESULT</b>
Acetamprid	ND ppb
Azoxystrobin	581 ppb
Bifenthrin	ND ppb
Brassinazole	ND ppb
Chlorpyrifos-ethyl	ND ppb
Chlorpyrifos-methyl	ND ppb
Clothianidin	44.7 ppb
Cyfluthrin	ND ppb
Cypermethrin	ND ppb
Cyproconazole	ND ppb
Deltamethrin	<5 ppb
Dimoxystrobin	ND ppb
Epoxiconazole	ND ppb
Fluconazole	ND ppb
Glufosinate	86.7 ppb
Glyphosate	3850 ppb
Imidacloprid	ND ppb
Isavuconazole	ND ppb
Lambda-cyhalothrin	<5 ppb
Metconazole	ND ppb
Orysastrobin	ND ppb
Permethrin	ND ppb
Posaconazole	ND ppb
Propiconazole	726 ppb
Pyraclostrobin	ND ppb
Ravuconazole	ND ppb
Tebuconazole	2330 ppb
Thiabendazole	39700 ppb
Thiacloprid	ND ppb
Thiamethoxam	26.0 ppb

Trifloxystrobin	737 ppb
Uniconazole	ND ppb
Voriconazole	ND ppb

**Comments:**

ND: Not Detected

---

**Performed By:**

South Dakota Agricultural Laboratories  
 1335 Western Avenue  
 Brookings, South Dakota 57006  
 Phone: 605-692-7325  
 E-Mail: regina.wixon@sdaglabs.com

**Collected By:**

EA Eng Science and Technology  
 221 Sun Valley Blvd Suite D  
 Accounts Payable, Dan Bigbee  
 Lincoln, NE 68528  
 Phone: 402-817-7612  
 E-Mail: dbigbee@eaest.com

**Report Date: 2019-04-25****Final Report****South Dakota Agricultural Laboratories has examined the sample of**

Limfinite Package Id :	20190415-002
Lab Sample Id :	19PE001997
Customer Sample Id :	Wet Cake
Sample Description :	Cake
Date Collected :	2019-04-08
Date Received :	2019-04-15

ANALYTE	RESULT
Acetamprid	ND ppb
Azoxystrobin	1430 ppb
Bifenthrin	ND ppb
Brassinazole	ND ppb
Chlorpyrifos-ethyl	ND ppb
Chlorpyrifos-methyl	ND ppb
Clothianidin	112000 ppb
Cyfluthrin	ND ppb
Cypermethrin	ND ppb
Cyproconazole	ND ppb
Deltamethrin	ND ppb
Dimoxystrobin	ND ppb
Epoxiconazole	ND ppb
Fluconazole	ND ppb
Glufosinate	ND ppb
Glyphosate	ND ppb
Imidacloprid	485 ppb
Isavuconazole	ND ppb
Lambda-cyhalothrin	ND ppb
Metconazole	ND ppb
Orysastrobin	ND ppb
Permethrin	ND ppb
Posaconazole	ND ppb
Propiconazole	ND ppb
Pyraclostrobin	ND ppb
Ravuconazole	ND ppb
Tebuconazole	10400 ppb
Thiabendazole	55600 ppb
Thiacloprid	ND ppb
Thiamethoxam	30500 ppb

Trifloxystrobin	1750 ppb
Uniconazole	ND ppb
Voriconazole	ND ppb

**Comments:**

ND: Not Detected

---

**Performed By:**

South Dakota Agricultural Laboratories  
1335 Western Avenue  
Brookings, South Dakota 57006  
Phone: 605-692-7325  
E-Mail: regina.wixon@sdaglabs.com

**Collected By:**

EA Eng Science and Technology  
221 Sun Valley Blvd Suite D  
Accounts Payable, Dan Bigbee  
Lincoln, NE 68528  
Phone: 402-817-7612  
E-Mail: dbigbee@eaest.com

**Report Date: 2019-04-25****Final Report****South Dakota Agricultural Laboratories has examined the sample of**

Limfinite Package Id :	20190415-002
Lab Sample Id :	19PE001998
Customer Sample Id :	Seed Corn
Sample Description :	Corn
Date Collected :	2019-04-08
Date Received :	2019-04-15

ANALYTE	RESULT
Azoxystrobin	4830 ppb
Brassinazole	ND ppb
Cyproconazole	ND ppb
Dimoxystrobin	ND ppb
Epoxiconazole	ND ppb
Fluconazole	ND ppb
Isavuconazole	ND ppb
Metconazole	ND ppb
Orysastrobin	ND ppb
Posaconazole	ND ppb
Propiconazole	ND ppb
Pyraclostrobin	ND ppb
Ravuconazole	ND ppb
Tebuconazole	ND ppb
Thiabendazole	247000 ppb
Trifloxystrobin	ND ppb
Uroniconazole	ND ppb
Voriconazole	ND ppb

**Comments:**

\* ND: Not Detected

**United States Environmental Protection Agency  
Region 7  
300 Minnesota Avenue  
Kansas City, KS 66101**

**Date:** 04/24/2019

**Subject:** Transmittal of Sample Analysis Results for ASR #: 8209

Project ID: TJAAEEP4S

Project Description: Alt-En Ethanol Plant - NDEQ ASR

**From:** Margaret E.W. St. Germain, Chief  
Laboratory Technology & Analysis Branch  
Laboratory Services and Applied Sciences Division

**MARGARET  
ST. GERMAIN**

Digitally signed by  
MARGARET ST. GERMAIN  
Date: 2019.04.24 17:00:48  
-05'00'

**To:** Tabatha Adkins  
ENST/LTAB

Enclosed are the analytical data for the above-referenced Analytical Services Request (ASR) and Project. These results are based on samples as received at the Science and Technology Center. The Regional Laboratory has reviewed and verified the results in accordance with procedures described in our Quality Manual (QM). In addition to all of the analytical results, this transmittal contains pertinent information that may have influenced the reported results and documents any deviations from the established requirements of the QM.

Please ensure that you file this electronic (.pdf only) transmittal in your records management system. The Regional Laboratory will now retain all of the original hardcopy documentation (e.g. COC[s] and the R7LIMS field sheet[s], etc.) according to our ENST records management system.

Please contact us within 14 days of receipt of this package if you determine there is a need for any changes. Please complete the Online ASR Sample/Data Disposition and Customer Survey for this ASR as soon as possible. The process of disposing of the samples for this ASR will be initiated 30 days from the date of this transmittal unless an alternate release date is specified on the Online ASR Sample/Data Disposition and Customer Survey. It is critical that we receive your response in accordance to RCRA and the laboratory accreditation.

If you have any questions or concerns relating to this data package, contact our customer service line at 913-551-5295.

**Project Manager:** Tabatha Adkins**Org:** ENST/LTAB**Phone:** 913-551-7128**Project ID:** TJAAEEP4S**QAPP Number:** NDEQ**Project Desc:** Alt-En Ethanol Plant - NDEQ ASR**Location:** Mead**State:** Nebraska**Program:** Water Enforcement**Purpose:** Site Characterization**GPRA PRC:** 000E50

This screening/characterization event is in response to recent complaints of odor and other concerns.

Additional field contact is: Wade Gregson, NDEQ (402-601-1011).

Per NDEQ submitted ASR on 4/4/2019: This ASR is not part of a litigation hold at this time.

**Explanation of Codes, Units and Qualifiers used on this report****Sample QC Codes:** QC Codes identify the type of sample for quality control purpose.**Units:** Specific units in which results are reported.   = Field Sample

mg/L = Milligrams per Liter

% = Percent

ug/L = Micrograms per Liter

mg/kg = Milligrams per Kilogram

**Data Qualifiers:** Specific codes used in conjunction with data values to provide additional information on the quality of reported results, or used to explain the absence of a specific value.

(Blank) = Values have been reviewed and found acceptable for use.

J = The identification of the analyte is acceptable; the reported value is an estimate.

UJ = The analyte was not detected at or above the reporting limit. The reporting limit is an estimate.

U = The analyte was not detected at or above the reporting limit.

**ASR Number:** 8209**Sample Information Summary****04/24/2019****Project ID:** TJAAEEP4S**Project Desc:** Alt-En Ethanol Plant - NDEQ ASR

<b>Sample No</b>	<b>QC Code</b>	<b>Matrix</b>	<b>Location Description</b>	<b>External Sample No</b>	<b>Start Date</b>	<b>Start Time</b>	<b>End Date</b>	<b>End Time</b>	<b>Receipt Date</b>
1 - __	Solid	Wet cake sample			04/08/2019	11:00			04/09/2019
2 - __	Water	West Lagoon water sample			04/08/2019	12:35			04/09/2019
3 - __	Water	Overflow Lagoon water sample			04/08/2019	13:30			04/09/2019
4 - __	Solid	Seed corn sample			04/08/2019	13:30			04/09/2019

**Analysis      Comments About Results For This Analysis**

---

## 1    Mercury in Soil or Sediment

**Lab:** Region 7 EPA Laboratory - Kansas City, Ks.**Method:** EPA Region 7 RLAB Method 3121.23E**Basis:** Dry**Samples:** 1-\_\_      4-\_\_**Comments:**

(N/A)

## 1    Metals in Solids by ICP-AES

**Lab:** Region 7 EPA Laboratory - Kansas City, Ks.**Method:** EPA Region 7 RLAB Method 3122.3G**Basis:** Dry**Samples:** 1-\_\_      4-\_\_**Comments:**

Barium was J-coded in sample 1. Although the analyte in question has been positively identified in the sample, the quantitation is an estimate (J-coded) due to low recovery of this analyte in the laboratory matrix spike. The actual concentration for this analyte may be higher than the reported value.

## 1    Percent Solid

**Lab:** Region 7 EPA Laboratory - Kansas City, Ks.**Method:** EPA Region 7 RLAB Method 3142.9H**Basis:** N/A**Samples:** 1-\_\_      4-\_\_**Comments:**

(N/A)

## 1    TCLP Mercury in Soil

**Lab:** Region 7 EPA Laboratory - Kansas City, Ks.**Method:** EPA Region 7 RLAB Method 3121.23E applied to TCLP extracts**Basis:** N/A**Samples:** 1-\_\_      4-\_\_**Comments:**

## 1    TCLP Metals in Soil

**Lab:** Region 7 EPA Laboratory - Kansas City, Ks.**Method:** EPA Region 7 RLAB Method 3122.3G TCLP

**ASR Number:** 8209

**RLAB Approved Analysis Comments**

**04/24/2019**

**Project ID:** TJAAEEP4S

**Project Desc** Alt-En Ethanol Plant - NDEQ ASR

---

**Analysis      Comments About Results For This Analysis**

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**Basis:** N/A

**Samples:** 1-\_\_      4-\_\_

**Comments:**

1    Mercury in Water

**Lab:** Region 7 EPA Laboratory - Kansas City, Ks.

**Method:** EPA Region 7 RLAB Method 3121.23E

**Samples:** 2-\_\_      3-\_\_

**Comments:**

(N/A)

1    Metals in Water by ICP-AES

**Lab:** Region 7 EPA Laboratory - Kansas City, Ks.

**Method:** EPA Region 7 RLAB Method 3122.3G

**Samples:** 2-\_\_      3-\_\_

**Comments:**

Aluminum was J-coded in sample 2. Although the analyte in question has been positively identified in the sample, the quantitation is an estimate (J-coded) due to high recovery of this analyte in the laboratory matrix spike. The actual concentration for this analyte may be lower than the reported value.

Nickel was J-coded in sample 2. Although the analyte in question has been positively identified in the sample, the quantitation is an estimate (J-coded) due to low recovery of this analyte in the laboratory matrix spike. The actual concentration for this analyte may be higher than the reported value.

Antimony, Cadmium, Lead, Molybdenum, Selenium, Silver, Thallium, Titanium was each UJ-coded in sample 2. This analyte was not found in the sample at or above the reporting limit, however, the reporting limit is an estimate (UJ-coded) due to low recovery of this analyte in the laboratory matrix spike. The actual reporting limit for this analyte may be higher than the reported value.

1    TCLP Mercury in Water

**Lab:** Region 7 EPA Laboratory - Kansas City, Ks.

**Method:** EPA Region 7 RLAB Method 3121.23E applied to TCLP extracts

**Samples:** 2-\_\_      3-\_\_

**Comments:**

1    TCLP Metals in Water

**Lab:** Region 7 EPA Laboratory - Kansas City, Ks.

**ASR Number:** 8209

**RLAB Approved Analysis Comments**

**04/24/2019**

**Project ID:** TJAAEFP4S

**Project Desc** Alt-En Ethanol Plant - NDEQ ASR

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**Analysis      Comments About Results For This Analysis**

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**Method:** EPA Region 7 RLAB Method 3122.3G TCLP

**Samples:** 2-\_\_      3-\_\_

**Comments:**

Lead was UJ-coded in sample 2. This analyte was not found in the sample at or above the reporting limit, however, the reporting limit is an estimate (UJ-coded) due to low recovery of this analyte in the laboratory matrix spike. The actual reporting limit for this analyte may be higher than the reported value.

**ASR Number:** 8209**Project ID:** TJAAEEP4S**RLAB Approved Sample Analysis Results****04/24/2019****Project Desc:** Alt-En Ethanol Plant - NDEQ ASR

<b>Analysis/ Analyte</b>	<b>Units</b>	<b>1-__</b>	<b>2-__</b>	<b>3-__</b>	<b>4-__</b>
1 Mercury in Soil or Sediment					
Mercury	mg/kg	0.0112			0.00179
1 Metals in Solids by ICP-AES					
Aluminum	mg/kg	2420			7.5
Antimony	mg/kg	1.9 U			2.0 U
Arsenic	mg/kg	4.8 U			5.0 U
Barium	mg/kg	42.3 J			2.0 U
Beryllium	mg/kg	1.0 U			1.0 U
Cadmium	mg/kg	1.0 U			1.0 U
Calcium	mg/kg	2640			49.6 U
Chromium	mg/kg	3.4			2.0 U
Cobalt	mg/kg	1.7			1.0 U
Copper	mg/kg	13.6			2.4
Iron	mg/kg	3270			19.3
Lead	mg/kg	4.8 U			5.0 U
Magnesium	mg/kg	1610			991
Manganese	mg/kg	124			5.0 U
Molybdenum	mg/kg	1.9 U			2.0 U
Nickel	mg/kg	4.2			2.0 U
Potassium	mg/kg	3960			3730
Selenium	mg/kg	9.7 U			9.9 U
Silver	mg/kg	1.9 U			2.0 U
Sodium	mg/kg	694			49.6 U
Thallium	mg/kg	9.7 U			9.9 U
Vanadium	mg/kg	5.9			5.0 U
Zinc	mg/kg	262			18.4
1 Percent Solid					
Solids, percent	%	39.2			89.8
1 TCLP Mercury in Soil					
Mercury	mg/L	0.000440			0.000350
1 TCLP Metals in Soil					
Arsenic	mg/L	0.050 U			0.050 U
Barium	mg/L	0.674			0.016 U
Cadmium	mg/L	0.010			0.005 U
Chromium	mg/L	0.078			0.015 U
Lead	mg/L	0.050 U			0.050 U
Selenium	mg/L	0.066			0.050 U
Silver	mg/L	0.025 U			0.025 U
1 Mercury in Water					
Mercury	ug/L		0.250 U	0.250 U	
1 Metals in Water by ICP-AES					
Aluminum	ug/L		1580 J	60200	
Antimony	ug/L		50 UJ	50 U	
Arsenic	ug/L		25 U	25 U	
Barium	ug/L		164	727	
Beryllium	ug/L		3 U	3 U	

**ASR Number:** 8209**Project ID:** TJAAEFP4S**RLAB Approved Sample Analysis Results****04/24/2019****Project Desc:** Alt-En Ethanol Plant - NDEQ ASR

<b>Analysis/ Analyte</b>	<b>Units</b>	<b>1-__</b>	<b>2-__</b>	<b>3-__</b>	<b>4-__</b>
Cadmium	ug/L		3 UJ	3 U	
Calcium	mg/L		97.6	616	
Chromium	ug/L		37	96	
Cobalt	ug/L		10 U	18	
Copper	ug/L		77	1660	
Iron	ug/L		6640	67700	
Lead	ug/L		50 UJ	97.2	
Magnesium	mg/L		246	150	
Manganese	ug/L		1130	2080	
Molybdenum	ug/L		15 UJ	24	
Nickel	ug/L		39 J	130	
Potassium	mg/L		848	320	
Selenium	ug/L		50 UJ	111	
Silver	ug/L		25 UJ	25 U	
Sodium	mg/L		414	256	
Thallium	ug/L		50 UJ	50 U	
Titanium	ug/L		20 UJ	105	
Vanadium	ug/L		10 U	127	
Zinc	ug/L		3060	14900	
<b>1 TCLP Mercury in Water</b>					
Mercury	mg/L		0.000250 U	0.000530	
<b>1 TCLP Metals in Water</b>					
Arsenic	mg/L		0.050 U	0.050 U	
Barium	mg/L		0.122	0.070	
Cadmium	mg/L		0.005 U	0.005 U	
Chromium	mg/L		0.091	0.087	
Lead	mg/L		0.050 UJ	0.050 U	
Selenium	mg/L		0.050 U	0.050 U	
Silver	mg/L		0.025 U	0.025 U	

~419119

**CHAIN OF CUSTODY RECORD**  
ENVIRONMENTAL PROTECTION AGENCY REGION VII

## **PERSONNEL CUSTODY RECORD**

REL INQUISHED BY (PM/SAMPLER) <i>Wad Oregon</i> SEALED	DATE 4/8/19	TIME 1530	RECEIVED BY <i>Keith D</i> SEALED	DATE 4/8/19	TIME 1535	REASON FOR CHANGE OF CUSTODY To ship samples
REL INQUISHED BY (PM/SAMPLER) <i>Keith D</i> SEALED	DATE 4/8/19	TIME 1720	RECEIVED BY <i>Michele Roberts</i> SEALED	DATE 4/9/19	TIME 1100A	REASON FOR CHANGE OF CUSTODY Analy
REL INQUISHED BY (PM/SAMPLER) SEALED	DATE	TIME	RECEIVED BY SEALED	DATE	TIME	REASON FOR CHANGE OF CUSTODY
REL INQUISHED BY (PM/SAMPLER) SEALED	DATE	TIME	RECEIVED BY SEALED	DATE	TIME	REASON FOR CHANGE OF CUSTODY
WHITE ORIGINAL - EPATAR - YELLOW - EPA PROJECT MANUAL 7-EPA-9767 (R-47-*)						

# **Sample Collection Field Sheet**

**US EPA Region 7**  
**Kansas City, KS**

**ASR Number:** 8209    **Sample Number:** 1    **QC Code:** \_\_\_    **Matrix:** Solid    **Tag ID:** 8209-1-\_\_\_

**Project ID:** TJAAEERP4S  
**Project Desc:** Alt-En Ethanol Plant - NDEQ ASR  
    **City:** Mead  
**Program:** Water Enforcement

**Project Manager:** Tabatha Adkins  
**State:** Nebraska

**Location Desc:** Wet cake sample

**Storet ID:**

**External Sample Number:**

**Expected Conc:** \_\_\_\_\_ (or Circle One: Low Medium High)      **Date** \_\_\_\_\_ **Time(24 hr)** \_\_\_\_\_

**Latitude:** \_\_\_\_\_ **Sample Collection: Start:** 04/08/2019 11:00  
**Longitude:** \_\_\_\_\_ **End:** \_\_\_\_\_.\_\_\_\_\_.\_\_\_\_\_.\_\_\_\_\_.\_\_\_\_\_

## **Laboratory Analyses:**

<b>Container</b>	<b>Preservative</b>	<b>Holding Time</b>	<b>Analysis</b>
1 - 8 oz glass	4 Deg C	28 Days	1 Mercury in Soil or Sediment
1 - 8 oz glass	4 Deg C	180 Days	1 Metals in Solids by ICP-AES
1 - 8 oz glass	4 Deg C	180 Days	1 TCLP Metals in Soil
1 - 8 oz glass	None	28 Days	1 TCLP Mercury in Soil
0 -	4 Deg C	0 Days	1 Percent Solid

---

### **Sample Comments:**

(N/A)

**Sample Collected By:** NDEQ

**Sample Collection Field Sheet**  
**US EPA Region 7**  
**Kansas City, KS**

**ASR Number:** 8209    **Sample Number:** 2    **QC Code:** \_\_\_\_\_    **Matrix:** Water    **Tag ID:** 8209-2-\_\_\_\_\_

**Project ID:** TJAAEEP4S                                  **Project Manager:** Tabatha Adkins  
**Project Desc:** Alt-En Ethanol Plant - NDEQ ASR  
**City:** Mead    **State:** Nebraska  
**Program:** Water Enforcement

---

**Location Desc:** West Lagoon water sample

**Storet ID:**    **External Sample Number:**

**Expected Conc:**    (or Circle One: Low Medium High)                                  **Date**    **Time(24 hr)**

**Latitude:** \_\_\_\_\_    **Sample Collection: Start:** 04/08/2019                                  12:35

**Longitude:** \_\_\_\_\_    **End:** \_\_\_\_/\_\_\_\_/\_\_\_\_    \_\_\_\_:\_\_\_\_

---

**Laboratory Analyses:**

<b>Container</b>	<b>Preservative</b>	<b>Holding Time</b>	<b>Analysis</b>
1 - 1 Liter plastic bottle	5 mL of HNO <sub>3</sub> /L to pH<2	28 Days	1 Mercury in Water
1 - 1 Liter plastic bottle	HNO <sub>3</sub> acidify, 4 Deg C	180 Days	1 Metals in Water by ICP-AES
1 - 1 Liter plastic bottle	None	28 Days	1 TCLP Mercury in Water
1 - 8 oz glass	4 Deg C	180 Days	1 TCLP Metals in Water

---

**Sample Comments:**

(N/A)

**Sample Collected By:** NDEQ

# **Sample Collection Field Sheet**

**US EPA Region 7**  
**Kansas City, KS**

**ASR Number:** 8209    **Sample Number:** 3    **QC Code:** \_\_\_\_\_    **Matrix:** Water    **Tag ID:** 8209-3-\_\_\_\_\_

**Project ID:** TJAAEFP4S      **Project Manager:** Tabatha Adkins  
**Project Desc:** Alt-En Ethanol Plant - NDEQ ASR      **City:** Mead      **State:** Nebraska  
**Program:** Water Enforcement

**Location Desc:** Overflow Lagoon water sample

**Storet ID:** **External Sample Number:**

**Expected Conc:** \_\_\_\_\_ (or Circle One: Low Medium High)      **Date** \_\_\_\_\_      **Time(24 hr)** \_\_\_\_\_

**Latitude:** \_\_\_\_\_ **Sample Collection: Start:** 04/08/2019 **13:30**

**Longitude:** \_\_\_\_\_ **End:** \_\_\_\_\_

## **Laboratory Analyses:**

<b>Container</b>	<b>Preservative</b>	<b>Holding Time</b>	<b>Analysis</b>
1 - 1 Liter plastic bottle	5 mL of HNO <sub>3</sub> /L to pH<2	28 Days	1 Mercury in Water
1 - 1 Liter plastic bottle	HNO <sub>3</sub> acidify, 4 Deg C	180 Days	1 Metals in Water by ICP-AES
1 - 1 Liter plastic bottle	None	28 Days	1 TCLP Mercury in Water
1 - 8 oz glass	4 Deg C	180 Days	1 TCLP Metals in Water

---

**Sample Comments:**

(N/A)

**Sample Collected By:** NDEQ

**Sample Collection Field Sheet**  
**US EPA Region 7**  
**Kansas City, KS**

**ASR Number:** 8209    **Sample Number:** 4    **QC Code:** \_\_\_\_\_    **Matrix:** Solid    **Tag ID:** 8209-4-\_\_\_\_\_

**Project ID:** TJAAEFP4S                                  **Project Manager:** Tabatha Adkins  
**Project Desc:** Alt-En Ethanol Plant - NDEQ ASR                                  **City:** Mead    **State:** Nebraska  
**Program:** Water Enforcement

**Location Desc:** Seed corn sample

**Storet ID:**    **External Sample Number:**

**Expected Conc:**    (or Circle One: Low Medium High)                                  **Date**    **Time(24 hr)**

**Latitude:** \_\_\_\_\_    **Sample Collection: Start:** 04/08/2019                                  13:30

**Longitude:** \_\_\_\_\_    **End:** \_\_\_\_/\_\_\_\_/\_\_\_\_    \_\_\_\_:\_\_\_\_

**Laboratory Analyses:**

<b>Container</b>	<b>Preservative</b>	<b>Holding Time</b>	<b>Analysis</b>
1 - 8 oz glass	4 Deg C	28 Days	1 Mercury in Soil or Sediment
1 - 8 oz glass	4 Deg C	180 Days	1 Metals in Solids by ICP-AES
1 - 8 oz glass	4 Deg C	180 Days	1 TCLP Metals in Soil
1 - 8 oz glass	None	28 Days	1 TCLP Mercury in Soil
0 -	4 Deg C	0 Days	1 Percent Solid

**Sample Comments:**

(N/A)

**Sample Collected By:** NDEQ

# Attachment C

---

**From:** Pomajzl, Mark  
**Sent:** Monday, August 31, 2020 5:10 PM  
**To:** Thiesfeld, Joseph  
**Subject:** FW: Lab Test Results - 20191113-003  
**Attachments:** 20191113-003.pdf

Please put this in the AltEn file:

IIS: 84069  
PCS: NE0137634

Thank you

Mark Pomajzl  
Program Specialist  
Wastewater NPDES Compliance Unit

Nebraska Department of Environment and Energy  
1200 N St, Suite 400  
P.O. Box 98922  
Lincoln, Nebraska 68509-8922  
DIRECT: 402-471-2936 FAX: 402-471-2909 <http://deq.ne.gov/>

-----Original Message-----

From: regina.wixon@sdaglabs.com <regina.wixon@sdaglabs.com>  
Sent: Wednesday, December 04, 2019 2:17 PM  
To: Pomajzl, Mark <mark.pomajzl@nebraska.gov>  
Cc: regina.wixon@sdaglabs.com  
Subject: Lab Test Results - 20191113-003

Please find the results of analysis for your recent submission to South Dakota Agricultural Laboratories. These results are indicative of the sample(s) as received at the laboratory. We invite you to visit our website at <https://gcc02.safelinks.protection.outlook.com/?url=http%3A%2F%2Fwww.sdaglabs.com%2F&data=02%7C01%7Cjoseph.Thiesfeld%40nebraska.gov%7C1cd6308728714de9987d08d84dfa92e%7C043207dfe6894bf6902001038f11f0b1%7C0%7C0%7C637345086401211065&sdata=UN39MPJMrNioNGQA3apPxI2tN7aRYR6HfJ5eKRXnSyo%3D&reserved=0> for submission forms, definitions for symbols and abbreviations used in the report, interpretation documents, a complete listing of services and fees, sampling instructions and other information. We appreciate comments to improve our website and its usefulness to you.

Thank you very much for supporting our laboratory. If there is anything we can do to further assist you, please let us know.

South Dakota Agricultural Laboratories  
1335 Western Avenue  
Brookings, SD 57006  
Call: 605-692-7325



Fax: 605-692-7236

Email: regina.wixon@sdaglabs.com

**Performed By:**

South Dakota Agricultural Laboratories  
 1335 Western Avenue  
 Brookings, South Dakota 57006  
 Phone: 605-692-7325  
 E-Mail: regina.wixon@sdaglabs.com

**Collected By:**

Nebraska Department of Environment & Energy  
 NE Dept of Env & Energy 1200 N St  
 Lincoln ,NE 95922  
 Phone: 402-471-2936  
 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 : 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
Difenconazole	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
Epiconazole	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
Ipcaconazole	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
Destho-Prothioconazole	ppb	19PE008838	119	ND	ND	ND
Difenconazole	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
Ipcconazole	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
Uconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

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**Comments:**

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**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.

**Performed By:**

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 1335 Western Avenue  
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 Phone: 605-692-7325  
 E-Mail: regina.wixon@sdaglabs.com

**Collected By:**

Nebraska Department of Environment & Energy  
 NE Dept of Env & Energy 1200 N St  
 Lincoln, NE 95922  
 Phone: 402-471-2936  
 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 : 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
Destho-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
Destho-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
Ipcconazole	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
Uconiconazole	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

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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**

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**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**

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**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 pyrethrroids 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 pyrethrroids 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 : 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
Ipcconazole	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
Destho-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
Ipcconazole	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

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**Comments:**

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**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

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## 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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**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
Destho-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
Ipcconazole	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
Destho-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
Ipcconazole	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
Uronicazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

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**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).

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

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The pyrethrroids 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 pyrethrroids 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.

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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. Alfemess 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.

**Performed By:**

South Dakota Agricultural Laboratories  
 1335 Western Avenue  
 Brookings, South Dakota 57006  
 Phone: 605-692-7325  
 E-Mail: regina.wixon@sdaglabs.com

**Collected By:**

Nebraska Department of Environment & Energy  
 NE Dept of Env & Energy 1200 N St  
 Lincoln ,NE 95922  
 Phone: 402-471-2936  
 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 : 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
Destho-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
Ipcconazole	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
Destho-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
Uconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

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**Comments:**

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**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

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**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 pyrethrroids 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 pyrethrroids 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.

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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.

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#### **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.

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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 1/12/2019  
**Sampler Name(s) (Print):** Mark Pomajzl, Hillary Stoll, and Jason Windhorst

**Sampler Name(s) (Signature):** *Mark Pomajzl, Hillary Stoll, Jason Windhorst*

Sample ID	Sample Date	Sample Time	Sample Type*	Grab or Composite?	Media	Comments	Lab Use
North Lagoon - ALTEN	1/12/2019	WS	C	WW			
North Lagoon - ALTEN	1/12/2019	10:30 AM	WS	C	WW	10:30 AM & 3:45 PM	
West Lagoon - ALTEN	1/12/2019	1:45 AM	WS	C	WW	1:45 AM	
South Lagoon - ALTEN	1/12/2019	FB	N/A	DI			
Field Blank (Between North Lagoon and South Lagoon Sampling) - ALTEN	1/12/2019	11:00 AM	FB	N/A	DI	11:00 AM	
Field Blank (Between North Lagoon and South Lagoon Sampling) - ALTEN	1/12/2019	FB	N/A	DI			
North Lagoon Duplicate Sample - ALTEN	1/12/2019	10:30 AM	WSQ	C	WW	Duplicate retained from previous sample bottle for North lagoon	10:30 AM
Trip Blank - ALTEN	1/12/2019	-	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.

Date/Time:

9:00 AM

**Relinquished by:** *Mark Pomajzl - 1/12/19 ~ 1:30 PM*  
**Received by:** *Mark Pomajzl - 1/12/19 ~ 1:30 PM*

Date/Time:

9:00 AM

**Relinquished by:** *Mark Pomajzl - 1/12/19 ~ 1:30 PM*  
**Received by:** *Mark Pomajzl - 1/12/19 ~ 1:30 PM*

Date/Time:

9:00 AM

**Shipment Method:**

1 of 2

2019-11-13-003  
19PE008834-8839



## Pesticide Residue Sample Submission Form

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

2019-11-13-003  
19PE008834-8839

Atten: Waskewill, Mark (Mark.Pomajzl)

Name: Nebraska Department of Energy \*Sample ID: 15PV Atten: Waskewill, M.P.  
Address: Suite 400, The Atrium, 1202 N 5th Street, Lincoln, NE  
Zip: P.O.B. 95922 Phone: (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  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 received at SD Ag Lab  
Date 2019-11-13  
Received by Christina Schepker

Azoles	Azoxystrobin	Sulams	Fludioxonil
Brassinazole	Pyraclostrobin	Cloransulam	Flumioxazin
Cypriconazole	Oryxastrobin	Florasulam	Fluopyram
Difenoconazole	Trifloxystrobin	Flumetsulam	Fluthiacet methyl
Epiclonazole	Fluoxastrobin	Penoxsulam	Flutriafol
Fluconazole		Pyroxasulam	Fluopyrad
Ipcronazole	<b>PGR</b>		Fluaziflop
Isavuconazole	2, 4-D	<b>OP Scan</b> : \$262	Fomesafen
Itraconazole	2,4,5-T	Glyphosate \$212	Halosulfuron \$212
Metconazole	2,4,5 TP	Glufosinate \$212	Hexazinone
Posaconazole	2,4-DB		Indaziflam
Propiconazole	2,4-DP	<b>Individual Active Ingredients</b>	Indoxacarb
Prothiconazole	Bentazon	Abelectin	Iodosulfuron \$212
Prothiconazole	Bromacil	Acetamiprid	Isoxaflutole
<i>Metabolite 1</i>	Bromoxynil	Acifluorfen	Lactofen
Prothiconazole	Clopyralid	Aldicarb	Lincomycin
<i>Metabolite 2</i>	DCPA	Aldicarb sulfone	Linuron
Ravuconazole	Dicamba	Aldicarb sulfoxide	Malathion
Tebuconazole	Fluroxypyr	Aminocyclopyrachlor	Mandipropamid
Tetracconazole	MCPA	Aminopyralid \$212	Mesotrione
Thiabendazole	MCPP	Benzovendiflupry	Methiocarb
Uniconazole	Picloram	Bicyclopyprome	Methomyl
Voriconazole	Pyrasulfotole	Carbaryl	Nitenpyram
	Quinclorac	Carbofuran	Oryzalin
<b>Permethrins</b>	Triclopyr	Carfentrazone	Oxamyl
Bifenthrin		Chlorantraniliprole	Oxathiapiprolin
Cyhalothrin 1-2	<b>Pre-Emergents</b>	Chlorophacinone	Oxyfluorfen
Permethrin 1-2	Acetochlor	Chlorpyrifos-ethyl	Paraquat \$312
Cyhalothrin 1-4	Alachlor	Chlorpyrifos-methyl	Pinoxaden
Cypermethrin 1-4	Atrazine	Chlorosulfuron \$212	Prodiamine
Deltamethrin 1-2	Dimethenamid	Clethodim	Propachlor
<b>IMI</b>	Metolachlor	Clethodim sulfone	Propanil
Imazalil	Metrybuzin	Clethodim sulfoxide	Propazine
Imazamethabenz	Pendimethalin	Clomazone	Propoxur
Imazamox	Prometon	Cyanazine	Propoxycarbazone
Imazapir	Simazine	Diazinon	Pydiflumetofen
Imazapyr	<b>SU</b>	Diflufenzoxypr	Pyroxasulfone
Imazaquin	Chlorimuron	Dinotefuran	Quizalofop
Imazethapyr	Formasulfuron	Diquat \$312	Saflufenacil
<b>Neonics</b>	Mesosulfuron	Dithiopyr	S <sub>4</sub> -daxane
Acetamiprid	Metsulfuron	Diuron	Sulfameturon methyl
Clothianidin	Nicosulfuron	Diuron metabolite	Sulfentrazone
Thiacloprid	Primsulfuron	Ethalfluralin \$212	Tebuthiuron
Imidacloprid	Prosulfuron	Emamectin benzoate	Tembotrione
Thiamethoxam	Rimsulfuron	Fenoxaprop	Terbacil
Nitenpyram	Sulfometuron	Fipronil	Thien carbazone
	Sulfosulfuron	Fipronil sulfide	Thiophanate methyl
Dinotefuran	Thifensulfuron	Fipronil sulfone	Topramezone \$212
<b>Strobins</b>	Triasulfuron	Fluazinam	Trifluralin \$212
Dimoxys robin	Tribenuron	Flucarbazone	Trimethoprim
Picoxystrobin	Triflusulfuron		

# Attachment D

## UNITED STATES ENVIRONMENTAL PROTECTION AGENCY WASHINGTON, D.C. 20460



OFFICE OF  
CHEMICAL SAFETY  
AND POLLUTION PREVENTION

January 13, 2021

Kara Valentine  
Deputy Director of Air, Land and Energy  
Nebraska Department of Environment and Energy  
PO Box 98922  
1200 N St., Suite 400  
Lincoln, NE 68509-8922

Dear Ms. Valentine,

Thank you for your inquiry to the Environmental Protection Agency (EPA) concerning the wastewater discharge situation with a Nebraska ethanol production facility. The EPA was informed that water from the facility's wastewater containment ponds (referred to as lagoons) contains various concentrations of pesticides and has been applied to nearby fields in the past. The EPA is taking this opportunity to explain our process for assessing pesticide risk to human health and the environment and why the residues detected in the wastewater lagoons far exceed the registered application rates for which EPA has conducted safety assessments for pesticide products. Our assessments include estimating pesticide concentrations in drinking water by determining the potential for runoff to surface water and leaching into ground water. We would also point out that our discussion here is limited to the Federal Insecticide, Fungicide and Rodenticide Act (FIFRA) and does not include an analysis of any possible implications under other statutes that may be implicated.

In accordance with FIFRA, the EPA registers a pesticide when it determines that it will not cause unreasonable adverse effects on humans or the environment, while taking into account the economic, social, and environmental costs and benefits of the use of the pesticide. Under FIFRA, the EPA is charged with balancing risks posed by the use of a pesticide against its benefits. The EPA must determine if the benefits in light of its use outweigh the risks in order for the EPA to register a pesticide.

In evaluating a pesticide registration application, the EPA assesses a wide variety of exposure information (i.e., where and how the pesticide is used) and studies concerning environmental fate (i.e., how the chemical will move in the environment) and toxicity (i.e., effects on humans and other non-target organisms) to determine the likelihood of adverse effects (i.e., risk) from exposures associated with the proposed use of the product. Risk assessments are developed to

evaluate the environmental fate of the pesticide compound, as well as how it might affect a wide range of non-target organisms including humans and terrestrial and aquatic wildlife (plants and animals). On the basis of these assessments, the EPA evaluates and approves language for each pesticide label to ensure the directions for use and safety measures are appropriate to mitigate any potential unreasonable risk. In this way, the pesticide label communicates essential limitations and mitigations that are necessary to prevent unreasonable adverse effects. It is a violation of FIFRA to use a pesticide in a manner inconsistent with its labeling.

The EPA's evaluation of environmental fate studies determines how fast and by what means a pesticide degrades, what chemicals it breaks down to, how much of the pesticide or its breakdown chemicals will travel from the application site, and where they will accumulate in the environment. The studies also assess how the pesticide breaks down in water, soil, and from exposure to light; how easily it evaporates in air; and how quickly it travels through soil. The EPA uses these tests to develop estimates of pesticide concentrations in the environment. The typical sources of pesticide exposure to humans are through food and drinking water. Most of the foods we eat have been grown with the use of pesticides and pesticide residues may be present inside or on the surfaces of these foods. Some pesticides applied to farmland or other land structures can make their way to the ground water or surface water systems that feed drinking water supplies. Humans may also be exposed to pesticides by inhalation of dust blown from treated fields.

The EPA's ecological risk assessments evaluate the likelihood that exposure to one or more pesticides may cause harmful ecological effects. The effects can be direct (e.g., fish die from a pesticide entering waterways, or birds do not reproduce normally after ingesting contaminated fish). Some of the impacts on animals that EPA evaluates are the short- and long-term effects of varying amounts of pesticide exposure to insects and other invertebrates, fish, and birds. For plants, the EPA evaluates how toxic the pesticide is to plants, how the pesticide affects a seed's ability to germinate and emerge, as well as the plant's health and vigor as it grows.

According to the analysis conducted in November 2019 by the South Dakota Agricultural Laboratories for the Nebraska Department of Environment and Energy, the pesticides detected in the wastewater lagoons include neonicotinoid insecticides and fungicides in the strobiluron and triazole classes, as well as two herbicides. EPA concludes that applying this water to nearby fields is likely to result in application of these compounds to farmlands at rates that far exceed the registered application rates for which EPA has conducted safety assessments for products containing these pesticides. Some of these pesticides are known to leach and may contaminate groundwater. Some may be persistent, and runoff will contaminate aquatic ecosystems. Additionally, there are systemic pesticides in these samples and based on the high levels detected, they can be taken up into plant tissues and result in levels of residues in nectar and pollen that may harm pollinators or in leaves or other plant parts that are consumed by birds and mammals. Information on the profile and characteristics specific to each of the chemicals in the wastewater is available at: <https://iaspub.epa.gov/apex/pesticides/f?p=chemicalsearch:1>

The EPA has a comprehensive and robust assessment process for assessing the environmental effects of a chemical used as a pesticide, but the wastewater produced by the Nebraska ethanol facility represents a level of contamination that has no uniformity or limit on the number and

amount of pesticides present. The EPA cannot conclude that discharging this water onto land will not result in unreasonable adverse effects on humans or the environment.

Sincerely,

**MICHAEL  
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Ed Messina, Esq., Acting Director  
Office of Pesticide Programs

Cc DeAndre Singletary, Region 7