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DEPT. OF ENVIRONMENT AND ENERGY

Site-Specific Sampling Plan

Stockpiled Alt-En Wet-cake/Dry Distiller Grain Waste Composite Sampling Procedures

March 10, 2021

Final Version



SCOPE AND APPLICABILITY

The site-specific sampling plan is designed to highlight the procedures necessary to collect a minimum of three composite samples of the wetcake/dry distiller grain waste that is stockpiled at various location on the Alt-En Ethanol Plant's property in Mead, NE. This plan utilizes procedure from the Nebraska Department of Environment and Energy (NDEE) Generic Quality Assurance Project Plan for the Superfund Site Assessment and Section 128(a) Assessment Program, dated January 2016 and Standard Operating Procedure (SOP) for Waste Characterization, REM-SFA-15, Version 1, dated February 2021. As needed, equipment and sampling materials will be decontaminated in accordance with the NDEE Field Decontamination SOP, REM-SFA-17, Version 1, dated February 2021.

This limited environmental sampling event is intended to supplement the data that is currently available in an effort to characterize the waste which is derived from a digester process at the ethanol plant. The feed product for this waste stream is seed corn that has been treated with a variety of agricultural chemicals that include pesticides, herbicides, and fungicides. These waste samples will be sent to the South Dakota Agricultural Laboratories (SDAL), at 1335 Western Avenue, Brookings, SD 57006.

The sampling team is anticipated to be two people. The sampling team will announce their arrival at the facility and coordinate with the plant manager to collect composite samples from existing waste piles on the facility property. The sampling team may be accompanied by facility personnel in order to confirm the locations of the stockpiled waste and to identify best routes for accessing the waste piles, as needed. Due to COVID-19 restrictions, the sampling team will wear masks and practice social distancing according to agency protocols.

EQUIPMENT AND MATERIALS

The equipment and materials that are anticipated to be used to accomplish this sampling event are listed below. Additional equipment and materials may be deemed necessary when conducting the sampling activities. The following equipment and materials will be collected before deploying to the site:

- Camera (optional, pictures may be taken using cellphones)
- Chain-of-custody documentation
- Distilled water
- Field logbook
- Global Positioning System (GPS) unit
- Health and Safety Plan / Safety Plan
- Materials required for decontamination (per NDEE's field decontamination SOP):
 - Distilled water
 - Isopropyl alcohol
 - Non-phosphate laboratory detergent (Alconox, liquid Dawn, etc.)

- Measuring wheel and/or tape measures (optional, GPS coordinates may suffice for measuring distances between locations/structures/markings devices that are used to demark sample collection points and for estimating waste pile dimensions)
- Packing tape
- Pens/markers
- Level D (modified) Personal Protective Equipment (PPE), including:
 - Hard hat, safety boots (i.e., steel toe, chemical resistant, insulated), disposable sample handling gloves, safety glasses, respirators (as deemed necessary), dust masks, etc.
- Pin flags and/or wooden stakes
- Plastic sheeting and/or tarp
- Sample containers prescribed by SDAL (composite sample volume is expected to be approximately 32 ounces in volume, in accordance with the analytical lab instructions)
- Sample labels
- Scoops, trowels, spoons, ladles
- Shipping materials (cooler with ice, large zipper storage bags, large garbage bags, package tape, cooler custody seal[s])
- Shovel, pickaxe, hoe (based on NDEE in-house inventory at time of deployment)
- Stainless steel bowl(s)
- Volume measuring device such as a measuring cup (8 liquid ounces)

PROCEDURES

PRECAUTIONS

The following precautions should be taken:

- **Sample Sequencing and Clean/Safe Zones:** The sampling team will identify and setup a practical clean zone for laying out sampling equipment and materials and to help avoid cross-contamination. It is advisable to use disposable tools and personal protective equipment to the extent possible such that decontamination is not necessary.
- **GPS of Sample Locations:** Do not collect GPS sample locations that increase exposure of the sampler to the waste material, such as walking on the waste.
- **Contaminated Waste:** Assume and treat sampled material as containing high levels of contaminants. Wear appropriate PPE during the actual sample collection process and follow appropriate decontamination procedures.
- **Equipment Cleanliness:** Once a piece of equipment has been decontaminated, be careful, protect it, keep it in a clean, and useable condition until needed.
- **Liquid Soaps:** Some soaps (Alconox, Dawn, etc.) that are used for decontamination of field equipment have been found to contain detectable levels of 1,4-Dioxane, an emerging groundwater contaminant. A project-specific decontamination method should be selected on a contaminant specific basis and may necessitate other methods to clean field equipment, such as using a natural soap such as Castile Soap, a natural-based detergent if 1,4-Dioxane is a contaminant of concern.
- **Investigation Derived Waste:** Disposable materials that are used for this sampling event are to be treated as solid waste based on quantity and waste characteristics.

- **Decontamination:** This plan has procedures that are based on NDEE's SOP for additional guidance for decontamination of sampling equipment.
- **Sample Collection:** Refer to NDEE SOP for sample container, preservation, and handling for additional guidance on sampling.
- **Safety Plan:** Refer to the Safety Plan for a hospital location, route map, and telephone information. In addition, calling 911 in the event of an accident should be considered.

WASTE PILE SAMPLING PROCEDURES

Identify the waste pile to be sampled and use GPS coordinates to document the location where samples are collected and record GPS coordinates for the correct benchmark point that is being described or otherwise identified by sample numbers or other nomenclature.

Waste Pile Identification, Size, and Location:

- If necessary, use a measuring wheel, tape measure, and/or GPS unit, to measure the perimeter and height of the waste "pile" that will be sampled.
- Photograph waste pile and record pile dimensions on field forms or in the field logbook.
- During active inspection of the pile note any changes in the texture, color, material composition, moisture content, etc., of the waste to be sampled.

Establish a Clean/Safe Zone and Review Sampling Objectives:

- Review the number of samples to be collected, sample composite techniques and the number of subsample aliquots to be collected for each composite sample.
- To the extent that the various sides and boundaries of the waste piles will be accessible, identify and mark approximate locations for each of the aliquots to be collected on the site map or waste pile location map/sketch. The goal is to collect up to ten (10) aliquots per composite sample. However, a minimum of five (5) aliquots must be collected in accordance with the NDEE Waste Characterization SOP guidance and procedures.
- Document sample and/or subsample locations in the field logbook accordingly.

Collect and composite subsamples:

1. As necessary, establish clean areas, sample processing areas, work/safe zone(s) and organize the equipment. Optionally, use a tarp or cut a section of plastic sheeting of approximately 4 feet x 4 feet or larger. Place the sheeting within walking distance of the waste pile, at the established clean/work area(s).
2. As needed, arrange the sample containers, sampler(s), and decontamination equipment on the plastic sheeting.
3. Don PPE (including gloves and safety glasses).
4. To the extent practical, remove surficial layer of stockpiled wet-cake sludge waste to a depth of up to 6-inches to expose and collect waste that has not been impacted by weather events.

5. Collect subsample aliquots at depth with a stainless-steel trowel. If practical and accessible, and to help collect a representative sample that allows for variation by waste pile depth, it may be necessary to collect a second aliquot at a deeper interval at any particular subsample location.
6. Use a stainless-steel container to collect approximately equal quantities of waste from each aliquot.
7. After all aliquots are collected for a particular composite sample, homogenize the sample using a decontaminated stainless-steel spoon, disposable trowel, or spatula.
8. Transfer the sample into sample containers using the spoon, trowel, or spatula.
9. Ensure that each sample container is properly labeled, noted on the chain-of-custody form, and placed in the sample cooler with ice (preferable in zip lock bags) or ice packs.
10. Decontaminate sampling equipment. Decontamination will be conducted in an area away from potential contaminants but not in a clean area.
 - a. Scrub the sampler to remove solid and liquid gross (visible) contamination.
 - b. Scrub the sampler in a wash container using appropriate brush(es), distilled water, and non-phosphate laboratory detergent.
 - c. In a second wash container rinse or spray off detergent/dirty water with distilled water.
 - d. Rinse or spray sampler a second time with distilled water.
 - e. Allow sampler to air dry.
 - f. Wrap sampler in aluminum foil or clean plastic sheeting, or store in a new zip-seal bag (size permitting) or clean, dedicated polyvinyl chloride or PTFE storage container.
 - g. Rinse sampler with distilled water immediately prior to re-use.
11. Collect and dispose of IDW materials in accordance with guidance and regulatory requirements.
12. Any remaining (unused) composite sample material will be placed back on the waste pile.

DOCUMENTATION

Documentation of field activities shall be presented in the form of site plan sketches, chain-of-custody, sample transmittal forms, NDEE field forms, and/or field logbooks. Photographs documenting sampling activities shall also be taken.

Field logbook documentation shall include latitude and longitude of sampling locations, date and time of sampling activities, as well as any unusual conditions or observations encountered during sampling.

Attachments:

- **Waste Pile Location Map**
- **Safety Plan**
- **SDAL Chain of Custody Form**
- **Completed Site-Specific QAPP Addendum Form**

AltEn LLC February 1, 2021 Site Visit Approximate Wetcake Area in Acres



Address: 11344 County Road 10, Mead NE
NDEQ ID: 84069
Program ID: NE0137634

0 200 400 800 1,200 1,600 2,000
Feet

NEBRASKA
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1335 Western Ave

South Dakota Agricultural Laboratories

Chain of Custody

Brookings, SD 57006

PH 605-692-7325 Fax 605-692-7326 www.sdaglabs.com

Contact information					Azole Screen	Neonics Screen	OP Screen	Strobins Screen	Abamectin
Company / Organization:		Address:							
Contact:		Email:							
Phone:		Special Instructions:							
Project Site / Sampling Event									
Date of Sample Collection									
Sample ID	Time Collected	Sample Media / Type	Number of Containers	Description / Notes					

Sample Type Codes	
VE –vegetation	CI- clothing
SO – soil	AN- animal
SW – swab	DDG – dried distiller's grains
WA – water	

Turn-around-times
Special charges apply for RUSH samples. Please contact the laboratory before sending.

Commercial Carrier			
Date/Time Relinquished to Carrier			
Number of Ice Chest(s) Shipped			
Relinquished By	Date & Time	Received By	Date & Time

NEBRASKA DEPARTMENT OF ENVIRONMENTAL QUALITY
SITE-SPECIFIC QUALITY ASSURANCE PROJECT PLAN (QAPP) ADDENDUM
SUPERFUND SITE ASSESSMENT & SECTION 128(a) ASSESSMENT PROGRAMS

Note: This Addendum supplements the Generic QAPP for Superfund Site Assessment and Section 128(a) Assessment activities (Revised January 2016) and includes documentation only for the specific site/project indicated. The Addendum is only necessary for projects with sampling requirements needed to accomplish site assessment activities.

1. SITE NAME AND LOCATION:

Name: Alt-En Ethanol Plant – Environmental Media Sampling (Wet-Cake Composite Samples)

Address or Other Location Identifier: Area of concern is located in the vicinity of Mead, NE

City: Village of Mead **County:** Saunders **State:** Nebraska **ZIP:** 68041

Site Point of Contact (POC) Name: Jim Borovich **POC Email:** jim.borovich@nebraska.gov

Telephone: 402-471-2223 (work/direct) **FAX:**

Directions to Site: From I-80, go north on Hwy 77, east on Hwy 66, north on Road 10 **Map(s) attached:** Yes

2. PROJECT MANAGEMENT AND PROJECT INFORMATION:

Distribution List (Check as appropriate and identify):

- | | |
|---|--|
| <input checked="" type="checkbox"/> NDEQ Section/Unit Supervisor(s): Mike Felix | <input checked="" type="checkbox"/> NDEQ QA Manager: Sam Capps |
| <input type="checkbox"/> NDEQ Fixed Lab / QC Coordinator: Wade Gregson | <input checked="" type="checkbox"/> NDEQ Project Manager(s): Jim Borovich |
| <input checked="" type="checkbox"/> Contractor Project Manager (Specify): N/A | <input checked="" type="checkbox"/> Other (Specify): Tom Buell, Div. Administrator |

2.1 Project and Task Organization (Check as appropriate and identify):

- | | |
|--|--|
| <input checked="" type="checkbox"/> NDEQ Project Manager: Jim Borovich | <input checked="" type="checkbox"/> NDEQ QA Manager: Sam Capps |
| <input checked="" type="checkbox"/> NDEQ QA/QC Coordinator: Wade Gregson | <input type="checkbox"/> Site Safety Officer: N/A |

2.2 Problem Definition and Background: (Check to indicate items are attached)

- | | |
|--|---|
| <input type="checkbox"/> Discussion of site-specific conditions | <input type="checkbox"/> Table indicating maximum concentrations detected |
| <input checked="" type="checkbox"/> Maps and figures (sample wet-cake stockpiled at plant) | <input type="checkbox"/> Table indicating concentrations of concern |
| <input type="checkbox"/> Table indicating number and types of field and QC samples to be collected | |
| <input type="checkbox"/> Discussion of laboratory deviations from latest version of EPA SOP 2440.5 (if applicable) | |

2.3 Project and Task Description/Type(s) (Check as appropriate):

- | | |
|---|---|
| <input type="checkbox"/> Pre-CERCLIS Site Screening Assessment (PCSSA) | <input type="checkbox"/> Preliminary Assessment (PA) |
| <input type="checkbox"/> Abbreviated Preliminary Assessment (APA) | <input type="checkbox"/> Site Inspection (SI) |
| <input type="checkbox"/> Expanded Site Inspection (ESI) | <input type="checkbox"/> Focused Site Inspection (FSI) |
| <input type="checkbox"/> Section 128(a) Assessment - Phase I Investigation | <input type="checkbox"/> Site Re-Assessment (SR or SI-2) |
| <input type="checkbox"/> Section 128(a) Assessment - Phase II Investigation | <input checked="" type="checkbox"/> Other (Specify and attach description): |

Collect a minimum of three (3) composite wet-cake samples from three different waste piles.

2.4 Quality Objectives and Criteria for Measurement Data: (Check appropriate boxes)

- | | | |
|---------------------|--|---|
| Accuracy: | <input type="checkbox"/> According to Generic Site Assessment QAPP | <input type="checkbox"/> Identified in attached table |
| Precision | <input type="checkbox"/> According to Generic Site Assessment QAPP | <input type="checkbox"/> Identified in attached table |
| Representativeness: | <input type="checkbox"/> According to Generic Site Assessment QAPP | <input type="checkbox"/> Identified in attached table |
| Completeness:* | <input type="checkbox"/> According to Generic Site Assessment QAPP | <input type="checkbox"/> Identified in attached table |
| Comparability: | <input type="checkbox"/> According to Generic Site Assessment QAPP | <input type="checkbox"/> Identified in attached table |

*A completeness goal of 100 % has been established for this project. However, a site disposition may still be possible from the remaining valid data.

Critical sample locations are included in (check all that apply):

- ☐ Attached Table(s) ☐ Attached Map(s) ☐ Work Plan ☒ Other (Describe): Sample locations will be based on safe access to existing stockpiled wet-cake byproduct from the plant's digester.

2.5 Special Training/Certification Requirements: (Check appropriate boxes)

- | | |
|---|--|
| <input checked="" type="checkbox"/> OSHA 40-hour (HAZWOPER) | <input type="checkbox"/> Direct Push Probe/Geoprobe Operator |
| <input type="checkbox"/> Mobile GC Field Analyst | <input type="checkbox"/> In-Field XRF Operator |
| <input type="checkbox"/> Water Well Monitoring Supervisor and/or Technician | <input type="checkbox"/> Water Well Drilling Contractor and/or Drilling Supervisor |
| <input type="checkbox"/> Pump Installation Contractor and/or Supervisor | <input type="checkbox"/> Drill Rig Operator |
| <input type="checkbox"/> Other (specify): | |

2.6 Documentation and Records Proposed For Project: (Check appropriate boxes):

- | | | |
|---|--|--|
| <input type="checkbox"/> Project Work Plan | <input checked="" type="checkbox"/> Log Book/Field Notes | <input type="checkbox"/> Drilling permit(s) |
| <input checked="" type="checkbox"/> Health and Safety Plan | <input type="checkbox"/> Field Sheets | <input checked="" type="checkbox"/> GPS Coordinates (optional) |
| <input checked="" type="checkbox"/> Site Maps/Figures | <input checked="" type="checkbox"/> Chain-of-Custody | <input type="checkbox"/> Licensed surveyor site map |
| <input type="checkbox"/> Site Sampling Map (attached) | <input checked="" type="checkbox"/> Site Photographs | <input type="checkbox"/> Calibration Records |
| <input type="checkbox"/> Property Access Agreement | <input type="checkbox"/> Site Videotapes | <input type="checkbox"/> ASTM Phase I ESA Requirements |
| <input type="checkbox"/> Property Ownership Records | <input type="checkbox"/> Utility Clearance Forms | <input type="checkbox"/> ASTM Phase II ESA Requirements |
| <input type="checkbox"/> Daily Tailgate Meeting Forms | <input type="checkbox"/> Other Documentation (Specify): | |
| <input type="checkbox"/> Reports, Deliverables, or Submittals Required (Specify): | | |

3. DATA GENERATION AND AQUISITION:**3.1 Sampling Process Design****A. General Sampling Approach (Check appropriate boxes):**

- | | |
|---|---|
| <input type="checkbox"/> Probability Sampling | <input checked="" type="checkbox"/> Judgmental Sampling |
|---|---|

Sampling Method:

- | | | |
|---|--|--|
| <input type="checkbox"/> Simple Random Sampling | <input type="checkbox"/> Stratified Sampling | <input type="checkbox"/> Systematic/Grid Sampling |
| <input type="checkbox"/> Ranked Set Sampling | <input type="checkbox"/> Adaptive Cluster Sampling | <input checked="" type="checkbox"/> Incremental/Composite Sampling |

B. Screening/Definitive Sampling (Check appropriate boxes):

- | |
|---|
| <input checked="" type="checkbox"/> Screening without Definitive Confirmation |
| <input type="checkbox"/> Screening with Definitive Confirmation. Confirmation sampling rate of _____% of field screening samples. |
| <input type="checkbox"/> Definitive Sampling |

C. Biased/Judgmental Sampling:

- | |
|---|
| <input type="checkbox"/> No (If No, explain the alternate sampling rationale and approach below): |
| <input checked="" type="checkbox"/> Yes (If Yes, the text below applies): |

Explain below:

The proposed sampling scheme will be judgmental. Judgmental sampling is the subjective (biased) selection of sampling locations based on available information, visual inspection, and professional judgment.

3.2 Analytical Methods and SOP Requirements (Specify all to be utilized):

Matrix	Sampling Method	Std. Operating Procedures	Sampling Equipment Proposed
<input type="checkbox"/> Soil			Hand Probe
<input type="checkbox"/> Soil-Gas			
<input type="checkbox"/> Groundwater			
<input type="checkbox"/> Surface Water			
<input type="checkbox"/> Sediment			
<input checked="" type="checkbox"/> Waste	Ag Chem Screening	REM-SFA-15 Waste Characterization	Disposable trowel, mixing basin, see SOP for list
<input type="checkbox"/> Leachate			
<input type="checkbox"/> Air			
<input type="checkbox"/> Other (specify below):			

3.3 Sample Handling and Custody Requirements (Check appropriate boxes):

- ☒ In accordance with Generic QAPP and SOPs ☒ Use Analytical Laboratory Chain of Custody Form(s)
☐ Other (specify):

3.4 Analytical Methods Requirements (Check appropriate box):

- ☐ Identified in Attached Table(s)/Lists ☒ Other (Describe): As Directed by NDEE Project Manager

3.5 Quality Control Requirements* (Check all appropriate boxes):

- ☐ Not Applicable ☒ In accordance with Generic QAPP
☐ Specific requirements (state):

Field QC Samples to be collected:

- | | | |
|---|--------------------------|----------------------------------|
| <input type="checkbox"/> Duplicates* | (frequency 1 per 20/day) | Prepared by: Field Personnel |
| <input type="checkbox"/> Trip Blanks (VOCs) | (frequency 1 per cooler) | Prepared by: Contract Laboratory |
| <input type="checkbox"/> Field Blanks* | (frequency 1 per day) | Prepared by: Field Personnel |
| <input type="checkbox"/> Equipment Rinse Blanks | (frequency) | Prepared by: (Select) |
| <input type="checkbox"/> Split Samples | (frequency) | Splits go to: |
| <input type="checkbox"/> Others (specify) | (frequency) | |

* The requirement for and frequency of field duplicates and field blanks must be established for each sampling event.

3.6 Instrument/Equipment Testing, Inspection, and Maintenance Requirements (Check appropriate box):

- ☒ Not Applicable ☐ In accordance with Generic QAPP
☐ Specific field or laboratory equipment requirements:
Instrument: Testing, Inspection, or Maintenance Frequency:
Instrument: Testing, Inspection, or Maintenance Frequency:
Instrument: Testing, Inspection, or Maintenance Frequency:
Critical Spare Parts Required:

3.7 Instrument/Equipment Calibration and Frequency (Check appropriate box):

- ☒ Not Applicable ☐ In accordance with Generic QAPP
☐ Specific field equipment requirements:
Instrument: Calibration Frequency:
Instrument: Calibration Frequency:
Instrument: Calibration Frequency:
Instrument: Calibration Frequency:

3.8 Inspection/Acceptance Criteria for Supplies and Consumables (Check appropriate box):

- ☒ Not Applicable ☐ In accordance with Generic QAPP
☐ Specific requirements (state):

3.9 Data Acquisition Requirements for Non-Direct Measurements (Check appropriate box):

- ☒ Not Applicable ☐ In accordance with Generic QAPP
☐ Specific requirements (state):

3.10 Data Management and Record Retention Requirements (Check appropriate box):

- ☐ In accordance with Generic QAPP ☒ Specific requirements (state): TBD/Screening level requirements

4. ASSESSMENT AND OVERSIGHT:

4.1A Assessment and Response Actions (Check all appropriate boxes):

- ☒ Peer Review ☒ Management Review ☐ Field Audit ☐ Lab Audit
☒ Those pertaining to analytical phases of the project will be in accordance with the contracted commercial Analytical Laboratory internal SOPs
☐ Specific requirements (state):

4.1B Corrective Action (Check appropriate box):

- ☐ In accordance with Generic QAPP ☒ Specific requirements (state): To be determined, if needed.

4.2 Reports to Management / Assessment Reporting (Check appropriate boxes):

- ☐ In accordance with Generic QAPP
☐ In accordance with ASTM E-1527-13 Phase I Environmental Site Assessment
☐ In accordance with ASTM E-1903-11 Phase II Environmental Site Assessment
☒ Specific requirements (state): TBD / screening level data validation and internal reporting requirements which will be based on the sampling results and other pertinent data being used to evaluate site conditions.

5. DATA VALIDATION AND USABILITY:

5.1 Data Review, Verification, and Validation Requirements (Check appropriate box):

- ☐ Data review and verification will be performed by Project Manager or QC officer delegate in accordance with Generic QAPP, with data validation conducted according to Generic QAPP.
☒ Data review, validation and verification will be performed as follows with data validation conducted according to alternate methods (describe): Contracted analytical laboratory will perform data validation and QA/QC review.

Field analysis utilized? No (If yes, memorandum, field analytical sheets, etc. from field analyst should be reviewed by Project Manager and/or Site QC Officer after completion of field analysis).

5.2 Verification and Validation Methods (Check appropriate box):

- ☐ In accordance with Generic QAPP ☒ Specific requirements (state): Analytical laboratory QA/QC/data validation review and internal QA/QC review process will be conducted.

5.3 Reconciliation with User Requirements—Data Quality Objectives (Check appropriate box):

- ☐ In accordance with Generic QAPP ☒ Specific requirements (state): TBD / screening level data validation and internal reporting requirements which will be based on the sampling results and other pertinent data being used to evaluate site conditions.

6. APPROVAL SIGNATURES & DATES:

Note: The QAPP Addendum will not be considered complete until all of the appropriate approval signatures indicated below are obtained. If the QAPP Addendum has to be revised after signatures (some or all) were obtained, new signatures will be required to properly finalize and approve the revised QAPP Addendum.

N/A	N/A	N/A
Contractor Project Manager	Signature	Date
(name)		

N/A	N/A	N/A
Contractor QA Officer/Designee	Signature	Date
(name)		

Jim Borovich		3/10/21
NDEQ Project Manager/Designee	Signature	Date
(name)		

Mike Felix		3/9/21
NDEQ Unit or Section Supervisor	Signature	Date
(name)		