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June 5, 2007

Mr. Chris Saari Wisconsin Department of Natural Resources 2501 Golf Course Road Ashland, WI, 54806

Dear Mr. Saari:

Subject: Field Pilot Test for DNT Degradation Former DuPont Barksdale Site Barksdale, WI

Enclosed please find 2 copies of the 2007 DNT Biopilot Field Study Design and Work plan dated June 2007 for your information and use. This workplan has been prepared to describe the procedures to be used to evaluate the laboratory work conducted at Georgia Tech during 2006. During this study, Georgia Tech demonstrated that DNT concentrations in soil could be reduced by 99% by adding air and water to the soils. It is DuPont's intention to begin construction immediately and run the pilot through the end of September 2007.

Should you have any comments or questions, please feel free to contact me at 502-217-1531.

Sincerely,

fred Nave

Brad Nave Project Director DuPont Corporate Remediation Group



2007 DNT BIOPILOT FIELD STUDY DESIGN AND WORK PLAN FORMER DUPONT BARKSDALE WORKS BARKSDALE, WISCONSIN

Date: June 2007

Project No.: 507625



CORPORATE REMEDIATION GROUP An Alliance between DuPont and URS Diamond

> 325 West Main Street Suite 1202 Louisville, Kentucky 40202

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1.0 INTRODUCTION

E.I. du Pont de Nemours and Company (DuPont) operated an explosive manufacturing facility for approximately 70 years in Barksdale, Wisconsin known as the DuPont Barksdale Works (site) (see Figure 1). In the mid-1980s, DuPont sold the site to Bretting Development Corp., which maintains the site for private recreation pursuits. The site is located on State Highway 13 in Barksdale, Wisconsin, approximately two miles north of the US Highway 2 and State Highway 13 junction.

In 1997, nitroamine and nitroaromatic organic compounds were identified in residential drinking-water wells adjacent to the site. As a result of the detections, DuPont has provided municipal drinking water to affected homes and has voluntarily conducted environmental site investigation activities to determine the nature of site-related compounds that are present at the site surface and in groundwater due to previous manufacturing operations. Sampling conducted during previous phases of the site investigation has indicated that the chemicals of concern in environmental media are nitroamine and nitroaromatic compounds, primarily the production materials trinitrotoluene (TNT) and dinitrotoluene (DNT).

DuPont has partnered with the Georgia Tech University (GA Tech) to develop an *in-situ* biodegradation approach to remediate DNT isomers in surficial soil. A laboratory pilot study conducted at GA Tech in 2006 confirmed that DNT could be degraded aerobically by naturally occurring bacteria. This field pilot will attempt to replicate the findings of the laboratory study by applying the study findings to actual field conditions.

1.1 Purpose

This document has been prepared on behalf of DuPont by the DuPont Corporate Remediation Group (CRG). The scope of this plan is to conduct an *in-situ* field study of the degradation of DNT isomers present in surficial soils (top 2 feet of soil horizon) at the site. This plan will document the design and implementation steps associated with conducting a multiple stage field study in the former Lydol House area.

The application of Key Learnings discovered during laboratory studies will be implemented during this field study in order to reach the project goal:

To determine if naturally occurring bacteria can produce a measurable decrease in DNT concentrations in a field setting and to provide evidence that DNT isomers have been mineralized.

In addition, the field study is designed to evaluate execution methodologies, infrastructure requirements, and estimate waste generated during full-scale operation.

1.2 Objectives

The objectives of the field study are as follows:

• Show a measurable degradation and mineralization of DNT isomers within surficial soil.

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- o Develop a better understanding of DNT degradation rates.
- o Identify key controllers for implementation to a full scale remediation application.

1.2.1 Measurable Degradation and Mineralization of DNT Isomers

Degradation of DNT isomers will be monitored through sampling of each treatment cell as prescribed under the established sampling protocols for each field study process phase (see Section 3.0). Target remediation levels have been developed to determine study success. The two identified remediation levels are based on progressively greater remediation requirements that allow soils to remain on-site as part of the final remedy.

Remediation Level 1 – 90% Reduction and Corrective Action Management Unit

A 90% reduction of DNT isomers would allow for the option to consolidate soils into an on-site Corrective Action Management Unit, subject to U.S. Environmental Protection Agency (EPA) approval. This option would require that soils be managed on-site through engineering controls, monitoring, and deed restrictions.

Remediation Level 2 – Unrestricted Recreational Use

A reduction of DNT concentrations to levels below the site-specific screening levels developed for unrestricted recreational use would require no soil removal, nor placement of deed restrictions on future use.

1.2.2 DNT Degradation Rates

DNT isomer degradation rates will be calculated from the analytical results associated with each field study process phase. Compilation of the analytical data will be used to develop degradation versus time curves, which can then be used to predict time requirements for achieving the remediation levels.

1.2.3 Full Scale Remediation Key Controllers

Several factors have been identified as important elements to the successful implementation of a full-scale remediation application. This field study will provide additional information as to the practicability of these elements.

Tilling

The primary limitation of biodegradation presently occurring is thought to be sequestration of DNT within the clayey soil. Through manual tilling of the surficial soils, it is believed that DNT will become readily available for degradation by naturally occurring bacteria.

Soil pH

DNT degradation products have a potential to drive soil pH downward to an acidic matrix. Monitoring and application of an agricultural grade lime will be implemented to maintain soil pH within a range of 6.5 to 7.5 (standard units). One treatment cell will

receive pH treatment exclusively during the initial stage (2007 season) of the field study; and, in subsequent stages, all cells will receive pH control based on the 2007 findings.

Water

The effect of water quantity will be addressed by applying a quantity of water equivalent to an additional 2 inches of rainfall per month. However, water will not be supplied when conditions of the treatment cell begin to retain water and present a risk of becoming an anaerobic system (see Table 1 and Figure 2 for details).

Ambient Temperature

Air temperature will be monitored on a continuous basis to determine the effect of ambient temperature on degradation rates across all treatment cells.

Impacts to Aquifer

To date, nitrate and nitrite ions have not been a concern at the site; however, the mineralization of DNT isomers will generate nitrate and nitrite compounds. Lysimeters will be installed within each treatment cell to monitor the production of these constituents through the collection and analysis of pore-space water. Piezometer 400 lies downgradient of the study location and will be monitored periodically for changes in nitrate and nitrite concentrations.

Overall Technique Improvements

In addition to the previously identified key controllers, the field study will allow for the development and testing of various field techniques. Technical improvements that are identified during the execution of the work will be documented at the end of each study stage and incorporated into future stages of this study.

2.0 CONCEPTUAL DESIGN

2.1 Location

The field study site is located in Barksdale Works Production Area C (PAC) (see Figure 1). PAC, located in the western central portion of the site between Main Drive and TNT Loop Road, housed the former Lydol (nitrated solvent naphtha - NSN) and Trivelene (DNT as a final product) Plants. The area contains one Lydol and three Trivelene production lines, which were operated between approximately 1913 and 1920.

PAC covers approximately 16 acres and is divided by West Gate Road, which crosses from east to west. North of West Gate Road the area is heavily wooded. The wooded portions of PAC have been heavily reconnoitered near production buildings and many of these building sites, including the Lydol House location, have been cleared of trees and brush during previous investigations.

The main drainage in PAC flows from west to east within the woods about 220 feet north of West Gate Road. North-to-south ditches feed into this drainage from process buildings throughout the wooded area. Drainage enters the area through culverts under Main Drive from Production Area E and Production Area U. Drainage leaves the area crossing West Gate Road to the southeast into Production Area O and Production Area R. Sediment accumulations and wash-outs around culverts leaving the area indicate that some erosion is occurring in the main ditch crossing PAC.

The soils at the Lydol House site are clay. Drilling at well PZ-40o, located 300 feet southeast of the former catch tank, indicated that the clay extends to about 24 feet below ground. Glacial till (dense silty gravel and sand with significant amounts of clay sized material) is located below the clay (see Figure 3). The upper groundwater surface in the area lies within the till at about 35 feet below ground. Groundwater levels interpolated from data at the nearest wells around the Lydol House (PZ-40o at 300 feet southeast, PZ-45o 800 feet north, and PZ-26s/d 900 feet southeast) indicate flow to the southeast toward Boyd Creek (see Figures 4 and 5).

Groundwater sampled from PZ-40o contained TNT [up to 0.047 micrograms per liter (μ g/L)], DNT (up to134 μ g/L), benzene, carbon tetrachloride, chloroform, methylene chloride, methyl nitrobenzene, 2-nitrotoluene, nitrobenzene, perchlorate and iron. Wells upgradient of this Use Area contained significantly more TNT and less DNT, while downgradient wells typically contained lower concentrations of each compound. The difference in DNT concentrations in the upgradient and downgradient wells indicates that a release of nitramine and nitroaromatic organic compounds may have occurred from PAC to groundwater.

2.2 Treatment Cells

This field study will determine the effects of aeration, pH control, and water quantity on the degradation of DNT.

Four treatment cells have been chosen as indicated below (see Figure 6):

- Treatment Cell #1 Tilling (initial), rainfall
 The cell identified as Treatment Cell #1 will be aerated upon initial startup of the study. The cell will receive no further treatment in terms of aeration, pH control, and/or water additives.
- Treatment Cell #2 Tilling (multiple), rainfall
 The cell identified as Treatment Cell #2 will be aerated upon initial startup and once per month during the In-Process phase of the field study. The cell will receive no further treatment in terms of pH control and/or water additives.
- Treatment Cell #3 Tilling (multiple), rainfall, lime
 The cell identified as Treatment Cell #3 will be aerated upon initial startup and once per month during the In-Process phase of the field study. The cell will receive pH control through the addition of an agricultural grade lime. The cell will receive no additional water other than rainfall.
- Treatment Cell #4 Tilling (multiple), rainfall, supplemental water The cell identified as Treatment Cell #4 will be aerated upon initial startup and once per month during the In-Process phase of the field study. The cell will receive supplemental amounts of water to maintain constant moisture content in addition to the rainfall. The cell will not receive treatment for soil pH control.

2.3 Operational Schedule

The field study will be operated during the 2007 construction season and additional seasons as determined by the project team. An average construction season at the site lasts from mid-May through September, or as the weather permits.

The 2007 season will tentatively consist of a Pre-Startup phase to begin during the last week of May. The In-Process phase will tentatively begin on June 1 and run through September 30 or as the weather permits. The Post-Stage phase and winterization of the study will be conducted upon the conclusion of the In-Process phase during each study stage.

2.4 Operation Procedures

Three operational phases have been identified for implementation of the field study.

2.4.1 Pre-Startup Procedures

Treatment cells will be divided based on the size requirements listed in Section 3.0. Dividers will be used to reduce transfer of soil and water between cells. Each treatment cell location will be surveyed from a local monument and recorded.

Soil samples will be collected as prescribed in Section 3.1 for analysis by GA Tech. In addition, confirmation samples equal to 25 percent of the GA Tech samples (or three duplicate samples from each treatment cell) will be sent to Severn-Trent Laboratories – Denver.

After collection of soil samples, a motor powered tilling device will be used to aerate the top 2 feet of soil within each treatment cell. In addition, soil additives for pH control and supplemental water will be applied to Treatment Cell #3 and #4, respectively.

Lysimeters will be installed to a depth greater than 2 feet below ground surface within each treatment cell in accordance with the manufacturer's recommendations.

2.4.2 In-Process Procedures

Treatment cells will be managed during the construction season as prescribed in Section 2.2. Cells requiring continuous aeration will be tilled at the beginning of each month to a depth of 2 feet. Soil characteristics will be monitored every two weeks, and treatment will be applied to maintain the prescribed soil conditions. Analytical samples will be collected as defined in Section 3.2.

2.4.3 Post-Stage Procedures

Analytical samples will be collected at the end of each construction season in accordance with the Post-Stage protocol described in Section 3.1. Treatment cells will then be winterized by placing breathable liner over the cells, in order to prevent cross contamination during freeze/thaw and snow melt periods.

3.0 SAMPLING PROTOCOL

Based on calculations from soil samples taken during the pilot study at GA Tech, it was determined that 12 samples will need to be collected during the Pre-Startup and Post-Process phases of each stage in order to statistically detect a reduction of DNT isomers. Sample size was determined by taking the natural log of the pilot study analytical data and applying a two-sample t-test to determine a reduction of 0.5 units (lognormal mean that corresponds to a 39% reduction in the original units). In addition, four samples will be collected from each treatment cell on a monthly basis to determine a general trend line to model DNT reduction over time. The In-Process sampling results will also be used to show any deviations from a linear reduction and/or to show stoppage in the degradation progress.

Treatment cells will be divided into a 2 by 6 grid with grids measuring 3.3 by 5 feet. Discrete samples will be collected in accordance with the site's Quality Assurance Plan (QAP) from the center of each grid for the Pre-Startup and Post-Stage sampling events. Four in-process sampling grids will be chosen randomly from the same 12 grid locations.

3.1 Pre-Startup and Post-Process Sampling

Twelve discrete soil samples will be collected from each treatment cell and shipped to GA Tech for analysis. In addition, verification samples will be sent to Severn-Trent Laboratories – Denver. Samples will be analyzed for nitramine and nitroaromatic organic compounds. In addition, Tentatively Identified Compounds (TICs) also may be identified and quantified. Soil parameters will be quantified by laboratories for comparison to field measurements.

Field measurements of soil pH along with potassium, nitrogen, and phosphorous concentrations will be collected on a bimonthly basis. In addition pore-water will be analyzed for nitrate and nitrite concentrations. These parameters will be measured using field test kits.

3.2 In-Process Sampling

Four discrete soil samples will be collected from each treatment cell and shipped to GA Tech for analysis. Samples will be analyzed for nitramine and nitroaromatic compounds. In addition, TICs also may be identified and quantified. Soil parameters will be quantified by laboratories for comparison to field measurements.

Field measurements of soil pH along with potassium, nitrogen, and phosphorous concentrations will be collected on a bimonthly basis. In addition, pore-water will be analyzed for nitrate and nitrite concentrations. These parameters will be measured using field test kits.

3.3 Weather Monitoring

Ambient conditions will be monitored on a daily basis for the site. The United States Geological Survey (USGS) real-time water data station for Whittlesey Creek (USGS 040263205) will be used to monitor area rainfall events. The Whittlesey Creek station is located at latitude 46°35'40", longitude 90°57'47", in SE 1/4 NW 1/4 sec.35, T.48 N., R.5 W., Bayfield County, Hydrologic Unit 04010301, at Cherryville road, 3.7 mi west of the courthouse in Ashland. This unit monitors a 37.6-square-mile drainage area through a water-stage recorder and crest-stage gauge (USGS, 2007). In addition, a local water gauge will be installed at the study location and monitored on a bimonthly (every other week) basis. The Weather Channel's Internet site will be used to monitor area temperatures and precipitation events. In addition, a local thermometer will be installed at the study location and monitored thermometer will be installed at the study location and monitor.

4.0 SITE MANAGEMENT

4.1 Housekeeping

Equipment staging, sample processing, and decontamination will be conducted at a secured location on the grounds of the former Barksdale Works.

All work areas including the sample processing, decontamination pad and staging areas will be cleaned up at the end of each shift.

Equipment will be left in place at the end of a work shift only if work at that location has not been completed and will be continued the following day. Otherwise, all equipment will be stored in the vicinity of the decontamination pad when not in use.

4.1.1 Treatment Study Derived Waste Management

The following types of generated wastes are anticipated as a result of the outlined activities:

- o Personal protective equipment (PPE)
- o Decontamination water
- o Groundwater and/or pore-space water from the field study treatment cells
- o Soils generated during sample preparation for on-site and laboratory analysis

Prior to initiating field activities, the following will be done:

- o Determine potential waste classifications.
- o Determine analyses required to confirm waste classification.
- o Identify disposal options for each identified waste stream.

All wastes generated via field activities will be properly containerized in the appropriate DOT-approved container, marked with the accumulation date and nature of the waste, and retained on site pending completion of the waste classification. All wastes will be appropriately labeled prior to shipment to a DuPont- and EPA-approved treatment, storage, and disposal facility. Any subsequently required reporting will be performed by DuPont.

4.1.2 Contamination Control

When possible, laboratory-cleaned or disposable sampling equipment will be used. In addition, dedicated sampling equipment will be used to minimize the need for field decontamination. However, in the event that field decontamination is necessary, the following procedures will be followed:

- 1. Wash with a perchlorate-free detergent and tap water.
- 2. Rinse with distilled/deionized water.

- 3. Rinse a second time with distilled/deionized water.
- 4. Completely air dry.

PPE specified in the project Health and Safety Plan will be worn during decontamination activities.

4.2 Health and Safety

All activities will be conducted as per the site specific Health and Safety Plan (HASP) and/or all applicable project-specific HASPs and addenda.

5.0 PROJECT CONTACTS

5.1 Project Team and Roles

Brad Nave (DuPont CRG) – Project Director
Mark Vetter (URS Diamond) – Project Manager
Erin Mack (DuPont CRG) – Technical Lead
Marcus Dudley (URS Diamond) – Design Lead
Joe Hughes (GA Tech University) – Technical and Analytical Support
Jim Spain (GA Tech University) – Technical Support
Lisa Schatzman-Rygiel (URS Diamond) – Regional Health & Safety Manager
Betsy Bishop (URS Diamond) – Waste Management Specialist
Steve Larson (DuPont CRG) – Analytical and Statistical Support
Nancy Grosso (DuPont CRG) – Hydrogeology Support
Cary Pooler (URS Diamond) – Technical and Resourcing Support
Sharon Nordstrom (URS Diamond) – Analytical Support
Jon Hammerberg (URS Diamond) – Field and Technical Support
Chris Curran (URS Corporation) – Design Support

6.0 SCHEDULING

Deliverable	Due Date						
Field Study Design & Work Plan	June 1, 2007						
System Construction	May 31, 2007						
System Startup	June 4, 2007						
System Shutdown	September 30, 2007						
Phase I Findings Report	November 22, 2007						

7.0 REFERENCES

- USGS 2007. http://waterdata.usgs.gov/nwis/dv/?site_no=040263205&PARAmeter_cd=00060. Accessed 3/30/2007.
- USGS 2007. http://waterdata.usgs.gov/nwis/dv/?site_no=040263491&PARAmeter_cd=00060. Accessed 3/30/2007.

The Weather Channel 2007.

http://www.weather.com/weather/local/54806?lswe=54806&lwsa=WeatherLocalUndecla red&from=whatwhere. Accessed 3/30/07.

TABLE 1

HISTORICAL AREA PRECIPITATION DATA

Table 1. Historical Area Precipitation Data

Month	1999	2000	2001	2002	2003	2004	2005	2006	2007	Average
January		0.00	0.00	0.00	0.00	0.00	0.00		0.00	0.00
February		0.00	0.00	0.00	0.00	0.00	0.00		0.00	0.00
March		0.00	0.00	0.00	0.00	0.84	0.00		1.51	0.34
April			8.80	1.72	1.59	3.61	1.64	1.88	1.15	3.09
May	2.48		3.68	3.27	6.18	4.73	2.81	4.07		3.86
June	5.46		4.22	1.78	3.01	2.38	6.83	2.00		3.95
July	9.43		4.43	6.83	5.51	2.95	4.42	9.00		5.60
August	3.64		3.15	4.77	1.40	4.14	3.39	1.10		3.42
September	3.56	2.14	1.75	5.00	4.00	2.83	3.99	2.50		3.32
October	1.01	1.42	0.22	0.01	0.10	2.64	12.21	2.41		2.52
November	0.00	2.68	0.00	0.00	0.00	0.00				0.45
December	0.00	0.00	0.00	0.00	0.00	0.00				0.00

Precipitation measured in inches of rainfall.

Stage Average (in)	4.07 < Season lasts June 1 through September 30
Width (in)	120
Length (in)	240
Rainfall (in)	2 < 50% increase in average rainfall
Conversion (cin/gal)	0.0043
Gallons Required	247.68 < gallons of supplied water per month

Data collected from Whittlesey Creek USGS monitoring station #040263205. 2006 data collected from North Fish Creek monitoring station (USGS 040263941). Whittlesey Creek equipment was not functioning. FIGURE 1

LYDOL AND TRIVELENE LINES (AREA PAC)

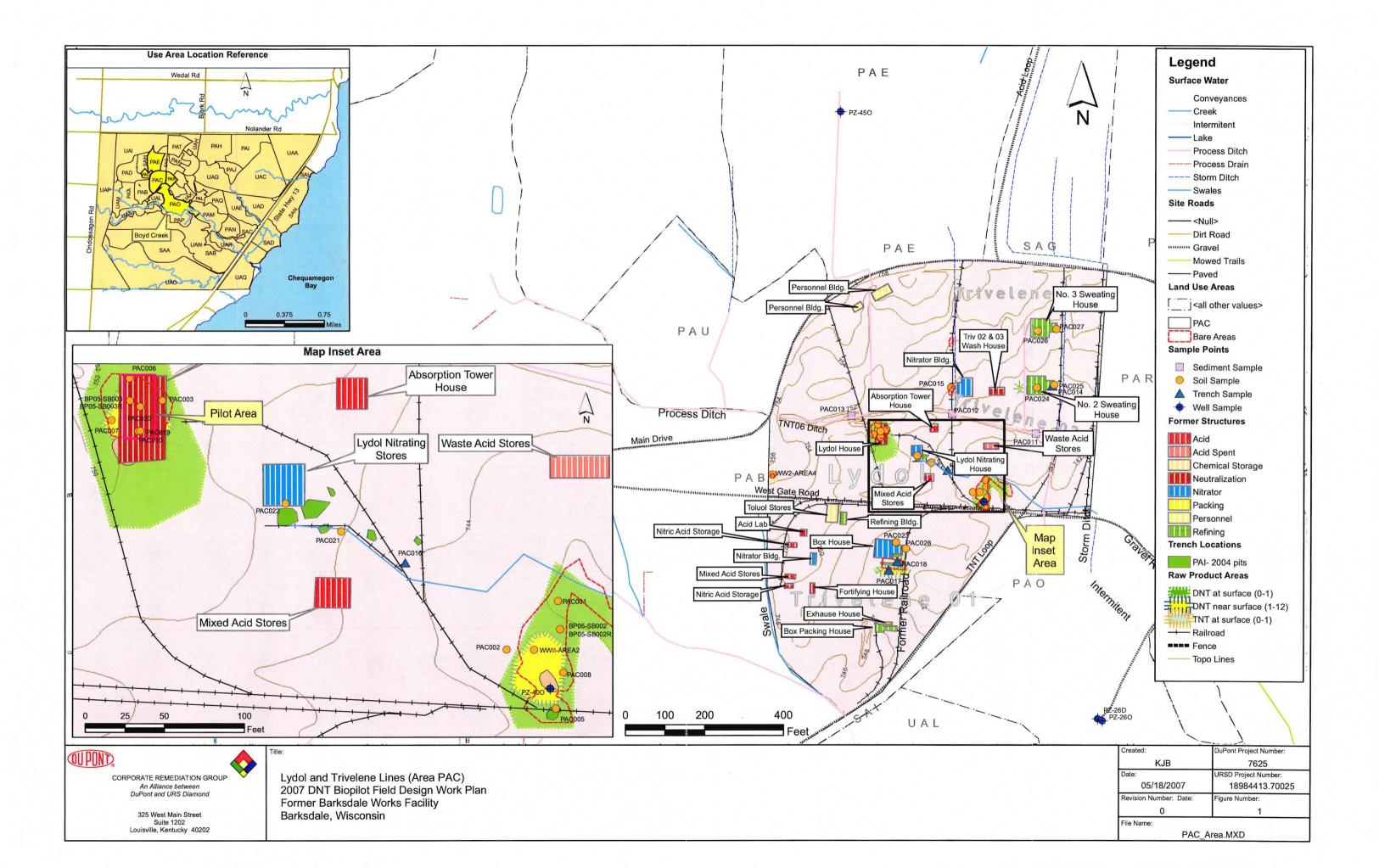


FIGURE 2

HISTORICAL AREA PRECIPITATION DATA

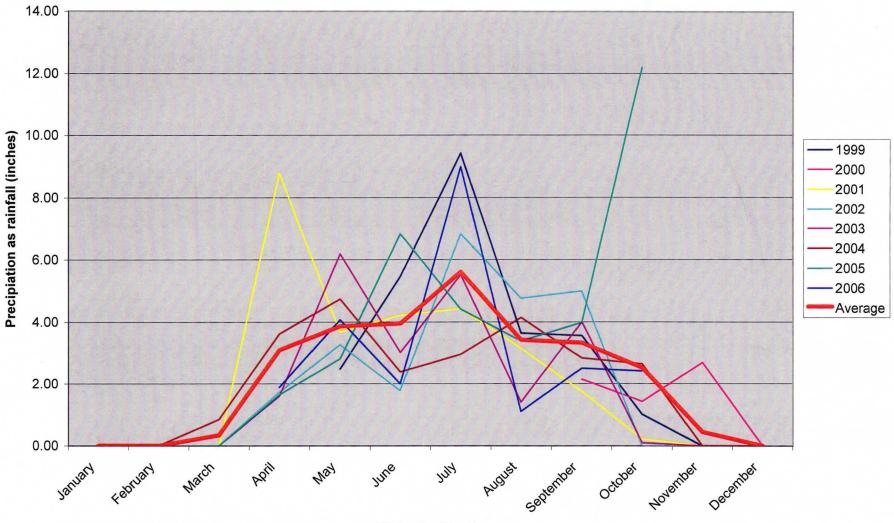


Figure 2. Historical Area Precipitation Data

Calendar Month

FIGURE 3

SOIL BORING LOG PZ-400

State of Wisconsin Department of Natural Resources

SOIL BORING	LOG	INFORMATION
Form 4400-122		Rev. 7-98

Rev.	7-98

Route To:

Watershed/Wastewater Remediation/Redevelopment Waste Management Other 🗌

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I hereby certify that the information on this form is true and correct to the best of my knowledge.

Signature	Firm	E.I. DuPont De Nemours & Company	Tel:
			Fax:

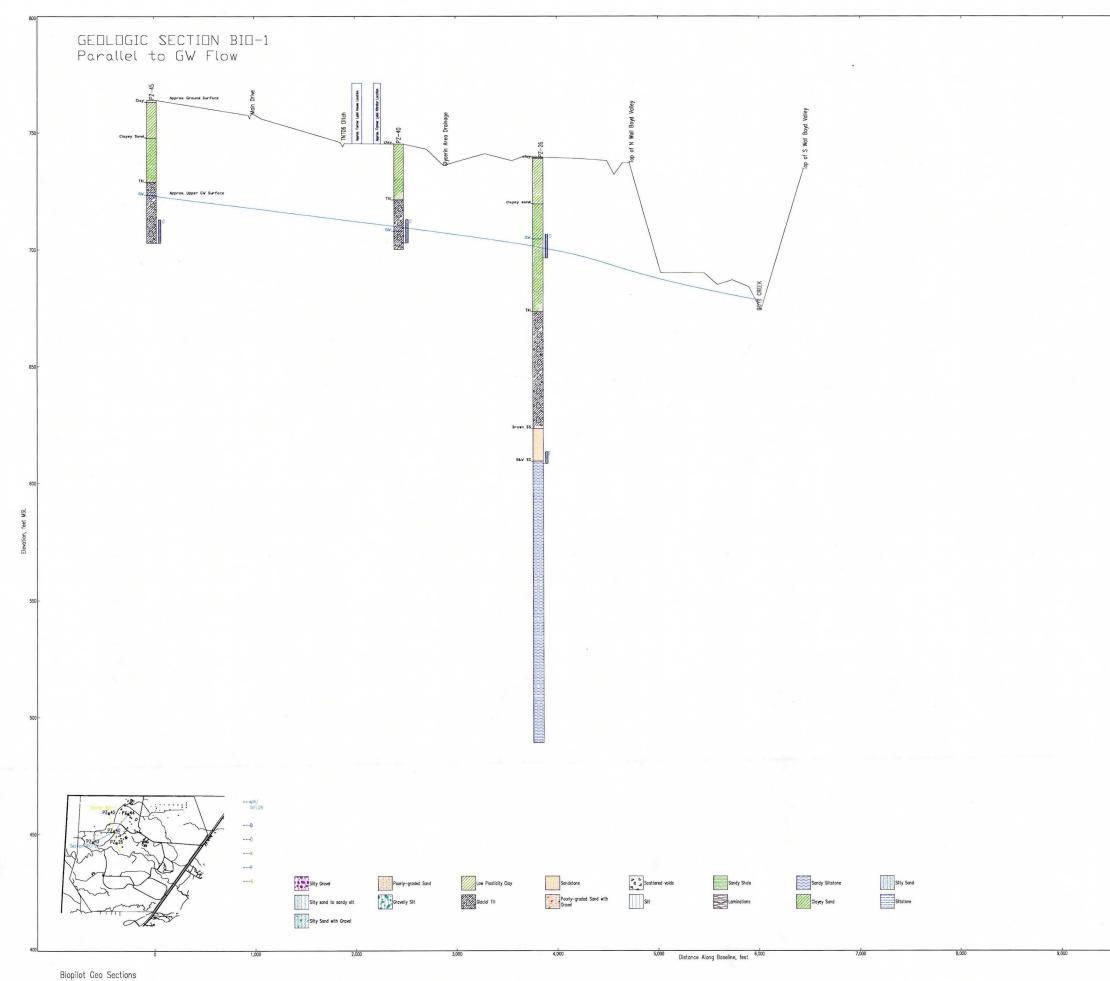
This form is authorized by Chapters 281, 283, 289, 291, 292, 293, 295, and 299, Wis. Stats. Completion of this form is mandatory. Failure to file this form may result in forfeiture of between \$10 and \$25,000, or imprisonment for up to one year, depending on the program and conduct involved. Personally identifiable information on this form is not intended to be be used for any other purpose. NOTE: See instructions for more information, including where the completed form should be sent.

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er 'pe	n Att ered	Cour	In F	And Geologic Origin For	s		E	ý	essiv	at e		ity		ents
Number and Type	Length Att. & Recovered (in)	Blow Counts	Depth In Feet	Each Major Unit	sc	Graphic Log	Well Diagram	Exspray	Compressive Strength	Moisture Content	Liquid	Plasticity Index	P 200	RQD/ Comments
Σ u	REL	B	Lă	Gray-brown silty SAND with some gravel (Till).		53	βã	Ĥ	S S	Σŭ	EE	<u>E</u> E	È.	<u> </u>
			È i	Gray brown sing states while some graves (Thi).										
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FIGURE 4

GEOLOGIC CROSS-SECTION PARALLEL TO GW FLOW

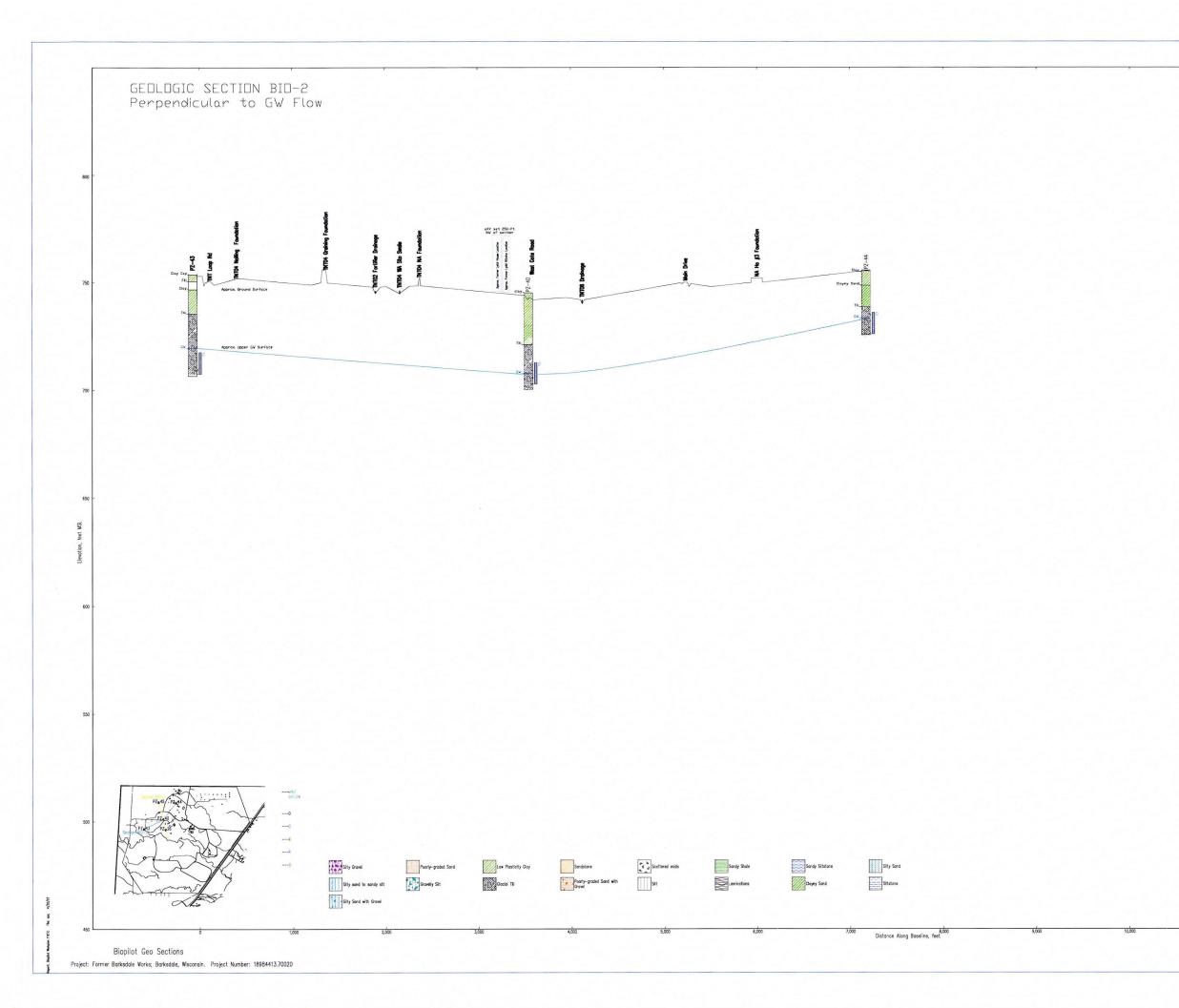


Project: Former Barksdale Works; Barksdale, Wisconsin. Project Number: 18984413.70020

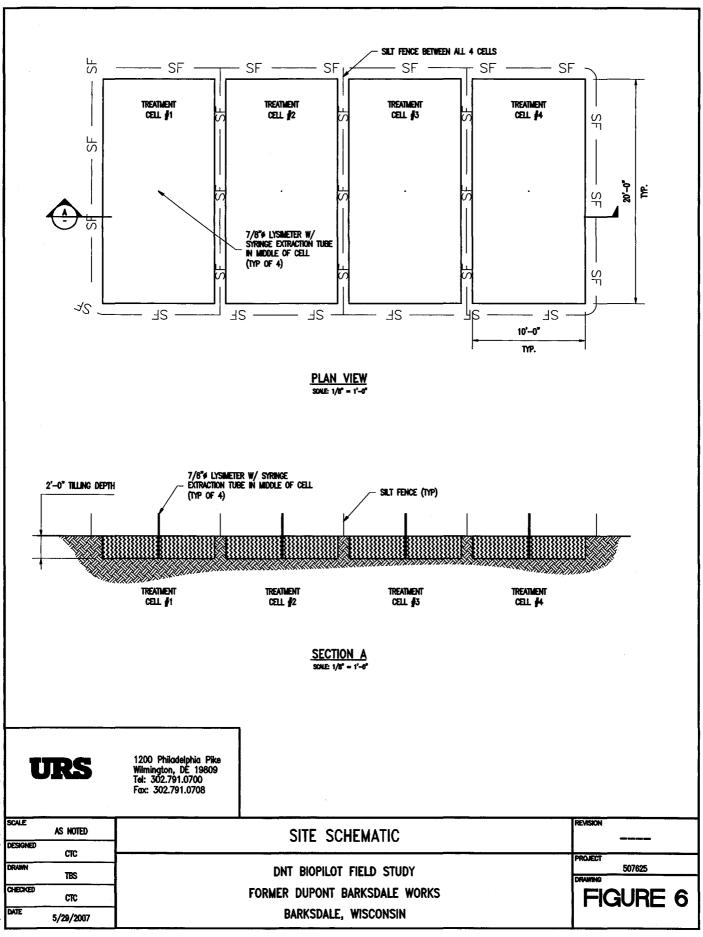
Section Begin East: 1,727,849 Section Begin North: 533,723 Section End East: 1,728,707 Section End North: 531,278

FIGURE 5

GEOLOGIC CROSS-SECTION PERPENDICULAR TO GW FLOW



Section Begin East: 1,726,861 Section Begin North: 531,935 Section End East: 1,729,116 Section End North: 534,016 FIGURE 6 SITE SCHEMATIC



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