

Technical Assistance, Environmental Liability Clarification or Post-Closure Modification Request

Form 4400-237 (R 12/18)

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Notice: Use this form to request a **written response (on agency letterhead)** from the Department of Natural Resources (DNR) regarding technical assistance, a post-closure change to a site, a specialized agreement or liability clarification for Property with known or suspected environmental contamination. A fee will be required as is authorized by s. 292.55, Wis. Stats., and NR 749, Wis. Adm. Code., unless noted in the instructions below. Personal information collected will be used for administrative purposes and may be provided to requesters to the extent required by Wisconsin's Open Records law [ss. 19.31 - 19.39, Wis. Stats.].

Definitions

"Property" refers to the subject Property that is perceived to have been or has been impacted by the discharge of hazardous substances.

"Liability Clarification" refers to a written determination by the Department provided in response to a request made on this form. The response clarifies whether a person is or may become liable for the environmental contamination of a Property, as provided in s. 292.55, Wis. Stats.

"Technical Assistance" refers to the Department's assistance or comments on the planning and implementation of an environmental investigation or environmental cleanup on a Property in response to a request made on this form as provided in s. 292.55, Wis. Stats.

"Post-closure modification" refers to changes to Property boundaries and/or continuing obligations for Properties or sites that received closure letters for which continuing obligations have been applied or where contamination remains. Many, but not all, of these sites are included on the GIS Registry layer of RR Sites Map to provide public notice of residual contamination and continuing obligations.

Select the Correct Form

This form should be used to request the following from the DNR:

- Technical Assistance
- Liability Clarification
- Post-Closure Modifications
- Specialized Agreements (tax cancellation, negotiated agreements, etc.)

Do not use this form if one of the following applies:

- Request for an **off-site liability exemption or clarification** for Property that has been or is perceived to be contaminated by one or more hazardous substances that originated on another Property containing the source of the contamination. Use DNR's Off-Site Liability Exemption and Liability Clarification Application Form 4400-201.
- Submittal of an Environmental Assessment for the **Lender Liability Exemption**, s 292.21, Wis. Stats., **if no response or review by DNR is requested**. Use the Lender Liability Exemption Environmental Assessment Tracking Form 4400-196.
- Request for an **exemption to develop on a historic fill site** or licensed landfill. Use DNR's Form 4400-226 or 4400-226A.
- **Request for closure** for Property where the investigation and cleanup actions are completed. Use DNR's Case Closure - GIS Registry Form 4400-202.

All forms, publications and additional information are available on the internet at: dnr.wi.gov/topic/Brownfields/Pubs.html.

Instructions

1. Complete sections 1, 2, 6 and 7 for all requests. Be sure to provide adequate and complete information.
2. Select the type of assistance requested: Section 3 for technical assistance or post-closure modifications, Section 4 for a written determination or clarification of environmental liabilities; or Section 5 for a specialized agreement.
3. Include the fee payment that is listed in Section 3, 4, or 5, unless you are a "Voluntary Party" enrolled in the Voluntary Party Liability Exemption Program **and** the questions in Section 2 direct otherwise. Information on to whom and where to send the fee is found in Section 8 of this form.
4. Send the completed request, supporting materials and the fee to the appropriate DNR regional office where the Property is located. See the map on the last page of this form. A paper copy of the signed form and all reports and supporting materials shall be sent with an electronic copy of the form and supporting materials on a compact disk. For electronic document submittal requirements see: <http://dnr.wi.gov/files/PDF/pubs/rr/RR690.pdf>

The time required for DNR's determination varies depending on the complexity of the site, and the clarity and completeness of the request and supporting documentation.

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Section 1. Contact and Recipient Information

Requester Information

This is the person requesting technical assistance or a post-closure modification review, that his or her liability be clarified or a specialized agreement and is identified as the requester in Section 7. DNR will address its response letter to this person.

Last Name	First	MI	Organization/ Business Name		
			Superior Refining Company LLC		
Mailing Address			City	State	ZIP Code
2407 Stinson Avenue			Superior	WI	54880
Phone # (include area code)	Fax # (include area code)	Email			
(715) 398-8434		matthew.turner@huskyenergy.com			

The requester listed above: (select all that apply)

- | | |
|---|--|
| <input checked="" type="checkbox"/> Is currently the owner | <input type="checkbox"/> Is considering selling the Property |
| <input type="checkbox"/> Is renting or leasing the Property | <input type="checkbox"/> Is considering acquiring the Property |
| <input type="checkbox"/> Is a lender with a mortgagee interest in the Property | |
| <input type="checkbox"/> Other. Explain the status of the Property with respect to the applicant: | |

Contact Information (to be contacted with questions about this request)

Select if same as requester

Contact Last Name	First	MI	Organization/ Business Name		
Turner	Matthew		Superior Refining Company LLC		
Mailing Address			City	State	ZIP Code
2407 Stinson Avenue			Superior	WI	54880
Phone # (include area code)	Fax # (include area code)	Email			
(715) 398-8434		matthew.turner@huskyenergy.com			

Environmental Consultant (if applicable)

Contact Last Name	First	MI	Organization/ Business Name		
Carney	Lynette		Barr Engineering Company		
Mailing Address			City	State	ZIP Code
325 S Lake Avenue, Ste 700			Duluth	MN	55803
Phone # (include area code)	Fax # (include area code)	Email			
(218) 529-7141		lcarney@barr.com			

Section 2. Property Information

Property Name			FID No. (if known)	
Superior Refining Company LLC			816009590	
BRRTS No. (if known)		Parcel Identification Number		
02-16-581317				
Street Address		City	State	ZIP Code
2407 Stinson Avenue		Superior	WI	54880
County	Municipality where the Property is located	Property is composed of:		Property Size Acres
Douglas	<input checked="" type="radio"/> City <input type="radio"/> Town <input type="radio"/> Village of	<input type="radio"/> Single tax parcel <input checked="" type="radio"/> Multiple tax parcels		250

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1. Is a response needed by a specific date? (e.g., Property closing date) Note: Most requests are completed within 60 days. Please plan accordingly.

- No Yes

Date requested by: _____

Reason: _____

2. Is the "Requester" enrolled as a Voluntary Party in the Voluntary Party Liability Exemption (VPLE) program?

- No. **Include the fee that is required for your request in Section 3, 4 or 5.**
 Yes. **Do not include a separate fee.** This request will be billed separately through the VPLE Program.

Fill out the information in Section 3, 4 or 5 which corresponds with the type of request:

Section 3. Technical Assistance or Post-Closure Modifications;

Section 4. Liability Clarification; or Section 5. Specialized Agreement.

Section 3. Request for Technical Assistance or Post-Closure Modification

Select the type of technical assistance requested: [Numbers in brackets are for WI DNR Use]

- No Further Action Letter (NFA) (Immediate Actions) - NR 708.09, [183] - **Include a fee of \$350.** Use for a written response to an immediate action after a discharge of a hazardous substance occurs. Generally, these are for a one-time spill event.
- Review of Site Investigation Work Plan - NR 716.09, [135] - **Include a fee of \$700.**
- Review of Site Investigation Report - NR 716.15, [137] - **Include a fee of \$1050.**
- Approval of a Site-Specific Soil Cleanup Standard - NR 720.10 or 12, [67] - **Include a fee of \$1050.**
- Review of a Remedial Action Options Report - NR 722.13, [143] - **Include a fee of \$1050.**
- Review of a Remedial Action Design Report - NR 724.09, [148] - **Include a fee of \$1050.**
- Review of a Remedial Action Documentation Report - NR 724.15, [152] - **Include a fee of \$350**
- Review of a Long-term Monitoring Plan - NR 724.17, [25] - **Include a fee of \$425.**
- Review of an Operation and Maintenance Plan - NR 724.13, [192] - **Include a fee of \$425.**

Other Technical Assistance - s. 292.55, Wis. Stats. [97] (For request to build on an abandoned landfill use Form 4400-226)

- Schedule a Technical Assistance Meeting - **Include a fee of \$700.**
- Hazardous Waste Determination - **Include a fee of \$700.**
- Other Technical Assistance - **Include a fee of \$700.** Explain your request in an attachment.

Post-Closure Modifications - NR 727, [181]

- Post-Closure Modifications: Modification to Property boundaries and/or continuing obligations of a closed site or Property; sites may be on the GIS Registry. This also includes removal of a site or Property from the GIS Registry. **Include a fee of \$1050, and:**
- Include a fee of \$300 for sites with residual soil contamination; and
- Include a fee of \$350 for sites with residual groundwater contamination, monitoring wells or for vapor intrusion continuing obligations.

Attach a description of the changes you are proposing, and documentation as to why the changes are needed (if the change to a Property, site or continuing obligation will result in revised maps, maintenance plans or photographs, those documents may be submitted later in the approval process, on a case-by-case basis).

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Skip Sections 4 and 5 if the technical assistance you are requesting is listed above and complete Sections 6 and 7 of this form.

Section 4. Request for Liability Clarification

Select the type of liability clarification requested. Use the available space given or attach information, explanations, or specific questions that you need answered in DNR's reply. Complete Sections 6 and 7 of this form. **[Numbers in brackets are for DNR Use]**

"Lender" liability exemption clarification - s. 292.21, Wis. Stats. [686]

❖ **Include a fee of \$700.**

Provide the following documentation:

- (1) ownership status of the real Property, and/or the personal Property and fixtures;
- (2) an environmental assessment, in accordance with s. 292.21, Wis. Stats.;
- (3) the date the environmental assessment was conducted by the lender;
- (4) the date of the Property acquisition; for foreclosure actions, include a copy of the signed and dated court order confirming the sheriff's sale.
- (5) documentation showing how the Property was acquired and the steps followed under the appropriate state statutes.
- (6) a copy of the Property deed with the correct legal description; and,
- (7) the Lender Liability Exemption Environmental Assessment Tracking Form (Form 4400-196).
- (8) If no sampling was done, please provide reasoning as to why it was **not** conducted. Include this either in the accompanying environmental assessment or as an attachment to this form, and cite language in s. 292.21(1)(c)2.,h.-i., Wis. Stats.:
 - h. The collection and analysis of representative samples of soil or other materials in the ground that are suspected of being contaminated based on observations made during a visual inspection of the real Property or based on aerial photographs, or other information available to the lender, including stained or discolored soil or other materials in the ground and including soil or materials in the ground in areas with dead or distressed vegetation. The collection and analysis shall identify contaminants in the soil or other materials in the ground and shall quantify concentrations.
 - i. The collection and analysis of representative samples of unknown wastes or potentially hazardous substances found on the real Property and the determination of concentrations of hazardous waste and hazardous substances found in tanks, drums or other containers or in piles or lagoons on the real Property.

"Representative" liability exemption clarification (e.g. trustees, receivers, etc.) - s. 292.21, Wis. Stats. [686]

❖ **Include a fee of \$700.**

Provide the following documentation:

- (1) ownership status of the Property;
- (2) the date of Property acquisition by the representative;
- (3) the means by which the Property was acquired;
- (4) documentation that the representative has no beneficial interest in any entity that owns, possesses, or controls the Property;
- (5) documentation that the representative has not caused any discharge of a hazardous substance on the Property; and
- (6) a copy of the Property deed with the correct legal description.

Clarification of local governmental unit (LGU) liability exemption at sites with: (select all that apply)

- hazardous substances spills - s. 292.11(9)(e), Wis. Stats. [649];
- Perceived environmental contamination - [649];
- hazardous waste - s. 292.24 (2), Wis. Stats. [649]; and/or
- solid waste - s. 292.23 (2), Wis. Stats. [649].

❖ **Include a fee of \$700, a summary of the environmental liability clarification being requested, and the following:**

- (1) clear supporting documentation showing the acquisition method used, and the steps followed under the appropriate state statute(s).
- (2) current and proposed ownership status of the Property;
- (3) date and means by which the Property was acquired by the LGU, where applicable;
- (4) a map and the ¼, ¼ section location of the Property;
- (5) summary of current uses of the Property;
- (6) intended or potential use(s) of the Property;
- (7) descriptions of other investigations that have taken place on the Property; and
- (8) (for solid waste clarifications) a summary of the license history of the facility.

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Section 4. Request for Liability Clarification (cont.)

Lease liability clarification - s. 292.55, Wis. Stats. [646]

❖ **Include a fee of \$700 for a single Property, or \$1400 for multiple Properties and the information listed below:**

- (1) a copy of the proposed lease;
- (2) the name of the current owner of the Property and the person who will lease the Property;
- (3) a description of the lease holder's association with any persons who have possession, control, or caused a discharge of a hazardous substance on the Property;
- (4) map(s) showing the Property location and any suspected or known sources of contamination detected on the Property;
- (5) a description of the intended use of the Property by the lease holder, with reference to the maps to indicate which areas will be used. Explain how the use will not interfere with any future investigation or cleanup at the Property; and
- (6) all reports or investigations (e.g. Phase I and Phase II Environmental Assessments and/or Site Investigation Reports conducted under s. NR 716, Wis. Adm. Code) that identify areas of the Property where a discharge has occurred.

General or other environmental liability clarification - s. 292.55, Wis. Stats. [682] - Explain your request below.

❖ **Include a fee of \$700 and an adequate summary of relevant environmental work to date.**

No Action Required (NAR) - NR 716.05, [682]

❖ **Include a fee of \$700.**

Use where an environmental discharge has or has not occurred, and applicant wants a DNR determination that no further assessment or clean-up work is required. Usually this is requested after a Phase I and Phase II environmental assessment has been conducted; the assessment reports should be submitted with this form. This is not a closure letter.

Clarify the liability associated with a "closed" Property - s. 292.55, Wis. Stats. [682]

❖ **Include a fee of \$700.**

- Include a copy of any closure documents if a state agency other than DNR approved the closure.

Use this space or attach additional sheets to provide necessary information, explanations or specific questions to be answered by the DNR.

Section 5. Request for a Specialized Agreement

Select the type of agreement needed. Include the appropriate draft agreements and supporting materials. Complete Sections 6 and 7 of this form. More information and model draft agreements are available at: dnr.wi.gov/topic/Brownfields/lgu.html#tabx4.

Tax cancellation agreement - s. 75.105(2)(d), Wis. Stats. [654]

❖ **Include a fee of \$700, and the information listed below:**

- (1) Phase I and II Environmental Site Assessment Reports,
- (2) a copy of the Property deed with the correct legal description.

Agreement for assignment of tax foreclosure judgement - s.75.106, Wis. Stats. [666]

❖ **Include a fee of \$700, and the information listed below:**

- (1) Phase I and II Environmental Site Assessment Reports,
- (2) a copy of the Property deed with the correct legal description.

Negotiated agreement - Enforceable contract for non-emergency remediation - s. 292.11(7)(d) and (e), Wis. Stats. [630]

❖ **Include a fee of \$1400, and the information listed below:**

- (1) a draft schedule for remediation; and,
- (2) the name, mailing address, phone and email for each party to the agreement.

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Section 6. Other Information Submitted

Identify all materials that are included with this request.

Send both a paper copy of the signed form and all reports and supporting materials, and an electronic copy of the form and all reports, including Environmental Site Assessment Reports, and supporting materials on a compact disk.

Include one copy of any document from any state agency files that you want the Department to review as part of this request. The person submitting this request is responsible for contacting other state agencies to obtain appropriate reports or information.

- Phase I Environmental Site Assessment Report - Date: _____
- Phase II Environmental Site Assessment Report - Date: _____
- Legal Description of Property (required for all liability requests and specialized agreements)
- Map of the Property (required for all liability requests and specialized agreements)

Analytical results of the following sampled media: Select all that apply and include date of collection.

- Groundwater
- Soil
- Sediment
- Other medium - Describe: _____

Date of Collection: _____

- A copy of the closure letter and submittal materials
- Draft tax cancellation agreement
- Draft agreement for assignment of tax foreclosure judgment
- Other report(s) or information - Describe: _____

For Property with newly identified discharges of hazardous substances only: Has a notification of a discharge of a hazardous substance been sent to the DNR as required by s. NR 706.05(1)(b), Wis. Adm. Code?

- Yes - Date (if known): _____
- No

Note: The Notification for Hazardous Substance Discharge (non-emergency) form is available at:
dnr.wi.gov/files/PDF/forms/4400/4400-225.pdf.

Section 7. Certification by the Person who completed this form

- I am the person submitting this request (requester)
- I prepared this request for: _____
Requester Name

I certify that I am familiar with the information submitted on this request, and that the information on and included with this request is true, accurate and complete to the best of my knowledge. I also certify I have the legal authority and the applicant's permission to make this request.


Signature

6/14/19
Date Signed

Environmental Technologist
Title

(715) 398-8434
Telephone Number (include area code)

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Section 8. DNR Contacts and Addresses for Request Submittals

Send or deliver one paper copy and one electronic copy on a compact disk of the completed request, supporting materials, and fee to the region where the property is located to the address below. Contact a DNR regional brownfields specialist with any questions about this form or a specific situation involving a contaminated property. For electronic document submittal requirements see: <http://dnr.wi.gov/files/PDF/pubs/rr/RR690.pdf>.

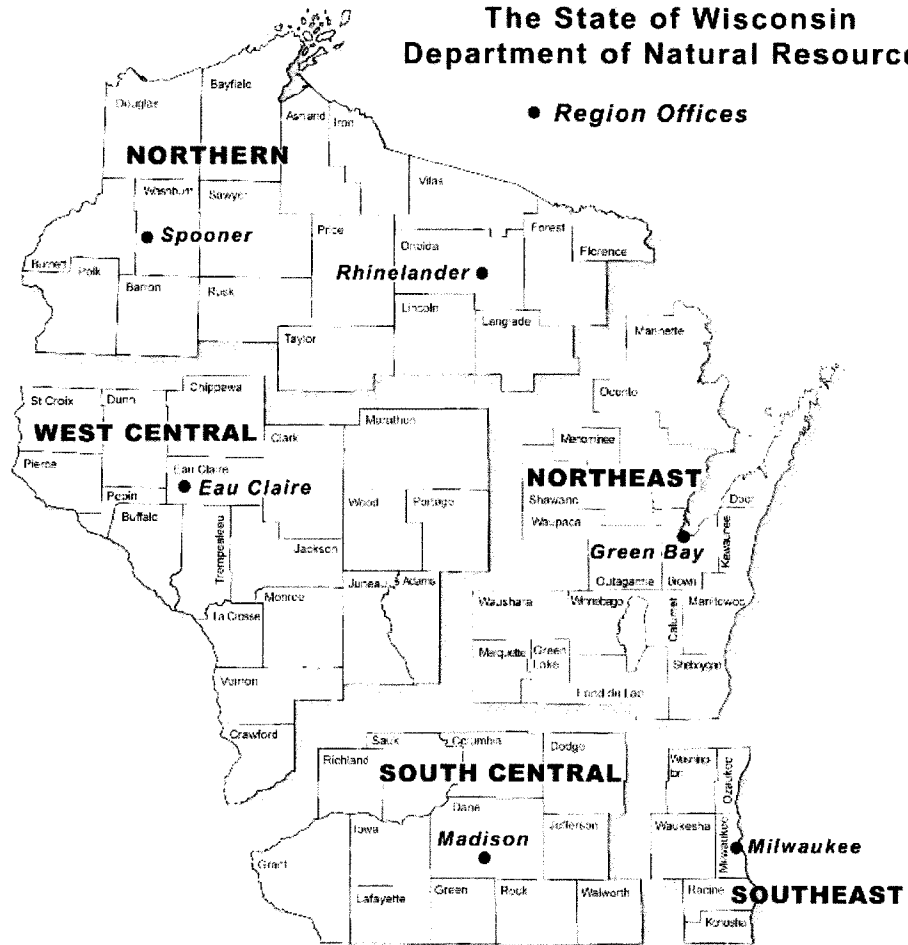
DNR NORTHERN REGION
Attn: RR Program Assistant
Department of Natural Resources
223 E Steinfest Rd Antigo, WI 54409

DNR NORTHEAST REGION
Attn: RR Program Assistant
Department of Natural Resources
2984 Shawano Avenue
Green Bay WI 54313

DNR SOUTH CENTRAL REGION
Attn: RR Program Assistant
Department of Natural Resources
3911 Fish Hatchery Road
Fitchburg WI 53711

DNR SOUTHEAST REGION
Attn: RR Program Assistant
Department of Natural Resources
2300 North Martin Luther King Drive
Milwaukee WI 53212

DNR WEST CENTRAL REGION
Attn: RR Program Assistant
Department of Natural Resources
1300 Clairemont Ave.
Eau Claire WI 54702



Note: These are the Remediation and Redevelopment Program's designated regions. Other DNR program regional boundaries may be different.

DNR Use Only			
Date Received	Date Assigned	BRRTS Activity Code	BRRTS No. (if used)
DNR Reviewer		Comments	
Fee Enclosed? <input type="radio"/> Yes <input type="radio"/> No	Fee Amount \$	Date Additional Information Requested	Date Requested for DNR Response Letter
Date Approved	Final Determination		



Site Investigation Work Plan

Superior Refinery April 26, 2018 Fire
BRRTs Number: 02-16-581317

Prepared for
Superior Refining Company LLC



June 2019

Site Investigation Work Plan

Superior Refinery April 26, 2018 Fire
BRRTs Number: 02-16-581317

Prepared for
Superior Refinery Company LLC



June 2019

Site Investigation Work Plan

June 2019

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1.0 Work Plan Objectives

This purpose of this work plan is to present the initial site investigation activities planned by Superior Refining Company LLC (SRC) in response to the April 26, 2018 Superior Refinery explosion and release of asphalt, Therminol® and # 6 fuel oil. The Wisconsin Department of Natural Resources (WDNR) requested this work plan in their letter dated September 18, 2018, following the transfer of the site from a WDNR spill program under NR 708 to an Environmental Repair Program (ERP) site under NR 716. Initial and interim actions in response to this release were immediately initiated by SRC and are ongoing. This work plan has been developed to review and investigate potential impacts that remain after the release under the requirements of NR 716 and, in particular, the site investigation scoping requirements in NR 716.07.

Since the Superior Refinery (Site) has been in operation for decades and has historical and/or unrelated ongoing monitoring, a phased investigation strategy is proposed that will allow for collection and evaluation of investigation data in the context of the larger Site setting. As a result, the initial phase of the SRC proposed site investigation work plan (SIWP) includes the following activities:

- Assess and characterize the condition of soil beneath pervious surfaces within the affected hydrocarbon release area(s);
- Determine the need for additional investigation, interim action and/or remedial action; and
- Collect information necessary to select additional interim and/or remedial action.

2.0 Site Description

Figure 1 provides a location map showing the Superior Refinery and the surrounding area using the USGS 7.5-minute topographic map (NR 716.09(2)(c)). Figure 2 provides an aerial image of the facility and property boundaries in relation to the surrounding features along with area private water supply wells located within 1,200 feet of the facility boundary (NR 716.07 (7)). Figure 3A and Figure 3B provide facility features and refining operational process area details (NR 716.09 (2) (c)).

Site Information: BRRTs Number: 02-16-581317
Facility Identification Numbers: 816009590
Superior Refinery Company LLC (SRC)
2407 Stinson Avenue
Superior, Wisconsin
Douglas County, Wisconsin
NW ¼, NW ¼ of Section 36, T49N, R14W
Latitude / Longitude: 46.690927 / 92.07179 (Facility Center)
WTM91 Coordinates: X: 361511, Y: 692726 (Facility Center)

Responsible Party: Superior Refining Company LLC (SRC)
Attn: Matt Turner, Environmental Technologist
2407 Stinson Avenue
Superior, WI 54880
Phone: (715) 398-8434
Email: matthew.turner@huskyenergy.com

Environmental Consultant: Barr Engineering Co.
Attn: Lynette Carney, Project Manager
325 South Lake Avenue, Suite 700
Duluth, MN 55802
Phone: (218) 529-7141
Email: lcarney@barr.com

3.0 Physical Setting

The information provided in this section outlines the physiographical and geological setting of the Site necessary to choose sampling methods and locations in accordance with the requirements of NR 716.09 (2) e.

3.1 Topography and Hydrology

The topography at the refinery slopes gently to the east. Surface elevations range from approximately 650 to 660 feet above mean sea level (MSL). The closest natural surface water body is Newton Creek, whose headwaters are located at the Newton Creek Impoundment shown on Figure 3A. The creek flows about 1.5 miles to Hog Island Inlet, which connects to Superior Bay. Stormwater retention and firewater ponds, along with two artificial wetlands for wastewater treatment plant discharge polishing, are located just northwest of the Newton Creek headwaters, near the intersection of Stinson Avenue and Bardon Avenue (Figure 3A).

Other than the process areas which have concrete cover, most of the refinery property is unpaved. Depending on time of year and topography, the depth to groundwater in the network monitoring wells ranges from less than 1.0 to greater than 5.9 feet below ground surface (bgs). The direction of shallow groundwater flow below the refinery is to the east toward Superior Bay.

3.1.1 Geology

Surficial geology in the region consists of Pleistocene-age glacial deposits of the Miller Creek Formation (Clayton, 1984). The Miller Creek Formation is composed of clayey glacial till, wave modified till, and glacial-lacustrine deposits. The glacial-lacustrine deposits are the uppermost surficial deposits in the region and were deposited in a water-logged state during high stages of Glacial Lake Duluth with subsequent isolated erosion and proglacial stream deposition associated with what is now incised Nemadji River channel (Clayton, 1984) located approximately $\frac{3}{4}$ -mile southeast of the facility.

The Miller Creek Formation overlies the Copper Falls Formation which is also a glacial till that is Pleistocene in age. The Copper Falls Formation contains sandy glacial till interbedded with sand and gravel deposited by melt-water streams (Clayton, 1984).

The regional bedrock geology consists of sandstone of the Precambrian-age Bayfield Formation. Depth to bedrock in the refinery area is greater than 150 feet (Young and Skinner, 1974).

Soil boring data previously collected at the Site indicates that a homogenous layer of red-brown lean to fat clay till is present across the refinery Site which extends to depths of at least 100 feet bgs (Gannett Fleming, 2014). No sand or silt lenses were reported to have been encountered within this clay layer. Desiccation and/or freeze/thaw fractures were describe to be commonly encountered in the approximately upper 7 feet of the clay till (Gannett Fleming, 2014).

Additional information regarding the regional geology was identified from nearby private water supply well construction logs obtained from the Wisconsin Geological and Natural History Survey (Appendix A). Locatable wells are shown on Figure 2. These area water supply wells ranged in depth from 108 feet bgs to 275 feet bgs. Logs indicate that red clay is present from ground surface to depths ranging from 85 to 170 feet. The wells in areas with a thinner clay layer are located near the Nemadji River at a lower surface elevation than the other wells. A hardpan layer was listed on all of the logs as being present below the clay. The thickness of the hardpan layer ranged from 5 to 120 feet. Several of the wells were drilled into the underlying sandstone formation with depths to bedrock ranging from 161 to greater than 260 feet bgs.

3.1.2 Hydrogeology

Data from previous groundwater monitoring reports associated with the facility indicate that the general groundwater flow direction at the refinery is to the east-southeast, with a horizontal gradient of approximately 0.003 (Gannett Fleming, 2018).

The median hydraulic conductivity of the clay is reported to be 2.4×10^{-7} centimeters per second (cm/sec), and the estimated groundwater velocity at the Site was reported at approximately 0.4 centimeters per year (cm/yr) (Gannett Fleming, 2014). The clay is almost entirely saturated, with the water table within 3 feet bgs. Because of the low permeability of the native clay, most wells installed at the refinery take several weeks to months before water levels stabilize, providing further evidence of the extremely low hydraulic conductivity of the clay till.

3.1.3 Potential Exposure Pathways

The potential exposure pathways for petroleum products in soil and groundwater are determined by the properties of the petroleum product and the characteristics of the geologic media. Because of the relatively impermeable surficial clay at the refinery, releases tend to migrate more horizontally along the ground surface. As outlined in the *Final Site Investigation/Remedial Action Plan (SI/RAP)* for this facility (Gannett Fleming, 2014), some vertical migration of petroleum is possible in the surficial air-filled desiccation fractures within the clay. However, once the contamination reaches the saturated conditions at the shallow groundwater table, it is not expected to penetrate the unfractured clay because of the high entry pressure (Bradbury et al., 1985). As a result, lateral subsurface migration of petroleum contamination is not considered a significant transport pathway. Therefore, the potential for petroleum compounds from this release to migrate beyond the estimated affected area is relatively low (Figure 5A).

In the event the petroleum hydrocarbon release enters a dissolved-phase in groundwater, transport will be with the flow of groundwater (i.e. the hydraulic gradient). As stated above, groundwater velocities in the clay are on the order of 0.013 feet per year (ft/yr). With the closest groundwater receptor Newton Creek located more than 1,000 feet down-gradient from the closest up-gradient boundary of the affected area, it would take more than 76,000 years for groundwater from the affected area to reach Newton Creek, assuming advective transport at groundwater velocity with no retardation or degradation.

Human exposure through direct or indirect contact with soil, groundwater, or vapor is also low. The low permeability of the clay significantly impedes the potential vapor migration of contaminants in the unsaturated zone. Additionally, the refinery has internal controls in place that further minimize potential direct contact exposure to impacted soil and groundwater. The refinery is surrounded by a 24-hour per day, 7-day per week security system that includes a barbed-wire chain-linked fence, video surveillance system, and security guards. These safeguards prevent the general public from accessing any refinery area. The refinery also has an internal safe work permit program that requires any employers or contractors to obtain a work permit prior to working in any refinery area. This permit system also includes a separate work instruction for soil excavation projects and defines the minimum project requirements, safe work practices, and control measures that are to be utilized for all trenching and excavation operations at the refinery.

4.0 Facility History

The information provided in this section addresses the requirements of NR 716.07 by providing a summary of the facility history and previous hazardous substance discharges, description of affected media, potential or known impacts to receptors and interim and immediate actions taken in response to this release.

4.1 Operational History

The Superior Refinery was constructed in 1951 by the Lake Superior Refinery Company and was sold to Murphy Oil USA, Inc. (Murphy) in 1958. Calumet Specialty Products Partners (Calumet) purchased the refinery from Murphy in October 2011. Effective November 8, 2017, Husky Superior Refining Holding Corp. (Husky Superior) purchased the refinery from Calumet and changed its legal name to Superior Refining Company LLC (SRC). The refinery temporarily ceased operation following an explosion and fire on April 26, 2018. SRC is currently focused on efforts to cleanup and rebuild the refinery. The facility is tentatively scheduled to be partially operational again in 2020 or early 2021 and reaching full operation in the second half of 2021.

The Superior Refinery is primarily a transportation fuels and asphalt production facility with an existing capacity of 50,000 barrels per day (bpd). Products produced at the refinery include liquefied petroleum gas (LPGs) (propane/butane/etc.), gasoline (sub-grade/mid-grade/premium/etc.), distillates (kerosene/diesel fuels/etc.), heavy oils (#6 fuel oil/slurry oil/etc.) and asphalt (multiple grades).

The refinery-related activities occupy an area of approximately 250 acres. The total land owned by SRC, including the refinery and adjacent property, is approximately 700 acres. The facility and area features are shown on Figure 2 and Figure 3A while the refining area detail is shown on Figure 3B. The area surrounding the Superior Refinery consists of primarily open and undeveloped land to the west, north and east. Further to the west is a rail yard and the Richard Bong Airport and further to the east are residential and commercial properties. Enbridge Energy's Superior Terminal is located to the south. SRC also owns three aboveground storage tanks (ASTs) located on approximately 17 acres south of the main refinery (Figure 2) adjacent to the Enbridge Terminal property.

4.2 History of Previous Releases

As required by NR 716.07 (3) this section provides a summary of the previous historical releases at the facility. Reportable releases of petroleum products to pervious surfaces at the refinery have been reported to the WDNR. These sites have either received closure from WDNR or require ongoing monitoring and/or cleanup. More details regarding individual historical release sites can be found in previously submitted correspondence to the WDNR (Gannett Fleming, 2014).

Past interim actions, site investigations and closures have followed the requirements of NR 708 and NR 716. To increase efficiency and streamline reporting for refinery release sites, a WDNR approved facility-wide SI/RAP (Gannett Fleming, 2014) was developed and became effective April 4, 2018. This SI/WP was also used as the basis for the Negotiated Agreement between SRC and the WDNR. In conjunction with the

Negotiated Agreement, a network of 23 wells and 8 piezometers for monitoring overall groundwater quality was established (Figure 4). Twice a year, starting in 2015, all wells and piezometers in the network are gauged (to check for non-aqueous phase liquids (NAPL), track seasonal changes in water levels, and prepare groundwater contour maps), and the perimeter wells and piezometers are purged and sampled for petroleum volatile organic compounds (PVOCs) plus naphthalene. As a result of the SI/RAP and associated Negotiated Agreement, the WDNR created a single, new refinery-wide ERP site designation (BRRTs Number 02-16-559511) that covers most releases that occur within the facility boundary.

According to information summarized in the SI/RAP (Gannett Fleming, 2014), the soil vapor exposure pathway has not been evaluated at any of the previously closed or currently active petroleum release locations. This decision was approved by the WDNR since these releases are located within, or adjacent to, the refinery’s tank farm and the only structures in these release areas are the ASTs. No structures designed for human occupancy are present within 30 feet of known areas of petroleum-contaminated soil or groundwater (WDNR, 2018) (Gannett Fleming, 2014). In addition, nearly all petroleum product pipelines are above grade, therefore, a vapor migration pathway of concern is not likely to exist.

4.3 April 2018 Explosion and Fire

An explosion and resulting fire occurred at the Superior Refinery on April 26, 2018 while shutting down for a refinery-wide maintenance turnaround. Debris from the initial explosion punctured asphalt storage Tank 101 resulting in a release of asphalt that later ignited, causing significant damage in the asphalt tank farm and also within multiple process units. The fire subsequently caused damage to piping that contained Therminol® and #6 fuel oil in the Asphalt Tank Farm, some of which was released at the approximate locations shown on Figure 5A. The fire was later extinguished on the same day using a combination of water and firefighting foam.

The estimated extent of the petroleum hydrocarbon release (asphalt, Therminol® and #6 fuel oil) to pervious surfaces has been identified as the affected area on Figures 5A and 5B. Some of the water used for firefighting efforts flowed to the north ditch along Stinson Avenue. This firefighting water contained small amounts of hydrocarbon residue and trace amounts of firefighting chemicals. The estimated release volumes related to hydrocarbon residue are summarized in the table below.

Substance Released	Source	Estimated Release Volume	Potential Contaminants of Concern
Asphalt	Damage to Tank 101	17,000 bbls	Petroleum Hydrocarbons
Therminol®	Damaged Piping	42 bbls	Petroleum Hydrocarbons
#6 Fuel Oil	Damaged Piping	11 bbls	Petroleum Hydrocarbons

bbls = barrels (1 bbl = 42 gallons)

4.4 Interim Actions

In response to this release, immediate and interim actions were initiated. Following the explosion, SRC closed the underflow weir located in the Stinson Avenue ditch. Once the fire was extinguished on April 26, 2018 and deemed safe to do so, SRC installed sand berms to prevent any additional hydrocarbons from leaving the facility and six (6) diesel powered pumps were placed adjacent to the Stinson Avenue weir which pumped the ditch flow material into the on-site stormwater and firewater containment ponds. Following containment, interim actions were initiated to address each of the released substances listed above. A brief summary of these actions and their current interim action status is summarized below. Interim action reports documenting the recovery, assessment, treatment and/or disposal of contaminated materials will be submitted separately to the WDNR as required by NR 708.15.

4.4.1.1 Asphalt, Therminol® and #6 Fuel Oil

Recovery of the combined asphalt, Therminol® and #6 fuel oil release was initiated shortly following the release event, once the site was deemed safe for entry. Therminol® and #6 fuel oil was first removed by vacuum truck prior to asphalt removal. Recovered hydrocarbons were re-inserted in to the refining process and contaminated water was routed to the onsite wastewater treatment plant for recovery and treatment prior to discharge. Following removal of surface liquids, the comingled asphalt and residual Therminol® and #6 fuel oil was excavated, collected, characterized and disposed of at an appropriate permitted off-site disposal facility.

The asphalt recovery efforts included some amount of soil removal from the tank farm area. The asphalt recovery efforts were completed on March 27, 2019 and will be documented in a separate interim action report to the WDNR in accordance with NR 708.15. In addition, SRC continues to contain and treat storm water that accumulates through the on-site wastewater treatment plant prior to discharge as authorized by the Superior Refinery's Waste Water Treatment Plant (WWTP) Permit No WI-0003085-08-0 with additional authorization provided under the WDNR general permit for petroleum contaminated water (Wisconsin Pollutant Discharge Elimination System (WPDES) Permit No. WI-0046531-06-0). Efforts associated with the immediate actions were documented in the SRC *Immediate Action Report* to the WDNR dated June 8, 2018 (SRC, 2018). Additional details regarding these efforts will be further documented in a separate interim action report to the WDNR in accordance with NR 708.15.

4.4.1.2 Other Released Substances

During the incident, aqueous film forming foam (AFFF) was used to combat the fire. While a relatively small amount of AFFF was used, it too was mobilized by firefighting water. AFFF contains the chemicals perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS). PFOA/PFOS impacts to the site have been, and continue to be addressed by treating the impounded firefighting water with both granular activated carbon and (as needed) ion-exchange resin treatment technologies, which have been successful in treating PFOS and PFOA to below detection limits (<10 parts per trillion).

5.0 Investigation Strategy

As described in Section 1.0, the objectives of this investigation are to assess the current conditions in the release area and to investigate migration pathways to assess the nature, degree and extent of contamination and determine the need for additional investigation or interim and/or remedial action. To do this, a phased investigation strategy will be implemented to collect data that can be evaluated with existing Site data to characterize chemicals of concern (COCs) related to the explosion/fire event, and perform an initial evaluation for potential migration pathways of these COCs. Once this initial investigation phase is completed, in the context of the Site's historical and ongoing environmental setting and monitoring activities, additional investigation phases can be designed to focus on the COCs, potential migration pathways and risk receptors associated with this release can be assessed, if necessary. This section has been developed in accordance with the requirements of NR 716.09 (2) (f).

5.1 Release Area Assessment

The April 26, 2018 incident and subsequent firefighting efforts resulted in the release of asphalt, Therminol[®], and #6 fuel oil to pervious ground surfaces. These products became comingled during the incident response and were retained in containment dikes, stormwater and fire water retention ponds and/or stormwater drainage features. For the purpose of this investigation, the release is identified as the extent of asphalt released in the asphalt tank farm containment dikes and the pervious gravel roads within the process area, including the extent of petroleum hydrocarbons carried by firefighting water which migrated beyond the asphalt release into low-lying areas and the Stinson Avenue ditch. The proposed investigation to characterize the release areas will only focus on the release of residual petroleum hydrocarbons to the limited portions of the process area where an impervious surface is not present (gravel roads or ditches) and the Stinson Avenue ditch. The estimated affected area of the release is shown on Figures 5A and 5B.

Emergency and interim actions have been completed to address the released materials and are summarized in Section 4.0. These actions included removing and disposing of product and, in some instances, up to 18 inches of asphalt-impacted soil from the asphalt tank farm area (based on visual observation) and some backfilling with clean aggregate. The soil removal interim action within the tank farm area will be further documented and characterized by the sampling planned as part of this investigation.

To assess the release area, this investigation will focus on characterization of shallow soils within 4 feet of the original surface, or the "direct contact zone" as defined by the WDNR. Soil borings and/or hand auger borings will be completed to investigate pervious surfaces such as grassy areas, gravel roads, asphalt tank farm containment area, and potential migration pathways along the Stinson Avenue Ditch. Soil samples will be collected from these areas for visual characterization and laboratory analysis. The proposed sample locations are shown on Figure 6A and Figure 6B.

Due to the Site setting and post-release actions already taken, assessment of ground water and vapor is not proposed at this time. Soil quality data collected during this first phase of investigation will be

evaluated to determine if the potential for impacts to groundwater and/or vapor is present, and additional investigations will be designed to focus on these media, if necessary.

Site stormwater is being addressed by ongoing surface water sampling and water treatment. Therefore, stormwater quality will not be evaluated as a part of this investigation phase. A summary of stormwater sampling and treatment information to date has been shared with the WDNR.

5.2 Laboratory Analysis

Hydrocarbon materials released during the explosion and fire include asphalt, Therminol®, #6 fuel oil. To determine the impact to soils in the release area by these products, samples will be analyzed for petroleum volatile organic compounds (PVOCs) and polycyclic aromatic hydrocarbons (PAHs). Sampling procedures, analytical methods and quality assurance are detailed in Sections 6.0 and 7.0. Data Evaluation

Analytical data from soil samples will be compared to Wisconsin groundwater and industrial residual contaminant level (RCL) screening criteria to evaluate risk to human health in worker safety during proposed construction activities and future property use scenarios.

If it is determined that soil quality in the release area poses a risk to human health or the environment, or that the extent of soils that pose this risk has not been determined, additional investigation and/or remedial actions will be proposed to address these risks. Since PAH compounds are widely found in the environment (Thiboldeaux, 2018), an evaluation of background concentrations of identified COCs may be included.

6.0 Methods

Field activities discussed in this section have been designed as an initial step to provide the necessary data for completion of the project objectives defined above. The shallow soil petroleum hydrocarbon investigation will be completed using a combination of soil borings and hand augers. Detailed descriptions of the planned investigation activities are presented below. This section has been developed in accordance with the requirements of NR 716.09 (2) (f).

6.1 Project Health and Safety Plan

A project health and safety plan (PHASP) will be prepared for the investigation.

6.2 Standard Operating Procedures

Appendix B provides the primary standard operating procedures (SOPs) that will be followed during this initial field investigation. Updates to this work plan and associated SOPs will be prepared as needed for each subsequent phase of investigation work.

6.3 Soil Borings

As part of the initial soil investigation, up to 18 soil borings will be completed in the refinery process area to evaluate shallow soil in pervious areas located within identified affected area (Figure 6A). Soil conditions will only be evaluated within the upper four feet of the ground surface (direct contact zone). The proposed sample locations presented on Figure 6A have been chosen to provide representative coverage of the pervious surface inside the process area which was in contact with the released products.

Soil borings will be advanced using a push probe. Soil will be field screened and soil samples will be collected from each of the borings as described below. Soil boring locations may vary from the planned locations (or be eliminated) depending on utility locations, accessibility in the field, or if surface or subsurface obstructions prevent boring completion.

Soil samples will be collected at continuous vertical intervals from all soil borings. These samples will be described in the field in accordance with the Universal Soil Classification System. Soils encountered will be described in accordance with ASTM-2488, *Standard Soil Practice for Description and Identification of Soils (Visual/Manual Method)*. Soil samples will be screened in the field for volatile organic vapors with a photoionization detector (PID). Additionally, soil samples will be inspected for other evidence of contamination such as staining, odors, discoloration, and/or sheen, and the observations documented on a soil boring log for each location. Depth to water, where encountered, will be recorded. Boreholes will be backfilled according to WDNR NR 141 requirements.

6.4 Hand Augers

Hand auger samples will be completed in area not accessible by a drill rig and from the north side of the Stinson Avenue ditch. Hand auger sample collection will follow applicable Barr Engineering Co. (Barr) SOPs (Appendix B).

Two hand auger samples will be collected from below the pipe rack located south of Tanks 86 and 87 (Figure 6A). Three hand auger samples will be collected from the north side of Stinson Avenue ditch to evaluate potential off-site petroleum hydrocarbon impacts. Since some areas within the ditch may be wet throughout the year, the samples will be collected from the north (SRC) side of the ditch above the water line. Soil samples will be collected from three representative locations along the ditch for characterization from depths of 0 to 18 inches bgs.

6.5 Soil Field Screening

The field screening techniques for soils are as follows: visual examination, distinguishable odor, headspace organic vapor screening, and oil sheen. The results of these four screening procedures will be used to screen soil samples for possible contamination.

A PID with a 10.6 eV lamp will be used to complete soil headspace screening for each sample interval in accordance with the applicable Barr SOP (Appendix B). The PID will be calibrated or checked against a known concentration of a calibration gas standard prior to collection of field measurements. Field representatives will document the field screening activities and measurements in a project-dedicated field logbook or on field log data sheets.

6.6 Soil Sample Collection and Analysis

Up to two representative soil samples from each of the process area soil borings / hand auger, or a maximum of 40 soil samples, will be submitted for PVOC and PAH laboratory analysis. From the soil borings / hand augers in the process area, one sample will be collected from 0-1.5 feet bgs (may include clean backfill from interim action in tank farm) and one will be collected from 3-4 feet bgs. One sample from each of the three off-site hand auger borings will be analyzed for PAH and PVOCs. Hand auger sampling, and decontamination procedures will follow applicable Barr SOPs (Appendix B).

A summary of the proposed sampling network including analytical methods and Quality Assurance/Quality Control (QA/QC) field samples is presented in Table 1. A summary of analytes, laboratory methods, method detection limits (MDL) and criteria is presented in Table 2. Soil sample collection, decontamination procedures, chain-of custody documentation, and transport of samples will follow applicable Barr SOPs (Appendix B).

Laboratory analyses will be performed by Pace Analytical in Minneapolis, Minnesota (Pace). Appropriate sample handling and documentation procedures, as described in Barr's SOP (Appendix B), will be followed.

6.7 Sample Labeling and Numbering

Soil boring/test pit/hand auger locations, composite sample locations, and/or sample type will be represented by abbreviated letter designators, followed by a unique location number. Samples will be labeled according to the location from which they are collected. Standard designators are as follows: SB = soil boring; HA = hand auger; TB = trip blank.

6.8 Field Records

All field activities and data will be recorded daily in a dedicated field notebook or on dedicated field data collection forms. The Barr field technician will record work times and dates, field data (soil boring logs, field screening results, field analytical data, sample depths, water levels, etc.), project health and safety information, internal Barr communications, client communications, decision-making processes and rationale, documentation of changes to the investigation scope, and any other observations or activities relevant to the project. Field investigation information will also be recorded as appropriate on the field forms.

6.9 Investigation Derived Waste

Plans for investigation-derived waste are being provided in accordance with NR 716.09 (2) (f) 7. Waste generated by this investigation will be disposed of in accordance with federal, state and local regulations and Barr's SOP: *Investigative Derived Waste*. It is anticipated that soil cuttings will be placed in the on-site soil disposal containment building (3-Sided Building).

6.10 Reporting

Investigation activities, analytical results and data evaluations will be summarized in an Investigation Report in accordance with NR 716.15. The report will summarize the data collected during the investigation phase and compare analytical results to State of Wisconsin risk-screening criteria relevant to the media and facility setting and to potential worker safety during proposed construction activities. The report will include the following elements: introduction; property setting; investigation results; QA/QC procedures and results; a preliminary risk-screening evaluation; conclusions; and recommendations. Soil boring and/or hand auger boring logs and a property map showing all sampling locations and soil conditions will be developed. Laboratory reports will also be attached to the report. Recommendations for future investigation work or response action plan development will be based on the results presented in the report.

7.0 Quality Assurance / Quality Control

7.1 Project Data Quality Objectives

The data and investigative information generated will be used to determine impacts to soil to determine the overall nature and extent of any potential risks to human health and environment at the Site. This section has been developed in accordance with the requirements of NR 716.09 (2) (f) 5 and 6. The data will satisfy the Property Data Quality Objectives (DQOs) presented below:

- Analytical results must accurately reflect the soil quality.
- Field collection of samples for risk-based evaluations will require a high level of data quality since the sampling will be used to determine the potential risks associated with the release.
- The field screening procedures will have an intermediate level of data quality, but will follow Barr's SOPs included in Appendix B, ensuring consistency and accuracy.
- Laboratory results must be of sufficient quality to demonstrate that the identified COCs either do or do not present risks to human health or the environment. In most cases, for COCs with established criteria, the MDL (also referred to as limits of detection (LOD) in the State of Wisconsin) will be lower than the appropriate risk-based values and applicable State criteria. In some cases, laboratory instrumentation limitations and sample matrix may result in final MDLs greater than the associated risk standard. Guidance on how to handle these situations will be evaluated on a case-by-case basis.

7.2 Quality Assurance Objectives

The laboratory analyses will be used for the determination of overall compliance with project objectives. Pace is a certified laboratory in the State of Wisconsin and will analyze the soil samples for PAHs and PVOCS. Pace will ensure the production of quality analytical data by overall quality assurance systems that are supported by documented quality control checks. The particular types and frequencies of quality control checks analyzed with samples are defined in the laboratory's SOPs and Quality Assurance Manual (QAM), which are available for review upon request. Laboratory acceptance criteria is included with each analytical report.

Quality assurance objectives (QAOs) have been established to ensure precision, accuracy, representativeness, comparability, and sensitivity (PARCCS) of laboratory analytical data and to meet the quality control (QC) acceptance criteria of analytical protocols in support of project needs. Overall, QAO procedures for field sampling, chain-of-custody, laboratory analysis, and reporting will provide the level of data required for determining the concentration of potential contaminants.

7.2.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions.

7.2.1.1 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of Relative Percent Differences (RPDs) for matrix spike/matrix spike duplicates (MS/MSD) and/or laboratory duplicates and will be analyzed as presented in Table 3. Laboratory precision criteria will be included in the laboratory's reports.

7.2.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value and measures bias in a measurement system.

7.2.2.1 Field Accuracy Objectives

Accuracy in the field is assessed through field equipment calibration and maintenance, use of field and trip blank samples, and through the adherence to sample handling, preservation and holding time requirements. Field equipment is tested and maintained when needed using manufacturers' recommendations.

Methanol trip blank samples are received from the laboratory with PVOC containers and are analyzed to determine the extent of potential PVOC contamination introduced during sample transport and handling. A methanol trip blank sample will be included in each PVOC cooler sent back to the laboratory. The results of field and trip blanks should not have a reportable concentration of any target analyte above its MDL (exceptions may be made for the common laboratory contaminants).

7.2.2.2 Laboratory Accuracy Objectives

Accuracy of laboratory results may be assessed using the analytical results of laboratory control samples/laboratory control sample duplicates (LCS/LCSD), MS/MSD samples, surrogate standards, and/or method blanks. The percent recovery (%R) for matrix spikes will be calculated using the following equation (for LCS and other laboratory-prepared samples, B is zero):

$$\%R = \frac{A - B}{C} \times 100$$

Where: A = The analyte concentration determined experimentally from the spiked sample

B = The background level determined by a separate analysis of the unspiked sample

C = The amount of the spike added

LCS, MS, and method blank samples will be analyzed as presented in Table 3. Laboratory accuracy criteria will be included in the laboratory's reports. The results of method blanks should not have a reportable concentration of any target analyte above its MDL (exceptions may be made for the common laboratory contaminants).

7.2.3 Representativeness

Representativeness is defined as a measure of the degree to which data accurately and precisely represents a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program to provide samples representative of Site conditions and proper laboratory protocol. The representativeness criteria will be satisfied by following the associated work plan and by the use of proper sampling techniques and appropriate analytical procedures. Sample collection procedures (Appendix B) will describe proper sample homogenization techniques for soil samples that will aid in ensuring a sample is representative of Site conditions.

7.2.4 Comparability

Comparability is defined as the confidence with which one set of data can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling methods, sample preparative procedures, analytical methods and holding times. Comparability will be satisfied by ensuring that the sample plan is followed and proper and consistent sampling techniques are used.

7.2.5 Sensitivity

Sensitivity expresses the methodology's and laboratory's ability to meet or exceed the applicable criteria. Sensitivity is dependent upon instrument sensitivity, sample matrix, and composition effects, and will be monitored by the laboratory. Laboratory sensitivity will be assessed by comparing the analytical MDLs to the applicable Site criteria. Actual MDLs achieved will depend on sample size available, sample matrix interferences, dilutions, and sample percent moisture. Laboratory MDLs are listed in Table 2 and are less than or equal to Site criteria with the exception of some groundwater RCL criteria as noted in the table.

7.3 Data Reporting

7.3.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of the measurements made in the field. Field documentation of well logs, boring logs, sample identifications, etc. will be contained in the final field reports.

7.3.2 Laboratory Data Reporting

Laboratory analyses reports will be submitted to Barr upon completion. Results will be reported to the MDL. The results between the MDL and limit of quantitation (LOQ) will be qualified ("j") indicating estimated concentrations. As part of their report, the laboratory may qualify (flag) their data for such items as concentration between the MDL and LOQ, estimated concentration due to poor spike recovery, or concentration of chemical also found in the laboratory method blank. The laboratory will perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the chain-of-custody, the report format shall consist of the following:

- Date of issuance
- Project name and number
- Condition of samples upon receipt at the laboratory
- Cross-referencing of laboratory sample to project sample identification numbers
- Sample collection and receipt date
- Laboratory analysis performed
- Reference method used for analysis
- Laboratory batch number
- Sample preparation and analysis dates
- Sample results (including units and percent moisture and/or solids data used in dry weight corrections, if applicable)
- Laboratory MDL and LOQ for each analyte
- Quality control data and acceptance criteria (including method blank results, laboratory control sample recoveries, MS/MSD recoveries and RPDs, surrogate standard recoveries, and/or laboratory duplicate RPDs, if applicable)
- Discussion and/or qualification of any laboratory quality control checks which failed to meet acceptance criteria
- Discussion and/or qualification of any holding times that were not met

-
- Data qualifier definitions
 - Discussion of technical problems or other observations which may have created analytical difficulties
 - Any deviations from intended analytical strategy
 - Signature of the laboratory project manager

7.4 Data Review

Analytical and data review procedures will be in accordance with Barr's SOPs for data evaluation which are included in Appendix B. Data quality evaluation procedures will use the QC acceptance limits specified in the laboratory reports. The specific requirements which will be checked during data evaluation (where applicable) are:

- Holding times
- Preservation
- Blank data
- Laboratory control sample data
- Matrix spike data
- Surrogate data
- Duplicate sample data

The data reviewer will identify any out-of-control data points and data omissions and interact with the laboratory to correct data deficiencies. Upon completing data review, the data reviewer will provide any qualifiers and will indicate whether the data are usable as reported, usable as an estimated concentration, or unusable.

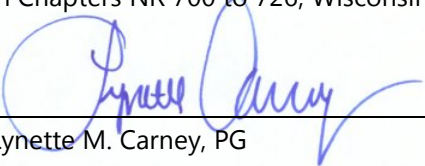
The electronic data deliverable (EDD) sample data will be verified against the laboratory hard copy report by a Barr data technician to verify that the results in the EDD and the hardcopy report accurately reflect the data collected. The EDD will be entered into a Barr computer database and the data will be output in a spreadsheet format to be used in report data tables. Data tables are reviewed by the Barr project manager before the report is submitted to the WDNR.

8.0 Schedule

The investigation activities outlined above will begin within 60 days of receiving WDNR approval of this work plan. Following the collection of soil samples, laboratory analysis will be take approximately 2 weeks to complete. Within 90 days of receiving the final laboratory results, an investigation report will be prepared to summarize the results of the initial phase of investigation. This report will make recommendations for additional investigation, interim action or remedial action. Final schedules will be dependent on approval of this work plan by the WDNR, coordination with the contractors, weather conditions, and facility accessibility during the refinery rebuild activities.

9.0 Certifications

"I, Lynette M. Carney, hereby certify that I am a hydrogeologist as that term is defined in s. NR 712.03(1), Wisconsin Administrative Code, and that to the best of my knowledge, all of the information contained in this document is correct, and the document was prepared in compliance with all applicable requirements in Chapters NR 700 to 726, Wisconsin Administrative Code."



Lynette M. Carney, PG

6/14/2019
Date

1138
Reg. No.

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Tables

Table 1
Sample Network Summary
Site Investigation Work Plan
Superior Refinery April 2018 Fire
Superior, Wisconsin

Sample Type	Laboratory Analytical Parameters	Laboratory Method	Estimated Maximum Number of Investigative Samples ¹	Grab Sample	Composite Sample	Quality Assurance/Quality Control (QA/QC) Samples	
						Methanol Trip Blank (TB)	Total
Soil Borings	PAHs	EPA 8270 SIM	36	X			36
	PVOCs	EPA 8260	36	X		2	38
Hand Auger Samples	PAHs	EPA 8270 SIM	7	X			7
	PVOCs	EPA 8260	7	X		1	8

PAHs Polycyclic Aromatic Hydrocarbons
PVOCs Petroleum Volatile Organic Compounds

Field screening parameters at each sampling location will include visual, distinguishable odor, and soil organic vapor headspace.

¹Actual number of samples will be determined based on field observations and/or locations as described in Section 5 of the Work Plan.

Table 2
Soil Analytical Target Compounds, Methods, Minimum Detections Limits, and Criteria
Site Investigation Work Plan
Superior Refinery April 2018 Fire
Superior, Wisconsin

Analyte	CAS#	MDL/LOD (mg/Kg)	LOQ* (mg/Kg)	Wisconsin Not to Exceed Direct Contact Industrial RCLs (mg/kg)	Wisconsin Groundwater RCLs, DF=2 (mg/kg)
Polycyclic Aromatic Hydrocarbons (PAH) - EPA 8270 SIM					
1-Methylnaphthalene	90-12-0	0.000535	0.001783	72.7	--
2-Methylnaphthalene	91-57-6	0.000505	0.001683	3010	--
Acenaphthene	83-32-9	0.000409	0.001363	45200	--
Acenaphthylene	208-96-8	0.000495	0.001650	--	--
Anthracene	120-12-7	0.000468	0.001560	100000	196.9492
Benzo(a)anthracene	56-55-3	0.00108	0.003600	20.8	--
Benzo(a)pyrene	50-32-8	0.000687	0.002290	2.11	0.47
Benzo(b)fluoranthene	205-99-2	0.000373	0.001243	21.1	0.4781
Benzo(g,h,i)perylene	191-24-2	0.000633	0.002110	--	--
Benzo(k)fluoranthene	207-08-9	0.000845	0.002817	211	--
Chrysene	218-01-9	0.00136	0.004533	2110	0.1442
Dibenz(a,h)anthracene	53-70-3	0.000461	0.001537	2.11	--
Fluoranthene	206-44-0	0.000428	0.001427	30100	88.8778
Fluorene	96-73-7	0.000313	0.001043	--	--
Indeno(1,2,3-cd)pyrene	193-39-5	0.000670	0.002233	21.1	--
Naphthalene	91-20-3	0.000771	0.002570	24.1	0.6582
Phenanthrene	85-01-8	0.00192	0.006400	--	--
Pyrene	129-00-0	0.00153	0.005100	22600	54.5455
Petroleum Volatile Organic Compounds (PVOC) - EPA 8260					
1,2,4-Trimethylbenzene	95-63-6	0.0100	0.033346	219	1.3787
1,3,5-Trimethylbenzene	108-67-8	0.0080	0.026573	182	1.3787
Benzene	71-43-2	0.0028	0.009385	7.07	0.0051
Ethylbenzene	100-41-4	0.0027	0.009083	35.4	1.57
Methyl-tert-butyl ether	1634-04-4	0.0059	0.019831	282	0.027
Toluene	108-88-3	0.0122	0.040605	818	1.1072
Xylene, Total (calculated)	1330-20-7	0.0116	0.038814	260	3.96

CAS# - Chemical Abstracts Service Registry Number

DF = dilution factor

mg/Kg = milligram per kilogram

RCL = Residual Contaminant Level

MDL/LOD - Method Detection Limit/Limit of Detection

MDL studies are performed annually or more often as needed per method requirements and are subject to change. Reported values may vary based on initial mass, dilution factor, % moisture, and possible matrix interferences. Results will be reported on a dry weight basis.

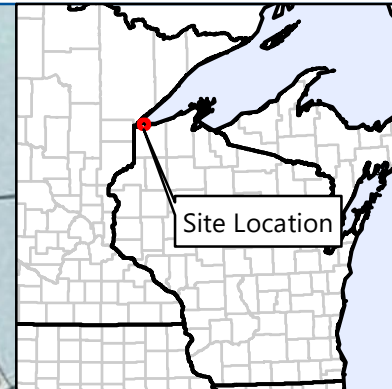
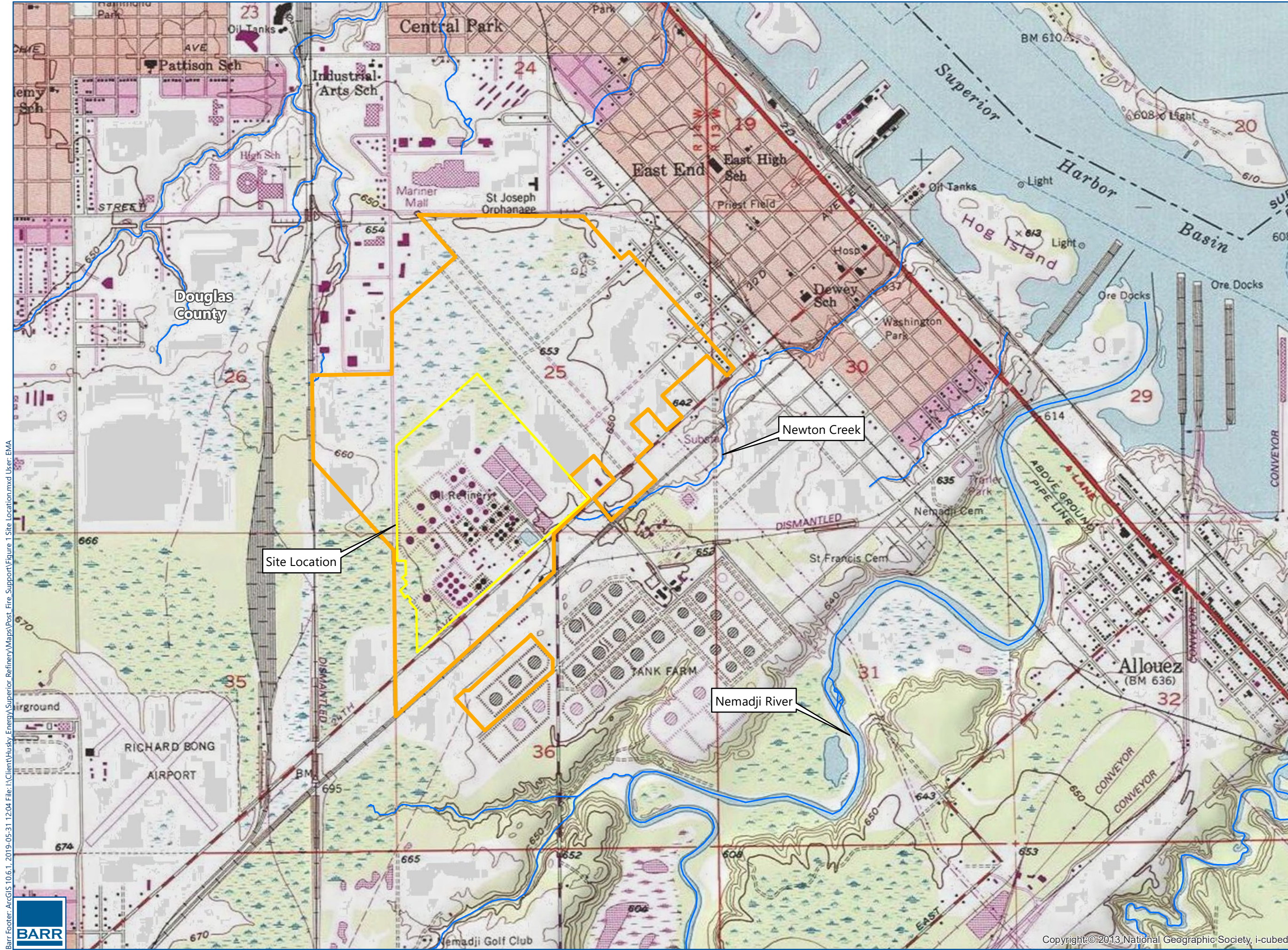
* LOQ - Limit of Quantitation - estimated values, approximately 10/3 of LOD, laboratory report will include actual value.

MDL/LOD values below criteria except where criteria are underlined.

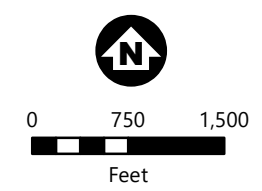
Table 3
Laboratory Quality Control Samples
Site Investigation Work Plan
Superior Refinery April 2018 Explosion and Fire
Superior, Wisconsin

Parameter	Frequency	Comments
Method Blank	1 per batch of 20 or fewer samples, with every analytical batch or as stated in the method, whichever is more frequent	Analyte-free media processed simultaneously with, and under the same conditions, as samples. Used to assess possible sources of laboratory contamination present at concentrations that may impact analytical results. Target analytes should not have a reportable concentration above the MDL.
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	1 LCS or 1 LCS/LCSD set per batch of 20 or fewer samples, with every analytical batch or as stated in the method, whichever is more frequent	Analyte-free media spiked with a known concentration of analyte processed with, and under the same conditions, as samples. Recovery is used to evaluate overall analytical method accuracy independent of sample matrix effects. If analyzed in duplicate, the calculated relative percent difference (RPD) is used to assess the overall analytical method precision.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	1 MS or 1 MS/MSD set per batch of 20 or fewer samples (may or may not be project samples)	A sample spiked with a known concentration of analyte processed with, and under the same conditions, in order to assess the accuracy of a method in a given sample matrix. If analyzed in duplicate, the calculated RPD is used to assess the precision of a method in a given sample matrix.
Laboratory Duplicate	1 per batch of 20 or fewer samples, where applicable	A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method. It may be a duplicate of a sample or a duplicate of a matrix spike.
Surrogates	Surrogates are added to each sample for organic analyses (blanks, spiked samples, project samples, QC samples) prior to sample extraction	Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Recovery is used to evaluate the analytical method efficiency.

Figures



- Approximate SRC Property Boundaries for Contiguous Operations
- Approximate Fenceline Boundaries for Refining-Related Activities







SITE LOCATION
 Superior Refining Company, LLC (SRC)
 Superior, WI
FIGURE 1

Barr Footer: ArcGIS 10.6.1, 2019-05-31 12:04 File: I:\Client\Husky Energy\Superior Refinery\Maps\Post_Fire_Support\Figure 1 Site Location.mxd User: EMA

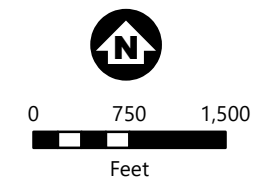


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-  Approximate Fenceline Boundaries for Refining-Related Activities
-  Surficial Water
-  Private Water Supply Well (Historical)
-  Private Water Supply Well (Post-1989)

Note: Private water supply well locations were obtained from WDNR (post-1989) and the Wisconsin Geological Survey database (pre-1989). Creek/River data from USGS.

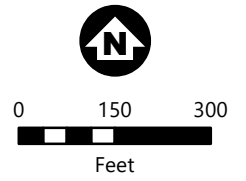


PROPERTY BOUNDARY
 Superior Refining Company, LLC (SRC)
 Superior, WI
FIGURE 2





- Approximate Fenceline Boundaries for Refining-Related Activities
- Process Area
- Process Unit Battery Limits

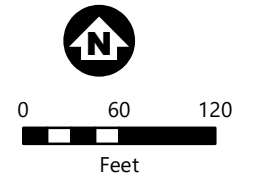


FACILITY & AREA FEATURES
 Superior Refining Company, LLC (SRC)
 Superior, WI
FIGURE 3A

Barr Footer: ArcGIS 10.6.1, 2019-05-31 15:11 File: I:\Client\Husky Energy\Superior Refinery\Maps\Post-Fire_Support\Figure 3B Refining Area Detail.mxd User: EMA



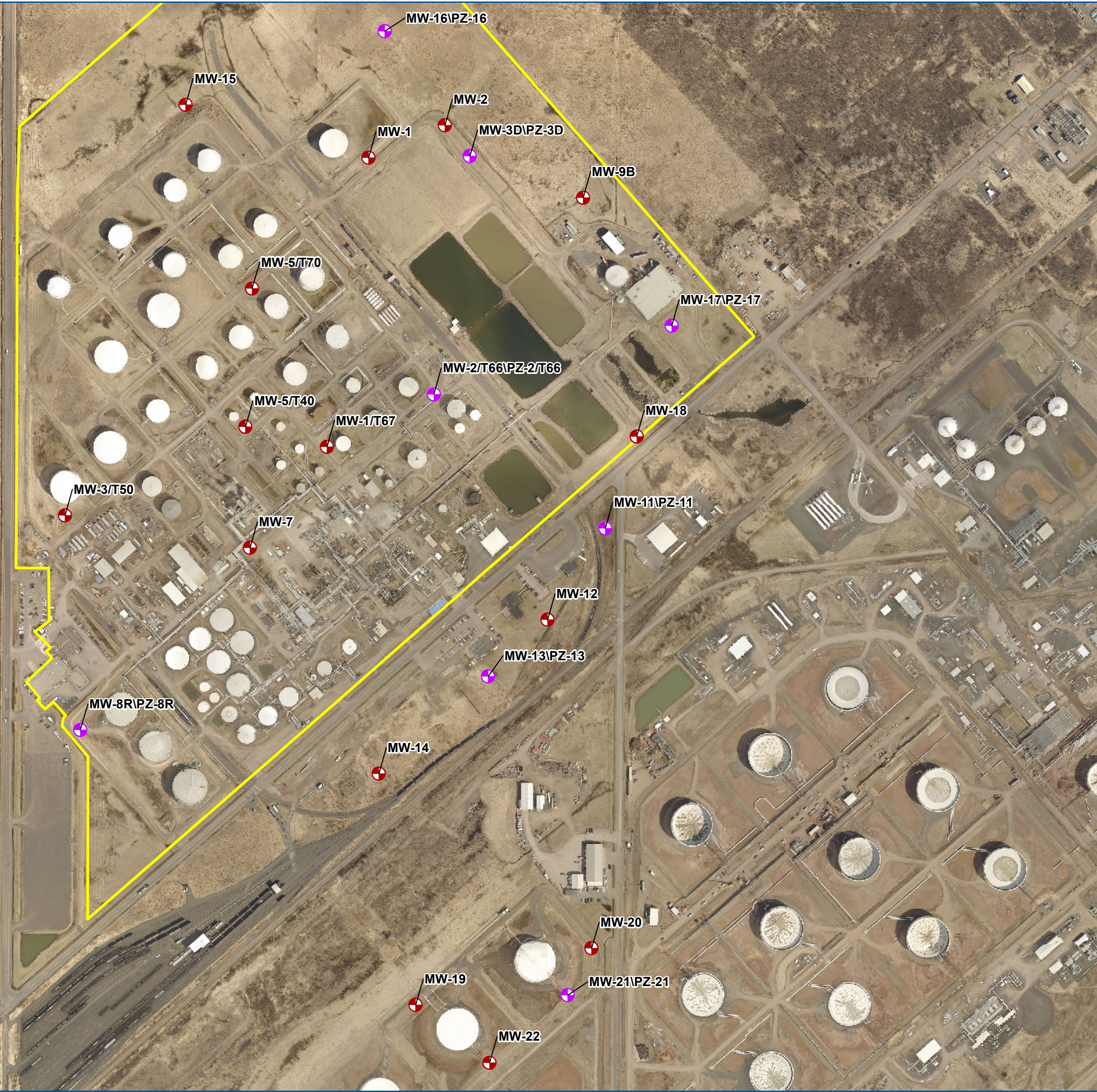
- Process Area
- Process Unit Battery Limits



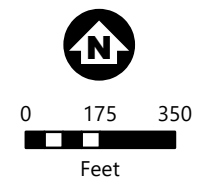
REFINING AREA DETAIL
Superior Refining
Company, LLC (SRC)
Superior, WI

FIGURE 3B





- Approximate Fenceline Boundaries for Refining-Related Activities
- Existing Monitoring Well
- Existing Monitoring Well & Piezometer Pair



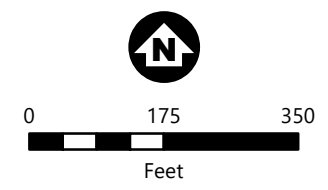
FACILITY MONITORING WELL LOCATIONS
Superior Refining Company, LLC (SRC)
Superior, WI
FIGURE 4

Barr Footer: ArcGIS 10.6.1, 2019-05-31 11:53 File: I:\Client\Husky Energy\Superior Refinery\Maps\Post_Fire_Support\Figure 5A Previous Surface Areas Affected by April 2018 Fire.mxd User: EMA



-  Approximate Fenceline Boundaries for Refining-Related Activities
-  Potentially Affected Pervious Surface
-  Stinson Ave Ditch
-  Culvert
-  Existing Monitoring Well
-  Existing Monitoring Well & Piezometer Pair

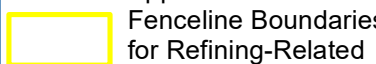
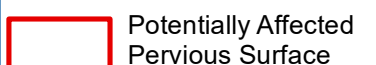
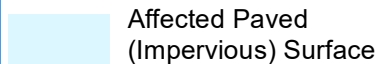
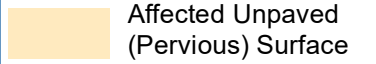


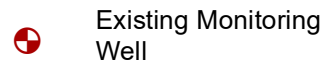
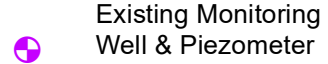
Note: Release extent based on information provided to Barr by SRC.



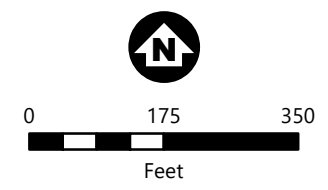
PERVIOUS SURFACE AREAS AFFECTED BY APRIL 2018 FIRE Superior Refining Company, LLC (SRC) Superior, WI
FIGURE 5A





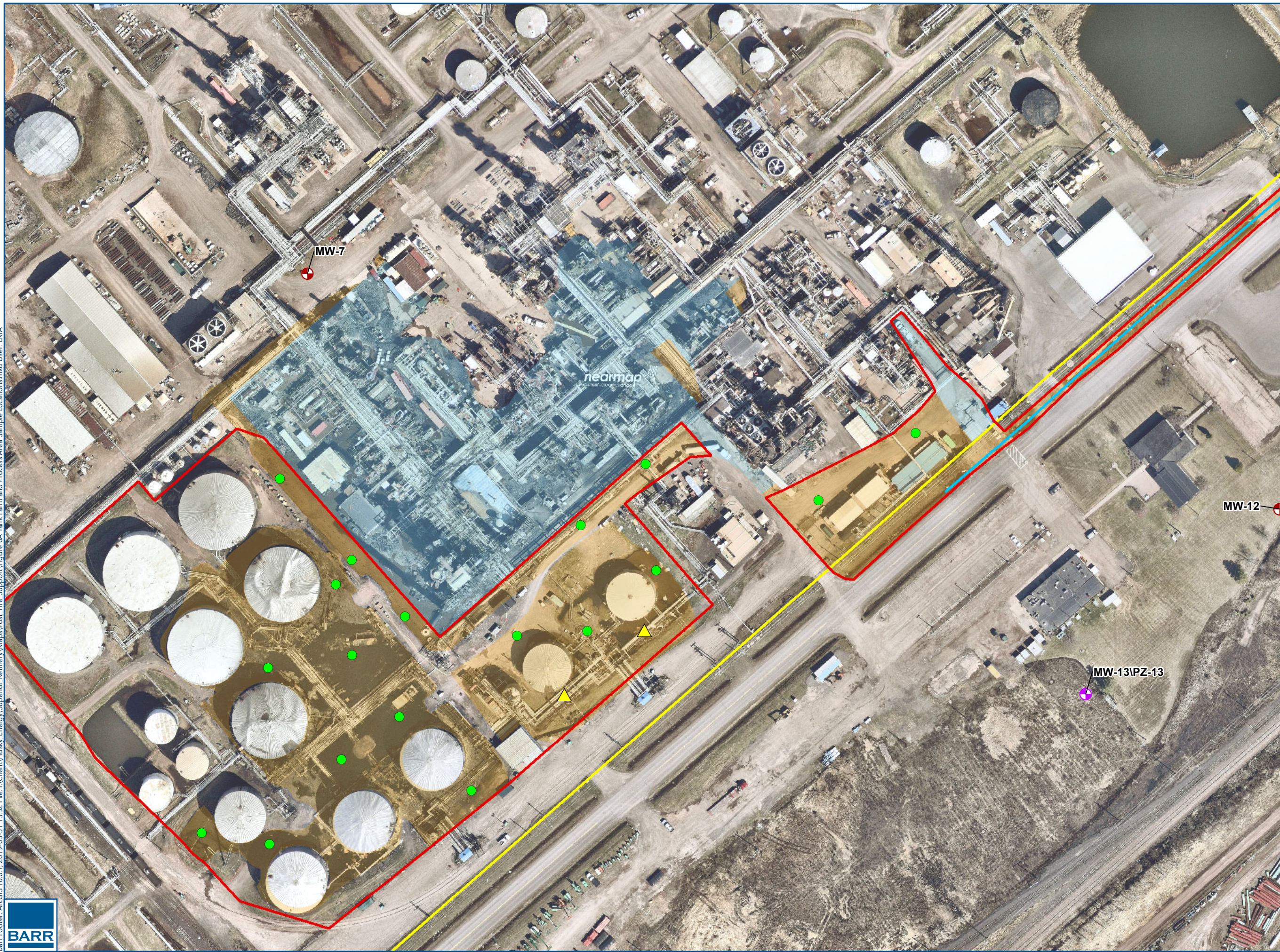
-  Approximate Fenceline Boundaries for Refining-Related Activities
-  Potentially Affected Pervious Surface
-  Affected Paved (Impervious) Surface
-  Affected Unpaved (Pervious) Surface
-  Stinson Ave Ditch
-  Culvert
-  Existing Monitoring Well
-  Existing Monitoring Well & Piezometer Pair










Note: Release extent based on information provided to Barr by SRC.



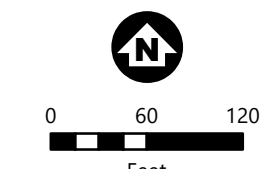
PAVED & UNPAVED SURFACES WITHIN AFFECTED AREA
Superior Refining Company, LLC (SRC)
Superior, WI
FIGURE 5B





-  Approximate Fence Line Boundaries for Refining-Related Activities
-  Potentially Affected Pervious Surface
-  Affected Paved (Impervious) Surface
-  Affected Unpaved (Pervious) Surface
-  Existing Monitoring Well
-  Existing Monitoring Well & Piezometer Pair
-  Stinson Ave Ditch
-  Proposed Soil Boring Location
-  Proposed Hand Auger Location










Note: Release extent based on information provided to Barr by SRC.



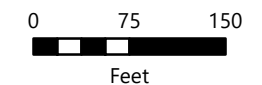
TANK FARM & PROCESS AREA SAMPLE LOCATIONS
Superior Refining Company, LLC (SRC)
Superior, WI
FIGURE 6A





-  Approximate Fenceline Boundaries for Refining-Related Activities
-  Potentially Affected Pervious Surface
-  Affected Paved (Impervious) Surface
-  Affected Unpaved (Pervious) Surface
-  Stinson Ave Ditch
-  Culvert
-  Existing Monitoring Well
-  Existing Monitoring Well & Piezometer Pair
-  Proposed Hand Auger Ditch Profile Sample Location

Note: Release extent based on information provided to Barr by SRC.



STINSON AVENUE DITCH
SAMPLE LOCATIONS
Superior Refining
Company, LLC (SRC)
Superior, WI
FIGURE 6B



Appendices

Appendix A

Area Well Construction Reports

TO THE WISCONSIN STATE BOARD OF HEALTH,
 WELL DRILLING DIVISION, MADISON, WIS.

WELL LOG, PREMISES DIAGRAM, and REPORT

For Official Record of the Board.

(TO BE USED FOR THAT PURPOSE ONLY)

Owner City of Superior Driller Marjorie Price
(If a joint ownership give name of responsible official. Also name of each individual holder on interest. Use a separate sheet and attach hereto.)
 Address Superior, Wis. Address Superior
 Date of Report July 27 1936
 Registration No. 232

Give below the location of the property on which well is drilled.
 If incorporated village or city Superior Name Superior Lot 5 Blk. 5 Street and No. Kenwood St. and Wisconsin
 If unincorporated hamlet _____ Name _____ County _____ Twp. _____ Highway _____
 If Lake Shore Plat _____ Name of Plat _____ Lots _____ Lot _____ Blk. _____ Street _____
 If Farm _____ County _____ Twp. _____ Sec. _____ Highway _____
 If School _____ County _____ Twp. _____ Sec. _____ District _____
 If other public building _____ Blk. _____ County _____ Twp. _____ Sec. _____
 Miscellaneous _____ Blk. _____ County _____ Twp. _____ Sec. _____

WELL LOG and REPORT

Screens, Seals, Grouts, etc.	Well Diagram (Each vertical line equals 1')	Kind of Casing, pipe, shoe, etc. (Each horizontal line equals 5')	Formations State if dry or water bearing	Record of FINAL Pumping Test
		4 in Driller's 3/4" pipe With 4 in forged steel shoe 168 ft Drill hole 3 3/4 in sandstone 68 ft	Clay Red 140 ft Hard pan 24 ft sandstone 71 ft shale solid	Duration of test. Hours <u>12</u> Pumping Rate. G. P. M. <u>4</u> Depth of pump in well. Ft. <u>108</u> Standing water-level (from surface.) Ft. <u>50</u> Water level when pumping Ft. <u>75</u> Water, End of test. Check: Clear <input checked="" type="checkbox"/> Cloudy _____ Turbid _____ Was well sterilized before test? Yes <input checked="" type="checkbox"/> No _____ Date <u>July 25 36</u> To which Laboratory was sample sent? _____ Date _____ Was the well sealed on completion? Yes <input checked="" type="checkbox"/> No _____ How high did you leave casing above grade? <u>14 in</u> Well was completed <u>July 27</u> 19 <u>36</u> Well Driller: <u>Marjorie Price</u> Signature (Be sure to complete the report on the reverse side)

Pipes shown in rock 3 ft

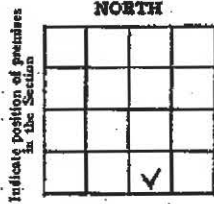
WGNHS ORIGINAL

PREMISES DIAGRAM

(See Rules)

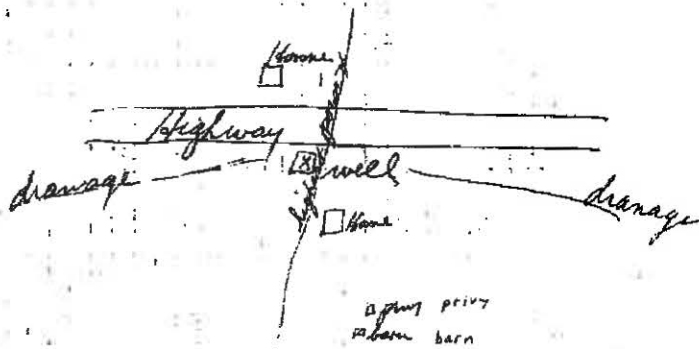
Draw a representative sketch of the premises on which this well is located, showing the location of the well with reference to buildings and possible sources of pollution. Indicate the condition of the surroundings by printing descriptive words like high, low, level, slope, lake, river, swamp, forest meadow, barnyard, cesspool, privy, sewer, etc., at their respective locations and show distance from the well on the sketch. Also show direction of the compass. See Part III for specimen Diagram.

REMARKS:

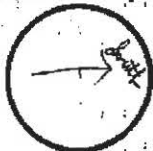


Sec 2 T4R3 (E) (W)

(Each division equals 10') (If more or less indicate: _____)



Show in circle the Direction of Compass



Note: Additional copies of this form may be obtained at 5c per copy in lots of 10 or more. Send remittance with order to State Board of Health, Well Drilling Division, Madison.

1899-2

County Douglas City Superior 48 Sec. 6 ¹⁴³⁶ Part 5
 (Office Record - Do not fill in)
 Section 6 T48N R13W

TO THE WISCONSIN STATE BOARD OF HEALTH,
 WELL DRILLING DIVISION, MADISON, WIS.

WELL LOG, PREMISES DIAGRAM, and REPORT

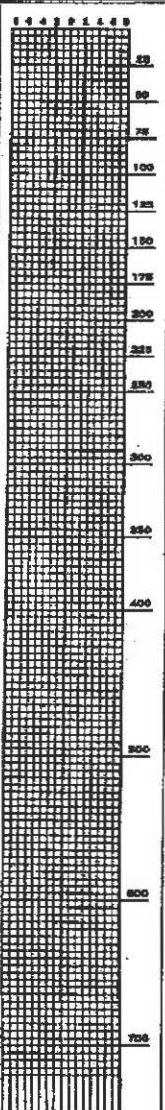
For Official Record of the Board.

(TO BE USED FOR THAT PURPOSE ONLY)

Owner City of Superior Driller Mastran Bros
 (If a joint venture, give name of responsible official. Also name of each individual holder an interest. Give percentage share and attach hereto.)
 Address 1422 Highhill
Superior Wis Date of Report Feb 27 1937
 (City, village, township, county) Registration No. 232

Give below the location of the property on which well is drilled.
 If incorporated village or city: Superior 27 E 18 St
 Name Lot Blk. Street and No.
 If unincorporated hamlet: Name County Town Highway
 If Lake Shore Plat: Name of Plat Lots Blk. Street
 If Farm: County Town Sec. Highway
 If School: County Town Sec. District
 If other public building: Kind County Town Sec.
 Miscellaneous: Kind County Town Sec.

WELL LOG and REPORT

Screen, Grate, Grouts, etc.	Well Diagram (Each vertical line equals 1')	Kind of Casing, Pipe, etc. (Each horizontal line equals 5')	Formations State if dry or water bearing	Record of FINAL Pumping Test
		<u>4 in special well pipe Drive shaft</u>	<u>Red clay 100 ft</u> <u>Hard pan 45 ft</u> <u>sand & gravel 15 ft</u> <u>coarse gravel 5 ft water bearing.</u>	Duration of test: <u>8 hr</u> Hours Pumping Rate: <u>10 gal</u> G. P. M. Depth of pump in well: <u>90 ft</u> Ft. Standing water-level (from surface): <u>55 ft</u> Ft. Water level when pumping: <u>55 ft</u> Ft. Water, End of test. Check: <u>Clear</u> Cloudy _____ Turbid _____ Was well sterilized before test? Yes <u>—</u> No _____ Date _____ To which Laboratory was sample sent? <u>Superior</u> Date _____ Was the well sealed on completion? Yes <u>—</u> No _____ How high did you leave casing above ground? <u>14 in</u> Well was completed <u>Aug 5</u> 19 <u>26</u> Well Driller: <u>Mastran Bros</u> Signature (Be sure to complete the report on the reverse side)

1900

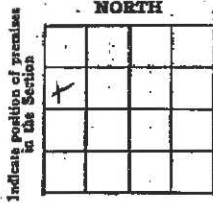
WGNHS ORIGINAL

PREMISES DIAGRAM

(See Rules)

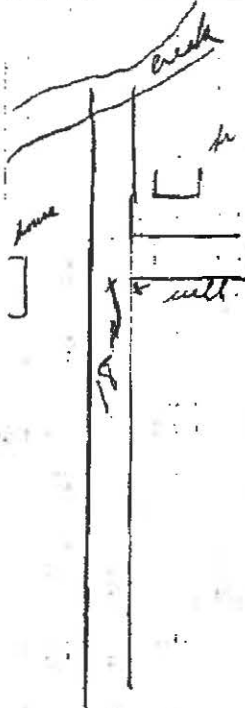
Draw a representative sketch of the premises on which this well is located, showing the location of the well with reference to buildings and possible sources of pollution. Indicate the condition of the surroundings by printing descriptive words like high, low, level, slope, lake, river, swamp, forest meadow, barnyard, cesspool, privy, sewer, etc., at their respective locations and show distance from the well on the sketch. Also show direction of the compass. See Part III for specimen Diagram.

REMARKS:



Scale 1/4" = 10' (If more or less indicate: _____)

(Each division equals 10') (If more or less indicate: _____)



Show in circle the Direction of Compass



Note: Additional copies of this form may be obtained at 5c per copy in lots of 10 or more. Send remittance with order to State Board of Health, Well Drilling Division, Madison.

1900-2

WELL CONSTRUCTOR'S REPORT TO WISCONSIN STATE BOARD OF HEALTH
See Instructions on Reverse Side

NW, NE,
Sec 1
T48N
R14W

1. County Douglas Town
Village
City
 2. Location Barber ave and 58 st Superior
Check one for city name
Name of street and number of premise or Section, Town and Range numbers
 3. Owner or Agent Lake Superior Fur Farm
Name of individual, partnership or firm
 4. Mail Address 3732 E 7 st
Complete address required
 5. From well to nearest: Building 4 ft; sewer _____ ft; drain _____ ft; septic tank _____ ft;
 dry well or filter bed _____ ft; abandoned well _____ ft.

6. Well is intended to supply water for: Fur Farm

7. DRILLHOLE:

Dia. (in.)	From (ft.)	To (ft.)	Dia. (in.)	From (ft.)	To (ft.)

8. CASING AND LINER PIPE OR CURBING:

Dia. (in.)	Kind	From (ft.)	To (ft.)
<u>6"</u>	<u>steel</u>	<u>0</u>	<u>140</u>

9. GROUT:

Kind	From (ft.)	To (ft.)

11. MISCELLANEOUS DATA:

Yield test: 50 Hrs. at 4 GPM.
 Depth from surface to water-level: 40 ft.
 Water-level when pumping: same ft.
 Water sample was sent to the state laboratory at:
owner on _____ 19____
City

10. FORMATIONS:

Kind	From (ft.)	To (ft.)
<u>Red clay</u>	<u>0</u>	<u>130</u>
<u>Muddy sand</u>	<u>130</u>	<u>135</u>
<u>Coarser sand</u>	<u>135</u>	<u>145</u>
<u>10 ft Johnson screen</u>		

Construction of the well was completed on:

Dec 1 1955

The well is terminated 12 inches above, below the permanent ground surface.

Was the well disinfected upon completion?
Yes No _____

Was the well sealed watertight upon completion?
Yes No _____

Signature Master Bros
Registered Well Driller

West North Ave
Complete Mail Address

Please do not write in space below

Rec'd _____ No. _____
 Ans'd _____
 Interpretation _____

10 ml _____ 10 ml _____ 10 ml _____ 10 ml _____ 10 ml _____
 Gas—24 hrs. _____
 48 hrs. _____
 Confirm _____
 B. Coli _____
 Examiner _____

2065

DEC 31 1945

WELL CONSTRUCTOR'S REPORT TO WISCONSIN STATE BOARD OF HEALTH

See Instructions on Reverse Side

- 1. County Douglas Town Superior
Village
City
- 2. Location NW-NE Sec 1-748-14W
- 3. Owner or Agent C A PALM
- 4. Address Superior Wis
- 5. From well to nearest: Building 3 ft; sewer 35 ft; drain — ft; septic tank 50 ft;
dry well or filter bed — ft; abandoned well — ft.
- 6. Well is intended to supply water for: Home

7. DRILLHOLE OR EXCAVATION:

Dis. (in.)	From (ft.)	To (ft.)
4"	0	145

8. CASING AND LINER PIPE OR CURBING:

Dis. (in.)	Kind	From (ft.)	To (ft.)
4"	Line Pipe	0	145
<u>2x48x60 gauge screen + packer</u>			

9. GROUT:

Kind	From (ft.)	To (ft.)
<u>None</u>		

10. FORMATIONS:

Kind	Thick-ness (ft.)	Total Depth (ft.)
Red CLAY	98	98
HARD sand	32	130
Britt. sand	10	140
CLEAN SAND	5	145

11. MISCELLANEOUS DATA:

Yield test: 10 Hrs. at 5 GPM.
Depth from surface to water: 57 ft.
Water-level when pumping: 65 ft.

Water sample sent to laboratory at
Superior on 1945

Signature Mastrian 1300 Wentworth Wis
Registered Well Driller Complete Mail Address

Construction of the well was completed on April 27 1945

The well is terminated 12 inches (above) ~~(below)~~ the permanent grade.

Was the well disinfected upon completion?
Yes No

Was the well sealed watertight upon completion?
Yes No

WELL CONSTRUCTOR'S REPORT TO WISCONSIN STATE BOARD OF HEALTH
See Instructions on Reverse Side

RECEIVED⁶
JAN 1 1965

1. County Douglas Town
Village
City Superior **SANITARY**
Check one and give **ENGINEERING**
2. Location Section 2, T48N, R14W
Name of street and number of premise or Section, Town and Range numbers
3. Owner or Agent Elmer Craft
Name of individual, partnership or firm
4. Mail Address 214 N 58 Superior, Wis
Complete address required
5. From well to nearest: Building 4 ft; sewer 45 ft; drain None ft; septic tank None ft;
 dry well or filter bed — ft; abandoned well — ft.
6. Well is intended to supply water for: Home

7. DRILLHOLE:

Dia. (in.)	From (ft.)	To (ft.)	Dia. (in.)	From (ft.)	To (ft.)
8	0	20	4	20	172

8. CASING AND LINER PIPE OR CURBING:

Dia. (in.)	Kind and Weight	From (ft.)	To (ft.)
4	Steel 14#	0	172

9. GROUT:

Kind	From (ft.)	To (ft.)
Mud	0	20

11. MISCELLANEOUS DATA:

Yield test: 16 Hrs. at 6 GPM.
 Depth from surface to water-level: 65 ft.
 Water-level when pumping: 80 ft.
 Water sample was sent to the state laboratory at:
Superior on 8-26 1964
City

10. FORMATIONS:

Kind	From (ft.)	To (ft.)
clay	0	130
hard pan	130	171
gravel	171	172

Construction of the well was completed on:

8-17- 1964

The well is terminated 10 inches
 above, below the permanent ground surface.

Was the well disinfected upon completion?

Yes X No —

Was the well sealed watertight upon completion?

Yes X No —

Signature [Signature]
 Registered Well Driller

[Signature]
 Complete Mail Address

Please do not write in space below

Rec'd _____ No. _____

Ans'd _____

Interpretation _____

2067

10 ml 10 ml 10 ml 10 ml 10 ml

Gas—24 hrs. _____

48 hrs. _____

Confirm _____

B. Coli _____

Examiner _____

WELL CONSTRUCTOR'S REPORT TO WISCONSIN STATE BOARD OF HEALTH
See Instructions on Reverse Side

RECEIVED

1. County Douglas Town Village City Superior
Check one and give name

2. Location Section 2, T48N, R14W JUN 25 1962
Name of street and number of premise or Section, Town and Range numbers

3. Owner or Agent Markus Fossnes SANITARY ENGINEERING
Name of individual, partnership or firm

4. Mail Address Superior Wis
Complete address required

5. From well to nearest: Building 6 ft; sewer _____ ft; drain None ft; septic tank _____ ft; dry well or filter bed _____ ft; abandoned well _____ ft. RECEIVED

6. Well is intended to supply water for: Home AUG 5 1962

7. DRILLHOLE:

Dia. (in.)	From (ft.)	To (ft.)	Dia. (in.)	From (ft.)	To (ft.)
8	0	20	4	20	218

8. CASING AND LINER PIPE OR CURBING:

Dia. (in.)	Kind and Weight	From (ft.)	To (ft.)
4	Steel 12	0	218

9. GROUT:

Kind	From (ft.)	To (ft.)
Mud	0	20

11. MISCELLANEOUS DATA:

Yield test: 12 Hrs. at 10 GPM.
Depth from surface to water-level: 65 ft.
Water-level when pumping: 60 ft.
Water sample was sent to the state laboratory at:
Superior on 8-29 1962
City

10. FORMATIONS:

Kind	From (ft.)	To (ft.)
clay	0	130
hardpan	130	200
clay	200	216
Sand + gravel	216	218

SANITARY ENGINEERING

Construction of the well was completed on:

8-25 1962

The well is terminated 12 inches above, below the permanent ground surface.

Was the well disinfected upon completion?
Yes No _____

Was the well sealed watertight upon completion?
Yes No _____

Signature Lyle Devier
Registered Well Driller

Wentworth Wis
Complete Mail Address

Rec'd _____ No. _____
Ans'd _____
Interpretation _____
2068

10 ml 10 ml 10 ml 10 ml 10 ml
Gas—24 hrs. _____
48 hrs. _____
Confirm _____
B. Coli _____
Examiner _____

WELL CONSTRUCTOR'S REPORT TO WISCONSIN STATE BOARD OF HEALTH DS-14-U
See Instructions on Reverse Side

1. County Douglas Town Superior
 Village City Superior
Check one and give name

2. Location SW, SE, SE, Sec. 2
T48N R14W East to Golf course
Name of street and number of premise or Section, Town and Range numbers

3. Owner or Agent Harold Olson
Name of individual, partnership or firm

4. Mail Address Superior sta B
Complete address required

5. From well to nearest: Building 18 ft; sewer none ft; drain none ft; septic tank none ft.
 dry well or filter bed none ft; abandoned well none ft.

6. Well is intended to supply water for: Home

7. DRILLHOLE:

Dia. (in.)	From (ft.)	To (ft.)	Dia. (in.)	From (ft.)	To (ft.)

8. CASING AND LINER PIPE OR CURBING:

Dia. (in.)	Kind and Weight	From (ft.)	To (ft.)
4"	steel pipe	0	220

9. GROUT:

Kind	From (ft.)	To (ft.)

11. MISCELLANEOUS DATA:

Yield test: 10 Hrs. at 7 GPM.
 Depth from surface to water-level: 60 ft.
 Water-level when pumping: same ft.
 Water sample was sent to the state laboratory at:
 _____ on _____ 19____
 City _____

10. FORMATIONS:

Kind	From (ft.)	To (ft.)
Red clay	0	120
Hardpan	120	220
Sandstone	220	240

RECEIVED
 FEB 3 1958
 ENVIRONMENTAL
 SANITATION

Construction of the well was completed on:

May 5 1957

The well is terminated 12 inches
 above, below the permanent ground surface.

Was the well disinfected upon completion?
 Yes No

Was the well sealed watertight upon completion?
 Yes No

Signature Mastran Bros
 Registered Well Driller

Wentworth Ave
 Complete Mail Address

Please do not write in space below

Rec'd _____ No. _____
 Ans'd _____
 Interpretation _____

10 ml 10 ml 10 ml 10 ml 10 ml
 Gas—24 hrs. _____
 48 hrs. _____
 Confirm _____
 B. Coli _____

2069

Examiner _____

WELL CONSTRUCTOR'S REPORT TO WISCONSIN STATE BOARD OF HEALTH
See Instructions on Reverse Side

RECEIVED

1. County Douglas Town Village City South Superior JAN 24 1948
Check one and give name
2. Location N.E. 1/4 of SE 1/4 of Sec. 2, T. 44N, R. 14W, P. 14
Name of street and number of premise or Sec. Tn. and R. numbers
3. Owner Paul Mues
Name of individual, partnership or firm
4. Mail Address South Superior
Complete address required
5. From well to nearest: Building 12 ft; sewer 60 ft; drain None ft; septic tank None ft;
 dry well or filter bed None ft; abandoned well None ft.
6. Well is intended to supply water for: Home

7. DRILLHOLE:

Dia. (in.)	From (ft.)	To (ft.)
<u>5</u>	<u>0</u>	<u>184</u>

10. FORMATIONS:

Kind	From (ft.)	To (ft.)
<u>Red clay</u>	<u>0</u>	<u>140</u>
<u>Red hard pan</u>	<u>140</u>	<u>184</u>
<u>clean water sand</u>	<u>184</u>	<u>187</u>

8. CASING AND LINER PIPE OR CURBING:

Dia. (in.)	Kind	From (ft.)	To (ft.)
<u>5</u>	<u>std pipe</u>	<u>0</u>	<u>184</u>
<u>3</u>	<u>screen</u>	<u>184</u>	<u>187</u>

9. GROUT:

Kind	From (ft.)	To (ft.)
<u>puddled clay</u>	<u>0</u>	<u>184</u>

11. MISCELLANEOUS DATA:

Yield test: 40 Hrs. at 7 GPM.

Depth from surface to water: 126 ft.

Water-level when pumping: 130 ft.

Water sample sent to laboratory at
Superior on July 15 1948

Construction of the well was completed on June 30 1948

The well is terminated 24 inches above, below the permanent ground surface.

Was the well disinfected upon completion?
 Yes No

Was the well sealed watertight upon completion?
 Yes No

Poplar Wis
 Complete Mail Address

Signature M. R. Long
 Registered Well Driller

WELL CONSTRUCTOR'S REPORT TO WISCONSIN STATE BOARD OF HEALTH

See Instructions on Reverse Side

1. County Douglas (Town Village City Superior Check one and give name

2. Location N-E 1/4 of S-E 1/4 of Sec 2 T 48 N R 14 W
Name of street and number of premises or Section, Town and Range numbers

3. Owner or Agent Edward Bak
Name of individual, partnership or firm

4. Mail Address Station Station B, Superior Wis
Complete address required

5. From well to nearest: Building 10 ft; sewer 12 ft; drain 25 ft; septic tank 80 ft;
dry well or filter bed 100 ft; abandoned well not ft.

6. Well is intended to supply water for: Tavern

7. DRILLHOLE:

Dia. (in.)	From (ft.)	To (ft.)	Dia. (in.)	From (ft.)	To (ft.)
8	0	20	4	160	265
6	20	160			

8. CASING AND LINER PIPE OR CURBING:

Dia. (in.)	Kind and Weight	From (ft.)	To (ft.)
4	Std. pipe 11 lb	0	251

9. GROUT:

Kind	From (ft.)	To (ft.)
packed clay	0	20

11. MISCELLANEOUS DATA:

Yield test: 16 Hrs. at 10 GPM.
 Depth from surface to water-level: 105 ft.
 Water-level when pumping: 110 ft.
 Water sample was sent to the state laboratory at:
Madison on July 5 1960
 City

10. FORMATIONS:

Kind	From (ft.)	To (ft.)
Red clay	0	140
Red Sandstone	140	245
sand	245	265

JUL 13 1960
ENGINEERING

Construction of the well was completed on:
June 25 1960
 The well is terminated 12 inches
 above, below the permanent ground surface.
 Was the well disinfected upon completion?
 Yes No
 Was the well sealed watertight upon completion?
 Yes No

Signature M. P. Long Registered Well Driller
Joseph W. ... Complete Mail Address

Rec'd JUL 6 1960 No. 23813

Ans'd _____

Interpretation SAFE—BACTERIOLOGICALLY

Gas—24 hrs. _____

48 hrs. _____

Confirm 0

B. Coli _____

Examiner _____

2071

County Douglas Twp. City Superior Sec. 30 Rm. 1437
 (Office Record - Do not fill in)
749 RBW 744 RBW ?
 SE Sec 30

TO THE WISCONSIN STATE BOARD OF HEALTH,
 WELL DRILLING DIVISION, MADISON, WIS.

WELL LOG, PREMISES DIAGRAM, and REPORT

For Official Record of the Board.

(TO BE USED FOR THAT PURPOSE ONLY)

Owner Park Board of City of Superior Driller Marion Pro
 (If a joint responsibility give name of responsible official. Also name of each individual holding an interest. See a separate sheet and attach hereto.)
 Address Superior Wis Address 714 Duquette Ave Superior Wis
 (City, village, township, county) Date of Report June 29 1937
 Registration No. 232

Give below the location of the property on which well is drilled.
 If incorporated village or city: City of Superior 4 E. 7th St
 If unincorporated hamlet _____
 If Lake Shore Plat _____
 If Farm _____
 If School _____
 If other public building _____
 Miscellaneous _____

WELL LOG and REPORT

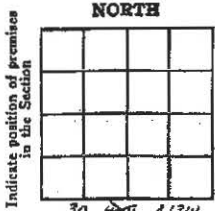
Screens, Seals, Grouts, etc.	Well Diagram (Each vertical line equals 1')	Kind of Casing, liner, shoe, etc. (Each horizontal line equals 5')	Formations State if dry or water bearing	Record of FINAL Pumping Test
6 ft Johnson Reel Brass screen 25' dia		Drops forged Liner shoe 6 in. Dutton Spool casing	Red clay 85 ft Lime sand to gravel to 102 ft gravel to 108 ft	Duration of test: Hours <u>10 hr</u> Pumping Rate: G. P. M. <u>60</u> Depth of pump in well: Ft. <u>25 ft</u> Standing water-level (from surface): Ft. <u>3 ft</u> Water level when pumping: Ft. <u>5 ft</u> Water, End of test. Check: Clear <input checked="" type="checkbox"/> Cloudy _____ Turbid _____ Was well sterilized before test? Yes <input checked="" type="checkbox"/> No _____ Date _____ To which Laboratory was sample sent? <u>Superior Wis</u> Date <u>June 29-37</u> Was the well sealed on completion? Yes <input checked="" type="checkbox"/> No <input checked="" type="checkbox"/> How high did you leave casing above grade? <u>Capped 1 ft below grade</u> Well was completed <u>June 29 1937</u> Well Driller: <u>Marion Pro</u> Signature. (Be sure to complete the report on the reverse side)
		Flowing Well flowing 8 gal per min 3 feet below Below grade at well and 7 ft about Pm level Pump house to be built when pumps to be installed		

PREMISES DIAGRAM

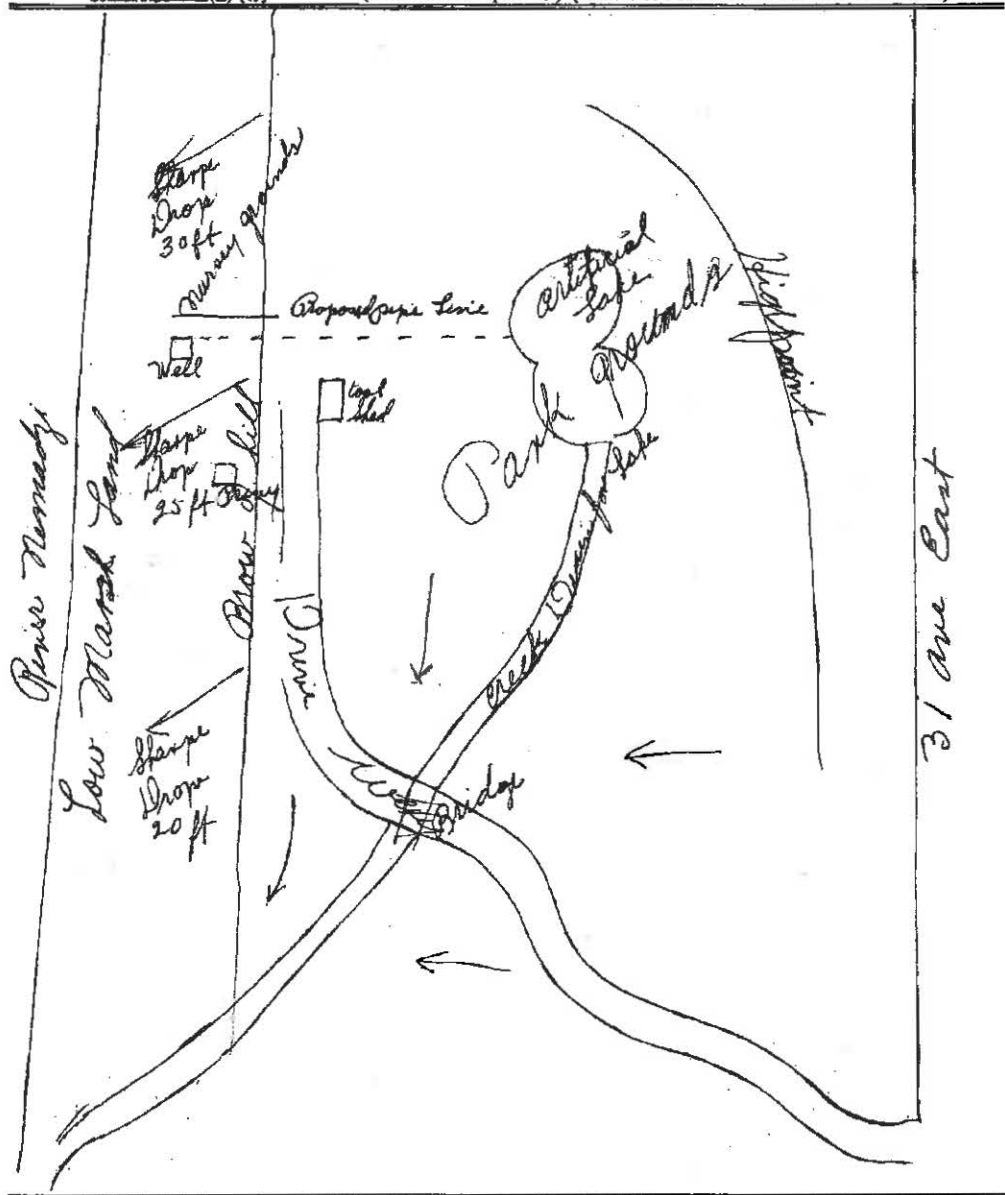
(See Rules)

Draw a representative sketch of the premises on which this well is located, showing the location of the well with reference to buildings and possible sources of pollution. Indicate the condition of the surroundings by printing descriptive words like high, low, level, slope, lake, river, swamp, forest meadow, barnyard, cesspool, privy, sewer, etc., at their respective locations and show distance from the well on the sketch. Also show direction of the compass. See Part III for specimen Diagram.

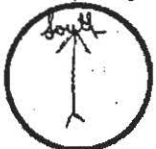
REMARKS :



Section ~~30~~ ³⁰ ~~13~~ ¹³ W (E) (W) T49N
 (Each division equals 10') (If more or less indicate: _____)



Show in circle the Direction of Compass



Note: Additional copies of this form may be obtained at 5c per copy in lots of 10 or more. Send remittance with order to State Board of Health, Well Drilling Division, Madison.

WELL CONSTRUCTION REPORT
WISCONSIN STATE BOARD OF HEALTH
WELL CONSTRUCTION DIVISION

✓ JAN 28 1943

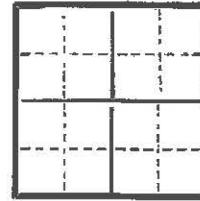
Note: Section 31 of the Wisconsin Well Construction Code, having the force and effect of law, provides that within thirty days after completion of every well the driller shall submit a report covering all essential details of construction to the State Board of Health on a form provided by the Board.

Owner Ray Zogen Driller Magstrain Bros
 Street or RFD 3928 E 17 st Post Office Westworth Wis
 Post Office Superior W 5 Date Jan 26-43 Permit No. 232

LOCATION OF PREMISES

Douglas County City of Superior Town

The square below represents a section of land divided into 40 acre tracts. Mark the position of the premises in the section.

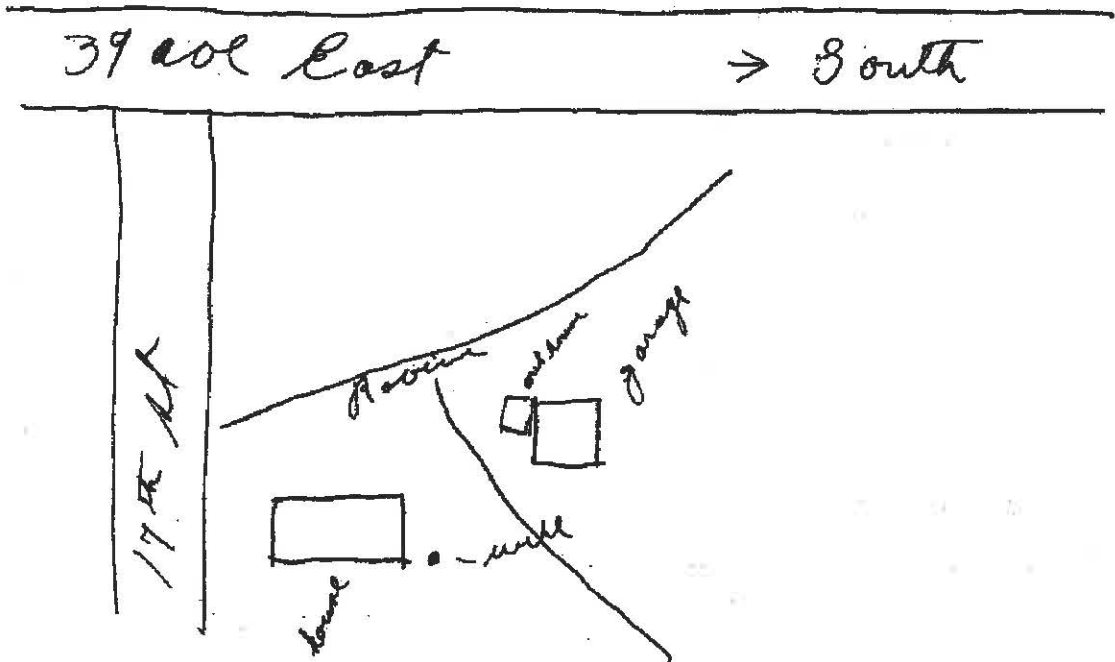


Sec. No. SE Sec 31
 Twp. No. T49N
 Range R13W E
W

Describe further by subdivision, plat, district, lake, lot.
Known as Albany Acres.
 block, nearest principal highway, etc., whichever apply.
at Albany

DIAGRAM OF PREMISES

See Well Construction Report bulletin. In making the diagram in the space below consider 10 ft. as the distance between lines. Be sure to indicate NORTH.



WELL LOG and REPORT

For method of making report, refer to bulletin entitled "Well Construction Report," 7-5-39.

In this column indicate the kind of casing, liner, shoe and other accessories used.

WELL DIAGRAM
Use a red line to show casing or liner pipe. Use black for drill or borehole.

In this column state the kind of formations penetrated, their thickness in feet and if water bearing.

Record of **FINAL** Pumping test

5" old Well
Reduced to
3 in.
special
Well pipe

pipe cut at
90 ft and
special packer
Installed →

Red line
denotes Rock

Inches Diameter		Depth
2 3 4 5 6 8 10 12 14 16 18		
		25
		50
		75
		100
		150
		172
		200
		400
		800
		1200

Draw the diagram to show the right half only

Old Well
to 140 ft
Hard pan
6 ft Lake Superior sandstone

Duration of test
Hours 10

Pumping rate
G.P.M. 6

Depth of pump in well. Ft. 90

Standing water-level (from surface)
Ft. 45

Water-level when pumping Ft. same

Water. End of test.
Clear
Cloudy
Turbid

Was the well sterilized?
Yes No

To which laboratory was sample sent?
Superior
Date

Was the well sealed on completion?
Yes No

How high did you leave the casing-pipe above grade?
1 ft

Well was completed
Date Oct 20 42

Well constructor
M. McKeown
Signature

2227-2

WELL CONSTRUCTOR'S REPORT TO WISCONSIN STATE BOARD OF HEALTH
See Instructions on Reverse Side

?
SE Sec 31
T 49N
R 13W

1. County Douglas Town Superior
Village City Check one and give name

2. Location Alloy Acres 3710 E 1st
Name of street and number of premise or Section, Town and Range numbers

3. Owner or Agent John Kurilla
Name of individual, partnership or firm

4. Mail Address Itasca St Superior Wis
Complete address required

5. From well to nearest: Building 10 ft; sewer ft; drain ft; septic tank ft;
dry well or filter bed ft; abandoned well ft.

6. Well is intended to supply water for: Home

7. DRILLHOLE:

Dia. (in.)	From (ft.)	To (ft.)	Dia. (in.)	From (ft.)	To (ft.)
4 1/2	0	165			

8. CASING AND LINER PIPE OR CURBING:

Dia. (in.)	Kind and Weight	From (ft.)	To (ft.)
4 1/2	Steel	0	165

9. GROUT:

Kind	From (ft.)	To (ft.)

11. MISCELLANEOUS DATA:

Yield test: 5 Hrs. at 5 GPM.
Depth from surface to water-level: 60 ft.
Water-level when pumping: same ft.
Water sample was sent to the state laboratory at:
by owner on 19
City

10. FORMATIONS:

Kind	From (ft.)	To (ft.)
Red clay	0	135
Kalpin	135	160

RECEIVED
JAN 14 1959
ENVIRONMENTAL
SANITATION

Construction of the well was completed on:

Sept 1 1958

The well is terminated 12 inches
 above, below the permanent ground surface.

Was the well disinfected upon completion?
Yes No

Was the well sealed watertight upon completion?
Yes No

Signature Mastran Bros
Registered Well Driller

West north 2nd
Complete Mail Address

Please do not write in space below

Rec'd _____ No. _____
Ans'd _____
Interpretation _____

10 ml _____ 10 ml _____ 10 ml _____ 10 ml _____ 10 ml _____
Gas—24 hrs. _____
48 hrs. _____
Confirm _____
B. Coli _____

Examiner _____

2228

WELL CONSTRUCTION REPORT
WISCONSIN STATE BOARD OF HEALTH
WELL DRILLING DIVISION

AUG 28 1941

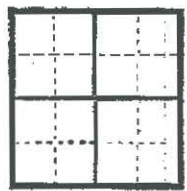
Note: Section 32 of the Wisconsin Well Drilling Sanitary Code, having the force and effect of law, provides that within thirty days after completion of every well the driller shall submit a report covering all essential details of construction to the State Board of Health on a form provided by the Board.

Owner William Kolb, 6 Driller Mastrin Bros
 Street or RFD Norman Maepube Post Office Westwortha Wis.
 Post Office Superior Date Feb 27/41 Permit No. 232

LOCATION OF PREMISES

Bayfield County Douglas City of Superior Town
Blk 12 Norman road Lot 27
 Describe further by subdivision, plat, district, lake, lot,
S 1/2 Sec 25 Sec 26?
 block, nearest principal highway, etc., whichever apply.

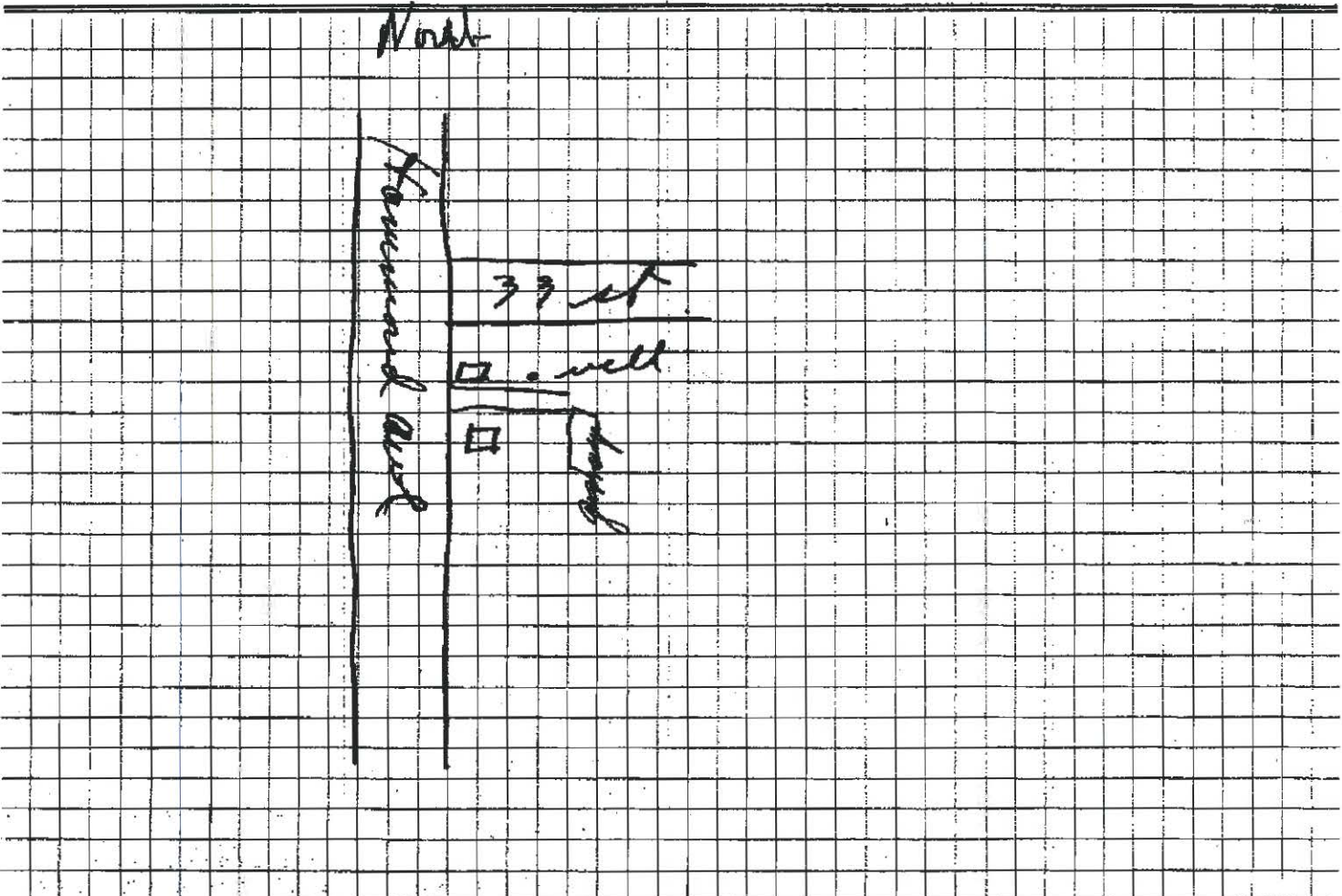
The square below represents a section of land divided into 40 acre tracts. Mark the position of the premises in the section.



Sec. 25 ^{26?}
 Twp. 49N
 Range 14 } W

DIAGRAM OF PREMISES

See discussion and illustration in Part III Well Drilling Code. In making the diagram in the space below consider 10 ft. as the distance between lines. Be sure to indicate NORTH.



WELL CONSTRUCTOR'S REPORT TO WISCONSIN STATE BOARD OF HEALTH
See Instructions on Reverse Side

RECEIVED
JAN 17 1954

See 36?
T49N
R14W

1. County Douglas Town Village City Check one and give name
2. Location City of Superior, Superior and Bedford ave
Name of street and number of premise or Section, Town and Range numbers
3. Owner or Agent Lake Head Pipeline Co
Name of individual, partnership or firm
4. Mail Address East End Superior Wis
Complete address required
5. From well to nearest: Building ✓ ft; sewer ✓ ft; drain ✓ ft; septic tank ✓ ft;
dry well or filter bed ✓ ft; abandoned well ✓ ft.
6. Well is intended to supply water for: Drinking

7. DRILLHOLE:

Dia. (in.)	From (ft.)	To (ft.)	Dia. (in.)	From (ft.)	To (ft.)

8. CASING AND LINER PIPE OR CURBING:

Dia. (in.)	Kind and Weight	From (ft.)	To (ft.)
4	standard	0	179

9. GROUT:

Kind	From (ft.)	To (ft.)

11. MISCELLANEOUS DATA:

Yield test: 5 Hrs. at 7 GPM.
Depth from surface to water-level: _____ ft.
Water-level when pumping: same ft.
Water sample was sent to the state laboratory at:
Superior on 19
city

10. FORMATIONS:

Kind	From (ft.)	To (ft.)
Red clay	0	135
Hard pan	135	175
water gravel	175	179

Construction of the well was completed on:

Oct 9 1953

The well is terminated 22 inches
 above, below the permanent ground surface.

Was the well disinfected upon completion?

Yes No _____

Was the well sealed watertight upon completion?

Yes No _____

Signature Wattman Bros
Registered Well Driller

Complete Mail Address Wattman Bros

Please do not write in space below

Rec'd _____ No. _____
Ans'd _____
Interpretation _____
22-14

10 ml _____ 10 ml _____ 10 ml _____ 10 ml _____ 10 ml _____
Gas—24 hrs. _____
48 hrs. _____
Confirm _____
B. Coli _____
Examiner _____

WISCONSIN UNIQUE WELL NUMBER
Source: WELL CONSTRUCTION **NK943**

State of Wi-Private Water Systems-DG/2 Form 3300-77A
 Department Of Natural Resources, Box 7921 (Rev 02/02)bw
 Madison, WI 53707

Property Owner **SMITH, KEN** Telephone Number **715-398-5559**
 Mailing Address **1811 42ND AVE E**
 City **SUPERIOR** State **WI** Zip Code **54880**

1. Well Location Depth **212** FT
 T=Town C=City V=Village Fire#
C of SUPERIOR

County of Well Location **NO** Co Well Permit No **W** Well Completion Date **May 27, 1999**

Street Address or Road Name and Number

Well Constructor License # Facility ID (Public)
THOMAS G BUTTERFIELD 555

Subdivision Name Lot# Block #

Address **14346W STATE RD 77** Public Well Plan Approval#
 City **HAYWARD** State **WI** Zip Code **54843** Date Of Approval
 Hicap Permanent Well # Common Well # Specific Capacity **2** gpm/ft

Gov't Lot or **NW** 1/4 of **NW** 1/4 of
 Section **6** T **48** N R **13** W

2. Well Type 1 (See item 12 below)
 1=New 2=Replacement 3=Reconstruction
 of previous unique well # _____ constructed in _____
 Reason for replaced or reconstructed Well?

3. Well Serves # of homes and or P (eg: barn, restaurant, church, school, industry, etc.) High Capacity: Well? **N** Property? **N**

1 1=Drilled 2=Driven Point 3=Jetted 4=Other

4. Is the well located upslope or sideslope and not downslope from any contamination sources, including those on neighboring properties? Y

- Well located in floodplain? **N**
 Distance in feet from well to nearest: (including proposed)
- | | | |
|---------------------------------|--|--------------------------------------|
| 1. Landfill | 9. Downspout/ Yard Hydrant | 17. Wastewater Sump |
| 8 2. Building Overhang | 10. Privy | 18. Paved Animal Barn Pen |
| 61 3. 1=Septic 2= Holding Tank | 11. Foundation Drain to Clearwater | 19. Animal Yard or Shelter |
| 4. Sewage Absorption Unit | 12. Foundation Drain to Sewer | 20. Silo |
| 5. Nonconforming Pit | 13. Building Drain | 21. Barn Gutter |
| 6. Buried Home Heating Oil Tank | 1=Cast Iron or Plastic 2=Other | 22. Manure Pipe 1=Gravity 2=Pressure |
| 7. Buried Petroleum Tank | 14. Building Sewer 1=Gravity 2=Pressure | 1=Cast iron or Plastic 2=Other |
| 8. 1=Shoreline 2= Swimming Pool | 15. Collector Sewer: ___ units ___ in. diam. | 23. Other manure Storage |
| | 16. Clearwater Sump | 24. Ditch |
| | | 25. Other NR 812 Waste Source |

5. Drillhole Dimensions and Construction Method

From		To	Upper Enlarged Drillhole	Lower Open Bedrock
Dia.(in.)	(ft)	(ft)		
4.0	surface	212	1. Rotary - Mud Circulation	
			2. Rotary - Air	
			3. Rotary - Air and Foam	
			4. Drill-Through Casing Hammer	
			5. Reverse Rotary	
			6. Cable-tool Bit n. dia	
			7. Temp. Outer Casing in. dia. depth ft. Removed?	
			Other	

8. Geology

Geology Codes	Type, Caving/Noncaving, Color, Hardness, etc	From (ft.)	To (ft.)
T_C_	TAN CLAY	0	10
R_C_	RED CLAY	10	27
T_C_	MED BRN CLAY (HARD) W/ SOFT	27	77
G_C_	GREY CLAY	77	104
HC	MED BRN CLAY (HARD)	104	167
R_Y_	RED SAND & GRAVEL	167	212

6. Casing Liner Screen

Dia. (in.)	Material, Weight, Specification	From (ft.)	To (ft.)
4.0	NEW P&E BLK WELDED ASTMA-53B 10:79 LB/FT SAWHILL	surface	212

9. Static Water Level **44.0** feet **B** ground surface
 A=Above B=Below

11. Well Is: 14 in. A Grade
 A=Above B=Below

Developed? **Y**
 Disinfected? **Y**
 Capped? **Y**

7. Grout or Other Sealing Material

Method	From (ft.)	To (ft.)	# Sacks Cement
MOUNDED	surface	20.0	2 S

10. Pump Test
 Pumping level **85.0** ft. below surface
 Pumping at **10.0** GP M **1.0** Hrs

12. Did you notify the owner of the need to permanently abandon and fill all unused wells on this property? N
 If no, explain **2 HOMES WERE ON ONE WELL**

13. Initials of Well Constructor or Supervisory Driller **TB** Date Signed **6/2/99**
 Initials of Drill Rig Operator (Mandatory unless same as above) **TB** Date Signed **6/2/99**

WISCONSIN UNIQUE WELL NUMBER
Source: WELL CONSTRUCTION **TF532**

State of WI-Private Water Systems-DG/2 Form 3300-77A
 Department Of Natural Resources, Box 7921 (Rev 02/02)bw
 Madison, WI 53707

Property Owner **ASHLEY, PAUL** Telephone Number **715-398-6597**
 Mailing Address **1521 E 4TH ST**
 City **SUPERIOR** State **WI** Zip Code **54880**
 County of Well Location **NO** Co Well Permit No **W** Well Completion Date **October 17, 2005**

1. Well Location Depth **220** FT
 T=Town C=City V=Village C of **SUPERIOR** Fire#

Well Constructor **MATT H LONG** License # **145** Facility ID (Public)
 Address **LONG'S WELL DRILLING** Public Well Plan Approval#
 City **POPLAR** State **WI** Zip Code **54864** Date Of Approval
 Hicap Permanent Well # Common Well # Specific Capacity **.3** gpm/ft

Street Address or Road Name and Number **39TH AVE E**
 Subdivision Name Lot# Block #

Gov't Lot or **SE** 1/4 of **SE** 1/4 of Section **31** T **49** N R **13** W

2. Well Type **1** (See item 12 below)
 1=New 2=Replacement 3=Reconstruction
 of previous unique well # _____ constructed in _____
 Reason for replaced or reconstructed Well?
1 1=Drilled 2=Driven Point 3=Jetted 4=Other

3. Well Serves # of homes and or **P** (eg: barn, restaurant, church, school, industry, etc.)
 High Capacity Well? **N** Property? **N**
 M=Munic O=OTM N=NonCom P=Private Z=Other X=NonPot A=Anode L=Loop H=Drillhole

4. Is the well located upslope or sideslope and not downslope from any contamination sources, including those on neighboring properties? **Y**
 Well located in floodplain? **N**
 Distance in feet from well to nearest: (including proposed)

1. Landfill	9. Downspout/ Yard Hydrant	17. Wastewater Sump
2. Building Overhang	10. Privy	18. Paved Animal Barn Pen
3. 1=Septic 2= Holding Tank	11. Foundation Drain to Clearwater	19. Animal Yard or Shelter
4. Sewage Absorption Unit	12. Foundation Drain to Sewer	20. Silo
5. Nonconforming Pit	13. Building Drain	21. Barn Gutter
6. Buried Home Heating Oil Tank	14. Building Sewer 1=Cast Iron or Plastic 2=Other	22. Manure Pipe 1=Gravity 2=Pressure
7. Buried Petroleum Tank	15. Collector Sewer: ___ units ___ in. diam.	23. Other manure Storage
8. 1=Shoreline 2= Swimming Pool	16. Clearwater Sump	24. Ditch
		25. Other NR 812 Waste Source

5. Drillhole Dimensions and Construction Method

From To		Upper Enlarged Drillhole	Lower Open Bedrock
Dia. (in.)	(ft)	(ft)	(ft)
8.5	surface	63	
6.0	163	220	

X - 1. Rotary - Mud Circulation
 - 2. Rotary - Air
 - 3. Rotary - Air and Foam
 - 4. Drill-Through Casing Hammer
 - 5. Reverse Rotary
 - 6. Cable-tool Bit n. dia
 - 7. Temp. Outer Casing in. dia. ___ depth ft. Removed?
 Other

8. Geology

Geology Codes	Type, Caving/Noncaving, Color, Hardness, etc	From (ft.)	To (ft.)
<u>C</u>	CLAY	0	144
<u>P</u>	HARDPAN	144	159
<u>PB</u>	HARDPAN/SANDSTONE MIX	159	161
<u>HN</u>	SANDSTONE/HARD & CLEAN	161	220

6. Casing Liner Screen Material, Weight, Specification From To
 Dia. (in.) Manufacturer & Method of Assembly (ft.) (ft.)

6.0	WHEATLAND A53 SCHD 40 WELD	surface	163
-----	----------------------------	---------	-----

Dia. (in.) Screen type, material & slot size From To

9. Static Water Level **35.0** feet **B** ground surface
 A=Above B=Below
11. Well Is: 24 in. A Grade
 A=Above B=Below
 Developed? **Y**
 Disinfected? **Y**
 Capped? **Y**

7. Grout or Other Sealing Material

Method	DRILLING MUD	From (ft.)	To (ft.)	# Sacks Cement
Kind of Sealing Material	QUIK GEL	surface	163.0	

10. Pump Test
 Pumping level **85.0** ft. below surface
 Pumping at **15.0** GP M **4.0** Hrs
12. Did you notify the owner of the need to permanently abandon and fill all unused wells on this property?
 If no, explain **NONE**
13. Initials of Well Constructor or Supervisory Driller **MHL** Date Signed **10/20/05**
 Initials of Drill Rig Operator (Mandatory unless same as above) Date Signed

WISCONSIN UNIQUE WELL NUMBER
Source: WELL CONSTRUCTION

TJ253

State of Wi-Private Water Systems-DG/2
 Department Of Natural Resources, Box 7921
 Madison, WI 53707

Form 3300-77A
 (Rev 02/02)bw

Property Owner **NEMADJI PUBLIC GOLF** Telephone Number - - -
 Mailing Address **5 N 58TH ST E**
 City **SUPERIOR** State **WI** Zip Code **54880**
 County of Well Location **NO** Co Well Permit No **W** Well Completion Date **July 19, 2006**

1. Well Location Depth **260** FT
 T=Town C=City V=Village
 C of **SUPERIOR** Fire# **5 N**

Well Constructor **KEITH R LIND** License # **4684** Facility ID (Public)
 Address **KEITH LIND WELL DRLG INC** Public Well Plan Approval#
 City **MAPLE** State **WI** Zip Code **54854** Date Of Approval
 Hicap Permanent Well # Common Well # Specific Capacity **.2** gpm/ft

Street Address or Road Name and Number
58TH ST
 Subdivision Name Lot# Block #

Gov't Lot or **NW** 1/4 of **NW** 1/4 of
 Section **1** T **48 N** R **14 W**

2. Well Type 1 (See item 12 below)
 1=New 2=Replacement 3=Reconstruction
 of previous unique well # _____ constructed in _____
 Reason for replaced or reconstructed Well?
 1. 1=Drilled 2=Driven Point 3=Jetted 4=Other

3. Well Serves # of homes and or POND
X (eg: barn, restaurant, church, school, industry, etc.) High Capacity Well? **N**
 Property? **N**
 M=Munic O=OTM N=NonCom P=Private Z=Other X=NonPot A=Anode L=Loop H=Drillhole

4. Is the well located upslope or sideslope and not downslope from any contamination sources, including those on neighboring properties? Y
 Well located in floodplain? **N**
 Distance in feet from well to nearest: (including proposed)

1. Landfill	9. Downspout/ Yard Hydrant	17. Wastewater Sump
2. Building Overhang	10. Privy	18. Paved Animal Barn Pen
3. 1=Septic 2= Holding Tank	11. Foundation Drain to Clearwater	19. Animal Yard or Shelter
4. Sewage Absorption Unit	12. Foundation Drain to Sewer	20. Silo
5. Nonconforming Pit	13. Building Drain	21. Barn Gutter
6. Buried Home Heating Oil Tank	14. Building Sewer 1=Cast Iron or Plastic 2=Other	22. Manure Pipe 1=Gravity 2=Pressure
7. Buried Petroleum Tank	15. Collector Sewer: ___ units ___ in. diam.	23. Other manure Storage
100 8. 1=Shoreline 2= Swimming Pool	16. Clearwater Sump	24. Ditch
		25. Other NR 812 Waste Source

5. Drillhole Dimensions and Construction Method

Dia.(in.)	From (ft)	To (ft)	Upper Enlarged Drillhole	Lower Open Bedrock
6.0	surface	231	- 1. Rotary - Mud Circulation	
			- 2. Rotary - Air	
			- 3. Rotary - Air and Foam	
6.0	231	260	X - 4. Drill-Through Casing Hammer	
			- 5. Reverse Rotary	
			- 6. Cable-tool Bit ___ in. dia	
			- 7. Temp. Outer Casing ___ in. dia. ___ depth ft. Removed?	
			Other	

8. Geology

Geology Codes	Type, Caving/Noncaving, Color, Hardness, etc	From (ft.)	To (ft.)
__C__	CLAY	0	130
__P__	HARDPAN	130	150
T_SU	DIRTY SAND-BROWN	150	156
__P__	HARDPAN	156	171
T_SU	DIRTY SAND-BROWN	171	175
G_C	GRAY CLAY	175	229
__G__	GRAVEL	229	231
__N__	SANDSTONE	231	260

6. Casing Liner Screen

Dia. (in.)	Material, Weight, Specification	From (ft.)	To (ft.)
6.0	PLAIN END WHEATLAND ASTM A53B .280 18.97	surface	231
Dia.(in.)	Screen type, material & slot size	From	To

9. Static Water Level **44.0** feet **B** ground surface
 A=Above B=Below
11. Well Is: 24 in. A Grade
 A=Above B=Below
 Developed? **Y**
 Disinfected? **Y**
 Capped? **Y**

7. Grout or Other Sealing Material

Method	From (ft.)	To (ft.)	# Sacks Cement
MOUNDED			
Kind of Sealing Material	From (ft.)	To (ft.)	# Sacks Cement
BENTONITE GRAN	surface	231.0	6 S

10. Pump Test
 Pumping level **200.0** ft. below surface
 Pumping at **35.0** GP M **3.0** Hrs
12. Did you notify the owner of the need to permanently abandon and fill all unused wells on this property?
 If no, explain **NA**
13. Initials of Well Constructor or Supervisory Driller **KL** Date Signed **7/30/06**
 Initials of Drill Rig Operator (Mandatory unless same as above) Date Signed

WISCONSIN UNIQUE WELL NUMBER
Source: WELL CONSTRUCTION **VE161**

State of WI-Private Water Systems-DG/2 Form 3300-77A
 Department Of Natural Resources, Box 7921 (Rev 02/02)bw
 Madison, WI 53707

Property Owner **ENBRIDGE ENERGY US** Telephone Number - -
 Mailing Address **10 BARDON AVE**
 City **SUPERIOR** State **WI** Zip Code **54880**
 County of Well Location **NO** Co Well Permit No **W** Well Completion Date **July 29, 2010**

1. Well Location Depth **260** FT
 T=Town C=City V=Village Fire#
C of SUPERIOR

Street Address or Road Name and Number
10 BARDON AVE
 Subdivision Name Lot# Block #

Well Constructor **KEITH R LIND** License # **4684** Facility ID (Public)
 Address **KEITH LIND WELL DRLG INC** Public Well Plan Approval#
 City **MAPLE** State **WI** Zip Code **54854** Date Of Approval
 Hicap Permanent Well # Common Well # Specific Capacity **2** gpm/ft

Gov't Lot or **NW** 1/4 of **SE** 1/4 of
 Section **36** T **49** N R **14** W

2. Well Type **1** (See item 12 below)
 1=New 2=Replacement 3=Reconstruction
 of previous unique well # _____ constructed in _____
 Reason for replaced or reconstructed Well?
1 1=Drilled 2=Driven Point 3=Jetted 4=Other

3. Well Serves # of homes and or **SHOP**
N (eg: barn, restaurant, church, school, industry, etc.) High Capacity: Well? **N**
 Property? **N**
 M=Munic O=OTM N=NonCom P=Private Z=Other X=NonPot A=Anode L=Loop H=Drillhole

4. Is the well located upslope or sideslope and not downslope from any contamination sources, including those on neighboring properties?
 Well located in floodplain? **N**
 Distance in feet from well to nearest: (including proposed)

1. Landfill	9. Downspout/ Yard Hydrant	17. Wastewater Sump
2. Building Overhang	10. Privy	18. Paved Animal Barn Pen
101 3. 1=Septic 2= Holding Tank	11. Foundation Drain to Clearwater	19. Animal Yard or Shelter
4. Sewage Absorption Unit	12. Foundation Drain to Sewer	20. Silo
5. Nonconforming Pit	13. Building Drain	21. Barn Gutter
6. Buried Home Heating Oil Tank	1=Cast Iron or Plastic 2=Other	22. Manure Pipe 1=Gravity 2=Pressure
7. Buried Petroleum Tank	14. Building Sewer 1=Gravity 2=Pressure	1=Cast iron or Plastic 2=Other
8. 2 1=Shoreline 2= Swimming Pool	15. Collector Sewer: ___ units ___ in . diam.	23. Other manure Storage
	16. Clearwater Sump	24. Ditch
		25. Other NR 812 Waste Source

5. Drillhole Dimensions and Construction Method

From (ft)	To (ft)	Upper Enlarged Drillhole	Lower Open Bedrock
8.8	surface	256	
6.0	256	260	

X - 1. Rotary - Mud Circulation
 - 2. Rotary - Air
 - 3. Rotary - Air and Foam
 - 4. Drill-Through Casing Hammer
 - 5. Reverse Rotary
 - 6. Cable-tool Bit ___ n. dia
 - 7. Temp. Outer Casing ___ in. dia. ___ depth ft.
 Removed?
 Other

8. Geology

Geology Codes	Type, Caving/Noncaving, Color, Hardness, etc	From (ft)	To (ft)
__C_	CLAY	0	141
__SU	DIRTY MUDDY SAND	141	145
__PG	HARD PAN & BOULDERS	145	171
__SU	MUDDY SAND	171	173
__PG	HARD PAN & BOULDERS	173	253
__SM	SILTY SAND	253	256
__NS_	FINE SAND	256	260

6. Casing Liner Screen

Dia. (in.)	Material, Weight, Specification	From (ft.)	To (ft.)
6.0	PLAINEND EXELL ASTM A53B .280 18.97	surface	256
3.0	Screen type, material & slot size 10 SLOT STAINLESS STEEL	256	260

9. Static Water Level
49.0 feet **B** ground surface
 A=Above B=Below

11. Well Is: 28 in. A Grade
 A=Above B=Below

Developed? **Y**
 Disinfected? **Y**
 Capped? **Y**

10. Pump Test
 Pumping level **94.0** ft. below surface
 Pumping at **8.0** GP M **4.0** Hrs

12. Did you notify the owner of the need to permanently abandon and fill all unused wells on this property?
 If no, explain **NA**

7. Grout or Other Sealing Material

Method	From (ft.)	To (ft.)	# Sacks Cement
PUMPING			
Kind of Sealing Material			
BENTONITE CUTTINGS	surface	256.0	S
CAVING FORMATION	256.0	260.0	S

13. Initials of Well Constructor or Supervisory Driller **KL** Date Signed **7/29/10**
 Initials of Drill Rig Operator (Mandatory unless same as above) Date Signed

WISCONSIN UNIQUE WELL NUMBER
Source: WELL CONSTRUCTION **VH933**

State of Wi-Private Water Systems-DG/2 Form 3300-77A
 Department Of Natural Resources, Box 7921 (Rev 02/02)bw
 Madison, WI 53707

Property Owner **ENBRIDE US ENERGY** Telephone Number - -
 Mailing Address 10 BARDON AVE
 City SUPERIOR State WI Zip Code 54880
 County of Well Location NO Co Well Permit No W Well Completion Date October 7, 2010

1. Well Location Depth **163** FT
 T=Town C=City V=Village Fire#
 C of SUPERIOR

Well Constructor BUTTERFIELD, TIM DRILLING INC License # 6900 Facility ID (Public)
 Address 395 REED ST Public Well Plan Approval#
 City SOMERSET State WI Zip Code 54025 Date Of Approval
 Hicap Permanent Well # Common Well # Specific Capacity gpm/ft

Street Address or Road Name and Number BARDON AVE
 Subdivision Name Lot# Block #

Gov't Lot or **NW** 1/4 of **SE** 1/4 of
 Section **36** T **49** N R **14** W

2. Well Type 1 (See item 12 below)
 1=New 2=Replacement 3=Reconstruction
 of previous unique well # _____ constructed in _____
 Reason for replaced or reconstructed Well?

3. Well Serves # of homes and or **BUILDING**
 N (eg: barn, restaurant, church, school, industry, etc.)
 High Capacity: Well? N Property? N

1 1=Drilled 2=Driven Point 3=Jetted 4=Other

4. Is the well located upslope or sideslope and not downslope from any contamination sources, including those on neighboring properties?
 Well located in floodplain? N
 Distance in feet from well to nearest: (including proposed)

1. Landfill	9. Downspout/ Yard Hydrant	17. Wastewater Sump
100 2. Building Overhang	10. Privy	18. Paved Animal Barn Pen
100 3. 1=Septic 2= Holding Tank	11. Foundation Drain to Clearwater	19. Animal Yard or Shelter
4. Sewage Absorption Unit	12. Foundation Drain to Sewer	20. Silo
5. Nonconforming Pit	13. Building Drain	21. Barn Gutter
6. Buried Home Heating Oil Tank	1=Cast Iron or Plastic 2=Other	22. Manure Pipe 1=Gravity 2=Pressure
7. Buried Petroleum Tank	14. Building Sewer 1=Gravity 2=Pressure	1=Cast iron or Plastic 2=Other
8. 2 1=Shoreline 2= Swimming Pool	15. Collector Sewer: ___ units ___ in. diam.	23. Other manure Storage
	16. Clearwater Sump	24. Ditch
		25025. Other NR 812 Waste Source FUEL TANK

5. Drillhole Dimensions and Construction Method

From		To	Upper Enlarged Drillhole	Lower Open Bedrock
Dia.(in.)	(ft)	(ft)		
6.0	surface	163	-- 1. Rotary - Mud Circulation -----	
			-- 2. Rotary - Air -----	
			-- 3. Rotary - Air and Foam -----	
			X -- 4. Drill-Through Casing Hammer	
			-- 5. Reverse Rotary	
			-- 6. Cable-tool Bit _____ n. dia -----	
			-- 7. Temp. Outer Casing _____ in. dia. _____ depth ft.	
			Removed?	
			Other	

8. Geology

Geology Codes	Type, Caving/Noncaving, Color, Hardness, etc	From (ft.)	To (ft.)
R_C_	RED CLAY	0	140
S	SAND	140	163

6. Casing Liner Screen

Dia. (in.)	Material, Weight, Specification	From (ft.)	To (ft.)
6.0	NEW P&E BLK WELDED 18.97 LB/FT ASTM-A53B IPSCO	surface	155
Dia.(in.)	Screen type, material & slot size	From	To
3.0	JOHNSON STAINLESS V-WIRE 10 SLOT	155	163

9. Static Water Level 50.0 feet B ground surface A=Above B=Below
11. Well Is: 24 in. A Grade A=Above B=Below
 Developed? Y
 Disinfected? Y
 Capped? Y

7. Grout or Other Sealing Material

Method	From (ft.)	To (ft.)	# Sacks Cement
MOUNDED			
Kind of Sealing Material			
BENTONITE	surface	20.0	2 S

12. Did you notify the owner of the need to permanently abandon and fill all unused wells on this property? N
 If no, explain N/A
13. Initials of Well Constructor or Supervisory Driller TB Date Signed 10/25/10
 Initials of Drill Rig Operator (Mandatory unless same as above) Date Signed

TO THE WISCONSIN STATE BOARD OF HEALTH,
 WELL DRILLING DIVISION, MADISON, WIS.

WELL LOG, PREMISES DIAGRAM, and REPORT

For Official Record of the Board.

(TO BE USED FOR THAT PURPOSE ONLY)

Owner Park Board of City of Superior Driller Masterson Bros
 (If a joint ownership give name of responsible official. Also name of each individual holding an interest. Use a separate sheet and attach hereto.)
 Address Superior Wis Address 714 Duquette Ave Superior Wis
 (City, village, township, county) Date of Report June 29 1937
 Registration No. 232

Give below the location of the property on which well is drilled.
 If incorporated village or city: City of Superior Blk. 4 Street and No. E. 7th St
 If unincorporated hamlet Name County Twp. Highway
 If Lake Shore Plat Name of Plat Lake Lot Sec. Blk. Street
 If Farm County Twp. Sec. Highway
 If School County Twp. Sec. District
 If other public building Kind County Twp. Sec.
 Miscellaneous Kind County Twp. Sec.

WELL LOG and REPORT

Screens, Seals Grouts, etc.	Well Diagram (Each vertical line equals 1')	Kind of Casing, liner, shoe, etc. (Each horizontal line equals 5')	Formations State if dry or water bearing	Record of FINAL Pumping Test
6 ft Johnson Reel Brass screen 25' dia		Drops forged Liner shoe 6 in Miller Spool casing	Red clay to 85 ft White sand to gravel to 102 ft gravel to 108 ft	Duration of test. Hours <u>10 hr</u> Pumping Rate. G. P. M. <u>60</u> Depth of pump in well. Ft. <u>25 ft</u> Standing water-level (from surface.) Ft. <u>3 ft</u> Water level when pumping Ft. <u>5 ft</u> Water. End of test. Check: Clear <input checked="" type="checkbox"/> Cloudy <input type="checkbox"/> Turbid <input type="checkbox"/> Was well sterilized before test? Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> Date _____ To which Laboratory was sample sent? <u>Superior Wis</u> Date <u>June 29-37</u> Was the well sealed on completion? Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> How high did you leave casing above grade? <u>Cased 1 ft below grade</u> Well was completed <u>June 29</u> 19 <u>37</u> Well Driller: <u>Masterson Bros</u> Signature. (Be sure to complete the report on the reverse side)
		Flowing Well flowing 9 gal per min 3 feet gravel to 102 ft Below grade at well and 7 ft about <u>Rein level</u> Pump house to be built when pumps to be installed		

Appendix B

Barr Standard Operating Procedures (SOPs)

Appendix B

Index of Standard Operating Procedures (SOP)

Site Investigation Work Plan Superior Refinery April 2018 Explosion and Fire Superior, Wisconsin

Contents:

Barr Engineering SOP Title
Field Screening Soil Samples
Collection of Soil Samples
Decontamination of Sampling Equipment
Collection and Disposal of Investigative Derived Waste
Documentation on a Chain-of-Custody Form
Domestic Transport of Samples to Laboratories within the USA - States and Territories
Routine Level SVOC, PAH, DRO, and TPH Data Evaluation
Routine Level VOC, GRO, and TPH Data Evaluation




Standard Operating Procedure Field Screening Soil Samples

Revision 8

April 9, 2019

Approved By:

John W. Juntilla		04/09/19
Print	Technical Reviewer	Date
Signature		

Terri A. Olson		04/09/19
Print	QA Manager	Date
Signature		

Review of the SOP has been performed and the SOP still reflects current practice.	
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

Field Screening of Soil Samples

1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedure for properly screening soil or sediment samples in the field. This procedure applies to field technicians responsible for field screening soil or sediment samples.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Screening techniques can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance, if applicable.
- Interferences on the test can be caused by any contaminant that can cause an oil sheen on water. The samples will be carefully observed for characteristic appearance or odors which may indicate a possible contaminant other than coal tar or petroleum substances.
- Sunlight and low temperatures may interfere with headspace development.
- Water and soil particles may interfere with PID and FID measurements.
- Decontamination of screening equipment is required to prevent cross-contamination.
- Contact the local one call system prior to digging to have public utilities identified at sampling locations. Privately owned underground utilities, if present, typically will not be identified by the one call system and contracting with a private utility locator may be necessary.

3.0 Responsibilities

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, SAP, etc.).

Experienced Field Technicians are responsible for the proper sample identification, field screening procedures, field equipment and calibration, quality control procedures, and documentation.

Equipment Technicians are responsible for maintaining equipment in working order and aiding in troubleshooting equipment issues.

The role of the Field Safety Representative is to oversee on-site safety activities.

4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent

sample contact with the skin and eyes. When screening soils contaminated with corrosive materials, emergency eye flushing facilities should be available.

Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

5.0 Equipment, Reagents, and Supplies

- Photoionization detector (PID)
- Flame ionization detector (FID)
- Squirt bottle with tap water
- Waterproof ink pen or pencil
- Polyethylene bags
- Chemical resistant gloves (e.g., nitrile)
- Stainless steel spoon
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

6.0 Procedure

The field screening techniques for soils are as follows: visual examination, odor, headspace organic vapor screening, and oil sheen. The results of these four screening procedures may be used to screen soil samples for possible contamination.

6.1 Calibration

The PID or FID shall be calibrated or checked against a known concentration of a calibration gas standard prior to collection of field measurements. Calibration of the PID or FID shall follow the recommended procedures as described in the manufacturer's operation manual or as per the applicable Barr SOP.

Regular calibration checks (bump tests) are expected to be performed by the field technician a minimum of once per day of use in the field. It is recommended that bump tests be conducted around mid-day and at the end of the day. More frequent bump testing may be completed if warranted by field conditions. The bump testing results should be recorded in the field log book or field log data sheets.

If problems occur during calibration, during bump tests, or if the unit will not stay calibrated, the field technician should document the issue in the field notes then contact the equipment technician or project manager for assistance.

6.2 Screening Techniques

The field screening techniques for soils are as follows: visual examination, odor, headspace organic vapor screening, and oil sheen. The results of these four screening procedures may be used to screen soil samples for possible contamination. To prevent sample cross-contamination, the screening equipment is carefully cleaned before and after working with each sample per Barr's SOP 'Decontamination of Sampling Equipment'.

6.2.1 Visual Examination

A visual examination of the soil sample will include noting any discoloration of the soil or visible oiliness or tar.

6.2.2 Odor

The field technician will note odor only if noticed incidentally while handling the soil sample. Field technicians will not unduly expose themselves to sample odors. Odor will be described as trace, light, moderate, or strong, and appropriate description of the type of odor, if evident.

6.2.3 Headspace Organic Vapor Screening

The polyethylene bag headspace method recommended by the Minnesota Pollution Control Agency will be used in the field to screen soils suspected to contain volatile organic compounds. The screening method is intended to be used in conjunction with other “real time” observations.

The following equipment is required to conduct headspace organic vapor screening: PID or FID, polyethylene bag, log book or record sheet, and appropriate PPE. Soil samples collected from a split-barrel sampler or a direct-push (i.e., Geoprobe) sample liner will be collected immediately after opening the barrel or liner. If the sample is collected from an excavation wall, soil pile, or backhoe bucket, it will be collected from a freshly exposed surface.

- Half-fill the bag with the sample to be analyzed using a stainless-steel spoon or a gloved hand and immediately seal it. Agitate the bag for 15 seconds and manually break up any soil clumps within the bag.
- Allow headspace development for approximately 10 minutes. The sample should be kept in a shaded area out of direct sunlight. Ambient temperatures during headspace development should be recorded. When ambient temperatures are below 50°F, headspace development should be conducted inside a heated vehicle or building. After completing the headspace development, agitate the bag for an additional 15 seconds.
- Quickly puncture the bag with the sampling probe of the PID or FID at a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particles.
- Record the highest PID or FID meter response as the headspace concentration. The maximum response will likely occur between 0 to 5 seconds.
- When using a FID, it may be necessary to correct for methane. In this case, take a reading first with the carbon filter, then without. This will require two duplicate bag samples. The second reading less the first is the headspace adjusted for methane. Adjusted readings less than zero are considered zero. Methane correction is not necessary if a PID is used.

6.2.4 Oil Sheen Test

The oil sheen or hydrocarbon test is a method used to immediately determine the approximate magnitude of coal tar or petroleum contamination in soil by observation of the sample in the field. The test is useful in soils which do not have a high binding capacity with petroleum compounds or polycyclic aromatic hydrocarbons (PAHs) (i.e., petroleum compounds or PAHs are free on the surface of the soil particles and can be released by a stream of water).

The equipment required to conduct the oil sheen test includes: a stainless-steel spoon, a squirt bottle filled with tap water, a log book or field log data sheet, and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan.

The procedure for conducting the oil sheen test consists of obtaining approximately 50 grams (about 30 cc) of representative soil with the spoon and then directing a stream of water onto the soil in the spoon with the squirt bottle until the soil is saturated and water begins to collect around the soil. The amount of oil sheen present on the water is determined by observation and the results of the test are reported as a magnitude of oil sheen observed: none, trace, light, moderate, heavy or rainbow. The test results, sample location, and observations of the sample’s appearance and odor are recorded in the log book or field log data sheet.

The specific soil types at the area of investigation should be accounted for when performing the oil sheen test. The best results are obtained in silts, sands, and/or gravels with low organic content. The results obtained from clay soils may appear deceptively low. Typical descriptions of each test result are provided in the table below.

Oil Sheen Test Result	Description
None	No sheen detected.
Trace	Possible or faint oil sheen observed (may not continue to generate sheen as additional water is added).
Light	Obvious sheen that may not cover entire water surface
Moderate	Definite oil sheen that covers entire surface, but "rainbow colors" not distinguishable.
Heavy	Definite oil film or product that does not display rainbow colors.
Rainbow	Definite oil sheen, film or product that displays rainbow colors.

6.3 Data Reduction/Calculations

No data reduction or calculations are associated with this procedure.

6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

7.0 Quality Control and Quality Assurance (QA/QC)

Field background readings are measured for the headspace organic vapor screening. PID and FID readings should be duplicated every 20 field samples.

8.0 Records

The field technician(s) will document the field screening activities and measurements in a project dedicated field logbook or on field log data sheets.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Field Sampling Report
- Field Log Data Sheet

Field documentation are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual."

Other Barr SOP subjects referenced within this SOP: PID and FID equipment, decontamination of sampling equipment, and investigative derived waste.

9.0 References

PID and FID operation manuals.



Standard Operating Procedure Collection of Soil Samples

Revision 9

March 20, 2019

Approved By:

Kevin McGilp  03/20/19

Print Technical Reviewer Signature Date

Terri Olson  03/20/19

Print QA Manager Signature Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

Collection of Soil Samples

1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the collection of a representative soil sample using a variety of methods (including compositing of discrete samples) and equipment depending on the depth and type of sample required. This procedure applies to the collection of soil samples for volatiles (VOC), semivolatiles (SVOC), general chemistry, and metals analyses.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Sample collection methods can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance.
- Inadequate homogenization of the samples, where applicable, can result in non-representative samples and results.
- Decontamination of sampling equipment is required to prevent cross-contamination.
- Contact the local one call system prior to digging to have public utilities identified at sampling locations. Privately owned underground utilities, if present, typically will not be identified by the one call system and contracting with a private utility locator may be necessary.
- If sampling for per- and polyfluorinated alkyl substances (PFAS), special consideration must be taken to avoid accidental contamination of environmental samples - see Barr's SOP 'Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples'.

3.0 Responsibilities

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, SAP, etc.).

Experienced Field Technicians are responsible for the proper sample identification, collection of samples, field screening procedures, field equipment and calibration, quality control procedures, and documentation.

Equipment Technicians are responsible for maintaining equipment in working order and aiding in troubleshooting equipment issues.

The role of the Field Safety Representative is to oversee on-site safety activities.

Project staff are responsible for ordering sample containers prior to the sampling event.

4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected

contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling soils contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

5.0 Equipment, Reagents, and Supplies*

- Sampling devices/tools
- Stainless steel mixing bowl and spoon
- Sample containers (method specific)
- Balance
- Coolers
- Plastic bags
- Chemical resistant gloves (e.g., nitrile)
- Paper towels/laboratory tissues
- Waterproof ink pen or pencil
- Ice
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

* See Barr's PFAS SOP for a list of prohibited and acceptable items.

6.0 Procedure

This section describes the procedure(s) for the sampling, handling, and delivery of soil samples.

6.1 Calibration

No specific calibration procedures are required for the actual sampling equipment; however, the calibration of the balance should be verified prior to use. Refer to the applicable Barr SOP.

6.2 Sampling

General considerations to be taken into account when planning and conducting sampling operations are the required sample weight, sample holding times, sample handling, and special precautions for trace contaminant sampling.

To prevent sample cross-contamination, the soil sampling equipment is carefully cleaned before initially sampling and after working at each sampling point per Barr's SOP 'Decontamination of Sampling Equipment'. A new, clean outer pair of disposable gloves will be worn for each sample location and sample containers are placed in separate plastic bags after collecting, preserving and tagging. Sample collection activities will typically proceed progressively from the least contaminated area to the most contaminated area (when known).

Depending on the project work to be done, soil samples will be collected for analysis by either a drilling apparatus (equipped with a split spoon or core barrel sampler), hand excavation (hand auger, trowel, or shovel), or direct-push (Geoprobe[®]) technology

- If a drilling apparatus was used, retrieve the split spoon or core barrel sampler from the desired sampling interval and open. If a liner (sleeve) is present and will not be sampled in the field, wrap the ends of the liner with heavy-duty aluminum foil, taking care to not pierce the foil. Tape the foil to the liner with duct tape to seal. Cover the ends of the liner with plastic caps or duct tape to

fully protect the foil and package for shipment to the laboratory. If a liner is being sampled in the field, open the liner to sample the soil.

- If hand excavating, dig with a trowel or shovel to the desired sampling interval and expose a fresh soil surface to sample. Collect a large sample on a shovel and bring it to the surface or collect the sample directly from the fresh soil surface. The hand excavation technique may be done from the bucket of a backhoe also.
- If direct-push (Geoprobe®) technology is used, soils are typically sampled following the subcontractor's soil sampling procedures. This method generally utilizes a direct-push soil boring rig, steel drive rods and a 2-inch outside diameter (O.D.) soil core sampler with a dedicated 1.75-inch inside diameter (I.D) removable acetate plastic sampler liner. The probe rods and sampling unit are driven to the desired sampling depth by the static weight of the carrier vehicle and hydraulic hammer percussion. Two, four, or five-foot sample cores are typically collected. The assembly is brought to the surface and the soil sample is exposed by cutting open the sampler liner.

In most investigations, the soil samples are field screened for moisture, odor, oil sheen, discoloration and the presence of organic soil vapors and classified in accordance with ASTM D-2488, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure). Refer to Barr's SOP 'Screening Soil Samples'.

The form 'Soil Sampling Guidelines' lists the analyses (in order of collection) and describes the preservation, container, and holding time for the most common sampling media (information can vary depending on the laboratory used). The container size, type, preservative, and holding time are important considerations in sample collection. Sample and container size must be adequate to meet laboratory requirements for quality control, split samples, or repeat analyses. The container type varies with the analysis required. Typically, the analytical laboratory will preserve the container before shipment, where applicable. Preservation and shelf life vary; contact the laboratory to determine if an on-hand container is still useful.

Both discrete and composite samples can be used for environmental investigations. A discrete sample is a sample that originated from a specific area at a specific time. The sample may be transferred directly from the sampler or sampling location to the sample container.

A composite sample is a collection of multiple temporary or discrete samples of the same medium that are combined, thoroughly homogenized, and treated as a single sample. Composite samples are valuable in characterizing a large area or volume of soil.

Note: Samples collected for analysis of volatile organic compounds (VOC) should not be homogenized or composited, due to aeration of the sample during mixing which may result in loss of VOC.

6.2.1 Volatile Organic Compounds (VOC)

If VOC or similar analyses (e.g., GRO, TPH as Gasoline) are being analyzed, these samples should be collected as soon as possible after the soil is removed from the ground from a representative area of the most undisturbed soil possible. Please refer to Barr's SOP 'Screening Soil Samples'. It is important to note that there are different containers and sampling media available for collecting a soil sample for VOC. Typically, the VOC sample is collected at a 1:1 weight ratio with a preservative. A coring device, such as a

Terra Core® or En Core® sampler, is the first choice for sampling. After VOC samples are collected, mix the remaining soil from the sampling locations/intervals prior to filling the rest of the sample containers.

Note: Analytical samples should not be collected from polyethylene bags used for field screening purposes.

6.2.1.1 Terra Core® Sampler

The Terra Core® Sampler is a single use device that is typically supplied with a 40 mL VOA (volatile organic analysis) vial containing preservative (e.g., methanol) and an unpreserved container for % moisture/% solids determination. To use the Terra Core®, make certain the plunger is aligned with, and seated in, the handle. Push the Terra Core® into freshly exposed soil until the sample chamber is filled. Depending on the Terra Core® sampler size, a filled chamber will deliver approximately 5 or 10 g of soil. If a 1:1 ratio of soil to preservative is needed, verify the correct size sampler is being used.

Wipe the outside of the sampler, check that the soil plug is flush with the mouth of the sampler, and remove any excess soil. Rotate the plunger 90° until it is aligned with the slots in the body. Extrude the sample into the appropriate container by pushing the plunger down. To provide a good sealing surface, wipe the container lip and screw threads to remove soil and immediately screw on the lid. If preservative is present in the container, swirl to immerse the sample. Record the sample ID on the container and package for shipment to the laboratory.

6.2.1.2 En Core® Sampler

The disposable En Core® sampler is a single use device that is pushed into the soil using a reusable En Core® T-handle. Two, 5 g samplers are typically supplied with an unpreserved container for percent moisture/percent solids determination. Hold the En Core® coring body and push plunger down until the small O-ring rests against the tabs so the plunger moves freely.

Depress the locking lever on the T-handle. Place coring body plunger end first into the open end of the T-Handle, aligning the slots on the coring body with the locking pins in the T-Handle. Twist coring body clockwise to lock pins in slots. Make certain that the sampler is locked in place.

Turn T-handle with T-up and coring body down. This will position the plunger bottom flush with bottom of coring body. Using T-handle, push sampler into soil until coring body is completely full. When full the small O-ring will be centered in the T-handle viewing hole. Remove excess soil from the coring body exterior.

Cap the coring body while it is still on the T-handle by pushing and twisting the cap over the bottom until grooves on locking arms seat over ridge on coring body. Remove the coring body from the T-handle and lock plunger by rotating extended plunger rod fully counterclockwise until wings rest firmly against tabs.

Attach the accompanying label and package for shipment to the laboratory.

6.2.1.3 Other

If no coring device is available, an estimate of the amount of soil needed to provide the desired weight can be determined. Place an extra laboratory container, disposable weigh boat, paper towel, or laboratory tissue on a balance pan. Using a stainless steel spoon, add the desired weight (10 g or 25 g) of a representative soil sample on the balance. Once the amount has been established, discard the soil used in the estimation and collect the sample as per form 'Soil Sampling Guidelines' or laboratory instructions.

If allowed by applicable regulations for VOC sample collection, the VOC aliquot may be weighed directly into the sample container by placing the pre-weighed sample container on the balance, taring the balance, then adding the appropriate amount of soil to the container to reach the desired aliquot weight. This should be done quickly to reduce the possible loss of VOCs.

6.2.2 Compositing Discrete Samples

Discrete samples, to be used for compositing, are stored at ≤ 6 °C until each individual sample is obtained. A minimum volume of soil obtained during discrete sampling will be dependent on the final analytical requirements for the composite sample and the laboratory requirements.

After discrete samples have been obtained, record the locations to be included in a final composited sample in the field documentation. Appropriate laboratory containers should be labeled with this final sample identifier and the date of collection.

Retrieve the samples selected for compositing from storage. One container from each discrete sample location should remain in storage in case individual sample confirmations are necessary. Empty the entire contents of each container into a stainless steel mixing bowl, removing any large debris or rocks, and mix thoroughly.

6.2.3 SVOC / General Chemistry / Metals

Using either a composited sample or a homogenized, discrete sample, fill the remaining containers in the order listed on form 'Soil Sampling Guidelines'. To reduce potential contamination, samples for PFAS should be collected first. See Barr's SOP 'Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples'. Typically, the soil is packed into the sample jars leaving no headspace. If allowed by applicable regulations, the WIDRO sample may be weighed directly into the sample container by placing the pre-weighed sample container on the balance, taring the balance, then adding the appropriate amount of soil to the container to reach the desired sample weight (~25 g).

Wipe the container lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

6.2.4 Handling

After collection, the samples should be handled as few times as possible. Samplers should use extreme care to ensure that samples are not contaminated. Immediately after samples are collected, they are bubble wrap or bagged and placed in a cooler containing bagged ice. Samples will be kept cold (≤ 6 °C, but not frozen) until receipt at the laboratory, where they are to be stored in a refrigerated area.

Note: Samples may need to be stored indoors in winter to prevent freezing.

6.2.5 Shipment/Delivery

Once the cooler is packed to prevent breaking of containers, the proper COC documentation is relinquished by the sampler, placed into a plastic bag, and included in the cooler.

Samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured.

Custody seals may be present, but at a minimum, the coolers must be taped shut to prevent the lid from opening during shipment.

The coolers must be delivered to the laboratory via hand or overnight delivery courier in accordance with Federal, State and Local transportation regulations and Barr's SOP 'Domestic Transport of Samples to the Laboratory'.

6.3 Data Reduction/Calculations

No data reduction or calculations are associated with this procedure.

6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

7.1 QA/QC Samples

QA/QC samples are defined in Barr's SOP 'Collection of Quality Control Samples'. The sampling frequency should be performed as written in the project scope of work and/or documentation (e.g., Work Plan, SAP, or QAPP).

7.2 Measurement Criteria

No specific criteria apply to the implementation of this SOP.

8.0 Records

The field technician will document the soil sampling event in a project dedicated field logbook or on field log data sheets. The analysis for each container, the number of bottles, and the laboratory used will be documented on the chain-of-custody record. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- COC
- Sample label
- Custody seal (if applicable)
- Field Sampling Report
- Field Log Data Sheet
- Soil Sampling Guidelines (includes sampling order, container, preservation, and holding time)

Field documentation and COC are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual."

Other Barr SOP subjects referenced within this SOP: screening soil samples, balance calibration, collection of QC samples, collection of PFAS samples, decontamination of sampling equipment, investigative derived waste, domestic transport of samples, and documentation on a COC.

9.0 References

USEPA Environmental Response Team. 2000. *SOP for Soil Sampling*.



Standard Operating Procedure Decontamination of Sampling Equipment

Revision 1

March 15, 2018

Approved By:

John W. Juntilla *John W. Juntilla* 03/15/18
Print Technical Reviewer Signature Date

Terri Olson *Terri A. Olson* 03/15/18
Print QA Manager Signature Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

Decontamination of Sampling Equipment

1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to define the process used for decontaminating environmental sampling-related equipment including pumps, meters, and materials coming into contact with actual sampling equipment or with sampling personnel. This procedure is applicable to all personnel who are collecting samples and/or decontaminating sampling and field equipment.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Equipment used once and discarded such as bailers, protective gear, and filtration devices are not part of this SOP.

3.0 Responsibilities

The equipment technician is responsible for ensuring field equipment has been thoroughly decontaminated and prepared for use out in the field. The field technician(s) are responsible for decontamination in the field at each individual sampling point and for ensuring adherence to any investigative derived waste (IDW) project-specific requirements set forth in a QAPP or SAP (if applicable).

The role of the Field Safety Representative is to oversee on-site safety activities.

4.0 Safety

Barr staff is responsible for implementing aspects of the job safely. Where available, refer to the appropriate Project Health and Safety Plan (PHASP) to determine the proper personal protection equipment (PPE) required when using this SOP. Barr staff is responsible for conducting all aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protection equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling soils contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

5.0 Equipment, Reagents, and Supplies

- Non-phosphorus detergent (e.g., Liquinox™)
- Scrub brush made of inert materials
- Oven
- Bucket
- Tap water
- Analyte-free water (e.g., distilled or deionized (DI) water, or equivalent)
- Kimwipes®, or equivalent
- Chemical resistant gloves (e.g., nitrile)
- Spray bottle
- Organic solvent (e.g. methanol)

6.0 Procedure

This section describes the procedure(s) for the decontamination of equipment used to sample water, soil, or air.

6.1 Calibration

Calibration is not applicable to this SOP.

6.2 Operation

Decontamination of sampling equipment will be performed before sampling and after working at each sampling point, if applicable.

6.2.1 Water Sampling Equipment

Equipment that does not contact sample water or the inside of the well should be rinsed with analyte-free water and inspected for remaining particles or surface film. If these are noted, repeat cleaning and rinse procedures.

Equipment that contacts sample water or the inside of the well should be cleaned (inside and outside where possible) with a non-phosphorus detergent solution applied with a spray bottle and/or scrub brush (if needed). Rinse with analyte-free water and containerize with other IDW if required by the SAP or QAPP and inspect for remaining particles or surface film. If these are noted, repeat cleaning and rinse procedures. Shake off remaining water and allow to air dry.

The internal surfaces of pumps and tubing that cannot be adequately cleaned by the above methods alone will also be cleaned by first circulating a non-phosphorus detergent solution through them followed by circulating analyte-free water. Special care will be exercised to ensure that the "rinse" fluids will be circulated in sufficient quantities to completely flush out contaminants and detergents.

When transporting or storing equipment after cleaning, the equipment will be stored in a manner that minimizes the potential for contamination.

6.2.2 Soil/Sediment Sampling Equipment

A variety of samplers (split-barrel, split-barrel with brass liners, piston sampler, backhoe, hand-auger, or shovel) may be used to retrieve soil from sampling locations. The soil sample will either be sealed within the sampler (e.g., collecting volatile samples) or the soil sample will be transferred to laboratory-supplied containers depending on the analysis to be conducted on the soil sample. The equipment required to transfer the soil from the sampler to the laboratory-supplied sample containers includes: stainless-steel

spoons or scoops and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the PHASP.

All soil sampling equipment, including split-barrels, stainless-steel spoons and scoops, will be carefully cleaned before and during sampling with a tap water and non-phosphorus detergent solution, using a brush if necessary to remove particulate matter and films. The equipment is then rinsed three times with tap water and/or three times with analyte-free water. Inspect equipment and repeat procedure if any residual soil or visible contaminants are present. Dry sampler with a Kimwipes®. Organic solvents (e.g., methanol) may be used to aid with desorbing organic material but should be kept to a minimum and must be collected and containerized if used.

At the completion of the work day, the samplers should be decontaminated following the procedure above and stored in a manner that minimizes the potential for contamination.

6.2.3 Air Sampling Equipment

For non-laboratory manifold equipment, methanol soak manifold components for a minimum of two hours. Remove from the methanol bath and place in an oven pre-heated to 90 °C and continue to heat manifold components for at least 3 hours or until interior and exterior surface inspections of the manifold components indicate that they are free of liquid methanol.

6.2.4 Handling

All equipment will be handled in a manner that minimizes cross-contamination between points. After cleaning, the equipment will be visibly inspected to detect any residues or other substances that may exist after normal cleaning. If inspection reveals that decontamination was insufficient, the decontamination procedures will be repeated.

6.3 Data Reduction/Calculations

No data reduction or calculations are associated with this procedure.

6.4 Disposal

IDW generated by this process will be disposed of in accordance with Federal, State and Local regulations and/or as required by project-specific SAP or Work Plan. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

7.1 QA/QC Samples

Decontamination procedures may be monitored through the use of an equipment blank which consists of analyte-free water processed through non-disposable or non-dedicated aqueous or solid sampling equipment after equipment decontamination and before field sample collection. The equipment blank is analyzed for the same parameters as the samples at a project specific frequency (e.g., one per twenty samples).

7.2 Measurement Criteria

Equipment blank results should be below the laboratory's method detection limit or reporting limit (depending on the data quality objectives).

8.0 Records

When required, the field technician(s) will document the field equipment decontamination procedures in a project dedicated field logbook or on field log data sheets.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation is listed in the applicable sample collection SOP.

Field documentation and COC are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual."

Other Barr SOP subjects referenced within this SOP: collection of samples and investigative derived waste.

9.0 References

ASTM. 2015. Standard Practice for Decontamination of Field Equipment Used at Waste Sites.





Standard Operating Procedure Collection and Disposal of Investigative Derived Waste

Revision 6

March 15, 2018

Approved By:

<u>John Juntilla</u>		<u>03/15/18</u>
Print	Technical Reviewer	Date
Signature		

<u>Terri Olson</u>		<u>03/15/18</u>
Print	QA Manager	Date
Signature		

Review of the SOP has been performed and the SOP still reflects current practice.	
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

Collection and Disposal of Investigative Derived Waste

1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to define the procedures for the collection and disposal of investigative derived waste (IDW) generated during field investigation activities. This procedure is applicable to sampling IDW which are materials containing pollutants derived during investigation activities including drill cuttings, drilling fluids, cleaning liquids, waste water, DNAPL, soil and rock samples, protective clothing and equipment, or any other items or materials which are exposed to, or may contain pollutants that must be characterized for off-site disposal.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- IDW can be contaminated with various hazardous substances, characterization may be necessary.

3.0 Responsibilities

The Barr Project Manager is responsible for determining whether any solid or liquid-phase product needs to be containerized and characterized for off-site disposal.

Experienced Field Technicians are responsible for the proper sample identification, collection and management of samples, documentation and sample transport to the laboratory.

The role of the Field Safety Representative is to oversee on-site safety activities.

Project staff are responsible for ordering sample containers prior to the sampling event.

4.0 Safety

Barr staff is responsible for conducting aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protection equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling material contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

5.0 Equipment, Reagents, and Supplies

- Applicable sampling equipment
- Weatherproof container labels
- Plastic garbage bags
- Chemical resistant gloves (e.g., nitrile)
- IDW containers
- Permanent markers
- Plastic covering

6.0 Procedure

The Barr Project Manager is responsible for determining if IDW can be left on-site or if it must be disposed of off-site. Two general objectives that will be considered when managing IDW are the minimization of IDW generation and managing the IDW consistent with the final remedy for the site. The extent to which the objectives can be met is dependent on the site-specific circumstances.

Any IDW that is required to be containerized will be containerized separately by media until laboratory data are received to determine the appropriate disposition of the materials. Containerization and disposal of personal protective equipment and/or other materials, if necessary, will be determined on a project by project basis and discussed in the project Sampling and Analysis Plan (SAP).

6.1 Calibration

Calibration is not applicable to this SOP.

6.2 Sampling

Representative samples will be collected, and/or composited, preserved, and handled following Barr's matrix specific sampling SOP. Sampling equipment will be cleaned following Barr's 'Decontamination of Sampling Equipment' SOP.

The samples must be delivered to the laboratory via hand or overnight delivery courier in accordance with all Federal, State and Local transportation regulations and Barr's 'Domestic Transport of Samples to the Laboratory' SOP.

6.3 Data Reduction/Calculations

Data reduction or calculations are not applicable to this SOP.

6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

7.1 QA/QC Samples

QA/QC samples are defined in Barr's SOP 'Collection of Quality Control Samples'. The sampling frequency should be performed as written in the project scope of work and/or documentation (e.g., Work Plan, SAP, or Quality Assurance Project Plan).

7.2 Measurement Criteria

Measurement criteria are not applicable to this SOP.

8.0 Records

The field technician will document the IDW sampling event on the field log data sheet and/or field notebook. They will also document the type and number of bottles on the chain-of-custody record, as appropriate. The analysis for each container and the laboratory used will be documented on the chain-of-custody record. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation is listed in the SOPs referenced in this procedure.

The field documents and COCs are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: collection of samples, collection of QC samples, decontamination of sampling equipment, domestic transport of samples, and documentation on a COC.

9.0 References

Environmental Protection Agency, 9345.3-03FS. January 1992. *Guide to Management of Investigation-Derived Wastes*



Standard Operating Procedure Documentation on a Chain-of-Custody Form

Revision 5

March 14, 2018

Approved By:

<u>Andrea Nord</u>	<u><i>Andrea Nord</i></u>	<u>03/14/18</u>
Print	Technical Reviewer Signature	Date
<u>Terri Olson</u>	<u><i>Terri A. Olson</i></u>	<u>03/14/18</u>
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.	
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

Documentation on a Chain-of-Custody Form

1.0 Scope and Applicability

The purpose of this procedure is to describe how to properly document information on a Chain-of-Custody (COC) form. A COC is a legally binding document that identifies sample identification, analyses required, and shows traceable possession of samples from the time they are obtained until they are introduced as evidence in legal proceedings. A Field Technician completes the information on the COC at the time he/she collects samples and the COC accompanies the samples during transport to a storage facility or to the laboratory for analysis.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- The SOP does not apply to sample aliquots that are only collected for field screening purposes.
- The SOP does not apply to samples remaining on-site.

3.0 Responsibilities

Experienced Field Technicians are responsible for the proper sample identification and for accurate and complete documentation on the COC.

4.0 Procedure

The COC is the most important sampling document; it must be filled out accurately and completely every time a sample is collected. The instructions below are specific to Barr's COC for air canisters and Barr's COC typically used for solid and liquid samples. The COC for air canisters is typically used when collecting soil gas, soil vapor, or air samples in an evacuated canister. The COC for solid and liquid samples is typically used when collecting matrices such as groundwater, surface water, drinking water, waste water, storm water, soil, sediment, oil, paint chips, bulk materials, etc. Information common to both chains-of-custody and specific to each COC are detailed below. Some of the information on a COC may be filled out ahead of time (e.g., report and invoice recipient details, project number, project name, project manager, purchase order number, etc.) while other information should be completed when sampling. Complete one COC or more as needed for each set of project samples. The COC should be completed prior to leaving the sampling location.

Laboratory supplied COCs may be used but may differ in the information captured. The use of a Barr COC is recommended as it allows for more efficient data processing within Barr's systems. If there are any questions, please contact a member of Barr's Data Quality team.

The laboratory receiving the samples will sign the COC, record the date and time of sample receipt, assign a laboratory work order number, document sample condition, and document whether custody seals were used and if they were intact.

4.1 Common Chain-of-Custody Information

- Barr office location managing the work.
- Two digit identification for the state or province the samples originated from/sampled in.
- COC numbered pages (e.g., 1 of 1).
- Report and invoice recipient information.
- Purchase order number (if applicable).
- Barr project name and number.
- Sample location.
- Sample collection date and time.
- Sample matrix abbreviation (see "Matrix Code" on COC).
- Analysis requested.
- Field Technician (i.e. sampler) name.
- Barr Project Manager and project Data Quality (DQ) Manager names.
- Laboratory name and location in which samples are to be relinquished.
- Requested due date.
- Signature of Field Technician (i.e. sampler) under the first 'relinquished by'.
- Signature of sample transferee.
- Date and time of sample transfers.
- Method of transport (UPS, FedEx, local courier, sampler, etc.).
- Air Bill number (if applicable).

4.2 Completing a Chain-of-Custody for Air Canisters

Lab deliverable contents (based on project needs).

- Canister serial # and size.
- Flow controller serial #.
- Initial and final vacuum measurement (record unit).
- Record both the start and stop time and calculate the total time.
- Matrix Code.
- PID reading (indicate if ppm or ppb).
- Sample comments (if any).

4.3 Completing a Chain-of Custody for Solid and Liquid Samples

- Sample start and stop depth (if applicable) and unit of measurement (meter, feet, inches, etc.).
- Information regarding whether to perform sample Matrix Spike (MS) and MS duplicate (MSD).
- Container preservative type (see "Preservative Code" on COC).

- Information regarding whether the sample was field filtered.
- Number of each container type and the total number of containers for the sample.
- Presence or absence of ice.

4.4 Distribution of the COC Pages

Page one (white copy) accompanies the sample shipment to the laboratory; page two (yellow copy) is the Field Technician's copy; and page three (pink copy) is submitted to a Barr Data Management Administrator for filing.

5.0 Quality Control and Quality Assurance (QA/QC)

The Field Technician should review the COC for accurate and complete documentation.

6.0 Records

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Chain-of-Custody for Air Canisters Form
- Chain-of-Custody Form

A copy of the COC is provided to a Barr Data Management Administrator for storage on the internal Barr network files.

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

United States Environmental Protection Agency. 2002. *Guidance for Quality Assurance Project Plans*. EPA QA/G-5.



Standard Operating Procedure Domestic Transport of Samples to Laboratories within the United States of America – States and Territories

Revision No. 2

April 11, 2016

Approved By:

<u>Andrea Nord</u>	<u>Andrea Nord</u>	<u>4/11/2016</u>
Print	QA Manager	Signature
		Date

Review of the SOP has been performed and the SOP still reflects current practice.	
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

Standard Operating Procedures for the Domestic Transport of Samples to the Laboratories within the United States of America – States and Territories

1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures necessary for personal delivery or shipment of samples from locations within the United States of America and its territories to analytical laboratories located within the United States of America and its territories. This procedure applies to the transportation of ground and surface water, soil, wipe, sediment, paint chip, debris, and air samples to the appropriate laboratory.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Maintaining proper sample temperatures (<6°C or ambient air temperature in accordance with the analytical method requirements) and delivering samples to the laboratory within 24 to 48 hours from collection are primary concerns.
- This procedure does not apply to the transportation of ground and surface water, soil, wipe, sediment, paint chip, debris, and air samples to laboratories outside of the United States of America – States and Territories.

3.0 Responsibilities

The field technician(s) shall ensure the security, temperature, and packaging of environmental samples during transport and shipment.

4.0 Safety

Barr staff is responsible for conducting all aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protection equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of two pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When samples may be contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

5.0 Equipment, Reagents, and Supplies

- Rigid Cooler
- Ziploc® baggies
- Absorbent Padding
- Ice
- Chain-of-custody Record
- Dangerous Goods in Excepted Quantities Label with the number "8" added indicating the hazard class. This label must be used for all coolers containing unused sample containers with corrosive preservative.
- Directional arrow labels may be used to ensure samples remain upright.
- Environmental Samples
- Bubble-wrap/bubble bags (inner packing material)
- Heavy bag for containing ice and preventing leakage of melted water
- Packing Tape
- Shipping Papers – if shipping via delivery service
- Dangerous Goods in Excepted Quantities Label with the number "3" added indicating the hazard class. This label must be used for all coolers containing methanol preservative

6.0 Procedure

6.1 Packaging of water, soil and sediment samples (requiring chilled preservation per the analytical method of analysis)

6.1.1 Packaging Samples

Place samples in a rigid cooler, pack glass containers in bubble wrap or other cushioning material to avoid breakage. (Note: Bubble-wrap is the preferred packing material.) Methanol sample containers must be placed in a Ziploc® Baggie to meet shipping requirements for preventing leaks.

Place samples and cushioning material in strong plastic bag with enough absorption padding to absorb all of the liquid in the packaging. Be sure to zip tie this bag shut.

Add enough ice to maintain a constant temperature at $< 6^{\circ}\text{C}$, (but not frozen) until the samples arrive at the laboratory. Package ice in double-lined bags to ensure sample labels will not be compromised, and the cooler(s) will not leak melt water.

Before sealing cooler, fill out the chain-of-custody form completely and include required copies with the samples (see Standard Operating Procedure for Documentation on a Chain-of-Custody).

Adhere two to three strips of packaging tape on the cooler from top to bottom, and adhere an additional strip of tape covering the gap between the lid and sides of cooler to seal the cooler to avoid leakage. Custody Seals must be adhered on the cooler if project quality assurance plan or sampling and analysis plan require them. The custody seal must be adhered to the crack of the lid and the side of the cooler to ensure the cooler lid has not been tampered with in transit. Be sure to attach the courier shipping label to the top of the cooler.

6.1.2 Labeling

A secondary label with the same information should also be attached with packaging tape to the cooler in event that the original label is damaged or destroyed during sample shipment.

When shipping samples preserved with methanol, the cooler must have a Dangerous Goods in Excepted Quantities label (see attachment 4) placed on the outside of the cooler. Be sure to add the number "3" to each label in permanent marker to indicate the hazard class being shipped.

Each cooler shall not exceed 500 mL of Methanol (16 vials, 30 mL of methanol per vial) and each vial shall not have more than 30 mL of methanol to meet the requirements of a dangerous good in excepted quantities. Acid/base preserved samples vials are often 40 mL or larger and do not qualify for excepted quantities.

When shipping UNUSED sample containers preserved with acids or bases, the cooler must have a Dangerous Goods in Excepted Quantities label (see attachment 4) placed on the outside of the cooler. Be sure to add the number "8" to each label in permanent marker to indicate the hazard class being shipped.

Directional arrow labels should also be attached to the cooler to insure the cooler remains upright during shipping. Directional arrow labels should be attached to the outside of the cooler to keep the cooler in an upright position during sample shipment.

6.2 Packaging of wipe, paint chip, debris, and air samples (requiring ambient air temperature per the analytical method of analysis)

6.2.1 Packaging Samples

Place the samples in a cooler or cardboard box in a manner that will avoid breakage.

Adhere two to three strips of packaging tape from top to bottom on the cooler or box. Fill out the chain-of-custody completely and include required copies with the samples (see Standard Operating Procedure for chain-of-custody record).

Custody Seals must be adhered over the lid if project quality assurance plan or sampling and analysis plan require them. The custody seal must be adhered to the crack of the lid and the side of the cooler or over the flaps of the box to ensure the container remained shut and has not been tampered with in transit.

6.3 Sample Storage

For samples requiring ice as a preservative, the samples will be bubble wrapped, bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain-of-custody documentation. The samples will be kept cold (< 6 °C, but not frozen) until receipt at the laboratory, where they are to be stored in a refrigerated area.

For samples that are stored at ambient air temperature, the samples (wipe, paint chip, debris, and air samples) will be placed in a baggie or shipping carton (i.e. cardboard box) and accompanied with the proper chain-of-custody documentation.

For all samples, custody seals may be present, but at minimum, the coolers must be taped shut with two to three straps of packing tape. All samples will be kept secured to prevent tampering. If sample coolers

are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over-night delivery courier in accordance with all Federal, State and Local shipping regulations.

Note: Samples may have to be stored indoors in winter to prevent freezing.

6.4 Shipping Considerations

6.4.1 Shipment/Delivery

Once the cooler is packed to prevent breaking of bottles, the proper chain-of-custody (COC) documentation is signed off, sealed in a plastic bag, and placed in the cooler.

All samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured.

Custody seals may be present, but at a minimum, the coolers must be taped shut to prevent the lid from opening during shipment.

The coolers must be delivered to the laboratory via hand or overnight delivery courier in accordance with all Federal, State and Local transportation regulations and Barr's SOP 'Domestic Transport of Samples to the Laboratory.

6.4.2 Transport/Delivery Options

Account for all samples before shipping and compare to the chain of custody (see Standard Operating Procedure for chain-of-custody record). Ship samples during times when the laboratory will be able to accept and quickly analyze them. Whenever possible, select mode of transport/delivery to ensure delivery to the laboratory will occur with ample EPA recommended holding time remaining for the specified analytical methods required for the samples. Avoid sending samples during holidays and weekends. All Federal, State and Local shipping regulations must be met.

Personal Delivery. The samples are delivered to the laboratory by the field technician(s). The chain-of-custody record is signed and dated by the laboratory representative.

Local Courier. The same procedures are followed as above; i.e., the chain-of-custody record is signed and dated and the top copy is sent with the samples. The cooler or box is then secured with packaging tape and a courier form is filled out for the designated laboratory. The cooler or box is then left in the services area for pickup.

Overnight Courier. Follow the procedures above, replacing the courier form with the overnight courier (examples Federal Express, United Parcel Service, Speedy Delivery) form. Date, project number, type of delivery desired, weight, and number of coolers or boxes should be included.

7.0 Quality Control and Quality Assurance (QA/QC)

Not Applicable

8.0 Records

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Chain-of-custody record

Chain-of-custody records are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: Standard Operating Procedure for chain-of-custody record

9.0 References

Barr Engineering Co. Most current version. *Quality Assurance Manual: Groundwater and Surface Water Sampling Procedures*

Minnesota Pollution Control Agency. January 1995. *Procedures for Ground Water Monitoring*



Standard Operating Procedure

Routine Level Semivolatile Organic Compounds (SVOC), Polycyclic Aromatic Hydrocarbons (PAH), Diesel Range Organics (DRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

Revision 6

January 19, 2016

Approved By:

<u>Michael Dupay</u>		<u>01/19/16</u>
Print	Technical Reviewer Signature	Date
<u>Terri Olson</u>		<u>01/19/16</u>
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

Routine Level Semivolatile Organic Compounds (SVOC), Polycyclic Aromatic Hydrocarbons (PAH), Diesel Range Organics (DRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of semivolatile organic compounds data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine SVOC (including PAHs and phenols), TPH at various carbon ranges (e.g., TPH as fuel oil, TPH as motor oil, TPH as jet fuel), and DRO data evaluation for analyses by the following technologies:

- Gas Chromatography/Flame Ionization Detector (GC/FID)
 - Method examples: EPA 8015, EPA 8100, WI DRO
- Gas Chromatography/Mass Spectrometry (GC/MS)
 - Method example: EPA 625, EPA 8270
- Gas Chromatography/Mass Spectrometry-Selective Ion Monitoring (GC/MS-SIM)
 - Method example: EPA 8270
- High Performance Liquid Chromatography (HPLC)
 - Method example: EPA 610, EPA 8310
- Methods above with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136, WI GRO method, and the *Test Methods for Evaluating Solid Waste (SW-846)* are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation				
Compound	Matrix	Temp.	Preservative	Maximum Hold Time
SVOC/PAH/TPH	Aqueous	≤6° C	Ice	7 days extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6° C	Ice	14 days extraction/ addl. 40 days analysis
DRO	Aqueous	≤ 6° C	Ice, HCl < 2 pH	7 days extraction/ 47 days collection to analysis
	Sediment/Soil	≤ 6° C	Ice	10 days solvent addition/ 47 days collection to extraction and analysis
TCLP SVOC	Various	--	NA	14 days TCLP extraction/ 7 days extraction/ addl. 40 days analysis

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an “h”. Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subjected to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG). Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. It may be easier to work with the raw data and/or convert the data to the same units for comparison purposes.

Table 2 – Guidelines for Blank Contamination	
Sample Result	Recommended Action for Associated Data
Non-detect	No action required
< 5x blank concentration	Qualify with ‘b’
≥ 5x blank concentration	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with ‘*’ (estimated value, QA/QC criteria not met) or ‘**’ (unusable value, QA/QC criteria not met).

4.3 Deuterated Monitoring Compounds (DMC) and Surrogates

DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are only used for the SVOC GC/MS analysis. *Table 3* presents the recommended DMCs with their associated target compounds.

Table 3 – DMC and Associated Target Compounds		
DMC (alphabetical)	Associated Target Compounds	
<i>2,4-Dichlorophenol-d₃</i>	2,4-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol	2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol 2,3,4,6-Tetrachlorophenol
<i>2-Chlorophenol-d₄</i>	2-Chlorophenol	
<i>2-Nitrophenol-d₄</i>	Isophorone	2-Nitrophenol
<i>4-6-Dinitro-2-methylphenol-d₂</i>	4,6-Dinitro-2-methylphenol	
<i>4-Chloroaniline-d₄</i>	4-Chloroaniline Hexachlorocyclopentadiene	3,3'-Dichlorobenzidine
<i>4-Methylphenol-d₈</i>	2-Methylphenol 4-Methylphenol	2,4-Dimethylphenol
<i>4-Nitrophenol-d₄</i>	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol	4-Nitrophenol 4-Nitroaniline
<i>Acenaphthylene-d₈</i>	Naphthalene 2-Methylnaphthalene 2-Chloronaphthalene	Acenaphthylene Acenaphthene
<i>Anthracene-d₁₀</i>	Hexachlorobenzene Atrazine	Phenanthrene Anthracene
<i>Benzo(a)pyrene-d₁₂</i>	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
<i>Bis-(2-chloroethyl) ether-d₈</i>	Bis-(2-chloroethyl) ether 2,2'-oxybis(1-chloropropane)*	bis(2-Chloroethoxy) methane
<i>Dimethylphthalate-d₆</i>	Caprolactum 1,1'-Biphenyl Dimethylphthalate Diethylphthalate	Di-n-butylphthalate Butylbenzylphthalate bis(2-ethylhexyl)phthalate Di-n-octylphthalate
<i>Fluorene-d₁₀</i>	Dibenzofuran Fluorene 4-Chlorophenyl-phenylether	4-Bromophenyl-phenylether Carbazole
<i>Nitrobenzene-d₅</i>	Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene	2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine

(Table 3 continued on next page)

Table 3 – DMC and Associated Target Compounds		
DMC (alphabetical)	Associated Target Compounds	
<i>Phenol-d₅</i>	Benzaldehyde	Phenol
<i>Pyrene-d₁₀</i>	Fluoranthrene Pyrene	Benzo(a)anthracene Chrysene
SIM DMC and Associated Target Compounds		
<i>Fluoranthene-d₁₀</i>	Fluoranthrene Pyrene Benzo(a)anthracene Chrysene Benzo(b)fluoranthrene	Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
<i>2-Methylnaphthalene-d₁₀</i>	Naphthalene 2-Methylnaphthalene Acenaphthylene Acenaphthene	Fluorene Pentachlorophenol Phenanthrene Anthracene

* = Bis(2-chloroisopropyl)ether

Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Other DMC or surrogates may be used by a laboratory based on their experience provided adequate chromatographic separations can be demonstrated. All samples (blanks, spiked samples, project samples, QC samples) should contain DMC or surrogates. If a sample does not contain DMC or surrogates or the method does not require surrogates (WI DRO), professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of DMC or surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each DMC or surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not reported, use guidance found in the NFG, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

For the WI DRO analysis, surrogates are not required by the method. If used, the method requires that the surrogates must not elute within the WI DRO window (C₁₀-C₂₈). If the laboratory report includes a surrogate spike recovery for WI DRO, use professional judgment to assess the data.

Table 4 includes guidance to evaluate the surrogate recovery where a single surrogate is analyzed.

Table 4 – Guidelines for Single DMC or Surrogate		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R > Upper Limit	Qualify with '**'	No qualification
%R < Lower Limit	Qualify with '**' or '**'', use professional judgment	
%R within Limits	No qualification	

'**' = reported value is estimated and QA/QA criteria were not met

'**'' = reported value is unusable and QA/QC criteria were not met

Table 5 includes guidance where multiple surrogates are analyzed per analytical fraction.

Table 5 – Guidelines for Multiple DMC or Surrogates		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with '**' or '**'', use professional judgment	
Two or more %R > Upper Limit	Qualify fraction with '**'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

'**' = reported value is estimated and QA/QA criteria were not met

'**'' = reported value is unusable and QA/QC criteria were not met

4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (20 or less samples of the same matrix - WI DRO requires an additional LCSD analyzed at the end of 20 samples).
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset (see Table 6 for guidance) and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use guidance found in the NFG. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 6 – Number of Suggested Target Compounds - LCS/LCSD and MS/MSD	
Number of Target Parameters	Number of Spiked Compounds
1-10 analytes	Spike all compounds
11-20 analytes	At least 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike at least 16 compounds

Table 7 – Guidelines for Laboratory Control Samples		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '**' or '**'', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met

** = Reported value is unusable and QA/QC criteria were not met

4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

The MS/MSD duplicate pairs may be substituted for laboratory duplicates.

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable

concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

Table 8 – Guidelines for Laboratory Duplicates	
% RPD	Recommended Action for Associated Data
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are ≤ 5x RL, no action is required
RPD > Upper Limit	Both results are > 5x RL, consider qualifying with '**'

* = Reported value is estimated and QA/QC criteria were not met

4.6 Field Duplicate Samples

Field duplicate samples (also known as “masked” or “blind” duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in ‘Definitions’ from Barr’s “Compendium of Data Quality Assessment Documentation” and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs ≤ 30% for aqueous samples and ≤ 40% for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples may contain all target compounds or a subset (see *Table 6*) and provide information about the effect of each samples’ matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples (does not apply to DRO in the WI method)
- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (>4x), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

Table 9 – Guidelines for Matrix Spikes		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R and RPD > Upper Limit	Qualify with '**'	No qualification
%R < Lower Limit	Qualify with '**' or '**'', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met

** = Reported value is unusable and QA/QC criteria were not met

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented also. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the

sample results, notification should be given to the appropriate data management personnel and/or project team members.

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7.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3.*

Environmental Protection Agency, *National Functional Guidelines for Superfund Organic Methods Data Review.*

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

Attachment 1 Revision History

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		IX	Added Table 10
		Attachments	Added Attachment 3
3.2	04/2011	Document Wide	Added analytical methods to applicability section.
		Attachments	Updated Attachment 1 and 2 to include current forms.
4.0	04/06/12	Document Wide	Major revision
5.0	0601//17/13	Cover page	Added Calgary office
		I	Added waste rock and drill cores to examples of product sample
		III, IV, V, VI, VII	Added 'project specific requirements' as possible criteria source
		VI	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		VI	Clarified field duplicate criteria as < one value and not a range
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Standard Operating Procedure

Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

Revision 6

January 15, 2016

Approved By:

<u>Michael Dupay</u>		<u>01/15/16</u>
Print	Technical Reviewer Signature	Date
<u>Terri Olson</u>		<u>01/15/16</u>
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of VOC, GRO, and TPH data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine VOC (including BTEX), GRO, and TPH (in the approximate gasoline carbon range, C₆-C₁₀) data evaluation for analyses by the following technologies:

- Gas Chromatography/Flame Ionization Detector (GC/FID)
 - Method examples: EPA 8015, WI GRO (GRO)
- Gas Chromatography/Photoionization Detector (GC/PID)
 - Method example: EPA 8021, WI GRO (PVOC)
- Gas Chromatography/Electrolytic Conductivity Detector (GC/ELCD)
 - Method example: EPA 8021
- Gas Chromatography/Mass Spectrometry (GC/MS)
 - Method example: EPA 624, EPA 8260
- Gas Chromatography/Mass Spectrometry-Selective Ion Monitoring (GC/MS-SIM)
 - Method example: EPA 8260
- Methods above with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136, WI GRO method, and the *Test Methods for Evaluating Solid Waste (SW-846)* are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation				
Compound	Matrix	Temp.	Preservative	Maximum Hold Time
VOC/PVOC	Aqueous	≤ 6 °C	HCl < 2 pH	14 days
	Aqueous	≤ 6 °C	Unpreserved	7 days
	Sediment/Soil	≤ 6 °C	1:1 soil:solvent (e.g., 10 g soil:10 mL MeOH in lab pre-weighed vial)	14 days
GRO (WI Method)	Aqueous	≤ 6 °C	HCl < 2 pH	14 days
	Sediment/Soil	≤ 6 °C	1:1 soil:solvent (e.g., 10 g soil:10 mL MeOH in lab pre-weighed vial)	21 days

(Table 1 continued on next page)

Table 1 – Recommended Holding Times and Preservation				
Compound	Matrix	Temp.	Preservative	Maximum Hold Time
TPH	Aqueous	≤ 6 °C	HCl or H ₂ SO ₄ < 2 pH	7 day extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6 °C	Zero headspace*	14 days extraction/ addl. 40 days analysis
TCLP	Various	≤ 6 °C	No preservative	14 days TCLP extraction/ addl. 14 days analysis

* = Alternatively, samples may be collected as per the VOC analysis.

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an “h”. Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subjected to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG) – laboratories should analyze a method blank at least once every 12 hours. Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Trip blanks should be placed in each transport cooler containing VOC sample containers prior to shipment into the field and remain with the associated VOC samples submitted to the laboratory for VOC analysis; including sample storage through analysis.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. It may be easier to work with the raw data and/or convert the data to the same units for comparison purposes.

Table 2 – Guidelines for Blank Contamination	
Sample Result	Recommended Action for Associated Data
Non-detect	No action required
< 5x blank concentration	Qualify with ‘b’
≥ 5x blank concentration	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*' (estimated value, QA/QC criteria not met) or '**' (unusable value, QA/QC criteria not met).

4.3 Deuterated Monitoring Compounds (DMC) and Surrogates

DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are only used for the VOC GC/MS analysis. Table 3 presents the recommended DMCs with their associated target compounds.

Table 3 –DMC and Associated Target Compounds		
DMC (alphabetical)	Associated Target Compounds	
<i>1,1,2,2-Tetrachloroethane-d₂</i>	1,1,2,2-Tetrachloroethane	1,2-Dibromo-3-chloropropane
<i>1,1-Dichloroethane-d₂</i>	trans-1,2-Dichloroethene 1,1-Dichloroethene	cis-1,2-Dichloroethene
<i>1,2-Dichlorobenzene-d₄</i>	Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene	1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene
<i>1,2-Dichloroethane-d₄</i>	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl-tert-butyl ether	1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
<i>1,2-Dichloropropane-d₆</i>	Cyclohexane Methylcyclohexane	1,2-Dichloropropane Bromodichloromethane
<i>1,4-Dioxane-d₈</i>	1,4-Dioxane	
<i>2-Butanone-d₅</i>	Acetone	2-Butanone
<i>2-Hexanon-d₅</i>	4-Methyl-2-pentanone	2-Hexanone
<i>Benzene-d₆</i>	Benzene	
<i>Chloroethane-d₅</i>	Dichlorodifluoromethane Chloromethane Bromomethane	Chloroethane Carbon disulfide
<i>Chloroform-d</i>	1,1-Dichloroethane Bromochloromethane Chloroform	Dibromochloromethane Bromoform
<i>Toluene-d₈</i>	Trichloroethene Toluene Tetrachloroethene Ethylbenzene	o-Xylene m,p-Xylene Styrene Isopropylbenzene
<i>trans-1,3-Dichloropropene-d₄</i>	cis-1,3-Dichloropropene trans-1,3-Dichloropropene	1,1,2-Trichloroethane
<i>Vinyl Chloride-d₃</i>	Vinyl chloride	

Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Other DMCs or surrogates may be used by a laboratory based on their experience provided adequate chromatographic separations can be demonstrated. All samples (blanks, spiked samples, project samples, QC samples) should contain DMCs or surrogates. If a sample does not contain DMC or surrogates or the method does not require surrogates (WI GRO), professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of the DMC or surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each DMC or surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not reported, use guidance found in the NFG, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

For the WI GRO analysis, surrogates are not required for GRO but are required for PVOC. The method minimum surrogate recovery is 80%; there is no method maximum recovery. Use professional judgment when evaluating surrogates for WI GRO samples.

Table 4 includes guidance to evaluate the surrogate recovery where a single surrogate is analyzed.

Table 4 – Guidelines for Single DMC or Surrogate		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R within Limits	No qualification	

'*' = reported value is estimated and QA/QA criteria were not met

** = reported value is unusable and QA/QC criteria were not met

Table 5 includes guidance where multiple surrogates are analyzed per analytical fraction.

Table 5 – Guidelines for Multiple DMC or Surrogates		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with '*' or '**', use professional judgment	
Two or more %R > Upper Limit	Qualify fraction with '*'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

'*' = reported value is estimated and QA/QA criteria were not met

** = reported value is unusable and QA/QC criteria were not met

4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (typically 20 or less samples of the same matrix - WI GRO requires an additional LCSD analyzed at the end of 20 samples)
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset (see *Table 6* for guidance) and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use guidance found in the NFG. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 6 – Number of Suggested Target Compounds - LCS/LCSD and MS/MSD	
Number of Target Parameters	Number of Spiked Compounds
1-10 analytes	Spike all compounds
11-20 analytes	At least 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike at least 16 compounds

Table 7 – Guidelines for Laboratory Control Samples		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met

** = Reported value is unusable and QA/QC criteria were not met

4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are

not calculated where data are already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

The MS/MSD duplicate pairs may be substituted for laboratory duplicates.

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

Table 8 – Guidelines for Laboratory Duplicates	
% RPD	Recommended Action for Associated Data
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are $\leq 5x$ RL, no action is required
RPD > Upper Limit	Both results are $> 5x$ RL, consider qualifying with '**'

* = Reported value is estimated and QA/QC criteria were not met

4.6 Field Duplicate Samples

Field duplicate samples (also known as “masked” or “blind” duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in ‘Definitions’ from Barr’s “Compendium of Data Quality Assessment Documentation” and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

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