WCEC
ENVIRONMENTAL CONSULTANTS

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May 24, 2024

Mr. Reed Miner
MT Department of Environmental Quality
655 Timberwolf Parkway, Suite 3
Kalispell, MT 59901-1215

Re: **Additional Corrective Action Work Plan 34852** for Kelly Rae's, Kalispell, MT, Facility ID# 15-06101, Release# 1850, WPID# 34852.

Dear Mr. Miner:

Enclosed for your review is the **Additional Corrective Action Work Plan 34852** for the Kelly Rae's facility located at 25 Batavia Lane in Kalispell, Montana.

Thank you for your time and consideration of this work plan. If you have any questions or concerns, please call or contact me via e-mail at mmorris@wcec.com.

Sincerely,

Myles Morris, PG
Senior Project Manager

Enclosure

ec: Manpreet Singh, Kalispell Properties, LLC; histarms01@yahoo.com

Additional Corrective Action Work Plan 34852

Kelly Rae's

25 Batavia Lane

Kalispell, MT 59901

Facility ID 15-06101, Release 1850

Prepared for:

Kalispell Properties, LLC

PO Box 776

Spokane Valley, WA 99016

Prepared by:

West Central Environmental Consultants, Inc.

1030 South Ave. W.

Missoula, MT 59801

May 24, 2024

WCEC Project No. 2404-1006

WCEC
West Central Environmental Consultants, Inc.

Nationwide Services
www.wcec.com

Environmental ■ Emergency Response ■ Industrial Services

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1.0 Introduction

West Central Environmental Consultants (WCEC) has prepared this Additional Corrective Action Work Plan for the Kelly Rae's facility (Facility ID 15-06101, Release 1850) located at 25 Batavia Lane in Kalispell, MT [Figure 1]. Additional corrective actions were requested by the Montana Department of Environmental Quality (MTDEQ) in correspondence dated March 28, 2024. The purpose of the scope of work included in this Work Plan is to design and implement an injection pilot test to assess the feasibility of in-situ treatment of petroleum contamination in soil and groundwater.

1.1 Site Location

The site is located in a rural area of Flathead County near the town of Batavia, approximately five miles west of Kalispell on Highway 2 [Figure 1]. The primary land use is agricultural with mixed residential and light commercial businesses. Adjacent downgradient properties include the Batavia Waterfowl Production Area (WPA) and the Smith Valley School. The approximate geographic coordinates are 48.174912, -114.422852. The Public Land Survey System (PLSS) description for the site is the SW/4, NE/4, Section 20, Township 28 North, Range 22 West.

The Kelley Rae's facility consists of a retail convenience store with a petroleum storage/distribution system that includes four aboveground storage tanks (ASTs) with underground piping, one underground storage tank (UST), and two pump islands [Figure 2]. Product storage capacity and type by tank for the current UST/AST system is as follows:

Tank 03 – Tag Number: 4505, Install Date: 02-01-1994; 6,000-gal, diesel, cathodically protected steel UST

Tank S6 – Tag Number: 5891, Install Date: 01-10-2019; 5,000-gal, dyed diesel, AST

Tank S7 – Tag Number: 5892, Install Date: 01-10-2019; 10,000-gal, diesel, AST

Tank S8 – Tag Number: 5893, Install Date: 01-10-2019; 15,000-gal, gasoline, AST

Tank S9 – Tag Number: 5894, Install Date: 01-10-2019; 5,000-gal, premium gasoline, AST

The convenience store is serviced by a transient non-community public water supply well (PWS ID# MT0003093) located north of the store [Figure 2]. The store also has an onsite septic system consisting of an 1,100-gallon septic tank connected to a mounded drain field in the northwest corner of the property.

1.2 Site Geology

The Kelly Rae's facility is situated in the Smith Valley which is principally drained by Ashley Creek. Regionally, groundwater flows from recharge areas in the surrounding mountains towards the center of the valley, discharging to Ashley Creek which generally flows northeast [LaFave, 2004]. At the Kelly Rae's facility, depth to shallow groundwater ranges from approximately 2 to 9 feet below ground surface (bgs) based on the data collected from previous groundwater monitoring events [WET, 2023]. The calculated local groundwater flow direction is to the southeast towards Ashley Creek. The nearest surface water body is an ephemeral oxbow channel of Ashley Creek located approximately 300 feet southeast of the facility [Figure 1]. The average groundwater hydraulic conductivity is 0.1 feet/day based on slug tests completed in 2017 [AWC, 2018].

The surficial geology of the Smith Valley predominately consists of Quaternary glacial and alluvial deposits. The more recent alluvial deposits (Qal) are underlain and interbedded with outwash (Qgo), till (Qgt), and ablation/ice contact sediments (Qgta/Qgi) associated with Pleistocene glaciation [Smith, 2004]. Lithological data obtained from historical soil remediation activities conducted at the facility indicates that the shallow subsurface primarily consists of fine-grained silts and clays to a depth of at least 8 feet bgs [AWC, 2013], [AWC, 2015], [WET, 2020]. This fine-grained material is underlain by a layer of sandy gravel with cobbles starting at a depth of approximately 8 to 12 feet bgs depending on location. The coarse-grained gravel interval is saturated with groundwater that appears to be under confined conditions based on observations of water infiltrating into the December 2018 remedial excavation [AWC, 2019].

1.3 Site Background

The current active release (Release 1850) was confirmed on January 12, 1994, during the closure and removal of two USTs that had been in place south of the store since 1981. The existing diesel UST (Tank 03) was installed on February 1, 1994, after the older USTs were removed [Figure 2]. Initial remedial investigations included the installation of four groundwater monitoring wells, labeled MW-N1 through MW-N4. Additional monitoring wells were installed in 2012 (MW-5 – MW-9), 2014 (MW10 – MW13), 2019 (MW-11R), and 2023 (MW-3NR). The cumulative data set derived from the various remedial investigations indicated that two distinct source areas were evident, one located near two ASTs established west of the store in 1965, and another centered around the dispenser island east of the store.

The two ASTs installed in 1965 were decommissioned and removed in 2018, providing access to contaminated soil underlying the containment basin for remedial excavation. A total of 534 cubic yards of impacted soil was removed from the site during the December 2018 excavation [AWC, 2019]. Monitoring well MW-11 was destroyed by excavation activities and ultimately replaced by monitoring well MW-11R in 2019 [WET, 2020]. New dual-compartment ASTs (Tank S6 – Tank S9) were constructed northwest of the store to replace the tanks removed in 2018 [Figure 2].

A soil boring investigation was conducted in October 2023 in the vicinity of the dispenser island east of the store [WET, 2024]. A total of seven soil boreholes were advanced during the investigation (SB-1 – SB-7). A replacement well for destroyed monitoring well MW-N3 (labeled as MW-3NR) was also drilled in October 2023, in the same location as soil borehole SB-7. As of the current date, groundwater has never been sampled from monitoring well MW-3NR and the well has not been surveyed for top of casing elevation.

2.0 Scope of Work

The scope of work requested by MTDEQ includes:

- Design and implement a pilot test to assess the feasibility of and provide necessary information for full-scale design of in-situ treatment of petroleum contamination in soil and groundwater.
    - Identify the product that will be used, the area that will be treated (laterally and vertically), the volume of the treatment product applied, application rate, etc.
    - Identify the criteria that will be measured during and after treatment to assess the radius of influence, the completeness of the product application, the effectiveness of the product and its application method, etc.

- Collect soil and groundwater data needed to assess the effectiveness of the treatment.
    - Identify the method, timing (e.g., pre-, post-treatment), and location of sample collection.
    - Identify the disposal method of soil cuttings (if applicable) and purge water.

- Analyze samples for petroleum constituents as required by the Montana Risk-Based Corrective Action Guidance for Petroleum Releases and other criteria as needed to assess the feasibility and effectiveness of treatment.

- Validate laboratory analytical data using DEQ's Data Validation Summary Form (DVSF) found online under the Guidance dropdown at the Petroleum Tank Cleanup Section (PTCS) webpage.

- Discuss ongoing WP tasks and results with DEQ's project manager; submit written agreed-upon WP modifications as required to complete the WP objectives.

- Prepare an updated Release Closure Plan (RCP), discuss the results with DEQ's project manager. DEQ expects the RCP to cover the Release Investigation, cleanup pilot test, and monitoring information. Use the RCP format found online under the Guidance dropdown at the PTCS webpage.

- Prepare and submit Cleanup Report detailing the results of the pilot test. The Report is expected to include all the content, tables, figures, and appendices outlined in the Report format.

- Use standardized DEQ WP and Report formats found online under the Forms dropdown at the PTCS webpage.

- Submit WP and Reports electronically following the PTCS submittal requirements found under the Guidance dropdown at the PTCS webpage.

2.1 Injection Pilot Test

Data from the various investigation and cleanup activities conducted at the facility were evaluated in the injection pilot test design, including cumulative soil and groundwater analytical results. Based on this data, WCEC recommends focusing the pilot test on the area with worst case soil and groundwater impacts documented near monitoring well MW-8. The proposed injection area is displayed on Figure 3.

WCEC will complete all notifications, coordination, and permitting required to initiate the pilot test injection at the facility. In addition to project oversight, on-site direction, and professional field services, WCEC will provide the necessary drill rig, personnel, and equipment required for the injection, including mixing tanks, pumps, hoses, and drill tooling. The injection borings will be advanced using a limited access Geoprobe 7822 direct push rig that is track mounted.

Prior to initiating the injection event, WCEC will submit an underground utility locate for identification of potential subsurface utilities in the drilling area. In addition to the one-call public utility locate, WCEC will subcontract a private utility locator to scan the injection area for private utilities such as electrical conduits, product piping, and storm drain infrastructure.

Four remediation amendments were evaluated for the injection pilot test, including in-situ chemical oxidation (ISCO) PersulfOx, Oxygen Release Compound (ORC), PetroBac Stimulant Bundle with Custom Blend Nutrients (CBN), and PetroFix colloidal activated carbon (CAC). ISCO PersulfOx is corrosive and not recommended for application near subsurface utilities. Additionally, the relatively moderate soil and groundwater concentrations recorded in the vicinity of MW-8 do not necessitate the usage of an aggressive ISCO compound. For these reasons, ISCO PersulfOx was rejected as a potential option for the pilot test. After reviewing the pros and cons of the remaining three remediation amendments, including cost effectiveness, WCEC recommends completing the injection pilot test using PetroFix™ colloidal activated carbon (CAC) manufactured by Regenesis.

2.2 PetroFix™ Injection Summary

Regenesis developed PetroFix™ as a dual function colloidal activated carbon (CAC) consisting of a water-based suspension of micron-scale activated carbon and electron acceptors intended for in-situ treatment of dissolved phase hydrocarbons. When mixed with water and injected into the subsurface at low pressures as designed, the PetroFix solution will enter the same conductive zones that are preferentially inhabited by the hydrocarbons targeted for treatment. Recommended horizontal spacing between injection borings is typically 4 to 6 feet, ensuring that all of the transmissive zones in the impacted area are covered. Any dissolved phase hydrocarbons migrating through these transport zones that encounter the PetroFix solution will be adsorbed onto the activated carbon particles. Subsequently, the electron acceptor amendment stimulates microbial activity resulting in biodegradation of the adsorbed hydrocarbon source mass and reactivation of the carbon particles for further hydrocarbon adsorption in the future.

PetroFix is delivered in 55-gallon poly drums containing approximately 41 gallons of concentrated CAC remediation fluid weighing approximately 400 pounds. Cage totes of PetroFix are also available as a delivery option with an approximate weight of 2,000 pounds, equivalent to 5 drums (205 gallons). The concentrated PetroFix fluid is intended to be diluted with a calculated volume of water to achieve the ideal solution ratio based on design criteria relating to site-specific contaminant characteristics and hydrogeologic conditions. For each drum of PetroFix, there is a 20-pound pail of electron accepter blend that must be added to the water mixing tank along with the contents of the PetroFix drum prior to injection. Slight agitation of the mixing tank via pump recirculation or an impeller mixer is sufficient to keep the diluted PetroFix solution and electron acceptor blend in suspension while injection activities are progressing.

Appendix A contains the PetroFix Injection Grid Application Summary derived from site-specific inputs. The proposed injection area is shown on Figure 3. Based on the design input criteria including current groundwater concentrations and lithological characteristics (>75% silt/clay), an estimated total of 16 injection borings will be installed with application of 400 pounds of PetroFix remediation fluid and 20 pounds of electron acceptor blend. Horizontal spacing of the injection borings will be approximately 5 feet with a targeted vertical injection interval of 5 to 8 feet bgs. The injection solution will be prepared on site in a 275-gallon poly tote by mixing 20 gallons of PetroFix and 10 pounds of nitrate/sulfate electron acceptor blend with 255 gallons of water. Approximately 34 gallons of the PetroFix solution will be injected into each boring via a pressure activated injection tip attached to 1.5-inch probe rods. A Geoprobe DP800 pump will be used to inject the PetroFix solution into the targeted horizon using a "bottom-up" method. Injection pressures will be maintained within the recommended range for application of PetroFix solution at approximately 20 to 50 pounds per square inch (psi).

WCEC will perform process monitoring activities throughout the injection event as a quality control measure to confirm that the injection point installations are completed as designed. Total injection mass will be evaluated on a continual basis and injected solution volumes will be adjusted as needed. Delivery techniques and pumping rates will be refined as the injection event progresses to ensure that the entire target interval in each borehole receives an equivalent dose of solution. Top of borehole blowouts will be closely monitored and measures will be put in place to minimize leakage and maximize downhole delivery.

Field indicators of PetroFix influence in the subsurface will be monitored including visual confirmation of PetroFix distribution in soil cores retrieved with the direct push Geoprobe. Groundwater from monitoring wells MW-3NR and MW-8 will be inspected for the presence of PetroFix as signified by black coloration of the water. Semi-quantitative estimates of PetroFix concentrations in groundwater will be calculated using the CAC field test kit provided by Regenesis to further assess the degree of injectate distribution. Downhole pressure transducers will also be deployed in monitoring wells MW-3NR and MW-8 to enable instantaneous groundwater elevation measurements to be recorded while the injection is ongoing for radius of influence (ROI) calculations. Field parameter data will be collected from the wells both pre- and post-injection to assess potential changes in groundwater chemistry related to the injection.

At the completion of injection activities, all boreholes will be properly abandoned using chipped bentonite and resurfaced as appropriate to match the surrounding area, either concrete or asphalt. Borehole locations will be mapped relative to other site features using a survey grade GPS unit.

2.3 Groundwater Monitoring

One pre-injection and two post-injection groundwater monitoring events will be conducted to adequately evaluate groundwater concentrations before and after the PetroFix treatment. The two post-injection groundwater monitoring events will be performed on a semi-annual basis at 6 months and 12 months post-injection to assess the effectiveness of the treatment in reducing groundwater concentrations and monitor for potential rebound. Groundwater samples will be obtained from monitoring wells MW-3NR, MW-5, MW-6, MW-7, MW-8, MW-9, and MW-11R during each of the monitoring events. Any monitoring wells containing >100 mg/L CAC will be re-developed prior to sampling according to Regenesis guidance documents. If necessary, sample vials for VPH analysis will be treated with a flocculant compound (aluminum sulfate) to remove CAC from the water matrix.

Well sampling will be conducted according to WCEC Standard Operating Procedures (SOPs) and MTDEQ Guidance for low-flow sampling using a peristaltic pump for purging and sample collection [MTDEQ, 2018a]. Depth to water measurements will be recorded from the wells to provide an accurate potentiometric surface plot, flow direction, and gradient. Groundwater quality parameter data (conductivity, pH, salinity, dissolved oxygen, temperature, turbidity, and ORP) will be acquired during well purging using a flow through cell attached to the peristaltic pump. Purge water will be handled according to the MTDEQ Purge Water Disposal Flowchart.

Groundwater sample collection will be completed following stabilization of groundwater quality parameters. Groundwater quality parameter, purge, and stabilization data for each well are recorded in the field using WCEC's Well Sampling Form. If present, any accumulations of free product (FP) in the monitoring wells will be noted and FP thicknesses will be recorded. Groundwater samples will not be collected from any wells that contain a measurable thickness of FP.

Groundwater samples will be preserved with hydrochloric acid, packed on ice, and delivered to Energy Laboratories in Helena, Montana under chain of custody. All groundwater samples will be submitted for analysis of VPH and EPH constituents as outlined in MTDEQ guidance [MTDEQ, 2018b]. TEH fractions with PAHs analysis will be conducted if the EPH screen exceeds 1,000 µg/L. Groundwater samples will also be analyzed for intrinsic biodegradation parameters (IBIs) to assess microbial activity and evaluate the distribution of the PetroFix electron acceptor amendment.

2.4 Monitoring Well Survey

The horizontal location and top of casing elevation for monitoring well MW-3NR will be surveyed according to MTDEQ requirements. WCEC will obtain horizontal coordinates for the monitoring well using a Trimble Geo7X centimeter GPS referenced to a temporary control point set in Montana State Plane coordinates, US Survey Feet. A survey of the vertical well casing elevation will be completed to Fourth Order accuracy using a Nikon Ax-2s auto-level transit with a measurement precision of 0.01 feet. The top of casing elevation will be correlated to the North American Vertical Datum of 1988 (NAVD 88) using a GPS derived onsite control point and will be cross referenced for consistency with current elevations at existing monitoring wells.

2.5 Reporting

WCEC will prepare and submit an Interim Data Submittal (IDS) for the pre-injection and first post-injection groundwater monitoring events. Each IDS will include a discussion of the event, data, tables, and figures described in the MTDEQ Groundwater Monitoring Work Plan and Report Guidance for Petroleum Releases. Laboratory analytical data will be validated using DEQ's Data Validation Summary Form, with an individual Form appended for each laboratory analytical report.

Following receipt of results from the final post-injection groundwater monitoring event included in this work plan, WCEC will prepare and submit a Generic Applications Corrective Action Report (Report AR-07) detailing the results of the injection pilot test and all groundwater monitoring events. The report will include figures depicting historic, current, and planned site features, cumulative data tables for soil and groundwater, and appendices outlined in the AR-07 Report format guidance as appropriate for the scope of work completed. The Release Closure Plan (RCP) will be updated to include data collected during the pilot test and a thorough evaluation and discussion with recommendations for further corrective actions will be presented in the RCP and AR-07 Report.

3.0 Project Timeline & Costs

WCEC recommends conducting the PetroFix injection pilot test during low groundwater conditions in October 2024, pending review by MTDEQ and the Petroleum Tank Release Compensation Board (PTRCB). The attached *Estimated Costs – Additional Corrective Action Work Plan 34852* spreadsheet and *PTRCB Groundwater Sampling & Unit Cost Worksheet* detail anticipated project costs to complete the MTDEQ required scope of work.

3.1 Planned Workflow

WCEC anticipates completing the scope of work outlined in this work plan during four individual field events with completion and reporting milestones as follows:

Event 1 – September 2024: Pre-injection groundwater monitoring and sampling event.

Event 2 – October 2024: PetroFix injection during low groundwater conditions.

Interim Data Submittal (IDS) – January 2025: IDS for review by MTDEQ.

Event 3 – April 2025: Post-injection semi-annual groundwater monitoring and sampling event (6 months post-injection).

Interim Data Submittal (IDS) – July 2025: IDS for review by MTDEQ.

Event 4 – October 2025: Post-injection semi-annual groundwater monitoring and sampling event (12 months post-injection).

Groundwater Monitoring Report – January 2026: Report summarizing analytical results from pre- and post-injection groundwater monitoring events, includes complete documentation of the October 2024 PetroFix injection field processes and performance monitoring.

WCEC will implement the remedial actions proposed in this work plan pending review from MTDEQ and PTRCB.

4.0 References

Applied Water Consulting, LLC. (AWC, 2013). *Additional Remedial Investigation Report for the Petroleum Release at Former Long's Main Stop*. April 12, 2013.

Applied Water Consulting, LLC. (AWC, 2015). *Additional Remedial Investigation Report for the Former Long's Conoco*. January 27, 2015.

Applied Water Consulting, LLC. (AWC, 2018). *Groundwater Sampling and Hydraulic Conductivity Assessment Report (AR-07) for the Petroleum Release at the Former Long's Main Stop (Kelly Rae's)*. February 14, 2018.

Applied Water Consulting, LLC. (AWC, 2019). *Petroleum Contaminated Soil Remedial Excavation at Kelly Rae's (Formerly Long's Main Stop)*. March 25, 2019.

LaFave, J.I. (LaFave, 2004). *Potentiometric Surface Map of the Deep Aquifer, Kalispell Valley: Flathead County, Montana*. Montana Bureau of Mines and Geology: Groundwater Assessment Atlas 2, Part B, Map 2.

Montana Department of Environmental Quality. (MTDEQ, 2018a). *Groundwater Sampling Guidance.* March 6, 2018.

Montana Department of Environmental Quality. (MTDEQ, 2018b). *Montana Risk-Based Corrective Action Guidance for Petroleum Releases*. May 2018.

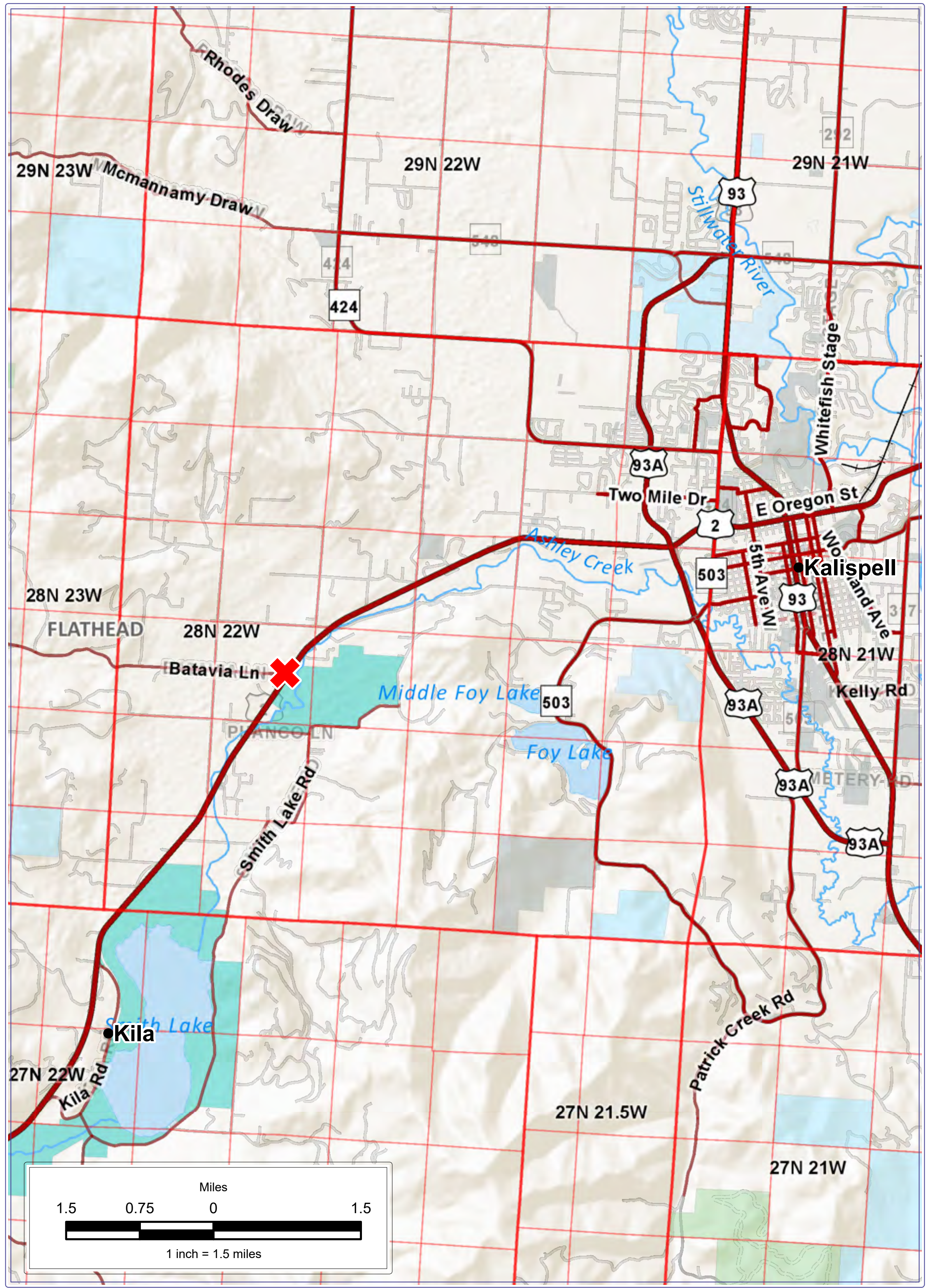
Smith, L.N. (Smith, 2004). *Surficial Geologic Map of the Upper Flathead River Valley (Kalispell Valley) Area, Flathead County, Northwestern Montana*. Montana Bureau of Mines and Geology: Groundwater Assessment Atlas 2, Part B, Map 6.

Water & Environmental Technologies. (WET, 2020). *Soil Boring, Monitoring Well Installation, and Groundwater Monitoring Report for Kelly Rae's (Former Longs Conoco)*. November 11, 2020.

Water & Environmental Technologies. (WET, 2023). *Groundwater Monitoring Interim Data Submittal for the Petroleum Release at Kelly Rae's*. December 13, 2023.

Water & Environmental Technologies. (WET, 2024). *Remedial Investigation Report for Kelly Rae's*. March 1, 2024.

Figures



N

Site Location

Site Location

Kelly Rae's
25 Batavia Lane
Kalispell, MT

DRAWN BY: MM
DATE: 05/14/24
SCALE: 1:12,000

WCEC
ENVIRONMENTAL CONSULTANTS

PROJECT NUMBER: 2404-1006
IMAGE SOURCE: ESRI BASEMAPS

FIGURE 1

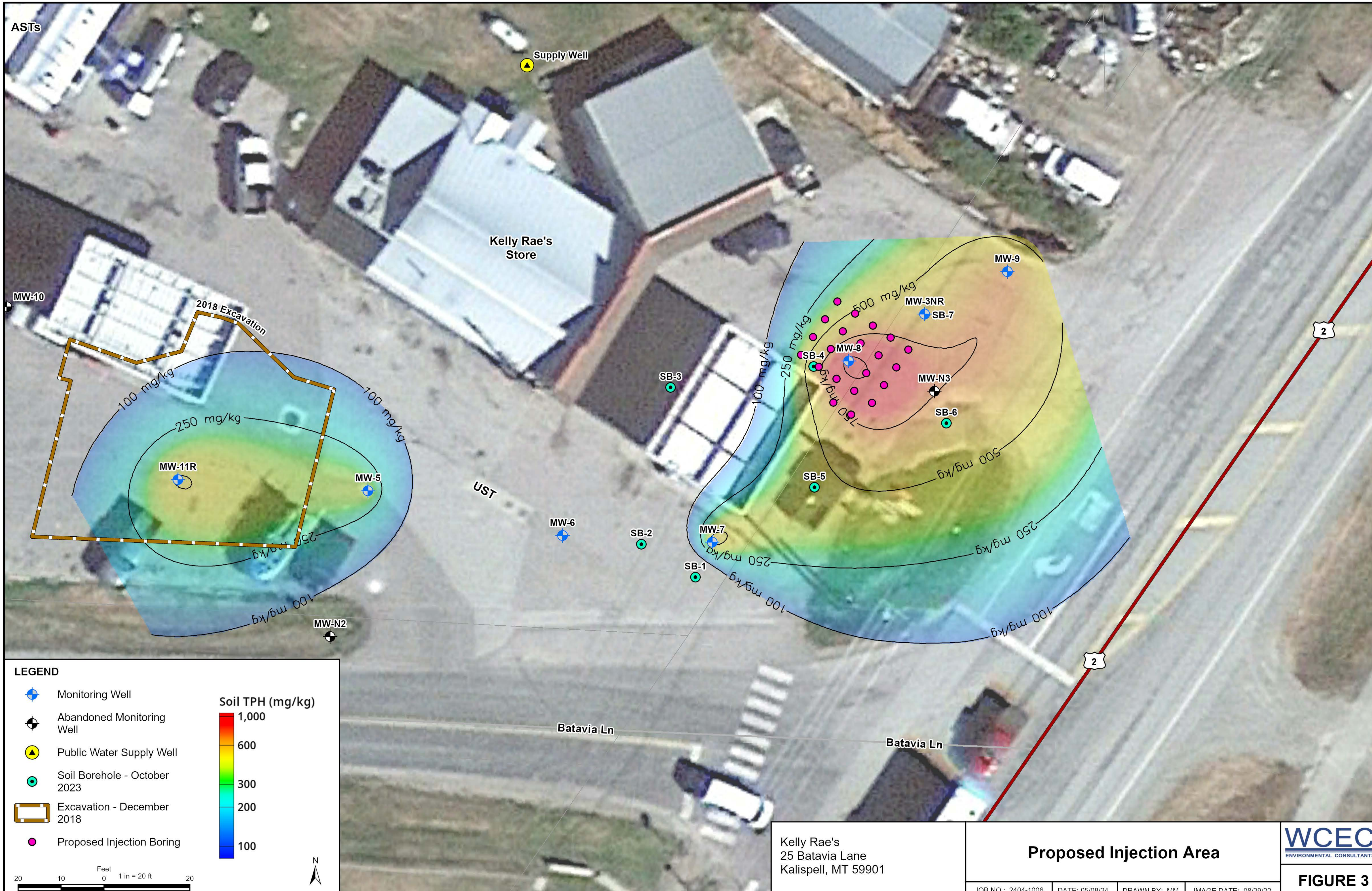
Kelly Rae's
25 Batavia Lane
Kalispell, MT 59901

Site Details

JOB NO.: 2404-1006 | DATE: 05/15/24 | DRAWN BY: MM | IMAGE DATE: 08/29/22

WCEC ENVIRONMENTAL CONSULTANTS

FIGURE 2



Kelly Rae's
25 Batavia Lane
Kalispell, MT 59901

Proposed Injection Area

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FIGURE 3

JOB NO.: 2404-1006 | DATE: 05/08/24 | DRAWN BY: MM | IMAGE DATE: 08/29/22

Appendix A

PetroFix™ Injection Grid Application Summary

Injection Grid Application Summary

Kelly Rae's

Pilot Test

PetroFix Amount	400 lb
Electron Acceptor	20 lb
Treatment Surface Area	400.0 ft^2
Delivery Points	16
Point Spacing	5.0 ft
Top of Treatment Interval	5.0 ft bgs
Bottom of Treatment Interval	8.0 ft bgs
Vertical Treatment Interval Thickness	3.0 ft
Treatment Volume	44 yd^3
PetroFix Dose	9.0 lb/yd^3

Total Volume	549 gal
Product Volume	41 gal
Water Volume	508 gal
Injection Volume/Point	34 gal
Inject Volume/Vertical ft	11 gal
Product/Point	2.6 gal
Water/Point	31.8 gal
Soil Type	>75% silt/clay
Effective Pore Volume Fill %	41%

Mix Tank Volume*	275.0 gal
Dilution Factor*	13.42
PetroFix per Mix Tank	20 gal
Water per Mix Tank	255 gal
Electron Acceptor per Mix Tank	10 lb
Total Batches Required	2.0

Specific Area Notes

Native Soil Type: >75% silt/clay

Reported Ground Water Concentrations (µg/L)

Benzene	69	Naphthalenes	662
Toluene	35	MTBE	0
Ethylbenzene	220	TPH-GRO	2,940
Xylenes	341	TPH-DRO	890
Trimethylbenzenes	0	**Sum of Dissolved Concentrations:**	**4,492**

Kelly Rae's

LAST UPDATED
05.23.24

Project Location
25 Batavia Lane
Kalispell, MT, 59901
UNITED STATES

Areas

Name	Size	Type	Dose	Product
Pilot Test	400.0sq.ft	Grid	9.0lbs/cy	400lbs

Total PetroFix Amount	**400 lbs**
Electron Acceptor	**20.0 lbs**
Product Cost	**$2,228.00**
Estimated Shipping Cost	**$401.00**
Estimated Project Cost	**$2,629.00**

*We use 18% of product cost as a good approximation for shipping cost.

Appendix B

Estimated Costs – Additional Corrective Action Work Plan 34852

PTRCB Groundwater Monitoring & Sampling Unit Cost Worksheet

REGENESIS®

Colloidal Activated Carbon Flocculation Method Validation

PetroFIX® Remediation Fluid

PLUMESTOP® Liquid Activated Carbon

Description and Contaminant Analytical Impacts

Test Vial Settling Agent

Figure 1 – Two vials containing 2,000 mg/L of CAC. The recommended dose of settling agent was also added to the vial on the right, resulting in clear water.

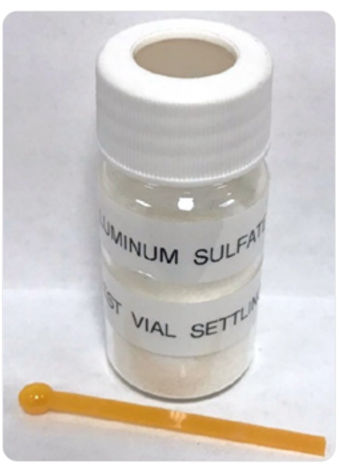


Figure 2 – ALUM kit with dosing scoop

After application of REGENESIS® colloidal activated carbon (CAC) products, groundwater and occasionally the water in monitoring wells will be impacted by the presence of suspended CAC. Within weeks or occasionally months, the CAC will fully attach to the aquifer matrix and the water will again become clear. The presence of CAC above approximately 100 mg/L (i.e.- water in a standard VOA vial too dark to see through) can have a negative impact on the methods and instruments used to quantify volatile organic compounds (VOCs) in water by standard methods like EPA 8260. To remove the CAC from analytical samples and permit VOC sampling, REGENESIS developed a method for flocculating suspended colloidal activated carbon directly in sampling vials **(Figure 1)**. For further information about the method and its use, please refer to the REGENESIS document: *Settling Agent for Test Vials*.

Verification of Settling Method: Effect on Aqueous VOC Concentrations

The objective for this method is to remove the suspended CAC from the water column while leaving the aqueous VOC concentration in the samples undisturbed. By adding a small amount of aluminum sulfate (ALUM) to a sample, the CAC will flocculate and fall to the bottom of the vial. This addition of a flocculant has minimal effect on any dissolved contaminants that may be present, as the ALUM does not affect the partitioning between the CAC and the water. A series of samples at various contaminant, ALUM, and CAC concentrations were evaluated to verify that ALUM does not cause changes in aqueous VOC concentrations.

About the Test Development

Effect of ALUM in the Absence of CAC

The presence of ALUM on the aqueous concentration for selected contaminants of interest was explored. Chlorinated volatile organic compounds and BTEX concentrations were tested after mixing with ALUM. Tetrachloroethylene, trichloroethylene, and cis-dichloroethylene were analyzed from 0.25 mg/L to 10 mg/L in the presence of 1 g/L ALUM. Analysis was performed via gas chromatography–mass spectrometry (GC-MS) using a head space sampling method. Samples were prepared in VOA vials with 40 mL of water and then spiked with CVOCs from a mixed 1000 mg/L stock to the concentrations listed. Alum was then added to each sample and the samples allowed to equilibrate for over 24 hours before aliquots were taken for analysis. The data showed that the addition of ALUM had a minimal effect on the contaminant levels vs control **(Table 1)**. The results were biased high and biased low with an average deviation of under 5% which is within the range of error for calibration and preparation. Benzene, toluene, ethylbenzene, and o-xylene were analyzed from 0.25 mg/L to 10 mg/L in the presence of 1 g/L ALUM via gas chromatography–mass spectrometry (GC-MS). Samples were prepared in VOA vials with 40 mL of water and then spiked with BTEX from a mixed 1000 mg/L stock to the concentrations listed. Alum was then added to each sample and allowed to shake for over 24 hours to equilibrate before aliquots were taken for analysis. The data showed that the addition of ALUM had a minimal effect on the contaminant levels vs control **(Table 3)**. The results were biased high and biased low with an average deviation of under 5% which is within the range of error for calibration and preparation.

Chlorinated Volatile Organic Compounds (CVOCs) with ALUM

Contaminant Concentration (mg/L)	Deviation from Control		
	PCE	TCE	Cis-DCE
0.25	-4.38%	-1.47%	4.32%
0.5	9.95%	0.76%	0.97%
2	-5.69%	-16.81%	-19.14%
5	5.87%	-4.82%	-8.62%
10	0.63%	0.38%	0.59%
Average	**-1.27%**	**-4.39%**	**-4.38%**

Table 1 – Effect of 1g/L ALUM on CVOC concentration analysis via GCMS.

Benzene, Toluene, Ethylbenzene, and O-Xylene with ALUM

Contaminant Concentration (mg/L)	Deviation from Control			
	Benzene	Toluene	Ethylbenzene	O-Xylene
1	0.24%	2.01%	3.77%	4.14%
2	-4.94%	-5.86%	-6.81%	-7.94%
5	-1.22%	-1.34%	-2.07%	-2.16%
10	-8.28%	-9.95%	-10.67%	-10.62%
Average	**-3.55%**	**-3.79%**	**-3.95%**	**-4.14%**

Table 3 – Effect of 1g/L ALUM on BTEX concentration analysis via GCMS.

Effect of ALUM in the Presence of CAC

Chlorinated volatile organic compound concentrations were analyzed with colloidal activated carbon after addition of ALUM to determine ALUM's effect on contaminant analysis. This experiment was performed using 500 mg/L CAC, 0.5 mg/L to 10 mg/L of CVOC, and 2 g/L ALUM in all samples with analysis via gas chromatography with an Electron Capture Detector (GC-ECD) by headspace. PCE and cis-DCE were selected to represent CVOCs as the contaminants with the highest and lowest sensitivity on GC-ECD respectively. To obtain 0.5 mg/L to 10 mg/L of contaminant in samples in the presence of CAC, isotherms were used to calculate the correct amount of neat contaminant to spike into each sample.

PCE and cis-DCE samples were prepared individually to match the isotherms calculated. Samples were prepared in VOA vials with 38 mL of 500 mg/L CAC and then injected with either neat PCE or cis-DCE to the concentrations listed. Then the samples were allowed to shake for 24 hours and then refrigerated for 24 hours to allow all contaminants to fully dissolve in solution. The samples were then injected with 2 mL of 35 g/L ALUM to obtain 2 g/L ALUM in the samples and allowed 2 hours to settle before aliquots were run on GC-ECD headspace. The data showed that the addition of ALUM had a minimal effect which caused the results to bias high for both contaminants **(Table 2)**. The data from this experiment support the notion that the addition of ALUM to flocculate CAC from aqueous samples may only slightly increase the measured CVOC concentrations, and that the use of ALUM as a settling agent is not leading to biased favorable results.

Chlorinated Volatile Organic Compounds (CVOCs) with Activated Carbon

Contaminant Concentration (mg/L)	Activated Carbon Concentration (mg/L)	Deviation from Control	
		PCE	Cis-DCE
0.25	500	29.41%	NA
0.5	500	34.87%	NA
2	500	31.47%	18.33%
5	500	8.3%	4.75%
10	500	NA	9.69%
Average		**26.01%**	**10.93%**

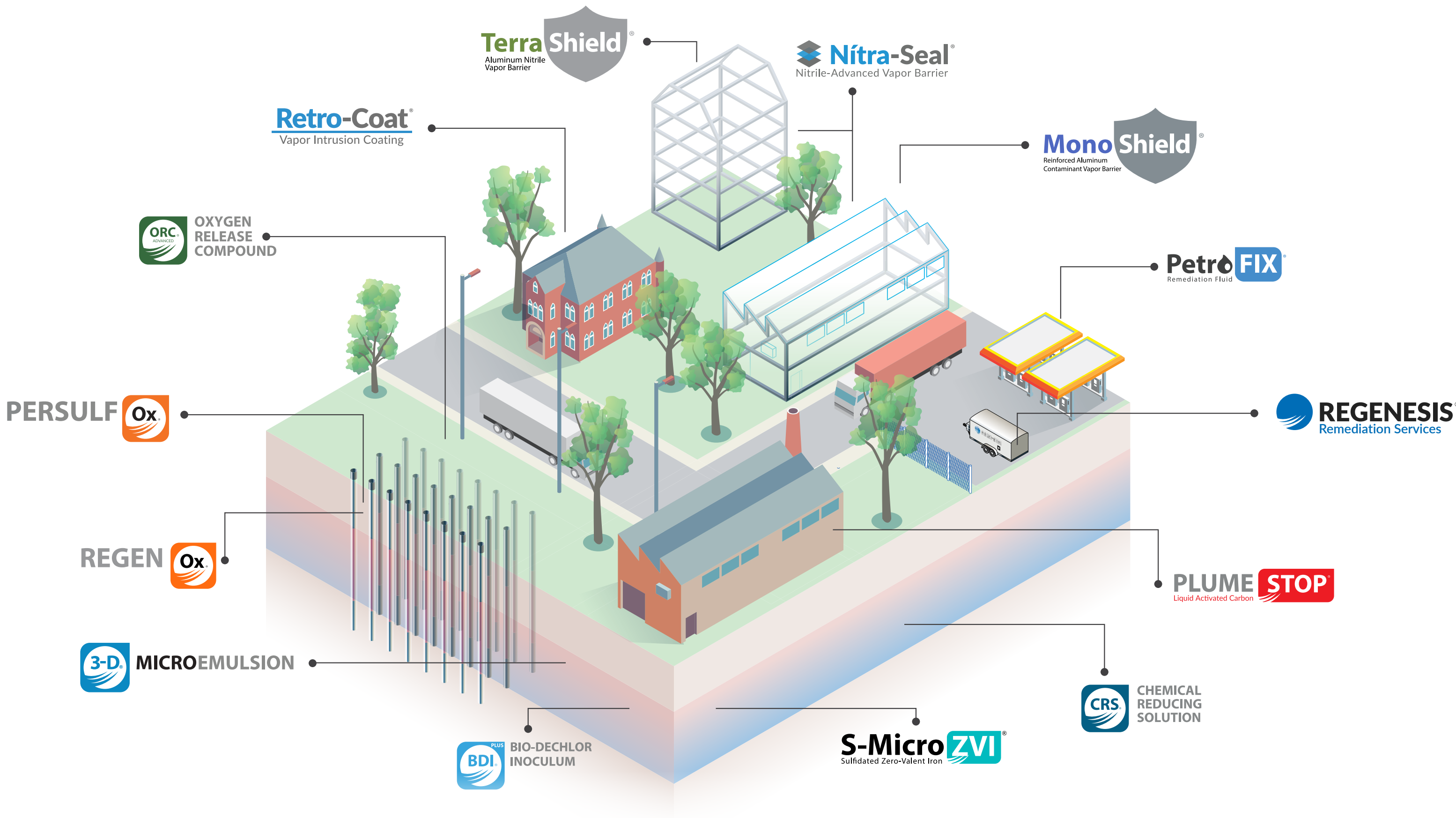
Table 2 – Effect of 2g/L ALUM on CVOC concentration analysis with 500 mg/L activated carbon analysis via GC-ECD.

Benzene and O-Xylene with Activated Carbon

Contaminant Concentration (mg/L)	Activated Carbon Concentration (mg/L)	Deviation from Control	
		Benzene	O-Xylene
1	500	24.31%	NA
2	500	2.99%	24.31%
5	500	2.87%	17.51%
10	500	15.09%	19.11%
Average		**11.32%**	**20.31%**

Table 4 – Effect of 2g/L ALUM on BTEX concentration analysis with 500 mg/L activated carbon analysis via GC-FID.

BTEX concentrations were analyzed with colloidal activated carbon after addition of ALUM to determine ALUM's effect on contaminant analysis. This experiment was performed using 500 mg/L CAC, 1 mg/L to 10 mg/L of BTEX, and 2 g/L ALUM in all samples with analysis via gas chromatography with a Flame Ionization Detector (GC-FID) by headspace. Benzene and o-xylene were selected to represent BTEX as the contaminants with the highest and lowest sensitivity on GC-FID respectively. To obtain 1 mg/L to 10 mg/L of contaminant in samples in the presence of CAC, isotherms were used to calculate the correct amount of neat contaminant to spike into each sample. Benzene and o-xylene samples were prepared individually to match the isotherms calculated. Samples were prepared in VOA vials with 38 mL of 500 mg/L CAC and then injected with either neat benzene or o-xylene to the concentrations listed. Then the samples were allowed to shake for 24 hours and then refrigerated for another 24 hours to allow all contaminants to fully dissolve in solution. The samples were then injected with 2 mL of 35 g/L ALUM to obtain 2 g/L ALUM in the samples and allowed 2 hours to settle before aliquots were run on GC-FID headspace. The data showed that the addition of ALUM had a minimal effect which caused the results to bias high for both contaminants **(Table 4)**. The data from this experiment support the notion that the addition of ALUM to flocculate CAC from aqueous samples may only slightly increase the measured BTEX concentrations, and that the use of ALUM as a settling agent is not leading to biased favorable results.



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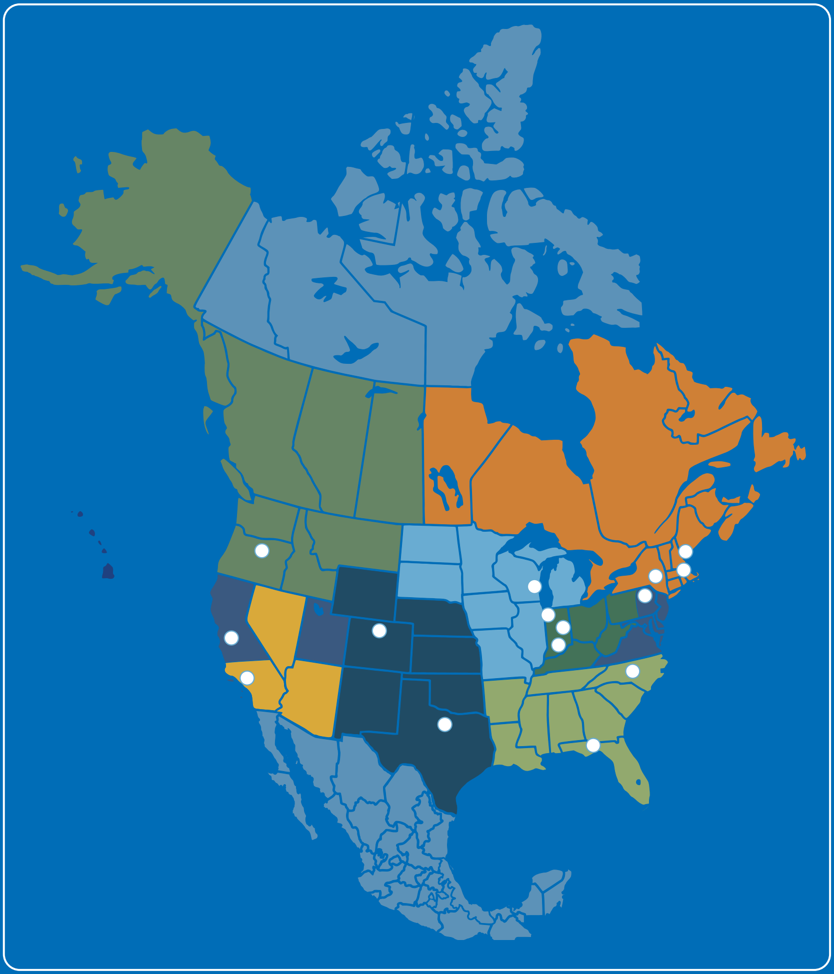

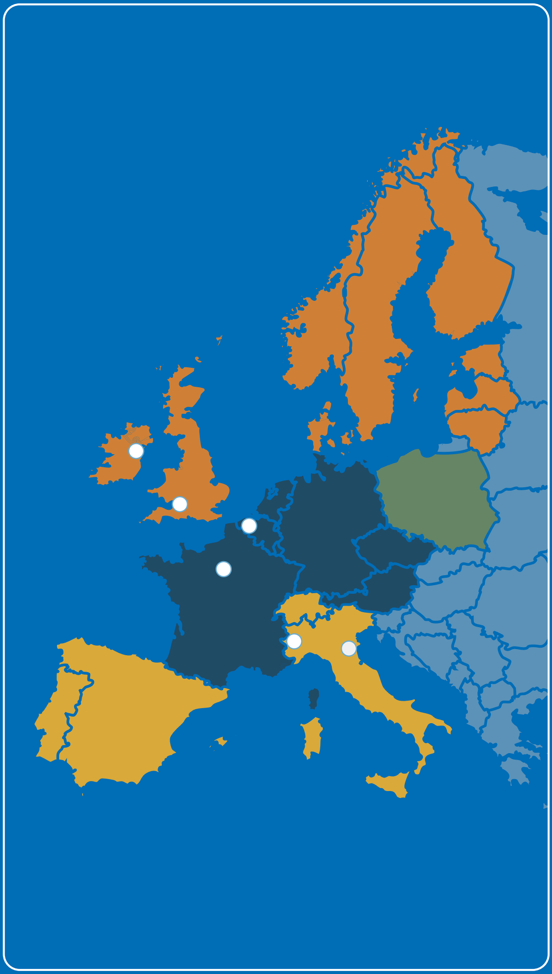

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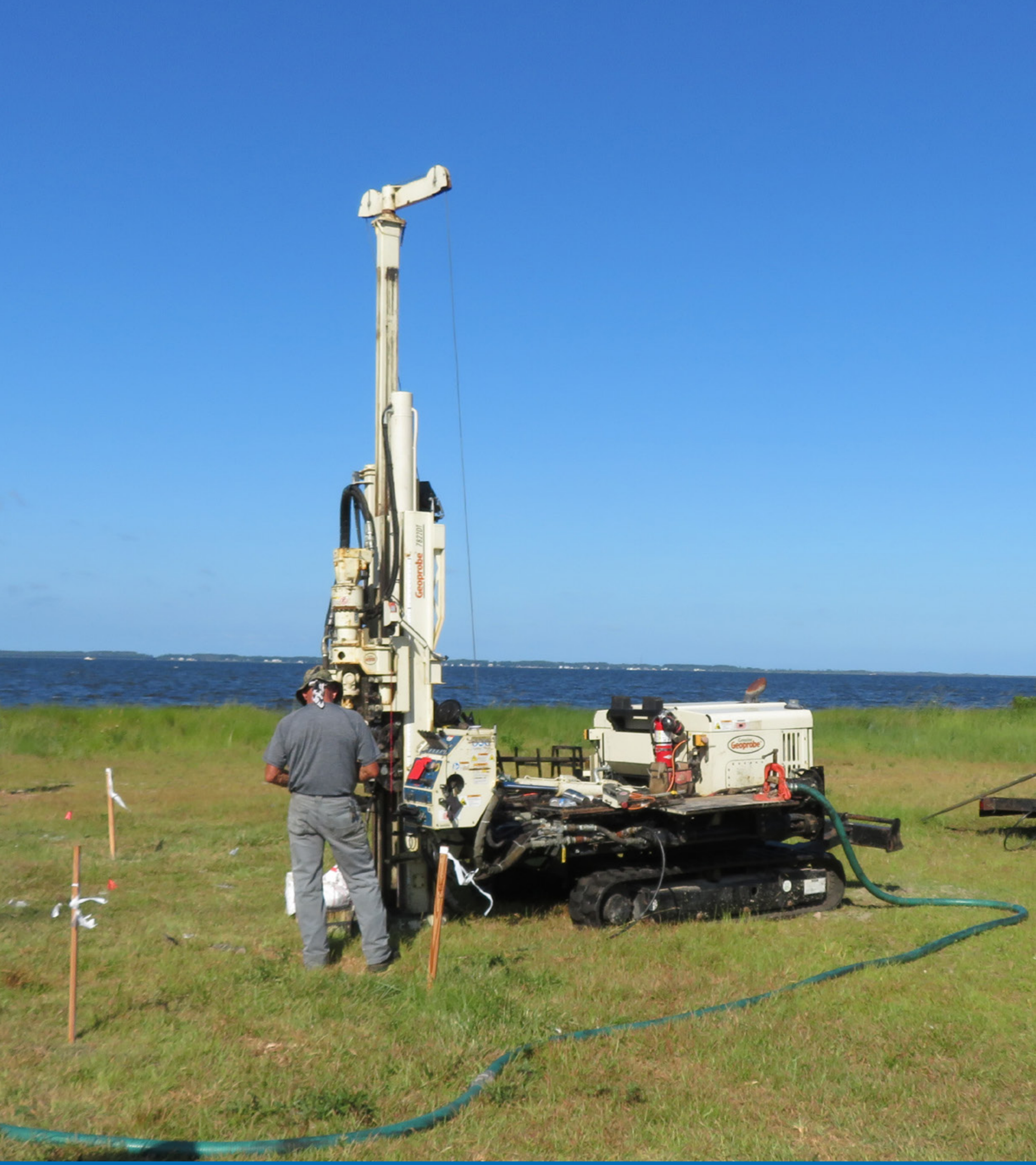

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