J.H. Campbell Generating Facility

Dry Ash Landfill and Cell 6 Expansion - Post-Closure Plan

Pursuant to:
40 CFR 257.104

Submitted to:

Consumers Energy Company
1945 W. Parnall Road
Jackson, Michigan USA 49201

Submitted by:

Golder Associates Inc.
15851 South US 27, Suite 50,
Lansing, Michigan, USA 48906
+1 517 482-2262

21451121

December 2021

CERTIFICATION

Professional Engineer Certification Statement [40 CFR 257.104(d)(4)]

I hereby certify that, having reviewed the attached documentation and being familiar with the provisions of Title 40 of the Code of Federal Regulations Section 257.104 (40 CFR Part 257.104), I attest that this Post-Closure Plan is accurate and has been prepared in accordance with good engineering practices, including the consideration of applicable industry standards, and with the requirements of 40 CFR Part 257.104.

Golder Associates Inc.

Signature

12/22/2021
Date of Report Certification

Jeffrey R. Piaskowski, PE
Name

6201061033
Professional Engineer Certification Number

STATE OF MICHIGAN
JEFFREY R. PIASKOWSKI
ENGINEER
No.
6201061033
LICENSED PROFESSIONAL ENGINEER

Table of Contents

TABLES

FIGURES

APPENDICES

1.0 INTRODUCTION

On April 17, 2015, the United States Environmental Protection Agency (USEPA) issued the Coal Combustion Residual (CCR) Resource Conservation and Recovery Act (RCRA) Rule (40 CFR 257 Subpart D) ("CCR RCRA Rule") to regulate the beneficial use and disposal of CCR materials generated at coal-fired electrical power generating complexes. In accordance with the CCR RCRA Rule, any CCR surface impoundment or CCR landfill that was actively receiving CCR on the effective date of the CCR RCRA Rule (October 19, 2015) was deemed to be an "Existing CCR Unit" on that date and subject to self-implementing compliance standards and schedules. Consumers Energy Company (CEC) currently operates the Dry Ash Landfill CCR unit (Dry Ash Landfill) at the J.H. Campbell Generating Facility (JH Campbell). JH Campbell is located in West Olive, Michigan as presented on Figure 1 – Site Location Map. The location of the Dry Ash Landfill is presented on Figure 2 – Site Map.

The JH Campbell Dry Ash Landfill was permitted as a Type III landfill by the Michigan Department of Natural Resources (MDNR) in 1993 and licensed under State of Michigan Part 115, Solid Waste Management of the Natural Resources and Environmental Protection Act, 1994 PA 451, as amended (Part 115). In March 2018, CEC submitted a permit upgrade request (Golder 2018) to the Michigan Department of Environment, Great Lakes, and Energy, Materials Management Division (EGLE) for the JH Campbell Dry Ash Landfill. The permit update request was submitted to comply with EGLE's Part 115 rules and the RCRA CCR Rule for lateral expansions of CCR units. The permit upgrade request was approved by EGLE in June 2018 (EGLE 2018).

On July 13, 2021 CEC submitted the JH Campbell Generating Facility – Dry Ash Landfill Expansion Construction Permit Application (CPA) (Golder 2021) to the Michigan Department of Environment, Great Lakes, and Energy (EGLE) Office of Waste Management and Radiological Protection. The CPA included lateral expansion of landfill cells 6-9 and a 42.9 acre vertical expansion to regain 532,000 cubic yards of airspace that was lost in the modification of the landfill floor grades in 2018. The CPA was submitted to comply with Part 115 and the RCRA CCR Rule for lateral expansions of CCR units. The permit upgrade request was granted by EGLE in a letter dated November 30, 2021 (EGLE 2021).

This written post-closure plan serves as the initial post-closure plan for the JH Campbell Dry Ash Landfill Cell 6 Expansion and an amended post-closure plan for the JH Campbell Dry Ash Landfill and Cell 5 Expansion. The post-closure plan was generated pursuant to 40 CFR 257.104(a) and describes the steps necessary to assure that integrity and effectiveness of the final cover is maintained over the 30-year post-closure care period. The Dry Ash Landfill is anticipated to be certified closed by December 31, 2040 with the post-closure care period lasting through December 31, 2070.

2.0 FACILITY CONTACT [40 CFR 257.104(d)(1)(ii)]

The post-closure point of contact for the Dry Ash Landfill at JH Campbell is:

Michelle Marion
1945 W. Parnall Road
Jackson, Michigan 49201
(517) 788-5824
michelle.marion@cmsenergy.com

3.0 MONITORING AND MAINTENANCE ACTIVITIES [40 CFR 257.104(d)(1)(i,iii)]

3.1 Site Maintenance [40 CFR 257.104(d)(1)(i)]

The following general site maintenance and monitoring will be conducted to ensure the integrity and effectiveness of the final cover system:

- Fertilizer will be applied if needed to improve vegetation quality where areas of stressed or poor-quality cover vegetation exist.
- Vegetative cover will be mowed as needed to restrict uncontrolled woody plant establishment on the cover for the remainder of the 30-year post-closure period (estimated through 2070). This includes mowing the side slopes around the perimeter of the Dry Ash Landfill.
- Areas of erosion, including erosion from run-off or vehicle use, will be repaired by restoring the thickness of the protective cover and topsoil and seeding as necessary upon discovery.
- Erosion repairs will utilize clean soils. Typically, repairs are expected to involve minor regrading, spreading of small amounts of additional soil, and reseeding. Areas of repeated erosion will be evaluated to determine if additional protection such as erosion blankets or riprap should be added.
- The groundwater monitoring system will be maintained in accordance with applicable requirements from 40 CFR 257.90 to 40 CFR 257.98.
- The leachate collection and removal system will be maintained in accordance with applicable requirements from 40 CFR 257.70(d).
- Differential settlement will be repaired as follows:
  - Minor differential settlement in which no ponding can occur or in which the subsurface drainage will not be compromised will be repaired by stripping topsoil, adding sandy soil, and replacing topsoil to attain a smooth surface before seeding.
  - If differential settlement has occurred to the extent that drainage is compromised, surface soils will be removed in the area to expose the geomembrane. The geomembrane will be cut back and sand added to attain the line grade. Geomembrane, protective soil, and topsoil will be replaced and seeded with repair certification maintained in the site files.

Areas requiring repair due to erosion or settlement will be identified during annual site inspections, which are detailed below in Section 3.2.

3.2 Periodic Inspection Requirements [40 CFR 257.104(d)(1)(i)]

Periodic site inspections verifying the integrity and effectiveness of the final cover system will be conducted throughout the 30-year post-closure period (estimated through December 2070). The inspections will be conducted no less frequently than quarterly for the first 5 years after closure certification to ensure that conditions at the facility are in compliance with plans, specifications and the CCR and Part 115 rules. After the final cover system is established at the end of 5 years, the inspections will be conducted no less than on an annual basis. When and if items requiring construction and/or maintenance are identified during an inspection, CEC will schedule and conduct repairs promptly while noting the risk associated with the deficiency. During site inspections, the inspector will walk the entire closed JHC Dry Ash Landfill area and document the problematic items on the Post Closure Inspection Checklist provided in Appendix A.

If maintenance is required, only low ground-pressure tire or track equipment should be utilized to correct the deficiencies on closed portions of the Dry Ash Landfill. Larger equipment can be used, but equipment loading cannot exert more than five pounds per square inch (psi) on the liner material.

If repairs to the geosynthetics (e.g., geomembrane, geotextile, etc.) are necessary, a certified geosynthetic installer must conduct the repairs under the direction of a quality assurance representative. Repairs will be documented in a report, and a copy will be placed in the site's operating record.

3.3 Site Use Restrictions [40 CFR 257.104(d)(1)(iii)]

Currently, the identified end use for the Dry Ash Landfill at JH Campbell has been limited to securing the area and maintaining the site as described in Sections 3.1 and 3.2. If the area is to be developed in the future, the integrity of the geomembrane cover liner will be confirmed with the proposed use; and institutional controls for maintaining the integrity of the geomembrane cover will be provided through an update to the Post-closure Plan. Once certified closed, CEC will ensure that the existing Restrictive Covenants recorded on February 12, 1999 and May 10, 2021 conform to the notation on the deed to the property required in 40 CFR 257.102(i).

To restrict the use of intrusive vehicles and activities at the site; fencing, gating, and procedures to prohibit access other than for inspections, maintenance, monitoring, and established easements will be followed.

3.4 Groundwater Monitoring

The groundwater monitoring system consists of fourteen groundwater monitoring wells that were established around the Dry Ash Landfill Cell 5 and Cell 6 Expansions. The groundwater monitoring well locations are provided on Figure 2 – Site Map.

A hydrogeological monitoring plan (HMP) was developed for the groundwater monitoring program at the JHC Dry Ash Landfill. The plan is included in Appendix B – Dry Ash Landfill Hydrogeological Monitoring Plan and includes direction on how to perform or acquire the following:

- Groundwater elevations
- Sample collection and handling procedures
- Equipment decontamination procedures
- Chain of custody control
- Sample preservation and shipment
- Quality assurance/quality control (QA/QC)
- Investigation derived waste (IDW)
- Field documentation
- Analytical suite and procedures
- Optional additional analyses
- Data evaluation

Once the CCR unit is certified closed, post-closure periodic groundwater samples will be collected at least semi-annually per the HMP and analyzed for 30 years for the following constituents in Table 3.4.1 – Groundwater Detection Monitoring Constituents.

Table 3.4.1 - Groundwater Detection Monitoring Constituents

Common Name	
Boron	Fluoride
Calcium	Sulfate
Chloride	Total Dissolved Solids (TDS)
pH	-

If a statistically significant increase over background levels for one or more of the constituents listed in Table 3.4.1 is detected during groundwater detection monitoring, then CEC will follow the procedures outlined in 40 CFR 257.93(h) and 257.94(e). If required by 40 CFR 257.94(e), an assessment groundwater monitoring program will be established meeting the requirements of 40 CFR 257.95 for the constituents presented in Table 3.4.2 – Groundwater Assessment Monitoring Constituents. The data will be presented in an annual groundwater monitoring and corrective action report per 40 CFR 257.90(e).

Table 3.4.2 - Groundwater Assessment Monitoring Constituents

Common Name		
Antimony	Chromium	Mercury
Arsenic	Cobalt	Molybdenum
Barium	Fluoride	Selenium
Beryllium	Lead	Thallium
Cadmium	Lithium	Radium 226 and 228 combined

The annual groundwater monitoring and corrective action reports will be:

- Maintained in the JH Campbell operating record per 40 CFR 257.105(h)(1)
- Submitted to EGLE per the notification requirement in 40 CFR 257.106(h)(1)
- Posted on a publicly accessible internet website per 40 CFR 257.107(h)(1)

If additional notification is warranted, CEC will notify appropriate parties per 40 CFR 257.106(h).

4.0 REFERENCES

Golder Associates Inc. March 2018. J.H. Campbell Dry Ash Landfill, Construction Permit Upgrade Request, Cells 5 through 9.

Golder Associates Inc. June 2021. J.H. Campbell Generating Facility – Dry Ash Landfill Expansion Construction Permit Application.

Michigan Department of Environmental Quality. June 2018. Construction Permit number 0299 - upgrade approval letter.

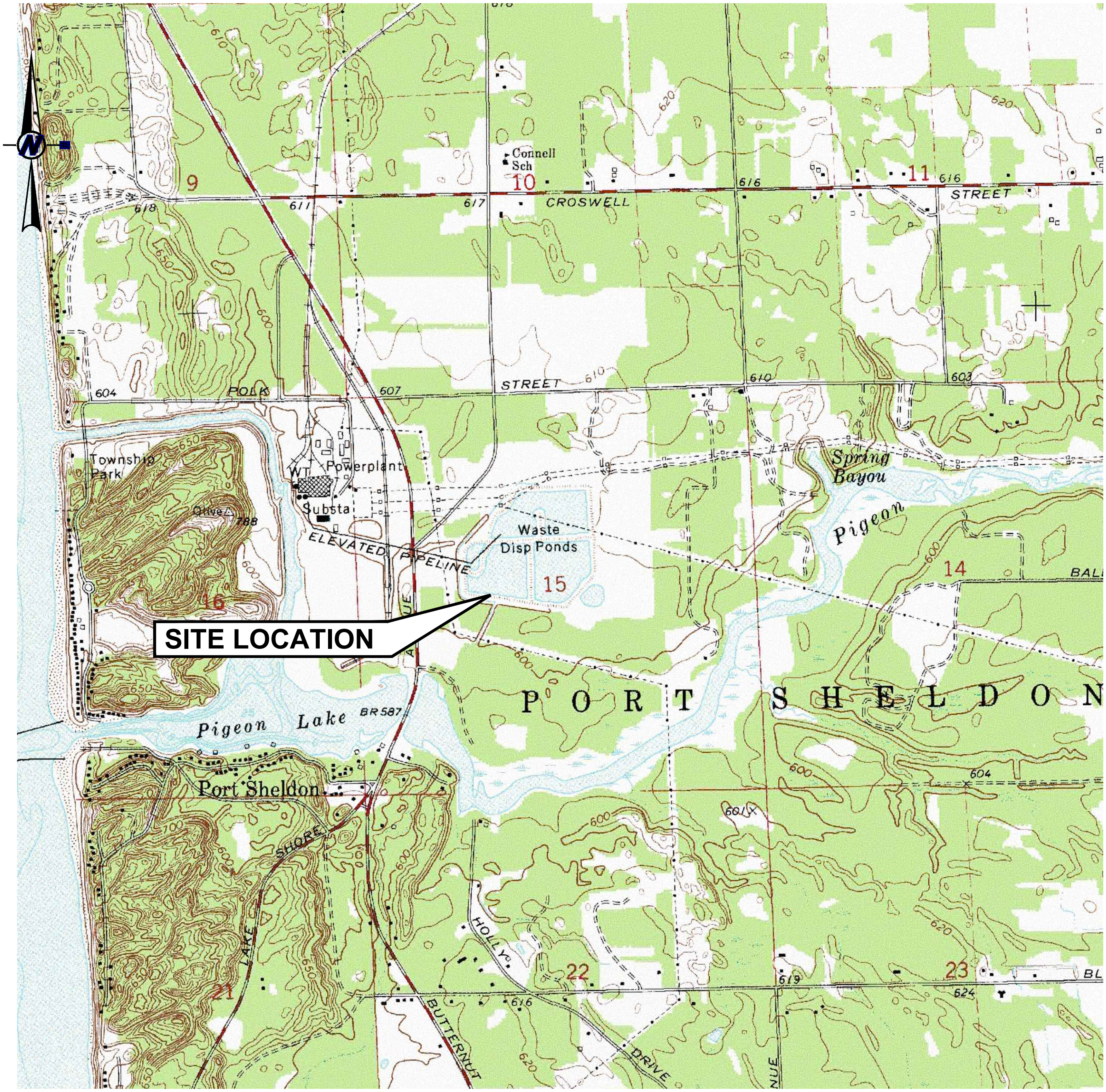
Michigan Department of Environment, Great Lakes, and Energy. November 30, 2021. Construction Permit Application Approval Letter and Permit.

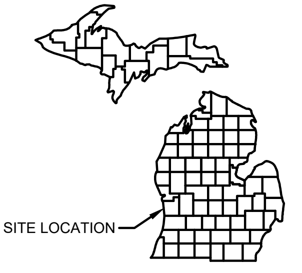
State of Michigan. 1994. Part 115, Solid Waste Management of the Natural Resources and Environmental Protection Act, PA 451, as amended.

TRC. November 2021. JH Campbell Power Plant Dry Ash Landfill Hydrogeological Monitoring Plan.

United States Environmental Protection Agency (USEPA). July 2018. Code of Federal Record Title 40 Part 257 Subpart D, as amended.

Figures





MICHIGAN COUNTIES
NOT TO SCALE

REFERENCE(S)

1. BASE MAP TAKEN FROM 7.5 MINUTE U.S.G.S. QUADRANGLES OF PORT SHELDON MICHIGAN, DOWNLOADED FROM MICHIGAN DNR WEBSITE JUNE 2016.

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1" = 2000' FEET

CLIENT
CONSUMERS ENERGY COMPANY
17000 CROSWELL ST.
WEST OLIVE, MI 49460

PROJECT
J.H. CAMPBELL GENERATING FACILITY
DRY ASH LANDFILL POST-CLOSURE PLAN

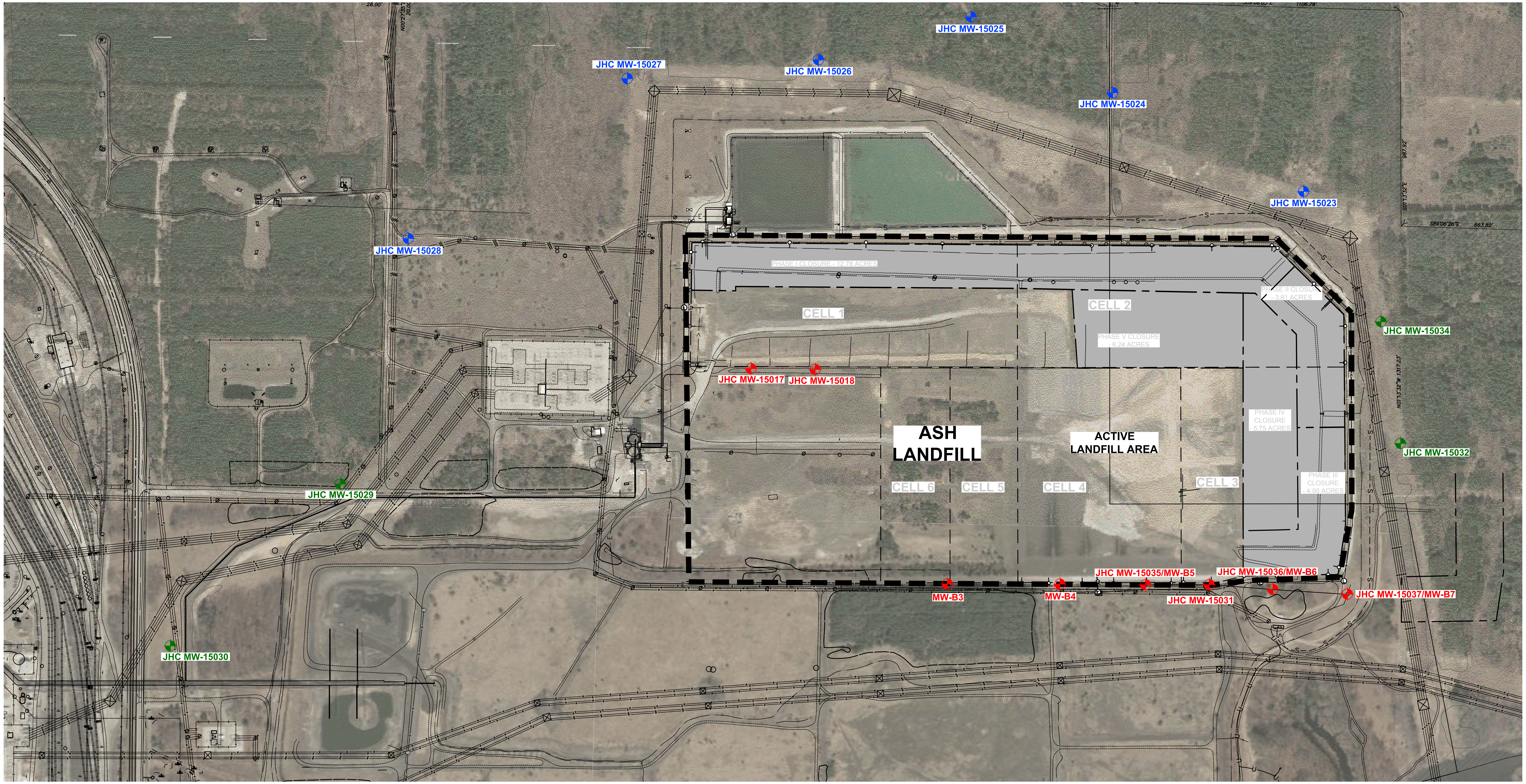
CONSULTANT

GOLDER

YYYY-MM-DD	2018-09-21
DESIGNED	JS
PREPARED	DJS
REVIEWED	JT
APPROVED	JRP

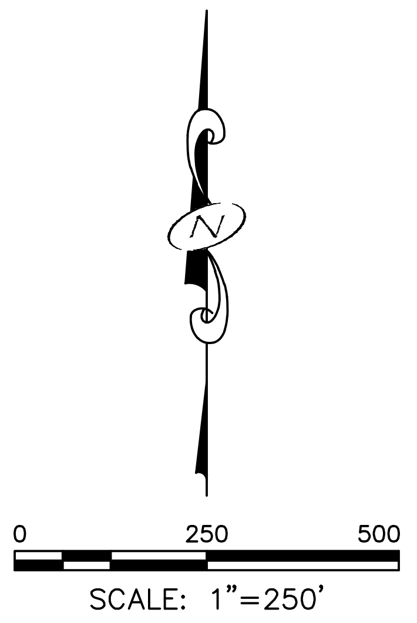
TITLE
SITE LOCATION MAP

PROJECT NO.	REV.	FIGURE
21451121	----	1



LEGEND

DOWN GRADIENT MONITORING WELL	🟥
UP GRADIENT MONITORING WELL	🟦
SIDE GRADIENT MONITORING WELL	🟩



SCALE: 1"=250'

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	REV	DATE	DESCRIPTION	BY	CHK	APP
REFERENCE DRAWINGS	REV	DATE	DESCRIPTION	BY	CHK	APP

REV	DATE	DESCRIPTION	BY	CHK	APP
C	12/9/2021	2021 UPDATE	DS	JP	JP
B	12/5/2018	2018 UPDATE	DS	JT	JP
A	10/6/2016	FILED IN OWNER'S OPERATING RECORD	AM	DS	JP

Consumers Energy

J.H. CAMPBELL ASH STORAGE FACILITY

EXISTING CONDITIONS

SITE MAP

UNIT#

SCALE 1" = 250'	DRAWING NO.		FIGURE 2	REV. C
JOB PT-02661				

APPENDIX A

Post Closure Inspection Checklist

Appendix A: JHC Dry Ash Landfill - Post Closure Inspection Checklist

Category	Survey Item	Frequency	OK?	Needs Action	Comments/Actions Needed
Entrance and Roadways	Entrance gate is present and lockable.	Yearly			
	Entrance is neat and attractive and access roads are properly graded and maintained.				
	Traffic control and information signs present, legible, and properly located.				
	All required permits and site use restrictions are posted.				
Landfill Slopes	Slopes are functioning as designed (no excessive settlement)	Quarterly for initial 5 years then Annually for the Closed Landfill			
	Toe of slope is dry and seepage is not observed.				
	Leachate seeps are not present on the landfill slopes.				
	Graded slopes are free of erosion and burrowing animals.				
	There is no exposed liner.				
	There is no ponding water on the landfill surface.				
	Final cover is free from signs of instability or failure.				
	Vegetation density and length is adequate.				
	Topsoil thickness is adequate.				
	Slopes are free from undesirable vegetation (trees, bushes, etc.)				
	Re-vegetation has been performed on disturbed areas to control erosion.				

Appendix A: JHC Dry Ash Landfill - Post Closure Inspection Checklist

Category	Survey Item	Frequency	OK?	Needs Action	Comments/Actions Needed
Facility Conditions	Shop/parking areas are free of petroleum stained soils.	Quarterly for initial 5 years then Annually for the Closed Landfill			
	Leachate manhole containment is free of water.				
	Leachate manhole water level is within acceptable limits.				
	Leachate pump and controls are operating properly.				
	There are no junk containers, equipment, or trucks, etc.				
	Surplus materials and equipment are stored neatly in an appropriate storage area, out of sight from main entrance.				
	Landfill coordinate system is in place and being used.				
	Liner edge markers are in-place and visible.				
	Groundwater monitoring points are clearly marked, accessible, adequately protected, and locked.				
	Al grassed areas are mowed and maintained.				
	All ditches are clean and performing as designed.				
	All let-down pipes/chutes are installed and functioning.				
	Silt basins, fences, barriers are in place as required and in good working conditions.				
	Visual examination of storm water outfalls has been performed as required by the permits.				
Monitoring Results	Environmental monitoring results do not exceed regulatory limits.	Quarterly			
	Groundwater monitoring results show concentrations within regulatory limits.				
	Storm water outfall sampling/testing has been performed as required.				
	Groundwater, surface water, and leachate monitoring has been performed as required and the report is in the Operating Record.				

Appendix A: JHC Dry Ash Landfill - Post Closure Inspection Checklist

Category	Survey Item	Frequency	OK?	Needs Action	Comments/Actions Needed
Reporting	Up-to-date and approved design, construction, and operating plans are filed in the Operating Record.	Yearly			
	Storm water Pollution Prevention Plan (SWPPP) is up-to-date and is filed on-site.				
	Latest update of Financial Assurance instruments are filed in the Operating Record and with EGLE.				
	Leachate collection system records are reviewed to evaluate the performance of these systems.				
	Records of cell construction are filed in the Operating Record.				
	Records of quantities and dates of leachate recirculation and/or disposal are filed in the Operating Record.				

Appendix A: JHC Dry Ash Landfill - Post Closure Inspection Checklist

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<tr><th>Category</th><th>Survey Item</th><th>Frequency</th><th>OK?</th><th>Needs Action</th><th>Comments/Actions Needed</th></tr>
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APPENDIX B

Dry Ash Landfill
Hydrogeological Monititoring Plan

Dry Ash Landfill Hydrogeological Monitoring Plan

JH Campbell Power Plant
West Olive, Michigan

October 2020; Revised November 2021

Prepared For:

Consumers Energy Company
1945 W. Parnall Road
Jackson, MI 49201

Prepared By:

TRC
1540 Eisenhower Place
Ann Arbor, Michigan 48108

Sarah B. Holmstrom, P.G.
Project Manager/Hydrogeologist

Graham Crockford, C.P.G.
Program Manager

Kristin Lowery, E.I.T.
Project Engineer

TABLE OF CONTENTS

TABLES

FIGURES

APPENDICES

1.0 Introduction

The JH Campbell (JHC) Dry Ash Landfill is a Type III solid waste disposal facility owned by Consumers Energy Company (Consumers Energy) located in West Olive, Michigan, which accepts coal ash from power plant operations (Figure 1). The Dry Ash Landfill was initially licensed over 20 years ago. The State of Michigan Waste Data System number is 395496.

TRC has prepared this Dry Ash Landfill Hydrogeologic Monitoring Plan (HMP) on behalf of Consumers Energy that serves as a revision to the Michigan Department of Environment, Great Lakes, and Energy (EGLE)[1]-approved HMP prepared for the Dry Ash Landfill in September 1996 (September 1996 HMP). In addition to the monitoring described in this HMP, groundwater monitoring is also currently performed at the Site in accordance with the Cells B-K monitoring described in the September 1996 HMP and an EGLE-approved Remedial Action Plan (RAP) and the Agreement for a Limited, Site-Specific, Criteria-Based Remedial Action LAND-WMD-2005-2, dated July 13, 2005. The original RAP, dated December 18, 2000, was revised on December 14, 2003, and revised again by a RAP Addendum dated January 31, 2008. On December 21, 2018, Consumers Energy and the EGLE executed Consent Agreement No. 115-01-2018. As outlined in Section 4.5 of the agreement, Consumers Energy submitted an updated, site-wide RAP to address groundwater across the entire solid waste disposal facility (including areas downgradient from the Dry Ash Landfill) on October 1, 2021 and will comply with criteria developed under Part 201 of the NREPA, as amended.

1.1 Background and Program Summary

On April 17, 2015, the United States Environmental Protection Agency (USEPA) issued the Coal Combustion Residual (CCR) Resource Conservation and Recovery Act (RCRA) Rule (40 CFR 257 Subpart D) (CCR Rule) to regulate the solid waste management of CCRs at electrical generating facilities. The CCR Rule, which became effective on October 19, 2015, applies to the Consumers Energy Dry Ash Landfill (Figure 2) located at the JHC Power Plant Site (the Site). Consumers Energy began monitoring groundwater at the Dry Ash Landfill in accordance with the CCR Rule in 2015, with the construction of a groundwater monitoring system in 2015, followed by the collection of background groundwater samples, the initiation of detection monitoring, and subsequent data evaluation and reporting. As discussed in the 2020 Annual Groundwater Monitoring and Corrective Action Report, the Dry Ash Landfill is currently in assessment monitoring pursuant to the CCR Rule.

In addition to the CCR Rule, the Dry Ash Landfill has been regulated under Part 115 of the Natural Resources and Environmental Protection Act (NREPA) PA 451 of 1994, as amended (Part 115) and monitored in adherence to the facility's EGLE-approved *Hydrogeological Monitoring Plan (HMP) for JH Campbell Ash Storage Facility, Consumers Power Company, Solid Waste Disposal Area, Coal Ash, Type III* (September 1996) and the aforementioned RAP. Groundwater quality data for the Dry Ash Landfill has been collected since 1992, during the baseline monitoring period including prior to construction of the landfill. Quarterly groundwater data have been provided to the EGLE in the quarterly hydrogeologic monitoring reports since

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[1] Effective Monday, April 22, 2019, the Michigan Department of Environmental Quality (MDEQ) became known as the Michigan Department of Environment, Great Lakes, and Energy.

ash placement began in early 1997.

After establishing the groundwater monitoring system and detection monitoring project pursuant to the requirements and schedule of §257.90 - §257.94, the State of Michigan enacted Public Act No. 640 of 2018 (PA 640) on December 28, 2018 to amend Part 115 of the NREPA. These amendments to Part 115 were developed to provide state of Michigan oversight of coal ash impoundments and landfills and to better align existing state solid waste management rules and statutes with the CCR Rule. This alignment would ensure compliance with the CCR standards through a state-approved permitting program that would be deemed to be “equivalent to” or “as protective as” through an administrative application that would be reviewed and authorized by U.S. EPA. Therefore, the basis for establishing a groundwater monitoring system and initiating detection and assessment monitoring will need to conform requirements for any licensed coal ash impoundment or landfill after December 28, 2018 with Part 115 amendments and the CCR Rule.

1.2 Purpose and Scope

The Dry Ash Landfill HMP is required for the construction permit application for proposed Cell 6 of the Dry Ash Landfill and a forthcoming operating license renewal in accordance with the provisions of the Michigan Part 115 Solid Waste Rules, as amended. The purpose of this HMP is to provide a means to comply with applicable monitoring requirements described in Part 115, as amended, and the self-implementing standards and schedules of the CCR Rules, until such a time as the USEPA recognizes Part 115, as amended, as an authorized permitting authority to regulate coal ash impoundments and landfills in Michigan. The monitoring program was developed based on the hydrogeologic characteristics of the site and surrounding area and the known and potential influence of the surface impoundment on the hydrogeologic system.

The methodologies outlined in this HMP are consistent with applicable regulations, general federal and state guidance, TRC’s and Consumer Energy’s Standard Operating Procedures (SOPs), and industry standards.

1.3 Requirements

As part of the construction permit application process, Consumers Energy is required to submit an HMP in compliance with Part 115 Rule 299.4905 to the EGLE. Revisions to Part 115 as amended by PA 640, in particular Section 11511a(3) and Section 11512(a)(1), require an approved HMP that complies with 299.4440 to 299.4445, if applicable, and 299.4905 to 299.4908 of the Part 115 Rules prior to issuing a solid waste operating license to a coal ash impoundment or landfill. A copy of the approved HMP will be placed in Consumers Energy’s JH Campbell Plant Operating Record and used to monitor groundwater quality at the Dry Ash Landfill.

Consumers Energy will notify the Director of the EGLE (“Director” or their designee) of any plans to modify monitoring points or schedules described in this HMP. Such changes will be implemented upon approval by the Director and documented in the operating record.

1.4 Site Overview

The JH Campbell Plant is a coal fired power generation facility located in West Olive, Michigan, in Section 15, Township 06N, Range 16W, Port Sheldon Township, Ottawa County. The power plant site is on the eastern shore of Lake Michigan. The solid waste management area, which includes the Dry Ash Landfill, is bordered by the Pigeon River on the south, 156th Avenue on the east, and Croswell Street to the north with Lakeshore Drive bisecting the site from north to south. The power generating plant consists of three coal fired electric generating units located on the western side of the site and the CCR disposal area is on the east side of the site, east of Lakeshore Drive. Figure 1 is a site location map showing the facility and the surrounding area.

1.5 Geology/Hydrogeology

The Dry Ash Landfill is located north-northwest of the Pigeon River and/or Spring Bayou and northeast of Pigeon Lake. The subsurface materials encountered at the JH Campbell site generally consist of approximately 40 to 60 ft of poorly graded, fine-grained lacustrine sand, which makes up the uppermost aquifer encountered in the vicinity of the Landfill. The aquifer is underlain by a laterally extensive clay-rich till, which serves as the confining base of the aquifer and prevents vertical groundwater flow. Groundwater is typically encountered at depths ranging from around 7 to 35 ft bgs and generally flows to the south-southeast across the site, with a southwesterly groundwater flow component on the west edge of the site.

The geology and hydrogeology of the site and surrounding area is further detailed in the *Dry Ash Landfill Hydrogeological Report, JH Campbell Power Plant, West Olive, Michigan* (Hydrogeological Report) (TRC, October 2020, Revised June 2021).

2.0 Groundwater Monitoring Program

Rule 299.4905(1)(a) states that an HMP shall include a groundwater monitoring well system that is in compliance with the provisions of R 299.4906. The groundwater monitoring program presented in this section of the HMP has been designed specific to the Dry Ash Landfill. A description of the hydrogeologic characteristics of the site and surrounding area is provided in the Hydrogeological Report.

Several other monitoring wells are present at the facility and are used for routine monitoring throughout the site in accordance with the RAP, Pond A HMP and the CCR Rule and some wells are utilized in multiple programs (Figure 2). Data from the other site wells will be utilized as needed to supplement the groundwater flow interpretation and/or nature and extent evaluations, if needed.

2.1 Groundwater Monitoring Well Network

A groundwater monitoring system has been established for the Dry Ash Landfill in accordance with R 299.4906 and the CCR Rule §257.91, which consists of 14 monitoring wells (six background monitoring wells and eight downgradient monitoring wells) that are screened in the uppermost aquifer. The monitoring well locations are shown on Figure 2. Monitoring well specifications and locations relative to groundwater flow direction (i.e. upgradient, downgradient, or side gradient) are included in Table 1.

MW-B3 and MW-B4 are located south of Cells 4 and 5 and future Cell 6. These monitoring wells were previously part of routine Part 115 monitoring programs and have not been previously monitored as part of the CCR Rule. As such, Consumers Energy will need to establish a new baseline dataset in compliance with this HMP (using unfiltered samples and updating the list of constituents analyzed). Baseline sampling will commence subsequent to the approval of this HMP.

2.1.1 Static Water Level Monitoring

A subset of monitoring wells will be used for static water level monitoring as indicated on Table 1. These wells are located side-gradient of the Dry Ash Landfill and as such, are not representative of groundwater quality downgradient of the Dry Ash Landfill. Static water level measurements will be collected at these wells to facilitate evaluation of groundwater flow conditions.

2.2 Monitoring Well Construction

Table 1 provides a summary of monitoring well locations, construction, and elevation information. Soil boring logs and monitoring well construction details are provided in Appendix A. All monitoring wells completed on-site are clearly labeled and visible throughout the year. Protective covers are installed for each monitoring well.

2.3 Monitoring Parameters and Sampling Frequency

2.3.1 Detection Monitoring

Groundwater monitoring will be conducted quarterly for the parameters listed in Section 11511a(3)(c) – Detection Monitoring Constituents. Once a minimum of 8 quarterly monitoring events have been completed, Consumer's Energy will evaluate the data to determine if semi-annual groundwater sampling would be as equally representative of groundwater conditions as is quarterly sampling. If semi-annual sampling is deemed adequate, Consumers Energy will prepare a demonstration for the reduction in sampling frequency to the EGLE for approval. Table 2 provides a detailed summary of the monitoring parameters and frequency for the groundwater monitoring program. Per Section 11511a(3)(e) of the Part 115 amendment, groundwater samples collected for metals analysis will not be field filtered.

2.3.2 Assessment Monitoring

In addition to the HMP monitoring, groundwater monitoring will be conducted quarterly for the parameters listed in Section 11519b(2) – Assessment Monitoring Constituents for a period of two years subsequent to EGLE approval of the HMP, with the exception of radium, which will continue to be analyzed on a semiannual basis. After completion of two years of quarterly monitoring (i.e., 8 quarterly monitoring events), Consumers Energy will petition the EGLE to reduce the sampling frequency for non-detected constituents to semiannually. If data indicate that a reduction to semiannual monitoring is appropriate at a time sooner than two years or for constituents with limited detections, Consumers Energy will submit a request to EGLE for approval prior to making the change. Table 2 provides a detailed summary of the monitoring parameters and frequency for the groundwater monitoring program. Per Section 11511a(3)(e) of the Part 115 amendment, groundwater samples collected for metals analysis will not be field filtered. Monitoring of the groundwater to surface water interface (GSI) pathway will continue to be completed in accordance with the existing RAP until it is replaced with the forthcoming site-wide RAP.

2.4 Static Water Level Monitoring

Measurement of static groundwater level data will be collected from all monitoring wells listed on Table 1 during each sampling event, prior to sampling, using the methods described in Section 4.3.2. The monitoring well locations are depicted on Figure 2.

3.0 Leachate Monitoring Program

In 2011, the EGLE determined the Dry Ash Landfill no longer met the Part 115 definition of a “monitorable unit” due to indications that certain constituents existed in some monitoring wells above background concentrations and Consumers Energy was not able to obtain approval of an alternate source demonstration (ASD). Based on this determination, groundwater monitoring results are not the only consideration in determining compliance with regard to the landfill unit’s impact on groundwater. Rather, the landfill’s secondary leachate collection system is considered to be a leak detection system, in addition to the groundwater data.

Consumers Energy will remove liquids from the primary collection system (PCS) and secondary collection system (SCS) to minimize the head on the PCS and SCS liner systems. In addition, the volumes of liquids removed from the SCS will be calculated monthly.

The leachate collection system will be inspected regularly to assure proper operation of the system in accordance with Part 115 Rule 299.4432(5).

3.1 SCS Volume Removal Data

The quantity of liquids removed from the Dry Ash Landfill SCS will continue to be monitored for compliance with Part 115 Rule 299.4432(3)(b)(i).

SCS volume removal data will be utilized to calculate SCS flow rates for the Dry Ash Landfill in gallons per acre per day (gpad). The response flow rate (RFR) for the Dry Ash Landfill is 25 gpad in accordance with R 299.4437(1)(b). SCS flow rate data will be evaluated in accordance with the provisions of Rule 299.4437. The flow rate will be compared against the RFR of 25 gpad and transmitted to the EGLE as part of the quarterly hydrogeologic monitoring report.

Since 2011, Consumers Energy has completed multiple engineering improvements to enhance the leachate collection system. The SCS volume has not exceeded the RFR of 25 gpad at any of the cells (Cells 1 through 5) in the past 16 months. However, to ensure that the system continues to operate effectively, Consumers Energy will continue to optimize the system operation and perform monthly flow rate evaluations. In the event that the RFR is exceeded, Consumers Energy will complete the following response activities for each cell:

1. Notify the director in writing within 7 days of the RFR being exceeded.
2. Submit a preliminary written assessment of the situation within 14 days of the determination that the RFR has been exceeded. The preliminary assessment needs to include the amount of liquids removed, the likely sources of the liquids, the possible locations, size and cause of any leaks, and the short term actions taken and planned to address the problem.
3. Determine the extent practicable the location, size, and cause of any leak.
4. Determine whether waste receipt should cease or be curtailed.
5. Determine whether waste should be removed from the landfill cell for inspection, repairs, or controls.
6. Determine whether the entire landfill unit should be closed.
7. Determine any other short them or long term actions to mitigate or stop any leaks.

3.2 Leachate Disposal

Leachate recovered from the Dry Ash Landfill will be disposed of in accordance with Rule 299.4432(6), and in compliance with Part 31 of NREPA. Leachate is pumped into two lined leachate retention ponds for storage and disposed of under NPDES Permit No. MI0001422.

3.3 Leachate Analytical Testing Requirements

3.3.1 Sample Location

Primary collection system (PCS) and SCS leachate is collected in individual sumps for each active landfill cell. Liquid levels in the sumps are managed as described in Section F of this Construction Permit Application. Samples will be collected from each SCS sump on a quarterly basis and from each PCS sump annually. The results will be submitted to the EGLE in the corresponding quarterly reports. The approximate sample collection locations are shown on Figure 2.

Leachate samples will be collected from the sumps of the constructed cells as follows:

- ***PCS Sample Locations:*** PCS sumps C1P, C2P, C3P, C4P, and C5P will be monitored to evaluate leachate from the active landfill cells. Additional PCS sample locations will be included as future cells are constructed.
- ***SCS Sample Locations:*** SCS sumps C1S, C2S, C3S, C4S, and C5S will be monitored when sample volume is available to evaluate leachate from the active landfill cells. Additional SCS sample locations will be included as future cells are constructed.

3.3.2 Leachate Analytical Testing Requirements

Consumers Energy will perform analytical testing of SCS and PCS liquids from the Dry Ash Landfill. Table 3 provides a detailed summary of the monitoring parameters and frequency for the leachate monitoring program. After completion of two years of quarterly monitoring (i.e., 8 quarterly monitoring events), Consumers Energy will petition the EGLE to reduce the sampling frequency for non-detected leachate constituents to annually.

Leachate samples will not be field filtered, in accordance with Table 3. Reporting limits are consistent with the monitoring plan for groundwater and are detailed on Table 3.

4.0 Field Sampling Procedures

The following sections address the methods and procedures associated with the installation of any future monitoring wells and collection and handling of samples at the Site in conformance with R 299.4906 and R 299.4907 of the Part 115 Rules. Any changes made to the monitoring network will be documented in the operating record and a notification will also be provided to the EGLE through the quarterly reporting or other communications, as appropriate.

4.1 Monitoring Well Installation and Development Procedures

If required, field personnel will install and develop monitoring wells in accordance with the procedures detailed below. Consumers Energy will obtain EGLE approval for all new or replacement wells prior to installation.

4.1.1 Monitoring Well Installation Procedures

Soil borings for monitoring well installation will typically be constructed using hollow stem auger or direct push methods. At new or replacement monitoring well locations, soil samples will be collected during drilling activities in order to log local geology and verify depth to groundwater prior to well installation. Soil samples will be collected with split spoon or tube samplers at intervals appropriate to the local geology, typically ranging from continuous to 1 sample per 5-foot interval. The field personnel will log and document geology and drilling details on standard field forms similar to the logs provided in Appendix A.

Following drilling activities, soil borings will be converted into monitoring wells at the designated monitoring locations. Monitoring well casings and screens will arrive at the site in the original factory packaging and will remain in the packaging until the casing and screen materials are installed in the borehole. Permanent monitoring wells will typically be constructed of 2-inch Schedule 40 PVC flush-threaded riser pipe and equipped with a 5-foot, 0.01-inch slot screen.

After the screen and well casing are lowered into the borehole, the filter sand will be backfilled around the well screen and casing to a depth of approximately 2 feet above the top of the screen. Approximately 2 feet of bentonite chips will be used to seal the annular space above the sand pack. Additional bentonite chips or a bentonite slurry will be used to seal the remainder of the annular space above the sand pack, to approximately 1 foot below the ground surface. The slurry, if used, will be placed using a tremie from the bottom up. The remaining borehole annulus will be grouted with cement/bentonite grout to within one foot of land surface and finished with a concrete pad.

Monitoring wells will be protected with a locking flush-mount or aboveground steel protective cover. Well caps will be left loose or will be vented to allow static water levels to remain equilibrated with ambient air pressure. The aboveground protective covers will be cemented in place around the PVC riser to stick up from the ground surface approximately 2 to 3 feet. The flush-mount protective covers will be installed flush with the ground surface, with the PVC riser finished below ground. The protective covers will be locked and clearly labeled for identification purposes. Monitoring well construction details will be recorded at the time of installation on well construction diagrams similar to the ones included in Appendix A.

4.1.2 Monitoring Well Development

Monitoring wells will be developed after the grout and well seal material has cured. Curing time will typically be 24 hours following well installation, although for shallow wells, where grout and well seal material is placed above the water table, well development may be performed immediately following well installation.

The development will remove fine particles around the well screen and filter pack to improve hydraulic communication between the well and the surrounding aquifer. Development will be accomplished using a pump or bailer to surge and purge the well. Development will be complete when the purge water turbidity measurements are less than 10 Nephelometric Turbidity Units (NTUs) as a goal. However, for wells that are screened in silt- or clay-rich units, this may be impractical. Development of these wells will be complete when the purge water is relatively clear (visibly) and free from suspended solids, after approximately five well volumes have been evacuated, or when well goes dry. Development notes will be recorded on the well construction diagrams.

4.1.3 Well Identification

Each permanent well installed will be identified as follows: JHC-MW-XXXXX. The first two digits of the variable -XX will be filled in with the last two digits of the calendar year during which the well was installed and the last three digits of the variable XXX will be filled in with the number of the well.

4.1.4 Well Decommissioning

Prior to making any changes in the monitoring well network, Consumers Energy will submit a request to EGLE for approval prior to making the change. Specific monitoring well decommissioning procedures will be provided to the EGLE for approval as part of the request.

4.2 Pre-Sample Procedures

The field personnel will obtain all necessary field sampling forms (examples provided in Appendix B) to complete the fieldwork. In addition, sample bottles, trip blanks, and deionized water will be obtained from the contract laboratory prior to sample collection. Potential contaminants from the sample bottles and rinse water will be minimized by obtaining these materials directly from the laboratory.

4.3 Groundwater Sampling Procedures

The following procedures will be used to collect groundwater monitoring data.

4.3.1 Monitoring Well Inspection

Prior to opening any groundwater monitoring well, field personnel will visually inspect the protective casing and the concrete collar for damage and wear. Visual observations of the integrity of the wells will be recorded in the field notes. Field personnel will notify Consumers Energy of any damage to the monitoring wells or protective casings. Field personnel will ensure that all wells are clearly and properly labeled and visible throughout the year. Any flushmount wells added into the program will be flagged with fiberglass driveway markers, or similar, to maintain visibility.

4.3.2 Static Water Levels

After visual inspection of the monitoring well, the well will be unlocked and static water level measurements will be recorded using an electronic water level meter accurate to 0.01 foot, from each on-site well prior to purging and sampling. All water level measurements will be recorded within a 24-hour time period to minimize temporal bias of measured groundwater elevation changes for the monitoring well network.

Field personnel will measure water levels from an identified reference point on the well casing and record the measurement in the field notes. Depth-to-water measurements from the top-of-casing (TOC) will be subtracted from the TOC elevation to determine the potentiometric elevation. The static water level meter will be cleaned prior to and between each monitoring well according to the decontamination procedures described below.

4.3.3 Groundwater Sample Collection and Handling

Groundwater samples will be collected from the monitoring wells following Low-Flow (Minimal Drawdown) Groundwater Sampling Procedures (US EPA, 1996), as detailed in the Groundwater Sampling Standard Operating Procedures (SOP) (Appendix B). Low flow sampling will commence with the installation of either a peristaltic, stainless-steel 12-volt submersible impeller pump or bladder pump to a depth representing the middle of the saturated screen interval. The background wells will be sampled first, followed by the downgradient wells. An appropriate length of polyethylene tubing will be connected to the pump discharge prior to pump placement. The discharge line will be connected to a flow-cell and multi-meter to collect water quality indicator parameters (described below) during well purging to determine water quality stabilization.

The pump will be operated at a flow rate that ensures low volatilization and low well disturbance. Water quality indicator parameters and depth to water will be recorded at 3 to 5-minute intervals during the purging process and recorded on the sampling worksheet provided in Appendix B. Purging and sampling will proceed at a low pumping rate, expected to be between approximately 0.1 and 0.5 liters per minute or less, such that the water column in the well is not lowered more than 0.3 feet below the initial static depth to water measurement. The subject well will be considered ready to sample when three consecutive water quality measurements meet the stabilization criteria presented below.

Parameter	Stabilization Criteria
pH	3 readings within +/- 0.1 standard units (SU)
Specific Conductance	3 readings within +/- 3% millisiemens per centimeter (mS/cm)
Temperature	For Information Only

Parameter	Stabilization Criteria
Turbidity	+/- 10% Nephelometric Turbidity Unit (NTU) variance between three consecutive readings and a turbidity less than 10 NTU[2]
Oxygen Reduction Potential (ORP)	3 readings within +/- 10 millivolts (mV)
Dissolved Oxygen (DO)	3 readings within +/- 0.3 milligrams per liter (mg/L)

If the well is dry, no attempt at sampling will be conducted, as the aquifer is not considered to have sufficient quantity at that location. Additionally, if the well is pumped dry during low-flow monitoring activity, the well will be left overnight to accumulate water, then a sample collected assuming the NTU criteria can be met or, if necessary, filter the sample as laid out in Section 4.5 below. The well, if pumped dry and allowed to recover, must be sampled within a 24-hour period. Prior to use, all equipment will be calibrated in accordance with the manufacturer's recommendations and the Groundwater Sampling SOP. Calibration information will be recorded in the field notes.

4.4 Leachate Sample Collection

If a primary collection system (PCS) or secondary collection system (SCS) leachate sample is required, a grab sample will be collected from the PCS or SCS sump. The sample will be screened with a water quality multi-meter and the pH, specific conductance, temperature, turbidity, ORP, and DO at the time of sample collection will be recorded in the field notes. The sample will be labeled, stored and transported to the laboratory using the same methodologies as outlined for the groundwater samples in Section 4.5.

4.5 Sample Preservation and Shipment

Samples will be collected immediately following stabilization of field parameters as set forth in in the preceding sections. Groundwater and leachate samples will be collected into the laboratory provided sample containers required for the analyses specified in the following section. The groundwater samples will be collected from the discharge tubing upstream of the water quality meter flow cell. Care will be taken to allow for a non-turbulent filling of laboratory containers. Routine samples will not be filtered in the field to provide a measure of total recoverable metals that will include both the dissolved and particulate fractions of metals as per Section 11511a(3)(e) of the Part 115 amendment.

If a more detailed understanding of the source of metals concentrations in groundwater is required for select monitoring wells, field filtered samples may be analyzed in addition to routine analysis. Field filtering may also be completed on highly turbid samples (greater than 10 NTU at stabilization). Field filtering will be completed using a 0.45-micron filter. If required, an attempt will be made to redevelop any monitoring wells that produce highly turbid prior to the subsequent sampling event. Where samples are filtered, a corresponding, unfiltered sample will also be collected.

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[2] If sample cannot be stabilized at less than 10 NTUs, filtering may be necessary as detailed in Section 4.5.

The samples will be labeled, stored and transported to the laboratory according to the Chain-of-Custody Procedures SOP presented in Appendix C. Following collection, samples will be immediately labeled, logged on the chain-of-custody, and placed in a cooler with ice. Sample coolers transported to the laboratory via overnight or next day air freight will be sealed with packing tape and a signed chain-of-custody seal. Sample coolers transported to the laboratory directly must be secured to ensure sample integrity is maintained. The samples will be packaged and shipped according to U. S. Department of Transportation and EPA regulations. The documentation of actual sample storage and transport will be by the use of chain-of-custody procedures. A laboratory provided chain-of-custody record will contain the dates and times of collection, receipt, and completion of all the analyses on a particular set of samples. The laboratory will return a copy of the chain-of-custody with the analytical report.

4.6 Quality Assurance/Quality Control (QA/QC)

Quality assurance/quality control (QA/QC) samples will be collected to ensure sample containers are free of analytes of interest, assess the variability of the sampling and laboratory methods, and monitor the effectiveness of decontamination protocols. As such, QA/QC samples will be collected on a site wide basis per sampling event rather than on a unit by unit basis. The following QA/QC samples will be collected during each groundwater sampling event:

- Field duplicates will be collected at a frequency of one duplicate sample per 10 groundwater samples per event. The field duplicates will be collected at the same time and in the same manner as the original sample. The duplicates will be labeled as a blind sample and noted on the sampling form of the designated well.
- Matrix spike/matrix spike duplicate (MS/MSD) samples will be collected at a frequency of one MS/MSD sample per 20 groundwater samples per event. Duplicate and MS/MSD samples will be collected from different monitoring wells.
- Field blanks[3] will be collected at a frequency of one field blank per 20 groundwater samples with at least one field blank collected from the Dry Ash Landfill.
- Equipment blanks will be collected at a frequency of one equipment blank per 20 groundwater samples per event. The equipment blank will be collected by pouring distilled or deionized water over the decontaminated static water level meter or sample tubing and into the laboratory supplied containers.

The QA/QC samples will be submitted on a separate chain-of-custody to the laboratory for the routine analyses specified in Section 2. The laboratory should provide adequate documentation of laboratory reporting and QA/QC procedures.

4.7 Equipment Decontamination Procedures

All non-dedicated equipment will be decontaminated prior to use and between samples. Non-dedicated equipment will include a water level meter and low flow sampling pump (submersible) (if used). Each item will be cleaned using distilled or deionized water, and when necessary, a non-phosphate detergent wash followed by a distilled or deionized water rinse.

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[3] Field blanks consist of analyte-free water exposed to the atmosphere during field sample collection. The water is containerized in an appropriate bottle with preservative for the analytical suite and shipped to the laboratory with the other field samples. The results are used to assess whether or not ambient/surrounding air conditions may have influenced analytical results.

When a peristaltic pump is used for low flow sampling, decontamination is not required, only replacement of the pump head tubing.

All dedicated equipment that cannot be reliably decontaminated, such as polyethylene tubing and bladders if a bladder pump is used for low-flow sampling, will be disposed of after removal from each sampling point.

The flow-cell and water quality multi-meter (sonde) will be decontaminated at the completion of low-flow sampling. All sample collection will occur upstream of this device and therefore will not affect groundwater sample analytical results.

4.8 Investigation Derived Waste (IDW)

All waste created during monitoring well sampling will remain on site. All purge water from the monitoring wells will be discharged back onto the ground near the well it was purged from in a manner that ensures it does not directly enter a surface water or drain.

4.9 Field Documentation

All information pertinent to the field activities and sampling efforts will be recorded in a log or notebook following the documentation procedures presented in Section 5.4 of the SOP in Appendix B. Example field logs are provided in the attachments to Appendix B. At a minimum, entries in the sample logs will include the following:

- Property details and location
- Type of sample (for example, groundwater, surface water, waste)
- Number and volume of samples taken
- Sampling methodology
- Date and time of collection
- Sample identification number(s)
- Field observations including weather
- Any field measurements made (for example, pH, temperature, water depth and air monitoring data)
- Personnel present

Records shall contain sufficient information so that the sampling activity can be reconstructed without relying on the collector’s memory. The sample logs will be preserved in electronic format.

5.0 Laboratory Analysis

All analyses will be performed within required hold times and analytical methods consistent with the data quality objectives of this HMP as presented in Tables 2 and 3. Laboratory Quality Assurance and Quality Control (QA/QC) procedures will be performed and documented in accordance with the Laboratory QA/QC Plan included in Appendix D. Additional QA/QC procedures, i.e., duplicate sample collection, field data forms, and chain-of-custody procedures, are described in Section 4 of this HMP.

6.0 Data Evaluation and Reporting

Consumers Energy will evaluate the groundwater data for each constituent included in the groundwater monitoring program using statistical methods that comply with R 299.4908 of the Part 115 Rules. The statistical evaluation will be conducted in accordance with the "Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities – Unified Guidance" USEPA, 2009 (Unified Guidance). The following sections describe the statistical data evaluation and reporting procedures.

6.1 Detection Monitoring Statistical Data Evaluation

Statistical data evaluation will be conducted for the detection monitoring constituents listed in Table 2 using the procedures in the *Groundwater Statistical Evaluation Plan* (Stats Plan) (TRC, October 2017, revised July 2020) included in Appendix E of this HMP.

In order to comply with the Part 115 amendments, background will be established for the Section 11511a(3) constituents not already included in the CCR Rule Appendix III (e.g., iron) throughout a minimum of four sampling events.

Background groundwater monitoring was conducted at the Dry Ash Landfill from December 2015 through August 2017 in accordance with the *JH Campbell Monitoring Program Sample and Analysis Plan* (SAP) (ARCADIS, 2016), pursuant to the CCR Rule, with the results from 2015 through 2017 documented in the *Annual Groundwater Monitoring Report for the JH Campbell Power Plant Dry Ash Landfill CCR Unit* (2017 Annual Report) (TRC, January 2018). Data collected to-date as part of the CCR Rule implementation will be used to the extent practical to inform this HMP and establish background groundwater conditions for the Dry Ash Landfill monitoring well system.

Per the CCR Rule and amended Part 115 Rules, if the detection monitoring program confirms a statistically significant increase (SSI) over background for one or more of the detection monitoring constituents, Consumers Energy will conduct assessment monitoring in compliance with §257.95 and R 299.4441 of the Part 115 Rules. Consumers Energy reported in the 2017 Annual Report that boron, calcium, chloride, sulfate, and total dissolved solids (TDS) were observed within groundwater at one or more downgradient monitoring well(s) with potential SSIs above background concentration levels. The Dry Ash Landfill is currently in assessment monitoring in accordance with the CCR Rule and has had no statistically significant levels exceeding the groundwater protection standards (GWPSs). Assessment monitoring statistical data evaluation is further discussed below.

6.2 Assessment Monitoring Statistical Data Evaluation

In April 2018, Consumers Energy initiated an Assessment Monitoring Program for the Dry Ash Landfill pursuant to Subpart 257.95 of the CCR Rule and consistent with R 299.4440 and R 299.4441 of Part 115, that included sampling and analyzing groundwater within the groundwater monitoring system for all constituents listed in Appendix IV of the CCR Rule and establishing groundwater protection standards (GWPSs) as described below.

Background will be established for the Section 11511a(c)(3) constituents and 11519b(2) constituents not already included in the CCR Rule (e.g., iron, copper, nickel, silver, vanadium, zinc) constituent lists after a minimum of four sampling events have been performed in compliance with this HMP.

In accordance with 40 CFR 257.95(h) and the Stats Plan, GWPSs were established for the Appendix IV constituents following the preliminary assessment monitoring event. The calculation of the GWPSs is documented in the *Groundwater Protection Standards* technical memorandum included in the 2018 *Annual Groundwater Monitoring Report for the JH Campbell Power Plant Dry Ash Landfill CCR Unit* (2018 Annual Report) (TRC, January 2019). GWPSs are used to assess groundwater quality downgradient from the Dry Ash Landfill by statistically comparing concentrations in the downgradient wells to the GWPSs for each Appendix IV constituent.

The Appendix IV GWPSs will be re-evaluated upon approval of this HMP to ensure compliance with Part 115. The lowest of the MCLs, RSLs, or applicable Part 201 RC will be the GWPSs unless the background concentration is greater than the MCL, RSL, or applicable Part 201 RC, in which case, the statistically-determined background value becomes the GWPS. For GWPSs that are established using background, tolerance limits are anticipated to be used to calculate the GWPS.

The *Unified Guidance* recommends that background data be updated every 4 to 8 measurements to account for any changes in background that may occur over time. It is anticipated that the background will be updated every two years, along with the resulting GWPS, consistent with the *Unified Guidance*

Consistent with the Unified Guidance, the preferred method for comparisons to a fixed standard are confidence limits. Confidence intervals will be established in a manner appropriate to the data set being evaluated, in accordance with the Stats Plan (Appendix E). An exceedance of the standard occurs when the 99 percent lower confidence level of the downgradient data exceeds the GWPS.

If Section 11519b(2) or 11511a(c)(3) constituents are detected at statistically significant levels exceeding the MCL or the GWPSs, Consumers Energy will take necessary next steps in accordance with Section 11519b(4) of the Part 115 amendments. In addition, Consumers Energy will continue implementing the monitoring program presented in this HMP.

6.3 Reporting

Analytical results and data reports as defined below will be submitted to the director no later than 30 days after the end of the calendar quarter in which the samples were obtained. Data reports will include the following:

- Statement of adherence to the approved HMP;
- Description of the sampling event;

- Tables of analytical results from the groundwater monitoring program screened against Part 201 criteria;
- Statistical data evaluation;
- Time-series plots for detection monitoring and assessment monitoring constituents at all 17 monitoring wells;
- Groundwater contour maps with summary of groundwater flow direction and rates;
- Alternate source demonstration(s) (if applicable);
- Laboratory analytical results and chain of custody information;
- Field forms; and
- Signature of certified professional.

7.0 References

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USEPA. April 2015. 40 CFR Parts 257 and 261. Hazardous and Solid Waste Management System: Disposal of Coal Combustion Residuals From Electric Utilities; Final Rule. 80 Federal Register 74 (April 17, 2015), pp. 21301-21501 (80 FR 21301).

USEPA. July 2018. 40 CFR Part 257. Hazardous and Solid Waste Management System: Disposal of Coal Combustion Residuals From Electric Utilities; Amendments to the National Minimum Criteria (Phase One, Part One); Final Rule. 83 Federal Register 146 (July 30, 2018), pp. 36435-36456 (83 FR 36435).

Tables

Table 1

Monitoring Well Network Summary

JH Campbell – Dry Ash Landfill Hydrogeological Monitoring Program

West Olive, Michigan

Well Location	Well Location	Static Water Level Monitoring	Detection Monitoring Program	Assessment Monitoring Program	Northing	Easting	TOC Elevation (ft)	Date Installed	Geologic Unit of Screen Interval	Well Construction	Screen Interval Depth (ft BGS)			Screen Interval Elevation (ft)		
Background																
JHC-MW-15023	Upgradient	√	√	√	521927.21	12638205.16	619.98	10/1/2015	Sand	2" PVC, 10 Slot	14.0	to	24.0	603.0	to	593.0
JHC-MW-15024	Upgradient	√	√	√	522366.01	12637322.68	616.62	10/1/2015	Sand	2" PVC, 10 Slot	7.0	to	17.0	606.8	to	596.8
JHC-MW-15025	Upgradient	√	√	√	522702.98	12636668.15	617.17	10/1/2015	Sand	2" PVC, 10 Slot	7.0	to	17.0	607.1	to	597.1
JHC-MW-15026	Upgradient	√	√	√	522495.09	12635971.82	618.04	10/2/2015	Sand	2" PVC, 10 Slot	8.0	to	18.0	607.1	to	597.1
JHC-MW-15027	Upgradient	√	√	√	522394.86	12635097.51	617.30	10/2/2015	Sand	2" PVC, 10 Slot	10.0	to	20.0	604.8	to	594.8
JHC-MW-15028	Upgradient	√	√	√	521646.20	12634105.34	613.80	10/2/2015	Sand	2" PVC, 10 Slot	8.0	to	18.0	603.0	to	593.0
JHC-MW-15029	Side Gradient	√			520503.52	12633774.30	610.95	10/5/2015	Sand	2" PVC, 10 Slot	8.0	to	18.0	600.1	to	590.1
JHC-MW-15030	Side Gradient	√			519760.83	12633044.37	607.17	10/5/2015	Sand	2" PVC, 10 Slot	4.0	to	14.0	600.1	to	590.1
JHC-MW-15032	Side Gradient	√			520779.28	12638667.93	614.29	10/6/2015	Sand	2" PVC, 10 Slot	13.0	to	23.0	598.3	to	588.3
JHC-MW-15034	Side Gradient	√			521335.83	12638568.90	615.97	10/6/2015	Sand	2" PVC, 10 Slot	11.0	to	21.0	601.9	to	591.9
Landfill																
JHC-MW-15017	Downgradient	√	√	√	521074.31	12635685.32	616.61	9/29/2015	Sand	2" PVC, 10 Slot	10.0	to	20.0	603.7	to	593.7
JHC-MW-15018	Downgradient	√	√	√	521075.54	12635979.61	617.02	9/29/2015	Sand	2" PVC, 10 Slot	10.0	to	20.0	604.3	to	594.3
JHC-MW-15031	Downgradient	√	√	√	520118.00	12637801.51	635.87	10/6/2015	Sand	2" PVC, 10 Slot	33.0	to	43.0	599.9	to	589.9
JHC-MW-15035/MW-B5	Downgradient	√	√	√	520112.93	12637510.26	634.28	3/14/2001	Sand	2" PVC, 10 Slot	33.0	to	43.0	599.5	to	589.5
JHC-MW-15036/MW-B6	Downgradient	√	√	√	520099.80	12638094.34	618.34	3/13/2001	Sand	2" PVC, 10 Slot	20.0	to	30.0	597.9	to	587.9
JHC-MW-15037/MW-B7	Downgradient	√	√	√	520083.04	12638436.69	616.06	8/29/2001	Sand	2" PVC, 10 Slot	23.0	to	28.0	591.3	to	586.3
MW-B3	Downgradient	√	√	√	520100.98	12636601.76	632.16	12/13/2007	Sand	2" PVC, 10 Slot	32.0	to	37.0	599.0	to	594.0
MW-B4	Downgradient	√	√	√	520109.01	12637117.83	636.16	5/23/2011	Sand	2" PVC, 10 Slot	40.0	to	45.0	593.8	to	588.8

Notes:

Survey conducted by Nederveld, November 2015

Elevation in feet relative to North American Vertical Datum 1988 (NAVD 88).

TOC: Top of well casing.

ft BTOC: Feet below top of well casing.

ft BGS: Feet below ground surface.

Table 2

Groundwater Monitoring Constituents and Analytical Program Summary
JH Campbell – Dry Ash Landfill HMP Monitoring Program
West Olive, Michigan

Part 115 Amendments - Public Act No. 640 of 2018

Section 11511a(3)(c) - Detection Monitoring Constituents

CONSTITUENT	MONITORED UNDER 40 CFR 257 SUBPART D	MONITORED UNDER 1996 HMP	ANALYTICAL METHOD	PRESERVATION[3]	HOLD TIME	REPORTING LIMIT (µg/L)	FILTER	FREQUENCY[4]
Boron	√	√	6010/6020	HNO_3, pH <2	180 days	20	no	Quarterly
Calcium	√		6010/6020	HNO_3, pH <2	180 days	1,000	no	Quarterly
Chloride	√		EPA 300.0	None, <6ºC	28 days	1,000	no	Quarterly
Fluoride[1]	√		EPA 300.0	None	28 days	1,000	no	Quarterly
Iron			6010/6020	HNO_3, pH <2	6 months	20	no	Quarterly
pH	√	√	Stabilized field measurement	NA	NA	0.1 standard units	no	Quarterly
Sulfate	√		EPA 300.0	None, <6ºC	28 days	2,000	no	Quarterly
Total Dissolved Solids	√		SM 2540C	None, <6ºC	7 days	20,000	no	Quarterly
Section 11519b(2) - Assessment Monitoring Constituents								
Antimony	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly
Arsenic	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly
Barium	√		6020	HNO_3, pH <2	180 days	5	no	Quarterly
Beryllium	√		6020	HNO_3, pH <2	180 days	1	no	Quarterly
Cadmium	√		6020	HNO_3, pH <2	180 days	0.2	no	Quarterly
Chromium, total	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly
Cobalt	√		6020	HNO_3, pH <2	180 days	6	no	Quarterly
Copper			6020	HNO_3, pH <2	6 months	1	no	Quarterly
Fluoride[1]	√		EPA 300.0	None, <6ºC	28 days	1,000	no	Quarterly
Lead	√		6020	HNO_3, pH <2	180 days	1	no	Quarterly
Lithium	√	√	6010/6020	HNO_3, pH <2	180 days	10	no	Quarterly
Mercury	√		7470	HNO_3, pH <2	28 days	0.2	no	Quarterly
Molybdenum	√	√	6020	HNO_3, pH <2	180 days	5	no	Quarterly
Nickel		√	6010/6020	HNO_3, pH <2	6 months	2	no	Quarterly
Selenium	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly
Silver			6020	HNO_3, pH <2	6 months	0.2	no	Quarterly
Thallium	√		6020	HNO_3, pH <2	180 days	2	no	Quarterly
Vanadium		√	6020	HNO_3, pH <2	6 months	2	no	Quarterly
Zinc			6020	HNO_3, pH <2	6 months	10	no	Quarterly
Radium 226 and 228 combined[2]	√		EPA 903.1/904.0	HNO_3, pH <2	None	1.00 picocurie per liter (pCi/L)	no	Semiannual

Notes:

HNO_3 – Nitric acid

NA – Not applicable

[1] *Listed in both detection and assessment monitoring constituent lists*

[2] *Requires a larger sample volume (minimum 2 liter)*

[3] *All samples will be cooled to 4° C as part of sample preservation.*

[4] *Frequency reduction to semiannual will be requested after completion of two years (8 quarterly monitoring events) subseqent to the HMP approval date.*

Table 3

Leachate Monitoring Constituents and Analytical Program Summary
JH Campbell – Dry Ash Landfill HMP Monitoring Program
West Olive, Michigan

Part 115 Amendments - Public Act No. 640 of 2018

Section 11511a(3)(c) - Detection Monitoring Constituents

CONSTITUENT	MONITORED IN SCS	MONITORED IN PCS	ANALYTICAL METHOD	PRESERVATION[1]	HOLD TIME	REPORTING LIMIT (µg/L)	FILTER	FREQUENCY	
								SCS	PCS
Boron	√	√	6010/6020	HNO_3, pH <2	180 days	20	no	Quarterly	Annually
Calcium	√	√	6010/6020	HNO_3, pH <2	180 days	1,000	no	Quarterly	Annually
Chloride	√	√	EPA 300.0	None, <6ºC	28 days	1,000	no	Quarterly	Annually
Fluoride[2]	√	√	EPA 300.0	None	28 days	1,000	no	Quarterly	Annually
Iron	√	√	6010/6020	HNO_3, pH <2	6 months	20	no	Quarterly	Annually
pH	√	√	Stabilized field measurement	NA	NA	0.1 standard units	no	Quarterly	Annually
Sulfate	√	√	EPA 300.0	None, <6ºC	28 days	2,000	no	Quarterly	Annually
Total Dissolved Solids	√	√	SM 2540C	None, <6ºC	7 days	20,000	no	Quarterly	Annually
Section 11519b(2) - Assessment Monitoring Constituents									
Antimony	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly	Annually
Arsenic	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly	Annually
Barium	√	√	6020	HNO_3, pH <2	180 days	5	no	Quarterly	Annually
Beryllium	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly	Annually
Cadmium	√	√	6020	HNO_3, pH <2	180 days	0.2	no	Quarterly	Annually
Chromium, total	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly	Annually
Cobalt	√	√	6020	HNO_3, pH <2	180 days	6	no	Quarterly	Annually
Copper	√	√	6020	HNO_3, pH <2	6 months	1	no	Quarterly	Annually
Fluoride[2]	√	√	EPA 300.0	None, <6ºC	28 days	1,000	no	Quarterly	Annually
Lead	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly	Annually
Lithium	√	√	6010/6020	HNO_3, pH <2	180 days	10	no	Quarterly	Annually
Mercury	√	√	7470	HNO_3, pH <2	28 days	0.2	no	Quarterly	Annually
Molybdenum	√	√	6020	HNO_3, pH <2	180 days	5	no	Quarterly	Annually
Nickel	√	√	6010/6020	HNO_3, pH <2	6 months	2	no	Quarterly	Annually
Selenium	√	√	6020	HNO_3, pH <2	180 days	1	no	Quarterly	Annually
Silver	√	√	6020	HNO_3, pH <2	6 months	0.2	no	Quarterly	Annually
Thallium	√	√	6020	HNO_3, pH <2	180 days	2	no	Quarterly	Annually
Vanadium	√	√	6020	HNO_3, pH <2	6 months	2	no	Quarterly	Annually
Zinc	√	√	6020	HNO_3, pH <2	6 months	10	no	Quarterly	Annually
Radium 226 and 228 combined[3]	√	√	EPA 903.1/904.0	HNO_3, pH <2	None	1.00 picocurie per liter (pCi/L)	no	Annually	Annually

Notes:

HNO_3 – Nitric acid

NA – Not applicable

[1] *All samples will be cooled to 4 °C as part of sample preservation.*

[2] *Listed in both detection and assessment monitoring constituent lists*

[3] *Requires a larger sample volume (minimum 2 liter)*

Table 3
Leachate Monitoring Constituents and Analytical Program Summary
JH Campbell – Dry Ash Landfill HMP Monitoring Program
West Olive, Michigan

Optional Additional Constituents									
Bicarbonate, carbonate, and total alkalinity	--	--	SM 2320B	None, <6ºC	14 days	10,000	no	Optional	Optional
Magnesium	--	--	6010/6020	HNO_3, pH <2	180 days	1,000	no	Optional	Optional
Sodium	--	--	6010/6020	HNO_3, pH <2	180 days	1,000	no	Optional	Optional
Potassium	--	--	6010/6020	HNO_3, pH <2	180 days	500	no	Optional	Optional

Notes:

HNO_3 – Nitric acid

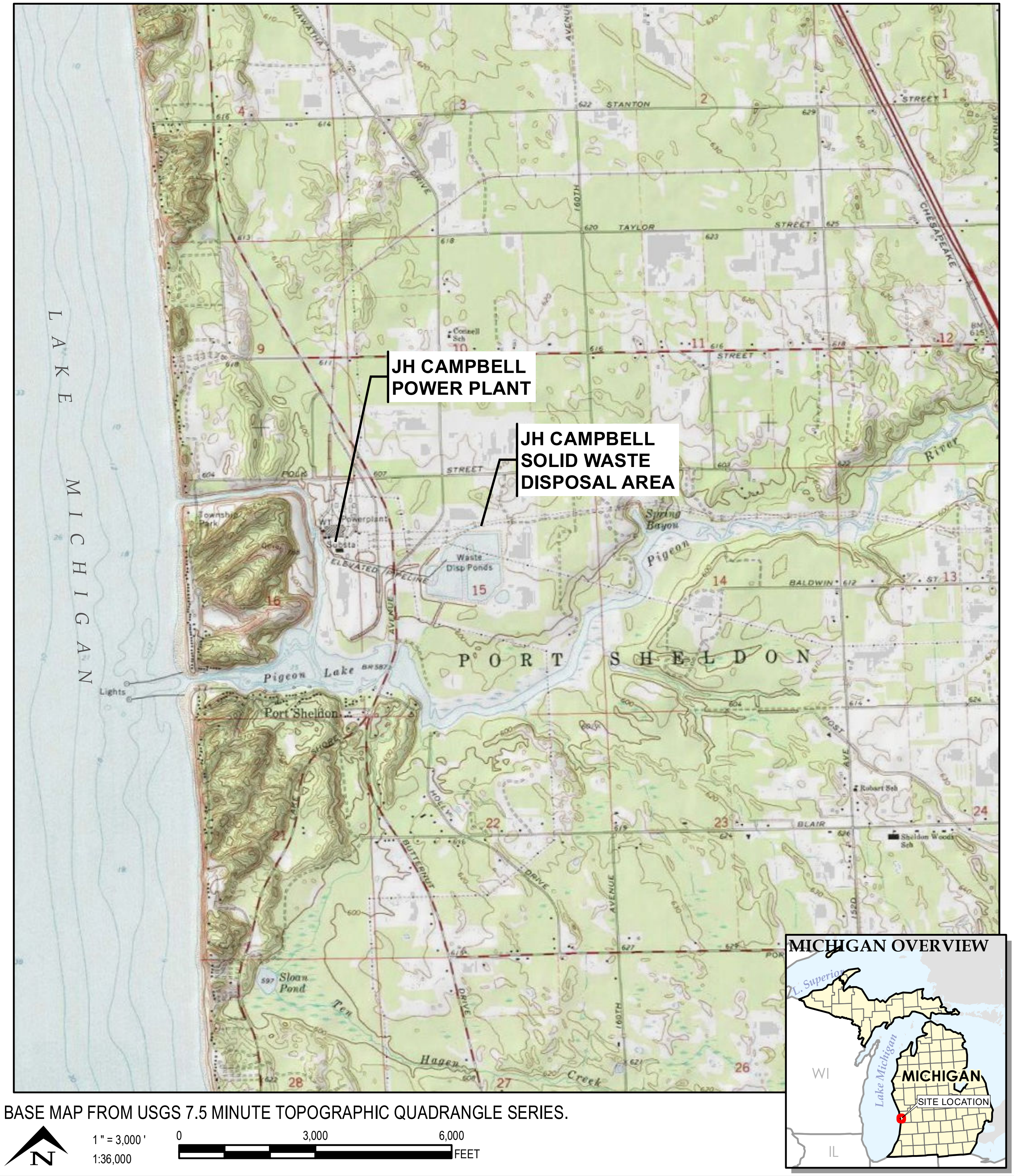
NA – Not applicable

[1] *All samples will be cooled to 4° C as part of sample preservation.*

[2] *Listed in both detection and assessment monitoring constituent lists*

[3] *Requires a larger sample volume (minimum 2 liter)*

Figures



BASE MAP FROM USGS 7.5 MINUTE TOPOGRAPHIC QUADRANGLE SERIES.

1540 Eisenhower Place
Ann Arbor, MI 48108-3284
Phone: 734.971.7080
www.trccompanies.com

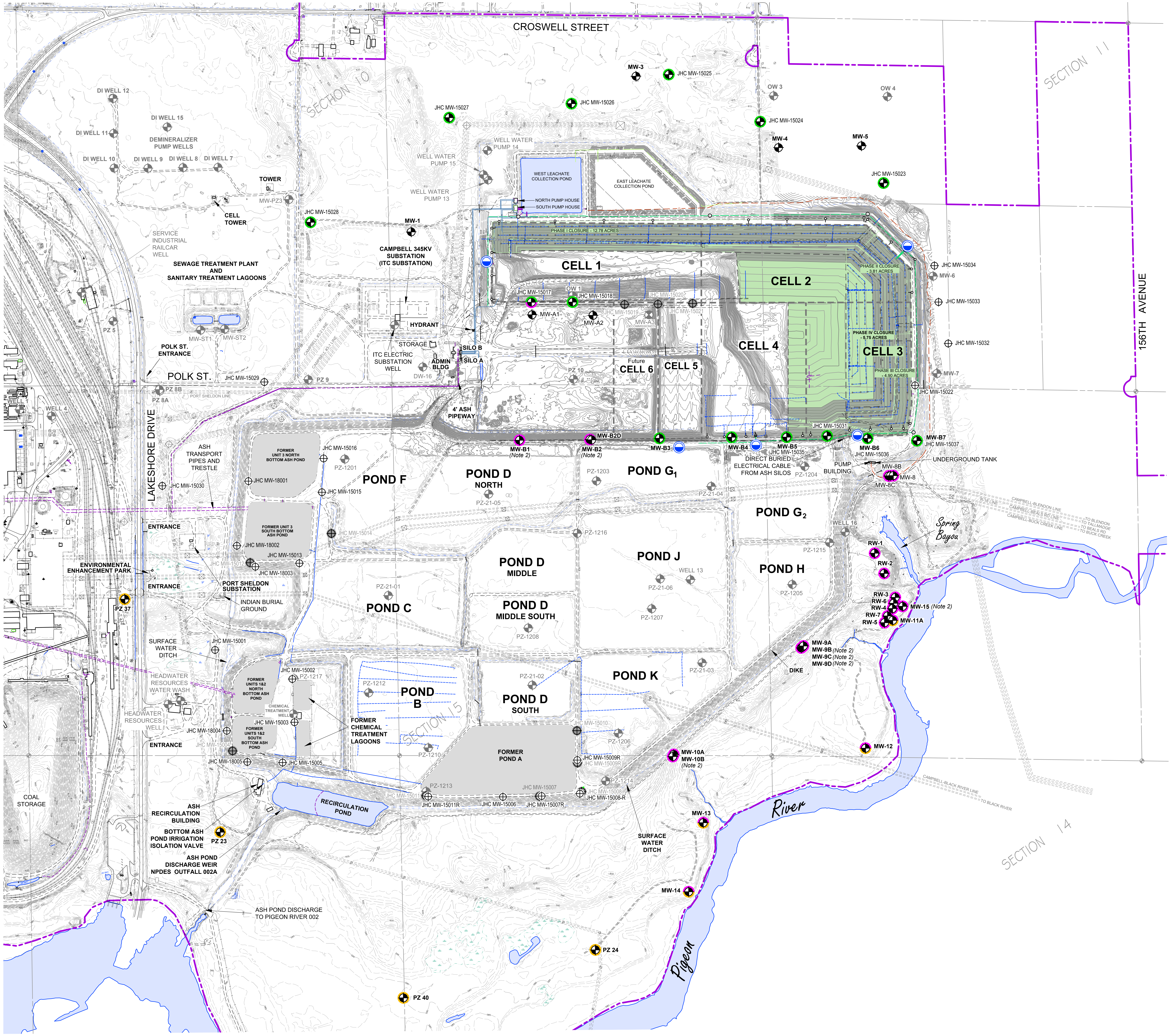
TRC - GIS

PROJECT:	CONSUMERS ENERGY COMPANY JH CAMPBELL POWER PLANT WEST OLIVE, MICHIGAN
TITLE:	SITE LOCATION MAP

DRAWN BY:	S. MAJOR
CHECKED BY:	B. YELEN
APPROVED BY:	S. HOLMSTROM
DATE:	JANUARY 2020
PROJ. NO.:	322174
FILE:	322174-001-022.mxd

FIGURE 1

E:\ConsumersEnergy\CCR_GW\2017_269767\322174-001-022.mxd -- Saved By: SMAJOR on 1/24/2020, 08:49:03 AM

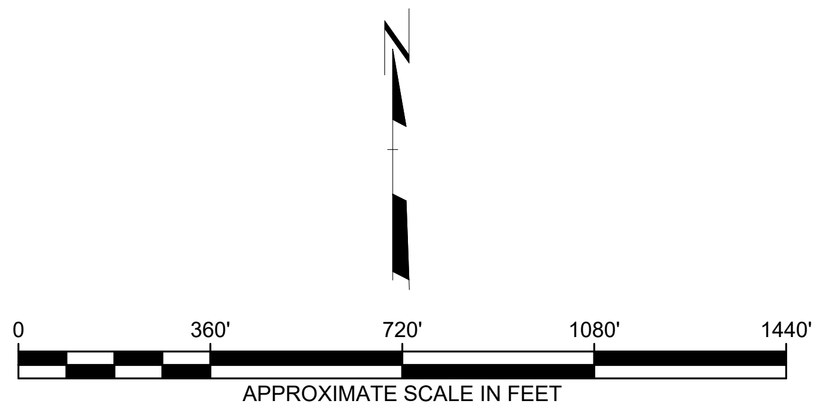


LEGEND

▬ ▬ ▬	APPROXIMATE PROPERTY BOUNDARY
— — —	SECTION LINE
┼┼┼┼	RAILROAD TRACKS
■■■■	CELL BOUNDARY
—x—x—x—	FENCELINE
— · · —	DITCH
═ ═ ═	UNPAVED ROAD OR DRIVE
≡≡≡≡	OVERHEAD POWER LINES
——E——	ELECTRIC LINE
— —S— —	SANITARY PIPE
——U——	UTILITY
— — —	LEACHATE PIPE
———	ASH PIPE
- - - -	PIPELINE
- - - -	IRRIGATION PIPE
🟦	WATER
🟩	FACILITY COVER
ᐝ	WETLAND
⌀	POLE
MW-1 ⊕	MONITORING WELL
MW-7 ⊕	WELL
JHC MW-15034 ⊕	RCRA MONITORING PROGRAM WELL
JHC MW-15004 ⊕	DECOMMISSIONED RCRA MONITORING PROGRAM WELL
MW-5 ⊕	DRY ASH LANDFILL HMP MONITORING WELL
PZ-23 ⊕	CELLS B-K HMP WELL
RW-1 ⊕	RAP MONITORING WELL
◒	APPROXIMATE SUMP SAMPLING LOCATION

NOTES

1. BASEMAP DEVELOPED FROM CONSUMERS ENERGY, "CAMPBASE.DWG", DATED 02/10/2014 AND NEDERVELD "CAMPBELL PLANT MONITORING WELLS-CCR MONITORING" , DATED 11/25/2015. PROVIDED BY CONSUMERS ENERGY.
2. RAP SENTINEL WELL



PROJECT:	CONSUMERS ENERGY JH CAMPBELL POWER PLANT WEST OLIVE, MICHIGAN		
TITLE:	SITE FEATURES MAP WITH TOPOGRAPHIC CONTOURS		
DRAWN BY:	D.STEHLE	PROJ. NO.:	367390.0001.01.03
CHECKED BY:	K.LOWERY	FIGURE 2	
APPROVED BY:	S.HOLMSTROM		
DATE:	NOVEMBER 2021		
FILE NO.:			367390.0001.01.03.02.dwg

TRC
1540 Eisenhower Place
Ann Arbor, MI 48108
Phone: 734.971.7080
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22x34 --- USER: ADragan --- ATTACHED XREF'S: CAMPBASE; Contours Misc; Contours Topo 2020-07-22 wo 7-8-9 bdrs --- ATTACHED IMAGES:
DRAWING NAME: \\annarbor-fp2\CADD\PJT\00_TRC\Consumers\JH Campbell\367390\0001\01\03\ 367390.0001.01.03.02.dwg --- PLOT DATE: November 04, 2021 - 2:33PM --- LAYOUT: FIG02B Site Features
Version: 2017-10-21

Appendix A
Soil Boring Logs and Well Construction Diagrams

Date Start: 9/29/15
Date Finish: 9/29/15
Drilling Company: Mateco Drilling
Driller's Name: John Pitsch
Drilling Method: Air Knife/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 12.0
Water Level Finish (ft. btoc.): 15.56

Northing: 521074.309
Easting: 12635685.32
Casing Elevation: 616.607

Borehole Depth (ft. bgs.): 20.0
Surface Elevation: 613.691

Descriptions By: A. Westhuis

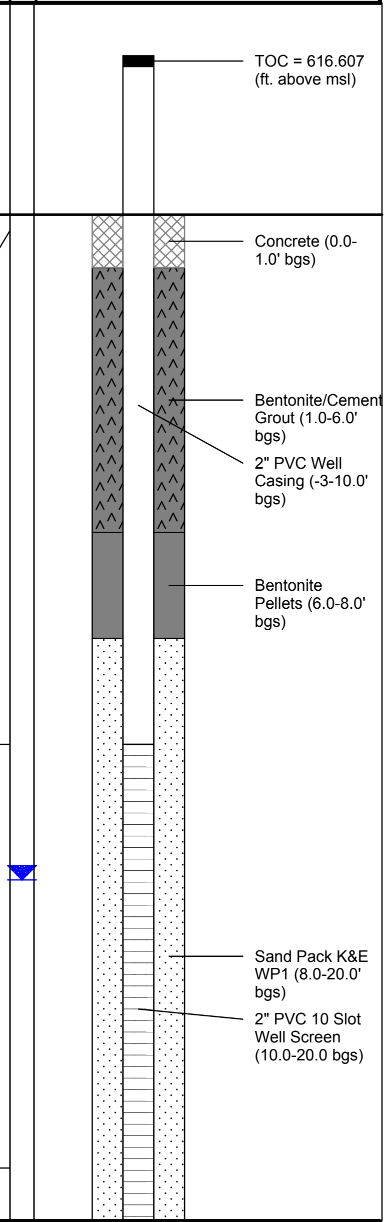
Well/Boring ID: **JHC MW-15017**

Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: 60 F Cloudy

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)	Well/Boring Construction
	615									
0								(0.0 - 0.3') Grass, Topsoil.		
								(0.3 - 10.0') SAND, fine to medium, subrounded; trace silt; poorly sorted; dry; yellowish brown (10YR 4/3).		
	610									
5		1	0.0- 10.0'	4.0	NA					
	605									
10								(10.0 - 18.0') SAND, fine, subrounded; trace silt; well sorted; moist to wet; pale brown (10YR 6/3). NOTE: Wet at 12.0'.		
	600									
15		2	10.0- 20.0'	7.0	NA					
								(18.0 - 20.0') SAND, medium, little fine, subrounded; trace silt; well sorted; wet; pale brown (10YR 6/3). NOTE: Little small pebbles, subrounded.		
	595									



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs = below ground surface
btoc = below top of casing

Air knife to 10.0' bgs.
Groundwater encountered at 12.0' bgs.
Water level at development was 15.56' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 596.00 feet

Date Start: 9/29/15
Date Finish: 9/29/15
Drilling Company: Mateco Drilling
Driller's Name: John Pitsch
Drilling Method: Air Knife/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 12.0
Water Level Finish (ft. btoc.): 15.56

Northing: 521074.309
Easting: 12635685.32
Casing Elevation: 616.607

Borehole Depth (ft. bgs.): 20.0
Surface Elevation: 613.691

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15017**

Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: 60 F Cloudy

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)	Well/Boring Construction
							⁚⁚			⁚⁚ ☰ ⁚⁚
20								End of boring at 20.0' bgs.		

Design & Consultancy for natural and built assets

Remarks: bgs = below ground surface
btoc = below top of casing

Air knife to 10.0' bgs.
Groundwater encountered at 12.0' bgs.
Water level at development was 15.56' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 596.00 feet

Date Start: 9/29/15
Date Finish: 9/29/15
Drilling Company: Mateco Drilling
Driller's Name: John Pitsch
Drilling Method: Air Knife/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 12.0
Water Level Finish (ft. btoc.): 16.29

Northing: 521075.536
Easting: 12635979.61
Casing Elevation: 617.022

Borehole Depth (ft. bgs.): 20.0
Surface Elevation: 614.262

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15018**

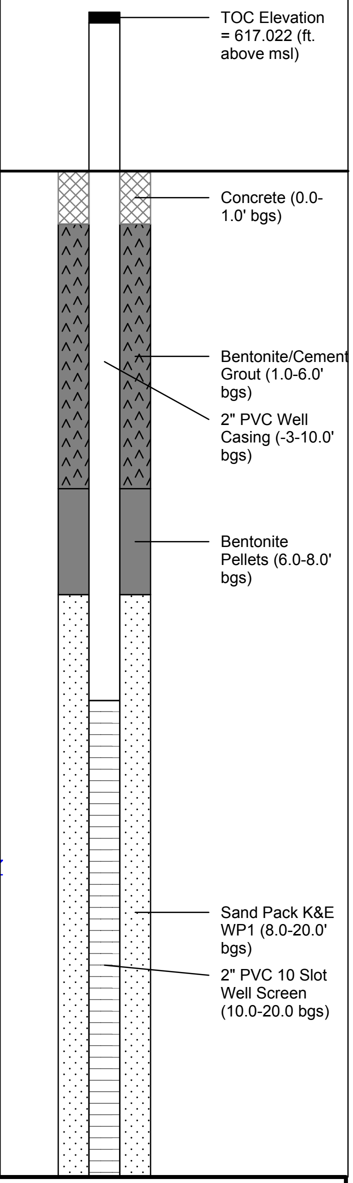
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: 60 F Cloudy

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
	615								
0								(0.0 - 0.3') Grass, Topsoil.	
								(0.3 - 1.0') ASH and SAND, very fine to fine, well sorted; dry; light gray (10YR 7/2). NOTE: Fill material.	
	610							(1.0 - 10.0') SAND, fine, subrounded; well sorted; dry; brownish yellow (10YR 6/6).	
5		1	0.0-10.0'	3.0	NA				
	605								
10								(10.0 - 17.0') SAND, fine, subrounded; trace silt; well sorted; moist to wet; very pale brown (10YR 7/3). NOTE: Wet at 12.0' bgs.	▼
	600								
15		2	10.0-20.0'	7.0	NA				
								(17.0 - 20.0') SAND, medium, little fine, subrounded; trace granules; subrounded; trace silt; well sorted; wet; very pale brown (10YR 7/4).	

Well/Boring Construction



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs = below ground surface
btoc = below top of casing

Air knife to 10.0' bgs.
Groundwater encountered at 12.0' bgs during drilling.
Water level at development was 16.29' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 600.45 feet

Date Start: 9/29/15
Date Finish: 9/29/15
Drilling Company: Mateco Drilling
Driller's Name: John Pitsch
Drilling Method: Air Knife/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 12.0
Water Level Finish (ft. btoc.): 16.29

Northing: 521075.536
Easting: 12635979.61
Casing Elevation: 617.022

Borehole Depth (ft. bgs.): 20.0
Surface Elevation: 614.262

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15018**

Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: 60 F Cloudy

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)	Well/Boring Construction
	595						⁘			⁘ ☰ ⁘
20								End of boring at 20.0' bgs.		

ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs = below ground surface
btoc = below top of casing

Air knife to 10.0' bgs.
Groundwater encountered at 12.0' bgs during drilling.
Water level at development was 16.29' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 600.45 feet

Date Start: 9/30/15
Date Finish: 9/30/15
Drilling Company: Mateco Drilling
Driller's Name: Dan Mourer
Drilling Method: Air Knife/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 21.0
Water Level Finish (ft. btoc.): 29.39

Northing: 520479.719
Easting: 12638430.24
Casing Elevation: 623.792

Borehole Depth (ft. bgs.): 33.0
Surface Elevation: 620.917

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15022**

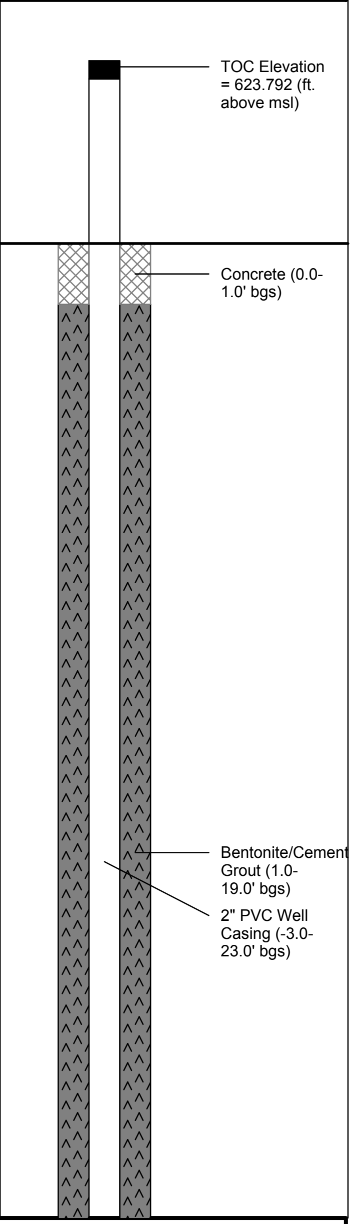
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Sunny, 65F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
0								(0.0 - 0.3') TOPSOIL; grass.	
	620							(0.3 - 10.0') SAND, very fine to fine, subrounded; trace silt; well sorted; dry to moist; dark yellowish brown (10 YR 4/6).	
5		1	0-10'	5	NA				
	615								
10								(10.0 - 21.0') SAND, fine, subrounded; little very fine sand; trace granules, subrounded; trace silt; well sorted; moist to wet; yellowish brown (10 YR 5/6).	
	610								
15		2	10-20'	4	NA				
	605								

Well/Boring Construction



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs= below ground surface
btoc = below top of casing

Air knife to 10.0' bgs.
Groundwater not encountered during drilling.
Water level at development was 29.39' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 594.34 feet

Date Start: 9/30/15
Date Finish: 9/30/15
Drilling Company: Mateco Drilling
Driller's Name: Dan Mourer
Drilling Method: Air Knife/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 21.0
Water Level Finish (ft. btoc.): 29.39

Northing: 520479.719
Easting: 12638430.24
Casing Elevation: 623.792

Borehole Depth (ft. bgs.): 33.0
Surface Elevation: 620.917

Descriptions By: A. Westhuis

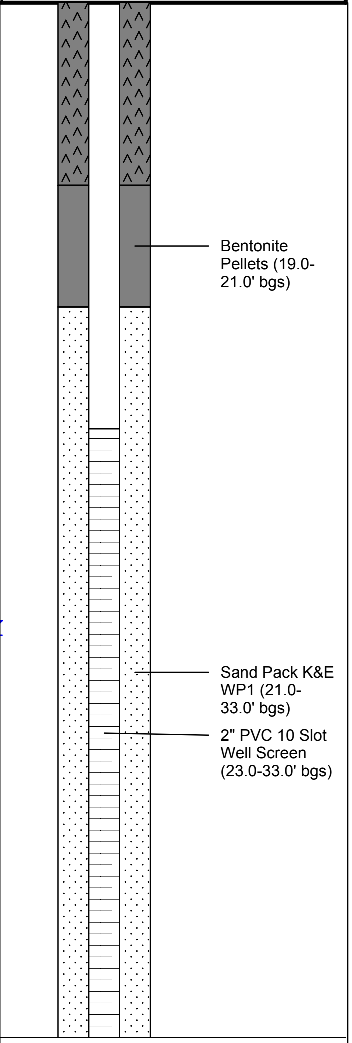
Well/Boring ID: **JHC MW-15022**

Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Sunny, 65F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)	Well/Boring Construction
20	600	3	20-30'	5	NA			(21.0 - 29.0') SAND, fine to medium, subrounded; trace granules, subrounded; well sorted; wet; yellowish brown (10 YR 5/4).		
25	595								▼	
30	590	4	30-33'	3	NA			(29.0 - 33.0') SAND, very fine to fine, subrounded; trace silt; well sorted; wet; yellowish brown (10 YR 5/4).		
35	585							End of boring at 33.0' bgs.		



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs= below ground surface
btoc = below top of casing

Air knife to 10.0' bgs.
Groundwater not encountered during drilling.
Water level at development was 29.39' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 594.34 feet

Date Start: 10/1/15
Date Finish: 10/1/15
Drilling Company: Mateco Drilling
Driller's Name: Dan Mourer
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): NA
Water Level Finish (ft. btoc.): NA

Northing: NA
Easting: NA
Casing Elevation: NA

Borehole Depth (ft. bgs.): 25.0
Surface Elevation: NA

Descriptions By: A. Westhuis

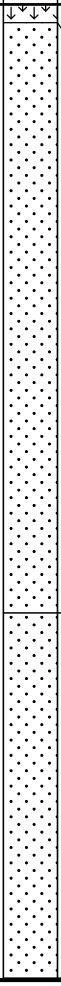
Well/Boring ID: **JHC-MW-15023**

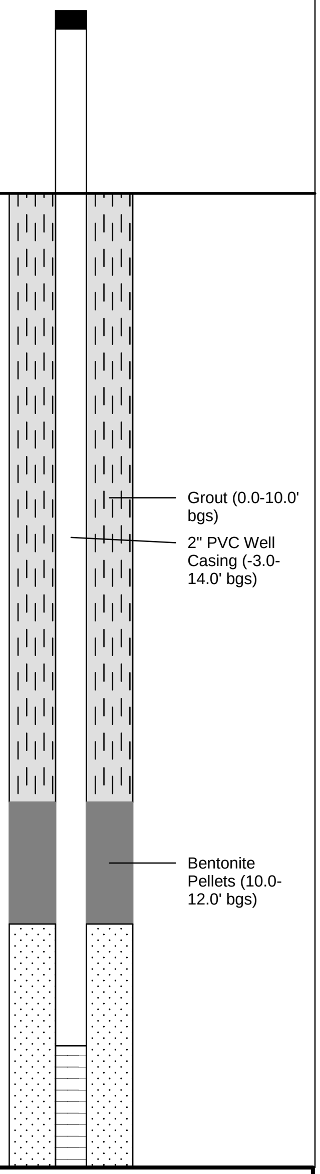
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Sunny, 60F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)	Well/Boring Construction
0	0							(0.0 - 0.3') TOPSOIL; grass.		
								(0.3 - 10.0') SAND, very fine to fine, subrounded; trace silt; well sorted; dry; brown (10 YR 4/3). Note: Trace wood fragments from 7.0 to 10.0' bgs.		
5	-5	1	0-10'	10	NA					
10	-10							(10.0 - 16.0') SAND, very fine to fine, subrounded; trace to little silt; well sorted; dry to moist; brownish yellow (10 YR 6/8).		
15	-15	2	10-20'	8	NA					





Remarks: bgs= below ground surface.

Hand auger to 10.0' bgs.
No odor or staining observed.

Date Start: 10/1/15
Date Finish: 10/1/15
Drilling Company: Mateco Drilling
Driller's Name: Dan Mourer
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): NA
Water Level Finish (ft. btoc.): NA

Northing: NA
Easting: NA
Casing Elevation: NA

Borehole Depth (ft. bgs.): 25.0
Surface Elevation: NA

Descriptions By: A. Westhuis

Well/Boring ID: **JHC-MW-15023**

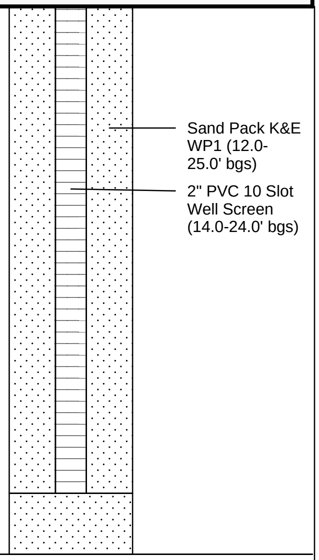
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Sunny, 60F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Stratigraphic Description	Water Level (ft. bgs.)
							(16.0 - 17.0') SAND, very fine to fine, subrounded; trace silt; well sorted; moist; yellow (10 YR 7/6).	
							(17.0 - 18.0') SAND, fine, subrounded; trace silt; well sorted; moist; brownish yellow (10 YR 6/6).	
20	-20						(18.0 - 21.0') SAND, very fine; little fine sand, subrounded; trace silt; well sorted; wet; pale brown (10 YR 6/3).	
		3	20-25'	4	NA		(21.0 - 25.0') SAND, medium; trace fine sand, subangular; trace granules, subangular; poorly sorted; wet; pale brown (10 YR 6/3).	
25	25						End of boring at 25.0' bgs.	
30	-30							
35	-35							

Well/Boring Construction



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Remarks: bgs= below ground surface.

Hand auger to 10.0' bgs.
No odor or staining observed.

Date Start: 10/1/15
Date Finish: 10/1/15
Drilling Company: Mateco Drilling
Driller's Name: Dan Mourer
Drilling Method: Air Knife/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 10.0
Water Level Finish (ft. btoc.): NA

Northing: 522366.013
Easting: 12637322.68
Casing Elevation: 616.617

Borehole Depth (ft. bgs.): 20.0
Surface Elevation: 613.787

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15024**

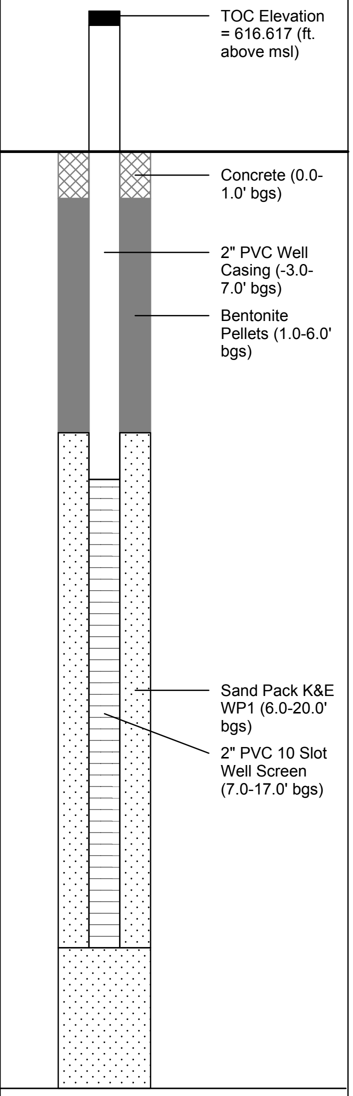
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Sunny, 60F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
	615								
0								(0.0 - 0.3') TOPSOIL; grass.	
								(0.3 - 10.0') SAND, fine, subrounded; trace silt; well sorted; dry to moist; brownish yellow (10 YR 6/6).	
	610								
5		1	0-10'	4	NA				
	605							NOTE: Wet at 10.0' bgs.	
10								(10.0 - 20.0') SAND, fine to medium, subrounded; trace to little very fine sand; trace silt; well sorted; wet; pale brown (10 YR 6/3).	
	600								
15		2	10-20'	9	NA				
	595								
20								End of boring at 20.0' bgs.	

Well/Boring Construction



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs= below ground surface.

Air knife to 10.0' bgs.
Groundwater encountered at 10.0' bgs during drilling.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 602.24 feet above mean sea level.

Date Start: 10/1/15
Date Finish: 10/1/15
Drilling Company: Mateco Drilling
Driller's Name: Dan Mourer
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 12.0
Water Level Finish (ft. btoc.): NA

Northing: 522702.978
Easting: 12636668.15
Casing Elevation: 617.167

Borehole Depth (ft. bgs.): 20.0
Surface Elevation: 614.137

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15025**

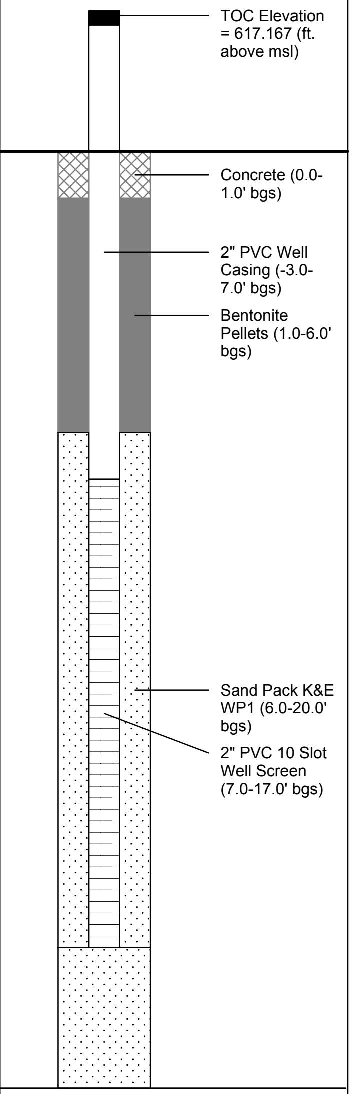
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Sunny, 60F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
	615								
0								(0.0 - 0.3') TOPSOIL; grass.	
								(0.3 - 5.0') SAND, fine, subrounded; trace silt; well sorted; dry; very pale brown (10 YR 7/3).	
	610								
5		1	0-10'	10	NA			(5.0 - 12.0') SAND, fine, subrounded; trace silt; well sorted; dry; brownish yellow (10 YR 6/6). Note: Color change to brownish yellow (10YR 6/8) at 6.0' bgs.	
	605								
10									
								(12.0 - 15.0') SAND, fine, subrounded; trace silt; well sorted; wet; pale brown (10 YR 6/3).	
	600								
15		2	10-20'	8	NA			(15.0 - 16.0') SAND, fine to medium, subrounded; trace coarse sand, subrounded; trace granules, subrounded; trace silt; well sorted; wet; pale brown (10 YR 6/3).	
								(16.0 - 20.0') SAND, very fine to fine, subrounded; little silt; well sorted; wet; pale brown (10 YR 6/3).	
	595								
20								End of boring at 20.0' bgs.	

Well/Boring Construction



Remarks: bgs= below ground surface.

Hand auger to 10.0' bgs.
Groundwater encountered at 12.0' bgs during drilling.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 603.36 feet above mean sea level.

Date Start: 10/2/15
Date Finish: 10/2/15
Drilling Company: Mateco Drilling
Driller's Name: Dan Mourer
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 12.0
Water Level Finish (ft. btoc.): NA

Northing: 522495.091
Easting: 12635971.82
Casing Elevation: 618.042

Borehole Depth (ft. bgs.): 20.0
Surface Elevation: 615.087

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15026**

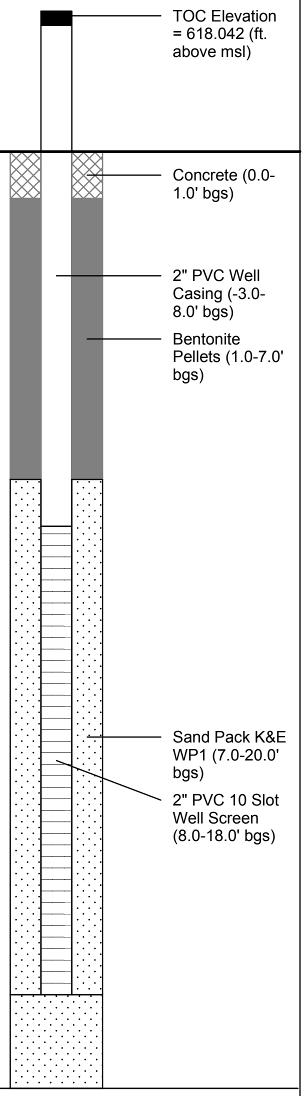
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Sunny, 45F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
0	615							(0.0 - 0.3') TOPSOIL; grass.	
								(0.3 - 3.0') SAND, fine, subrounded; trace medium sand, subrounded; trace silt; well sorted; dry; very pale brown (10 YR 7/3).	
								(3.0 - 8.0') SAND, fine, subrounded; trace silt; well sorted; dry; brownish yellow (10 YR 6/6).	
5	610	1	0-10'	10	NA				
								(8.0 - 12.0') SAND, fine, subrounded; little very fine sand, subrounded; trace silt; well sorted; dry; pale brown (10 YR 6/3) to brownish yellow (10YR 6/6).	
10	605								
								(12.0 - 20.0') SAND, very fine to fine, subrounded; trace silt; well sorted; moist to wet; pale brown (10 YR 6/3).	
15	600	2	10-20'	6	NA				
20	595							End of boring at 20.0' bgs.	

Well/Boring Construction



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Remarks: bgs= below ground surface.

Hand auger to 10.0' bgs.
Groundwater encountered at 12.0' bgs during drilling.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 602.32 feet above mean sea level.

Date Start: 10/2/15
Date Finish: 10/2/15
Drilling Company: Mateco Drilling
Driller's Name: Dan Mourer
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 13.0
Water Level Finish (ft. btoc.): 15.85

Northing: 522394.86
Easting: 1235097.51
Casing Elevation: 617.302

Borehole Depth (ft. bgs.): 20.0
Surface Elevation: 614.767

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15027**

Client: Consumers Energy

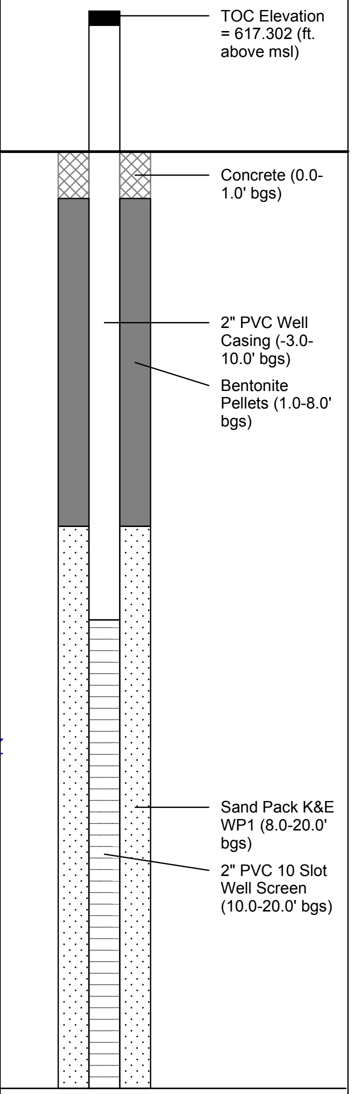
Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Sunny, 50F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample
0	615					
5	610	1	0-10'	10	NA	
10	605					
15	600	2	10-20'	8	NA	
20	595					

Stratigraphic Description	Water Level (ft. bgs.)
(0.0 - 0.3') TOPSOIL; grass.	
(0.3 - 2.0') SAND, very fine to fine, subrounded; trace silt; well sorted; dry; dark yellowish brown (10 YR 4/6).	
(2.0 - 6.0') SAND, very fine to fine, subrounded; trace silt; well sorted; dry; very pale brown (10 YR 7/3).	
(6.0 - 16.0') SAND, fine, subrounded; trace silt; well sorted; dry; yellow (10YR 7/6). Note: Wet at 13.0' bgs.	▼
(16.0 - 20.0') SAND, fine; trace medium sand, subrounded; well sorted; wet; pale brown (10 YR 6/3).	
End of boring at 20.0' bgs.	

Well/Boring Construction



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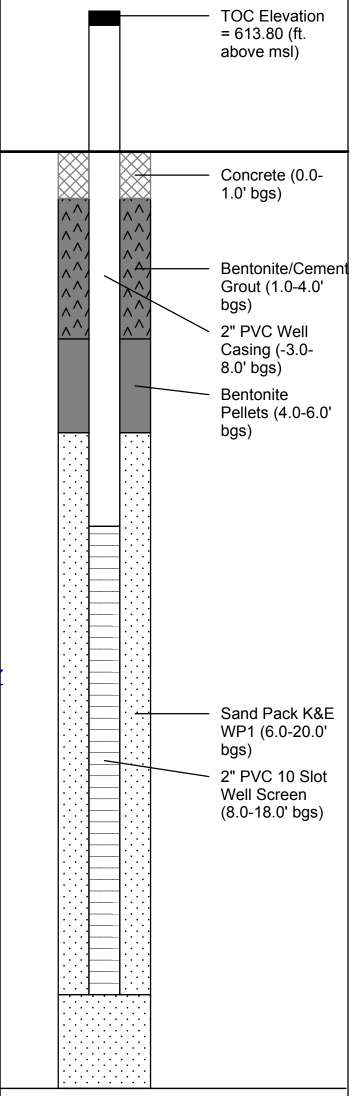
Remarks: bgs= below ground surface
btoc = below top of casing

Hand auger to 10.0' bgs.
Groundwater encountered at 13.0' bgs during drilling.
Water level at development was 15.85' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 601.04 feet

Date Start: 10/2/15 **Date Finish:** 10/2/15 **Drilling Company:** Mateco Drilling **Driller's Name:** Dan Mourer **Drilling Method:** Air knife/Sonic **Sampling Method:** Continuous **Rig Type:** Sonic **Water Level Start (ft. bgs.):** 9.0 **Water Level Finish (ft. btoc.):** 14.38	**Northing:** 521646.198 **Easting:** 12634105.34 **Casing Elevation:** 613.8 **Borehole Depth (ft. bgs.):** 20.0 **Surface Elevation:** 611.025 **Descriptions By:** A. Westhuis	**Well/Boring ID:** **JHC MW-15028** **Client:** Consumers Energy **Location:** JH Campbell Facility 1700 Crosswell Street Site A West Olive, MI 49460 **Weather Conditions:** Sunny, 60F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
0								(0.0 - 0.3') TOPSOIL; grass.	
	610							(0.3 - 5.0') SAND, very fine to fine, subrounded; trace silt; well sorted; dry; yellowish (10 YR 7/8).	
5	605	1	0-10'	10	NA			(5.0 - 9.0') SAND, fine, subrounded; trace silt; well sorted; dry to moist; pale brown (10 YR 6/3).	
10	600							(9.0 - 20.0') SAND, medium; trace to little very fine to fine sand, subrounded; trace silt; poorly sorted; moist to wet; pale brown (10 YR 6/3).	▼
15	595	2	10-20'	9	NA				
20	590							End of boring at 20.0' bgs.	

Well/Boring Construction



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs= below ground surface
btoc = below top of casing

Air knife to 10.0' bgs.
Groundwater encountered at 9.0' bgs during drilling.
Water level at development was 14.38' btoc.
No odor or staining observed.
Groundwater elevation measured on December 3, 2015 was 599.00 feet

Date Start: 10/5/15
Date Finish: 10/5/15
Drilling Company: Mateco Drilling
Driller's Name: Dan Mourer
Drilling Method: Air knife/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 5.0
Water Level Finish (ft. btoc.): 7.99

Northing: 519760.827
Easting: 12633044.37
Casing Elevation: 607.167

Borehole Depth (ft. bgs.): 20.0
Surface Elevation: 604.047

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15030**

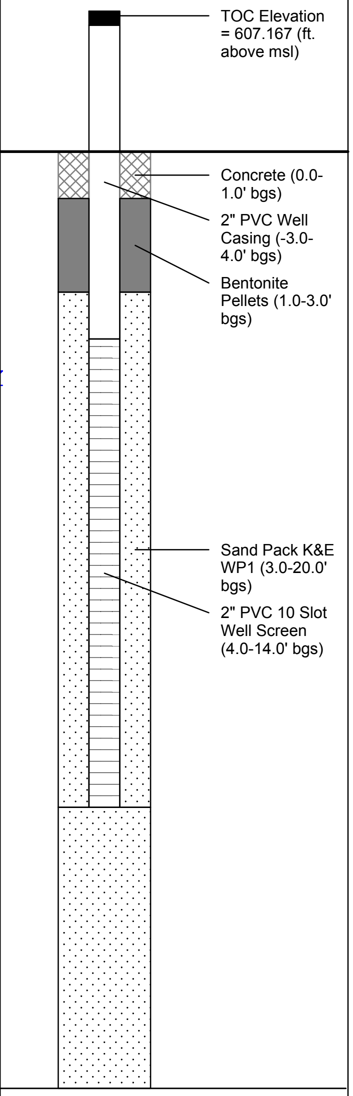
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Cloudy, Light Rain, 65F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
	605								
0								(0.0 - 0.3') TOPSOIL; grass.	
								(0.3 - 10.0') SAND, fine, subrounded; trace silt; well sorted; dry to moist; dark brown (10 YR 3/3) to very pale brown (10YR 7/3).	
	600								
5		1	0-10'	3	NA			NOTE: Wet at 5.0' bgs.	▼
	595								
10								(10.0 - 20.0') SAND, fine, subrounded; little medium sand, subrounded; trace silt; well sorted; wet; very pale brown (10 YR 7/3) to light gray (10YR 7/2).	
	590								
15		2	10-20'	6	NA				
	585								
20								End of boring at 20.0' bgs.	

Well/Boring Construction



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs = below ground surface
btoc = below top of casing

Air knife to 10.0' bgs.
Groundwater encountered at 5.0' bgs during drilling.
Water level at development was 7.99' btoc.
No odor or staining observed.
Groundwater elevation measured on December 3, 2015 was 599.65 feet

Project: DE000722.0003.00005 Template: ARCADIS_Analytical Boring-Well 2013_New Logo
Data File: JHC MW-15030.dat Date: 2/4/2016 Created/Edited by: A. DeGrandis/C. Jeffers

Date Start: 10/5/15
Date Finish: 10/6/15
Drilling Company: Mateco Drilling
Driller's Name: John Pitsch
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 36.0
Water Level Finish (ft. btoc.): 43.20

Northing: 520118.003
Easting: 12637801.51
Casing Elevation: 635.872

Borehole Depth (ft. bgs.): 45.0
Surface Elevation: 632.937

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15031**

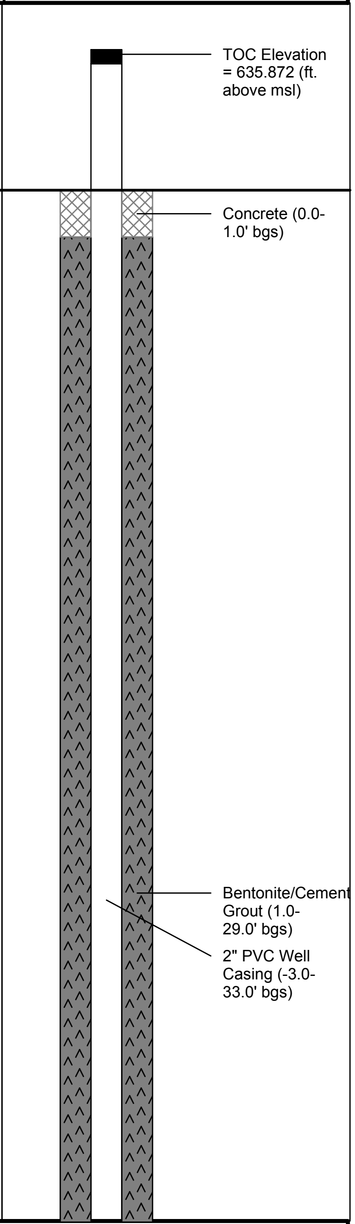
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Cloudy, 65F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
	635								
0		1	0-10'	10	NA			(0.0 - 0.3') TOPSOIL; grass.	
								(0.3 - 2.0') SAND, fine, subrounded; trace silt; well sorted; dry; dark grayish brown (10 YR 4/2).	
	630							(2.0 - 5.0') SAND, fine, subrounded; trace silt; well sorted; dry; very pale brown (10 YR 7/3).	
5	625							(5.0 - 10.0') SAND, fine, subrounded; trace silt; well sorted; dry; pale brown (10 YR 6/3).	
10	620	2	10-20'	6	NA			(10.0 - 19.0') SAND, fine, subrounded; trace silt; well sorted; moist; brown (10 YR 5/3).	
15	615								
20								(19.0 - 25.0') SAND, fine, subrounded; trace medium sand, subrounded; trace silt; well sorted; moist; yellowish brown (10 YR 5/4).	

Well/Boring Construction



Remarks: bgs = below ground surface
btoc = below top of casing

Hand auger to 10.0' bgs.
Groundwater encountered at 36.0' bgs during drilling.
Water level at development was 43.20' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 592.53 feet

Date Start: 10/5/15
Date Finish: 10/6/15
Drilling Company: Mateco Drilling
Driller's Name: John Pitsch
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 36.0
Water Level Finish (ft. btoc.): 43.20

Northing: 520118.003
Easting: 12637801.51
Casing Elevation: 635.872

Borehole Depth (ft. bgs.): 45.0
Surface Elevation: 632.937

Descriptions By: A. Westhuis

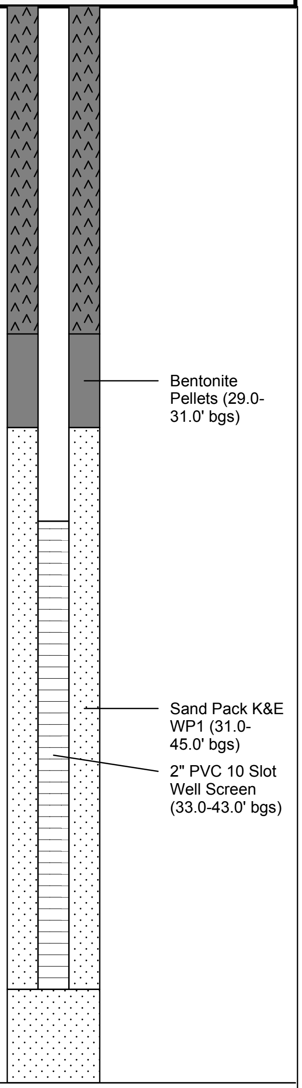
Well/Boring ID: **JHC MW-15031**

Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Cloudy, 65F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)	Well/Boring Construction
	610									
25		3	20-30'	8	NA			(25.0 - 36.0') SAND, very fine to fine, subrounded; trace silt; well sorted; very pale brown (10 YR 7/3).		
	605									
30										
	600									
35		4	30-40'	8	NA			(36.0 - 45.0') SAND, fine; little medium sand, subrounded; trace silt; well sorted; pale brown (10 YR 6/3). NOTE: Wet at 36.0' bgs.		
	595									
40									▼	
	590	5	40-45'	5	NA					
45								End of boring at 45.0' bgs.		
	585									



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs = below ground surface
btoc = below top of casing

Hand auger to 10.0' bgs.
Groundwater encountered at 36.0' bgs during drilling.
Water level at development was 43.20' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 592.53 feet

Date Start: 10/6/15
Date Finish: 10/6/15
Drilling Company: Mateco Drilling
Driller's Name: John Pitsch
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 16.0
Water Level Finish (ft. btoc.): 17.88

Northing: 520779.281
Easting: 12638667.93
Casing Elevation: 614.287

Borehole Depth (ft. bgs.): 25.0
Surface Elevation: 611.322

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15032**

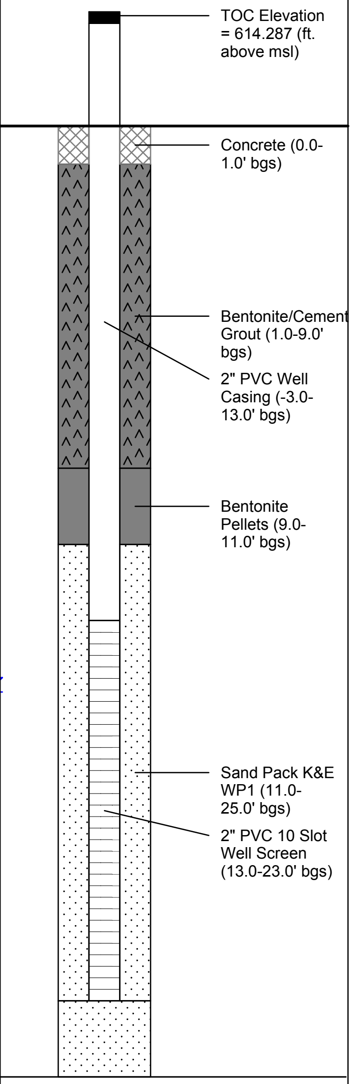
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Cloudy, 60F.

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
	615								
0								(0.0 - 0.3') TOPSOIL; grass.	
	610							(0.3 - 2.5') SAND, fine, subrounded; trace silt; well sorted; dry; brown (10 YR 4/3).	
								(2.5 - 11.0') SAND, fine, subrounded; trace silt; well sorted; dry; very pale brown (10 YR 7/3).	
5		1	0-10'	10	NA				
	605								
10									
	600							(11.0 - 21.0') SAND, fine, subrounded; trace silt; well sorted; moist to wet; pale brown (10 YR 6/3).	
15		2	10-20'	7	NA				▼
	595							NOTE: Wet at 16.0' bgs.	
20									
	590							(21.0 - 25.0') SAND, fine to medium, subrounded; trace silt; well sorted; wet; pale brown (10 YR 6/3).	
		3	20-25'	5	NA				
25								End of boring at 25.0' bgs.	
	585								

Well/Boring Construction



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs = below ground surface
btoc = below top of casing

Hand auger to 10.0' bgs.
Groundwater encountered at 16.0' bgs during drilling.
Water level at development was 17.88' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 595.41 feet

Date Start: 10/6/15
Date Finish: 10/6/15
Drilling Company: Mateco Drilling
Driller's Name: John Pitsch
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 19.0
Water Level Finish (ft. btoc.): 22.93

Northing: 521075.809
Easting: 12638598.12
Casing Elevation: 620.987

Borehole Depth (ft. bgs.): 30.0
Surface Elevation: 618.082

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15033**

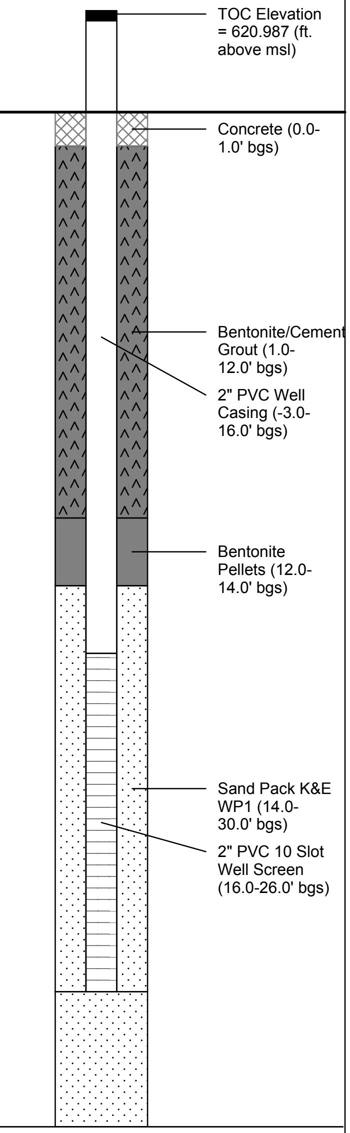
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Cloudy, 60F.

Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Stratigraphic Description	Water Level (ft. bgs.)
1	0-10'	10	NA		(0.0 - 0.3') TOPSOIL; grass. (0.3 - 0.8') SAND, fine, subrounded; trace silt; well sorted; dry; brown (10 YR 4/3). (0.8 - 2.0') SAND, fine, subrounded; trace silt; well sorted; dry; yellowish brown (10 YR 5/6). (2.0 - 10.0') SAND, fine, subrounded; trace silt; well sorted; dry; very pale brown (10 YR 7/3).	
2	10-20'	6	NA		(10.0 - 20.0') SAND, very fine to fine, subrounded; trace silt; well sorted; moist; yellowish brown (10 YR 5/4). NOTE: Wet at 19.0' bgs.	▼
3	20-30'	6	NA		(20.0 - 30.0') SAND, fine; little medium sand, subrounded; trace silt; well sorted; moist to wet; very pale brown (10 YR 6/3).	
					End of boring at 30.0' bgs.	

Well/Boring Construction



Remarks: bgs = below ground surface
btoc = below top of casing

Hand auger to 10.0' bgs.
Groundwater encountered at 19.0' bgs during drilling.
Water level at development was 22.93' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 598.05 feet

Project: DE000722.0003.00005 Template: ARCADIS_Analytical Boring-Well 2013_New Logo
Data File: JHC-MW-15033.dat Date: 2/4/2016 Created/Edited by: A. DeGrandis/C. Jeffers

Date Start: 10/6/15
Date Finish: 10/6/15
Drilling Company: Mateco Drilling
Driller's Name: John Pitsch
Drilling Method: Hand Auger/Sonic
Sampling Method: Continuous
Rig Type: Sonic
Water Level Start (ft. bgs.): 13.0
Water Level Finish (ft. btoc.): 16.87

Northing: 521335.834
Easting: 12638568.9
Casing Elevation: 615.972

Borehole Depth (ft. bgs.): 25.0
Surface Elevation: 612.902

Descriptions By: A. Westhuis

Well/Boring ID: **JHC MW-15034**

Client: Consumers Energy

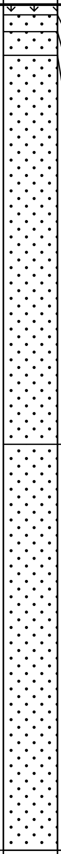
Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: Sunny, 60F.

DEPTH (feet bgs.)	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample
0-10	1	0-10'	10	NA	
10-20	2	10-20'	5	NA	
20-25	3	20-25'	5	NA	

DEPTH (feet bgs.): 0, 5, 10, 15, 20, 25, 30
ELEVATION: 615, 610, 605, 600, 595, 590, 585

Geologic Column



Stratigraphic Description

(0.0 - 0.3') TOPSOIL; grass.

(0.3 - 0.8') SAND, fine, subrounded; trace silt; well sorted; dry; brown (10 YR 4/3).

(0.8 - 1.5') SAND, fine, subrounded; trace silt; well sorted; dry; yellowish brown (10 YR 5/6).

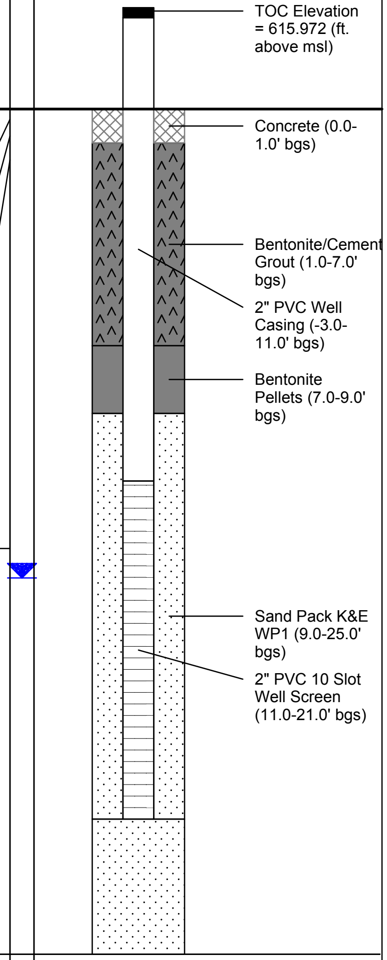
(1.5 - 13.0') SAND, fine, subrounded; trace silt; well sorted; dry; very pale brown (10 YR 7/3).

(13.0 - 25.0') SAND, fine; little medium sand, subrounded; trace silt; well sorted; moist to wet; pale brown (10 YR 6/3).

End of boring at 25.0' bgs.

Water Level (ft. bgs.)

Well/Boring Construction



Remarks: bgs = below ground surface
btoc = below top of casing

Hand auger to 10.0' bgs.
Groundwater not encountered during drilling.
Water level at development was 16.87' btoc.
No odor or staining observed.
Groundwater elevation measured on December 2, 2015 was 599.07 feet

Date Start: 3/14/01
Date Finish: 3/14/01
Drilling Company: EDC, Inc.
Driller's Name: Sean Smith
Drilling Method: Hollow Stem Auger
Sampling Method: Split Spoon
Rig Type: Hollow Stem Auger
Water Level Start (ft. bgs.): 36.0
Water Level Finish (ft. btoc.): NA

Northing: NA
Easting: NA
Casing Elevation: 634.89

Borehole Depth (ft. bgs.): 43.5
Surface Elevation: NA

Descriptions By: Rebecca J. Koepke

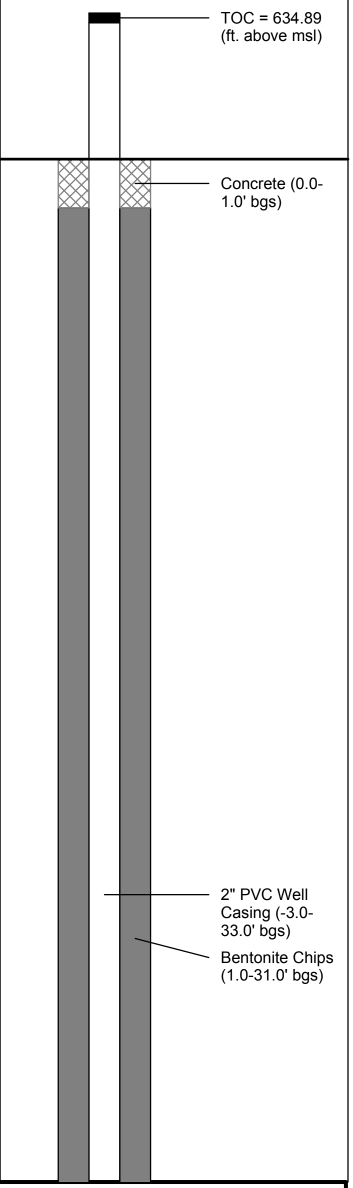
Well/Boring ID: **JHC MW-15035**

Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: NA

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)	Well/Boring Construction
	625									
0								(0.0 - 0.5') SANDY ORGANIC SOIl, very dark brown, very fine to fine, loose, moist.		
								(0.5 - 30.5') SAND, brownish yellow (10YR 6/8), poorly graded, fine, trace silt, round, loose, slightly moist.		
	620									
5		1	5.0-7.0'	0.9	NA					
	615									
10		2	10.0-12.0'	1.6	NA					
	610									
15		3	15.0-17.0'	1.6	NA					
	605									
20								NOTE: Brownish yellow (10YR 6/6)		



Remarks: bgs = below ground surface
btoc = below top of casing

Groundwater encountered at 36.0' bgs during drilling.
No odor or staining observed.

Date Start: 3/14/01
Date Finish: 3/14/01
Drilling Company: EDC, Inc.
Driller's Name: Sean Smith
Drilling Method: Hollow Stem Auger
Sampling Method: Split Spoon
Rig Type: Hollow Stem Auger
Water Level Start (ft. bgs.): 36.0
Water Level Finish (ft. btoc.): NA

Northing: NA
Easting: NA
Casing Elevation: 634.89

Borehole Depth (ft. bgs.): 43.5
Surface Elevation: NA

Descriptions By: Rebecca J. Koepke

Well/Boring ID: **JHC MW-15035**

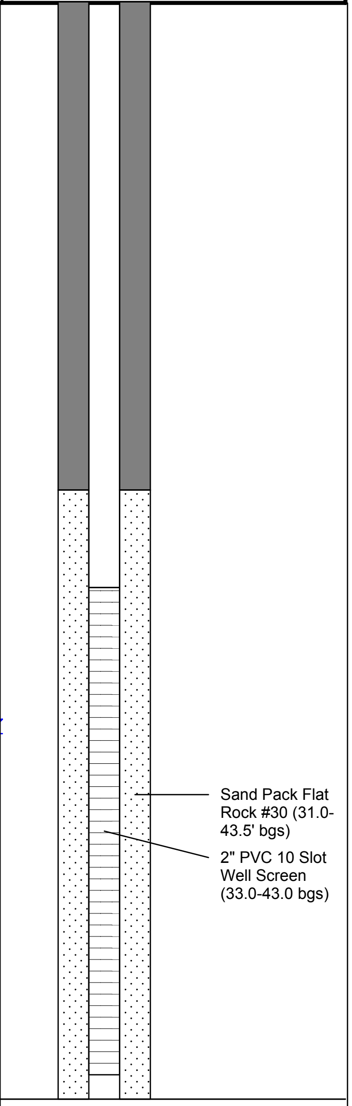
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: NA

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
		4	20.0-22.0'	1.8	NA				
	600								
25									
		5	25.0-27.0'	1.8	NA				
	595								
30								NOTE: Yellow (10YR 7/6)	
		6	30.0-32.0'	1.3	NA			NOTE: Very pale brown (10YR 7/4)	
	590								
35									
		7	35.0-37.0'	1.8	NA			NOTE: Wet at 36.0' bgs	▼
	585								
40									
								End of boring at 43.5' bgs.	
	580								
45									

Well/Boring Construction



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs = below ground surface
btoc = below top of casing

Groundwater encountered at 36.0' bgs during drilling.
No odor or staining observed.

Date Start: 3/13/01
Date Finish: 3/13/01
Drilling Company: EDC, Inc.
Driller's Name: Sean Smith
Drilling Method: Hollow Stem Auger
Sampling Method: Split Spoon
Rig Type: Hollow Stem Auger
Water Level Start (ft. bgs.): NA
Water Level Finish (ft. btoc.): NA

Northing: NA
Easting: NA
Casing Elevation: 615.90

Borehole Depth (ft. bgs.): 30.5
Surface Elevation: NA

Descriptions By: Rebecca J. Koepke

Well/Boring ID: **JHC MW-15036**

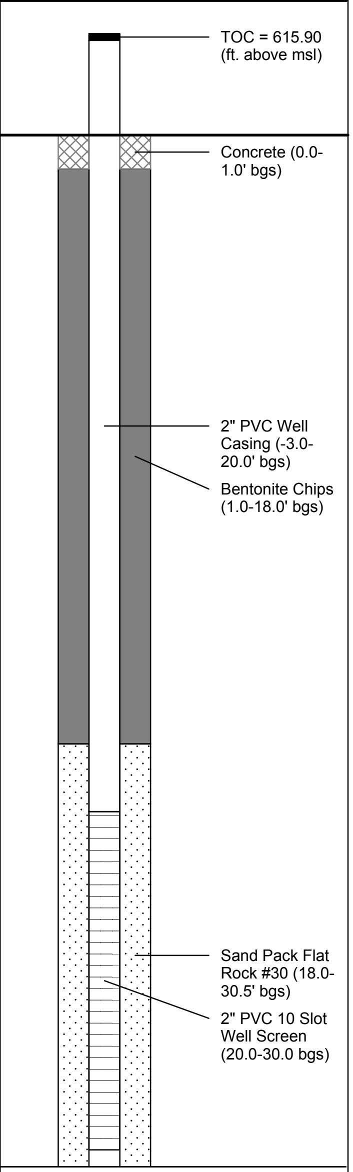
Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: NA

DEPTH (feet bgs.)	ELEVATION	Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample	Geologic Column	Stratigraphic Description	Water Level (ft. bgs.)
	625								
0								(0.0 - 0.5') SANDY ORGANIC SOIl, very dark brown, very fine to fine, loose, moist.	
								(0.5 - 30.5') SAND, very pale brown (10YR 7/4), poorly graded, fine, trace silt, round, loose, slightly moist.	
	620								
5		1	5.0-7.0'	0.9	NA				
	615								
10		2	10.0-12.0'	1.6	NA			NOTE: Very fine.	
	610								
15		3	15.0-17.0'	1.6	NA			NOTE: Very pale brown (10YR 8/3), fine.	
	605								
20		4	20.0-22.0'	1.8	NA			NOTE: Fine to medium, moist.	
	600								
25		5	25.0-27.0'	1.8	NA				
	595								
30									
								End of boring at 30.5' bgs.	

Well/Boring Construction



ARCADIS Design & Consultancy for natural and built assets

Remarks: bgs = below ground surface
btoc = below top of casing

No odor or staining observed.

Project: DE000722.0003.00006 Template: ARCADIS_Analytical Boring-Well 2013_New Logo
Data File: MW-15036.dat Date: 2/4/2016 Created/Edited by: Natural Resource Technology, Inc./A. Westhuis

Date Start: 8/29/01
Date Finish: 8/29/01
Drilling Company: EDC, Inc.
Driller's Name: Sean Smith
Drilling Method: Hollow Stem Auger
Sampling Method: Split Spoon
Rig Type: Hollow Stem Auger
Water Level Start (ft. bgs.): NA
Water Level Finish (ft. btoc.): NA

Northing: NA
Easting: NA
Casing Elevation: 613.42

Borehole Depth (ft. bgs.): 28.5
Surface Elevation: NA

Descriptions By: Rebecca J. Koepke

Well/Boring ID: **JHC MW-15037**

Client: Consumers Energy

Location: JH Campbell Facility
1700 Crosswell Street Site A
West Olive, MI 49460

Weather Conditions: NA

Sample Run Number	Sample/Int/Type	Recovery (feet)	PID Headspace (ppm)	Analytical Sample
1	0.0-2.0'	1.5	NA	
2	2.0-4.0'	1.96	NA	
3	4.0-6.0'	1.5	NA	
4	6.0-8.0'	1.4	NA	
5	8.0-10.0'	1.0	NA	
6	10.0-12.0'	1.2	NA	
7	12.0-14.0'	1.5	NA	
8	14.0-16.0'	1.2	NA	
9	16.0-18.0'	NA	NA	
10	18.0-20.0'	1.0	NA	
11	20.0-22.0'	1.6	NA	
12	22.0-24.0'	1.5	NA	
13	24.0-26.0'	1.5	NA	
14	26.0-28.0'	1.8	NA	

Stratigraphic Description

(0.0 - 1.0') SAND, semi compact, dark, organic, clayey, fine, moist.

(1.0 - 2.0') SAND, loose, tan, fine, trace silt, moist.

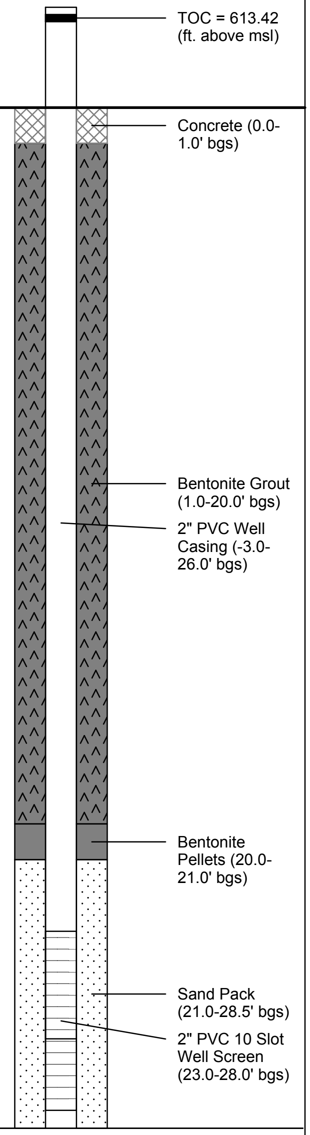
(2.0 - 3.0') SAND, medium compact, light brown, fine, trace clay, moist.

(3.0 - 28.5') SAND, loose, tan, fine, trace silt, moist.

NOTE: Wet at 22.0' bgs, grades to light brown.

End of boring at 28.5' bgs.

Well/Boring Construction



Remarks: bgs = below ground surface
btoc = below top of casing

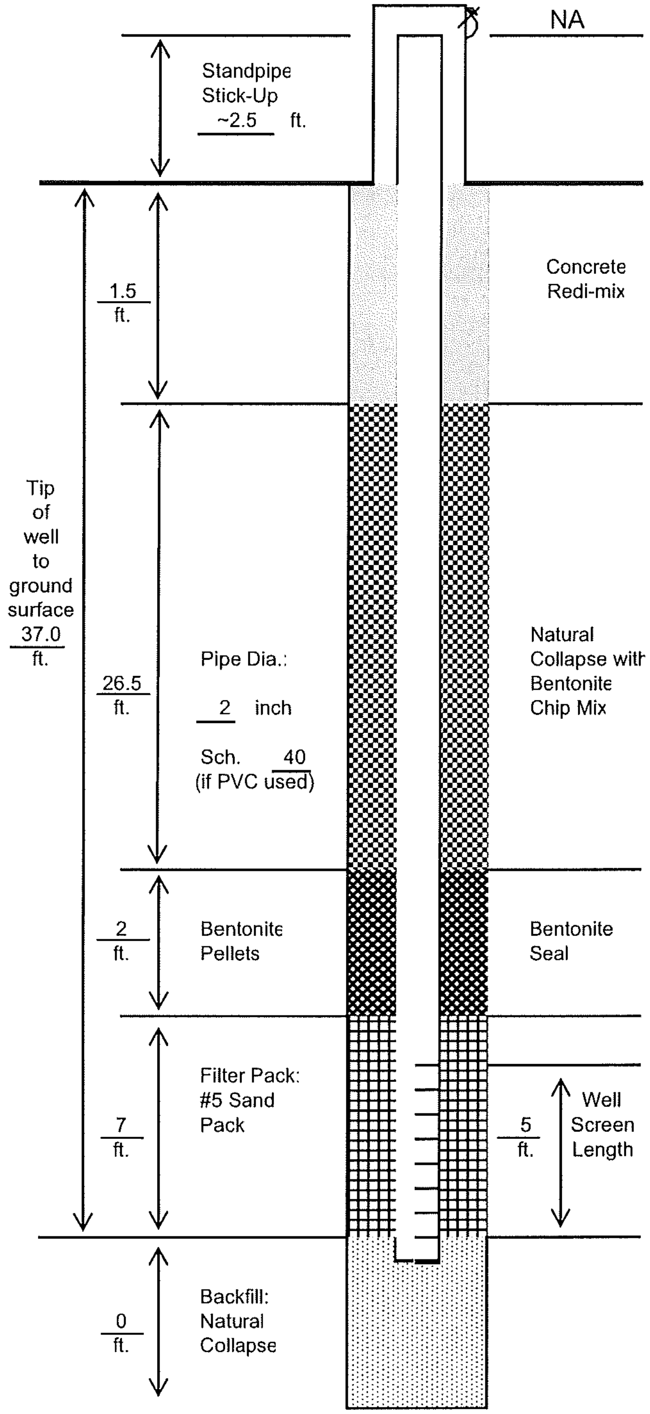
Groundwater encountered at 22.0' bgs during drilling.
No odor or staining observed.

WELL INSTALLATION DIAGRAM

PROJECT: Consumers Energy - JH Campbell - Well Installation STS PROJECT NO. 200706041

WELL NO.: **MW-B3** DATE INSTALLED: 12/13/07 DRILLERS: Shepler DRILL RIG: Hollow Stem Auger

NORTHING: 802.55 EASTING: 4997.50



Elevation Top of Well Pipe

1) Type of Pipe:

☒ PVC ☐ Galvanized

☐ Stainless ☐ Other

2) Type of Pipe Joint:

☒ Threaded ☐ Other

3) Type of Well Screen:

☒ PVC ☐ Galvanized

☐ Stainless ☐ Other

4) Screen Slot Size? 10

5) Type of Protector Pipe:

☐ None ☒ Steel Pipe with Lock

☐ Flushmount

6) Construction Details

A. Protective pipe, top elevation NA ft. MSL

B. Well casing, top elevation NA ft. MSL

C. Land surface, elevation NA ft. MSL

D. Surface seal, bottom ft. MSL or 1.50 ft. BGS

E. Bentonite seal, top ft. MSL or 28.00 ft. BGS

F. Filter pack, top ft. MSL or 30.00 ft. BGS

G Screen top ft. MSL or 32.00 ft. BGS

H. Well botton ft. MSL or 37.00 ft. BGS

I. Filter pack, bottom ft. MSL or 37.00 ft. BGS

J. Borehole, bottom ft. MSL or 37.00 ft. BGS

K. Borehole, diameter 8.0 inches

L. O.D. well casing 2.00 inches

WATER WELL DRILLING AND PUMP INSTALLATION WELLOGIC FIELD WORK SHEET

Well B-4

Well owner: ______
Permit number: ______
Drill rig operator: Randy Shepler
Job number: 110454
Date: 03/26/2007

Well depth: 45 ft.

Well type:
- ✓ new
- ___ dry hole
- ___ replacement

Well use:
- ___ household
- ___ public (well label & WSSN needed)
- ___ other Monitoring Well

Drilling method:
- ___ mud rotary
- ___ cable tool
- ___ other 4¼ Auger

Date completed: 03/26/07

CASING type:
- ✓ pvc
- ___ steel galvanized
- ___ steel black
- ___ other ______

Diameter: 2 in. to 45 ft. depth
___ in. to ___ ft. depth

Casing joint:
- ___ solvent welded
- ___ welded
- ___ threaded and coupled
- ___ other Flush Joint

Height: 3 ft. above grade

Casing fittings:
- ___ centralizer
- ___ drive shoe
- ___ shale packer
- ___ other ______

BOREHOLE diameter: 8 in. to 45 ft. depth
___ in. to ___ ft. depth

STATIC WATER LEVEL: N/A ft. below / above grade

WELLHEAD COMPLETION:
- ___ 12" above grade
- ___ pitless adapter
- ___ well house
- ___ other Exp. CAP.

NEAREST SOURCE OF CONTAMINATION:
Type ______ ft. ___ direction ______
Type ______ ft. ___ direction ______

SCREEN: installed ✓ yes ___ no

Well intake:
- ___ bedrock well
- ___ unscreened drift well

Filter pack: ✓ yes ___ no

Filter pack from: 38 ft. to 45 ft.

Filter type (brand): K+E #1

Screen material:
- ✓ pvc slotted
- ___ s.s. wirewrapped
- ___ other ______

Screen diameter: 2 in. ___ telescoped ✓ pipe size

Screen length: 5 ft. **Slot:** 10

Set between: 40 ft. and 45 ft.

Blank: ___ ft. above / below / other

Screen fittings:
- ___ k packer
- ___ bremer check
- ___ other Attached

GROUTING method:
- ___ pipe outside casing
- ___ driven dry granular
- ✓ other Chips

Number of bags: 1

Additives: None.

Grout material:
- ___ high solids bentonite
- ___ neat cement & bentonite
- ___ neat cement
- ___ other bentonite chips

Grouting depth: 4 ft. to 8 ft.

DEPTH TO WATER WHILE PUMPING:
___ ft. after ___ hrs. at ___ gpm

Yield test method: ___ air ___ bailer ___ test pump ___ other ______

PUMP installed: ___ yes ___ no

Pump installation only: ___ yes ___ no

Manufacturer ______

Model no ______ hp. ______

Drop pipe diameter: ______ in Length: ______ ft

Pump capacity: ______ gpm

Drawdown seal ___ yes ___ no

Pump type: ___ submersible
___ other ______

PRESSURE TANK installed: ___ yes ___ no

Tank buried: ___ yes ___ no

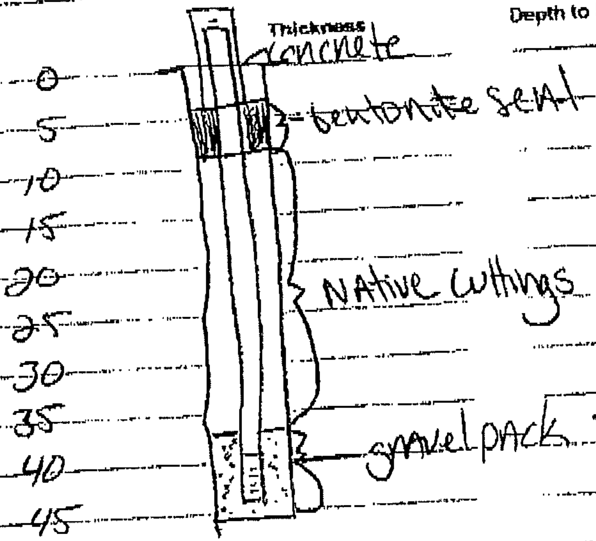
Tank type: ___ bladder/diaphragm
___ other ______

Manufacturer ______

Model no. ______

Total tank capacity: ______ gallons

GEOLOGY
Formation Description **Thickness** **Depth to Bottom:**



General remarks:

Top of Casing 634.48

N 803.015
E 5500.253
Ground 631.895

Engineering &
Environmental
Solutions, LLC

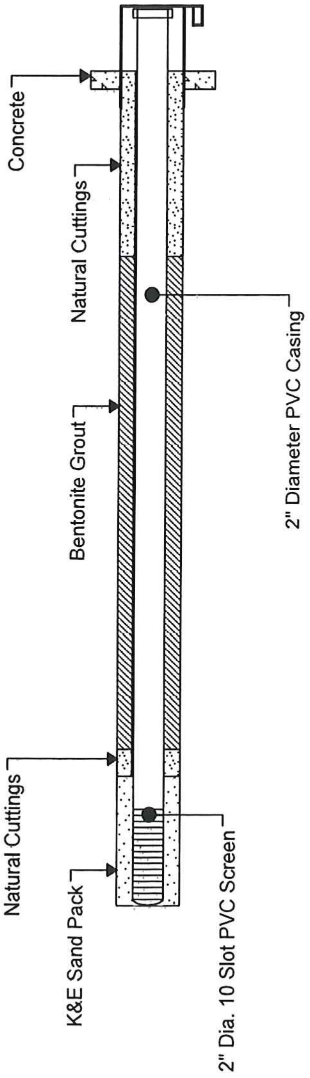
200 North Franklin Street
Suite 202
Zeeland, Michigan 49464

Log of Borehole: MW-B4R

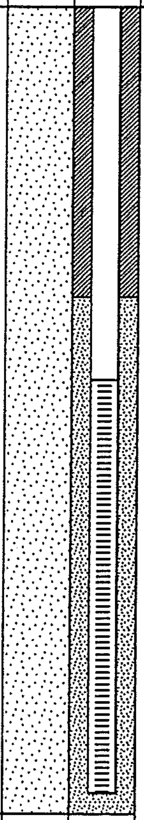
Project Name: Consumers Energy Company
Project Number: 005-11-001
Site Location: J.H. Campbell
Drilling Method: 4.25" OD HSA
Sampling Method: 2' Split Spoon
Ground Elevation (feet): 633.80
Top of Casing Elevation (feet): 636.16
Logged By: Kurt Van Appledorn
Start Date: 5-23-2011
End Date: 5-23-2011
Driller: EDAC
Crew Chief: Rick
Depth to Water (ft BGS during drilling): 36
Easting: 5514
Northing: 802
Comments: Original MW-B4 was abandoned in place by backfilling with bentonite grout

SUBSURFACE PROFILE				SAMPLE			
Depth (feet BGS)	Symbol	Description	Depth/Elev.	Sample Length (feet)	Recovery (feet)	Blow Counts	PID (ppb)
0		Ground Surface	633.8 0.0				
40		Brown (7.5YR/5/3) fine SAND, trace medium sand, wet.		2	1.3	2 2 4 6	
				2	1.0	7 14 18 23	
45		End of Boring	588.8 45.0				

Well Completion Details



Sheet: 1 of 1



Sample									Soil Properties					
Number and Type	Length Att. & Recovered (in)	Blow Counts	Depth in Feet	Soil/Rock Description And Geologic Origin For Each Major Unit	USCS	Graphic Log	Well Diagram	PID/FID	Compressive Strength	Moisture Content	Liquid Limit	Plasticity Index	P 200	RQD/ Comments
MW-B5 (5)	21	5/6 8/6	26											
			28											
			30	yellow (10YR 7/6)										
MW-B5 (6)	15	2/3 6/6												
			32											
			34		SP									
MW-B5 (7)	22	2/4 6/7	36	very pale brown (10YR 7/4) wet at approximately 36 feet below ground surface										
			38											
			40											
			42											
			44	*End of Boring at 43.5 feet below ground surface*										
			46											
			48											
			50											
			52											
			54											
			56											
			58											
			60											
			62											

Appendix B
Groundwater Sampling SOP

Title: **Groundwater Sampling**	Procedure Number: **ECR 009**
	Revision Number: **4**
	Effective Date: **August 2020**

Authorization Signatures

Darby Litz		Elizabeth Denly	
Technical Reviewer	Date	Environmental Sector Quality Director	Date
Darby Litz	8/4/20	Elizabeth Denly	8/4/20

TABLE OF CONTENTS

Page No.

ATTACHMENTS

Attachment A	Groundwater Field Parameter Stabilization Criteria for Selected Jurisdictions
Attachment B	Example Groundwater Field Data Records
Attachment C	SOP Fact Sheet
Attachment D	SOP Modifications for PFAS

1.0 INTRODUCTION

1.1 Scope & Applicability

This Standard Operating Procedure (SOP) was prepared to provide TRC personnel with general guidance in performing groundwater sampling activities. This SOP details equipment and sampling procedures for low-flow sampling, multi-volume purge sampling and passive diffusion bag sampling from monitoring wells. Various regulatory agencies and project-specific work plans may have specific requirements (e.g., equipment/instrument, flow rate, etc.) that may be applicable and take precedence, depending on the program.

The objective of groundwater sampling is to obtain a representative sample of water from a saturated zone or groundwater-bearing unit (i.e., aquifer) with minimal disturbance of groundwater chemistry. This requires that the sample being collected is representative of groundwater within the formation surrounding the well bore as opposed to stagnant water within the well casing or within the filter pack immediately surrounding the well casing.

1.2 Summary of Method

There are three general approaches to groundwater purging/sampling that can be used to obtain a representative groundwater sample for analysis: 1) the low-flow or micropurge method where the mixing of the stagnant water is minimized using low-flow pumping rates during the collection of the groundwater sample; 2) the multiple well volume removal approach in which the stagnant water is removed from the well and the filter pack prior to sample collection; and 3) the passive sampler procedure where water quality equilibration with the surroundings is achieved through deployment of the passive sampler for a sufficient amount of time prior to sampling.

For low-flow and multiple well volume removal, there are various types of equipment available to perform groundwater sampling. The most common of these are the submersible pump, peristaltic pump, and bailer. However, the equipment selected and the purge method used, if any, will depend on project goals, data quality objectives (DQOs), hydrogeologic conditions, and regulatory requirements. Care should be taken when choosing the sampling procedures and device(s), as some procedures have the potential to affect the representativeness of the sample more than others. For repeated monitoring events, the sampling methodology and operating equipment employed should be consistent to minimize potential variability due to sampling procedures. The type of sampling method utilized is dependent upon site-specific conditions and it is not within the scope of this document to recommend a specific methodology. For specialized sampling programs involving per- and polyfluorinated alkyl substances (PFAS), refer to Attachment D for further details. Information on applicability of sampling methods can be found on Interstate Technology & Regulatory Council (ITRC) and United States Environmental Protection Agency (EPA) websites.

1.3 Equipment

The following equipment is commonly used to collect groundwater samples from a monitoring well. Site-specific conditions may warrant the use of additional equipment or deletion of items from this list.

- Appropriate level of personal protective equipment (PPE) as specified in the site-specific Health and Safety Plan (HASP)
- Electronic water level indicator capable of measuring to 0.01 foot accuracy
- Oil/water interface probe
- Extra batteries for water level/interface probe
- Submersible pump with low-flow capabilities (less than 1 liter/min) constructed of inert materials (e.g., stainless steel and Teflon®), such as a bladder pump (with sufficient quantity of bladders, o-rings, grab plates, etc.)
- Peristaltic pump
- Source of power for use with submersible or peristaltic pump (e.g., 12-volt battery, compressor, generator, compressed gas tanks, etc.)
- Flow controller for use with submersible pump (varies depending on type of pump used)
- Bottom-filling bailer constructed of inert materials (i.e., polyethylene, polyvinyl chloride [PVC], stainless steel or Teflon®)
- Bailer cord or wire (recommended Teflon®-coated, stainless steel cable; bailer wire; or contaminant-free rope with a Teflon®-coated stainless steel leader to connect bailer and rope)
- Tubing (Teflon®, Teflon®–lined polyethylene, or high density polyethylene [HDPE], type dependent upon project objectives)
- Silicone tubing (only used for peristaltic pump head and/or flow-through cell connections)
- Water quality meter(s) capable of measuring parameters, such as pH, temperature, specific conductivity, oxidation-reduction potential (ORP), and dissolved oxygen (DO)
- Flow-through cell
- T-connector
- Turbidity meter
- Passive sampling device (and any device-specific accessories)
    - Passive diffusion bags (PDBs)
    - Tether (stainless steel cable or marine-grade polyethylene rope), well cap, and weights, unless already installed
    - Funnel (Fill kit)
    - PVC cable ties
    - Tool to cut cable ties
    - PVC discharge tubes
    - Tether reel
- Well lock keys
- Bolt cutters

- Appropriate tools for equipment and to open well box (e.g., socket wrench, pry bar, etc.)
- Containers with lids for purge water (i.e., 5-gallon buckets, drums, etc.)
- Stopwatch or timer
- Graduated measuring container appropriately sized to measure flow rate
- Sample bottle labels
- Laboratory-grade water (can request from lab – for equipment blanks)
- Chain-of-custody (COC) forms
- Sample cooler(s)
- Photoionization detector (PID) or flame ionization detector (FID) for well head monitoring
- Sample containers (may be supplied by the laboratory depending upon the regulatory program): The proper containers should be determined in conjunction with the analytical laboratory in the planning stages of the project. If not included in sample containers provided by laboratory, sample preservatives will need to be kept with sample containers, and added to sample containers prior to sample collection.
- Field book and/or Groundwater Field Data Record (multiple copies)
- Filtration equipment
- In-line filter (0.45 micron [µm]) or as otherwise required by the project-specific work plan.
- Bubble wrap/Bubble wrap bags
- Lint-free, non-abrasive, disposable towels (e.g., Kimwipes®)
- Indelible marking pens
- Plastic bags (e.g., Ziploc®)
- Ice
- Teflon® tape
- Plastic sheeting or large trash bags which can be cut open
- Umbrella, tent, or equivalent for shading equipment (particularly the flow-through cell) from sunlight or blocking rain
- Equipment decontamination supplies
- Container for bailing water out of water-logged road boxes or well vaults
- Map of well locations and well construction data
- Copy of field notes from previous sampling event for reference
- Project-specific work plan

1.4 Definitions

Bailer	A cylindrical device suspended from a rope or cable, which is used to remove water, non-aqueous phase liquid (NAPL), sediment or other materials from a well or open borehole. Usually equipped with some type of check valve at the base to allow water, NAPL, and/or sediment to enter the bailer and be retained as it is lifted to the surface. A bailer may be made in varying diameters; however a bailer that fits in a two-inch well is the most common. In some instances a < 1-inch diameter bailer (a.k.a. pencil bailer) is used for small diameter wells.
Borehole	A hole drilled into the soil or bedrock using a drill rig or similar equipment.
Dense Non-aqueous Phase Liquid (DNAPL)	Separate-phase product that is denser than water and, therefore, sinks to the bottom of the water column.
Depth To Water (DTW)	The distance to the groundwater surface from an established measuring point.
Drawdown	The response to purging/pumping a well resulting in the lowering of groundwater within the water column in the well or in a water-bearing zone.
FID	An instrument that uses a flame to break down volatile organic compounds (VOCs) into ions that can be measured.
Flow-Through Cell	The container used to immerse the multi-parameter probes in well purge water during pre-sampling well purging. The flow-through cell is usually made of transparent acrylic and is connected to the end of the discharge tubing creating an in-line, sealed container in which purge water circulates around the measurement probes. The discharge from the pump prior to the flow-through cell may be fitted with a check valve or T-connector for collection of water for turbidity measurement.
Flush Mount	The type of well completion where the riser terminates at or below grade. Flush-mounted wells are typically completed with a “curb box” which is an “at-grade” enclosure designed to protect the well riser.
Light Non-aqueous Phase Liquid (LNAPL)	Separate-phase product that is less dense than water and therefore floats on the surface of the water.

Monitoring Well	A well made from a PVC pipe, or other appropriate material, with slotted screen installed across or within a saturated zone. A monitoring well is typically constructed with a PVC or stainless steel pipe in unconsolidated deposits and with steel casing in bedrock.
PID	An instrument that uses an ultraviolet light source to break down VOCs into ions that can be measured.
Piezometer	A well made from PVC or metal with a slotted screen installed across or within a saturated zone. Piezometers are primarily installed to monitor changes in the potentiometric surface elevation.
Potentiometric Surface	A surface representing the hydraulic head of groundwater.
Protective Casing	The pipe installed around the well riser that sticks up from the ground (above-grade completions) or is flush with the ground (at-grade completions, e.g., curb box) in order to protect the well integrity. Protective casings are typically constructed of steel or aluminum and usually closeable with a locking cover/hasp to maintain well integrity between sampling events.
Recharge Rate	The rate at which groundwater returns to the water column in the well.
Separate-Phase Product	A liquid that does not easily dissolve in water. Separate-phase product can be more dense (i.e., DNAPL) or less dense (i.e., LNAPL) than water and, therefore, can be found at different depths in the water column.
Static Water Level	Level at which water resides in a well when the water level is at equilibrium with atmospheric pressure.
Well Cover	The cap or lid constructed at the end of the protective casing (above-grade completions) or flush-mounted curb box (ground surface completions) to secure access to the well. Well covers for stick-up wells are often equipped with a hasp to accommodate a padlock. Well covers for flush-mounted road boxes or vaults are opened and closed using a threaded bolt.
Well Filter Pack	A material composed of clean silica sand or sand and gravel of selected grain size and gradation that is placed in the annulus between the screened interval and the borehole wall in a well for the purpose of retaining and stabilizing the formation material.

Well Plug/Expansion Plug	The plug fashioned into a cap placed into the top of the well riser (e.g., J-Plug). Well plugs are usually designed with an expandable gasket that is activated by turning a locking wing nut or removable key latch, closing a snap cap or engaging a magnetic clutch cap to seal the well riser.
Well Riser	Sections of blank (non-slotted) pipe that extend from the well screen to or above the ground surface.
Well Screen	Pipe (typically PVC or stainless steel) used to retain the formation or filter pack materials outside of the well. The pipe has openings/slots of a uniform width, orientation, and spacing. The openings/slots can vary based on formation and filter pack material specifications.

1.5 Health & Safety Considerations

TRC personnel will be on site when implementing this SOP. Therefore, TRC personnel shall follow the site-specific HASP. TRC personnel will use the appropriate level of PPE as defined in the HASP.

The well head should be pre-screened using a PID/FID to avoid inhalation of contaminants venting from the well. If monitoring results indicate sustained elevated concentrations of organic contaminants, the level of PPE may need to be increased in accordance with the HASP or work could be conducted upwind of the well.

When present, special care should be taken to avoid contact with LNAPL or DNAPL. The use of an air monitoring program, as well as the proper PPE designated by the site-specific HASP, can identify and/or mitigate potential health hazards.

Implementing this SOP may require the use of reagents and/or compressed gases for the calibration and operation of field equipment. These substances may be hazardous and TRC personnel must appropriately handle, store, and dispose of them at all times. Skin contact with liquid from preserved sample bottles must be avoided as they may contain strong acids or bases. When filling bottles pre-preserved with acid (e.g., hydrochloric acid, nitric acid, sulfuric acid), vapors may be released and should not be inhaled. Do not allow bottles with acid to be exposed to elevated atmospheric temperatures or sunlight as this will facilitate fumes from the acids.

1.6 Cautions and Potential Problems

The following sections highlight issues that may be encountered and should be discussed with the Project Manager prior to mobilization into the field. Special care should be taken when sampling for PFAS. Please refer to Attachment D for details.

1.6.1 Pre-Sampling Issues

(a) Selection of equipment for groundwater sampling should consider multiple factors, including: DTW, well specifications (e.g., depth and length of well screen intervals), desired flow rate, possible weather conditions, type and concentration of contaminant(s), and remoteness/accessibility to the site. The benefits and limits of each type of groundwater sampling equipment should be fully reviewed during project planning or prior to mobilization if the project-specific work plan does not identify the required equipment. For example, peristaltic pumps are incapable of withdrawing water in wells in which the depth to water is greater than approximately 20-25 feet below ground surface (bgs).

(b) If the screen or open borehole is greater than 10 feet in length, consult the project-specific work plans for the target sampling interval. Generally, pumps are either placed in the middle of the saturated zone if the water level is below the top of the screen or in the middle of the screen interval if the water level is above the top of the screen.

(c) The need for redevelopment of the monitoring wells should be evaluated periodically in accordance with the project-specific requirements. This is assessed by comparing the measured total depth of the well with the constructed depth. If the measured depth is less than the constructed depth, this may indicate siltation of the well and/or the presence of an obstruction in the well. If it is determined that redevelopment is necessary, it should be performed in accordance with ECR SOP 006, *Well Development.* The time necessary for a well to restabilize after redevelopment will be determined on a project-specific basis and may depend on regulatory requirements.

(d) During the total well depth measurement, there is the potential for sediment, if present at the bottom of the well, to be disturbed, thereby increasing the turbidity of the groundwater. Therefore, the total well depth measurement should be collected the day prior to collecting groundwater samples, if possible.

(e) Use caution if using compressed gas cylinders (e.g., nitrogen, carbon dioxide) for purging/sampling of groundwater. Check for leaks around regulator connections by spraying soapy water on the connections. If a leak is discovered, the connection to the regulator should be disassembled, wrapped with Teflon® tape, and reconnected to the cylinder. If the leak continues, the regulator should be replaced. It should be noted that Department of Transportation (DOT) regulations apply to the transportation and handling of compressed gas cylinders (see 49 Code of Federal Regulations [CFR] 171). Never transport cylinders with the regulator attached. Replace the cylinder valve cover on the compressed gas cylinder before transport.

(f) All field personnel must be made aware of the water level measurement reference point being used for each well at a site (i.e., must be clearly marked) in order to ensure collection of comparable data between events.

(g) Bolt cutters may be necessary to remove rusted locks. Dipping rusted locks in a soapy solution may help with opening difficult locks. Oils and other products containing VOCs (e.g., WD-40) should not be used on locks as these compounds may cause contamination of water samples collected at the well. Replace cut locks and note in the field book.

(h) Prior to accessing the well, physical conditions around the well head should be assessed for situations that might result in cross-contamination or the introduction of foreign material/debris into the well. For example, flush-mounted wells may have water or road sand/salt/debris inside the curb box. Rodents and insects (e.g., bees, wasps) have been known to construct nests within the protective casing of a well. If bees, wasps, or other insects are encountered, insecticides should be used with caution as the chemicals may cause contamination of water samples collected at the well. If water or foreign material is introduced into the well, the Project Manager should be immediately notified.

1.6.2 General Purging and Sampling Issues

(a) Prior to installation of a submersible pump into a well, ensure that the tubing is properly sealed to the pump to avoid losing the pump down the well and to prevent escape of air or water from the pump, which could result in poor pump performance and the aeration of the well water. Do not do this by tugging on tubing. Never lower pumps into the well using only tubing; instead a security line attached to the pump is required to prevent potentially losing the pump down the well.

(b) A submersible pump should not be lowered to the bottom of the well to avoid stirring up any sediment at the bottom of the well and prevent getting the pump stuck (fine sediment accumulation in the bottom of the well can create a strong suction with a flat bottom pump such as a bladder pump, which may require jetting to retrieve the pump).

(c) Start with the lowest pumping rate possible and increase until a sustainable rate is reached. Avoid high pumping rates (> 1 liter/min), as this could lead to damage of the well filter pack, if present. Where practical and/or possible, refer to previous sampling events to establish consistent flow rates.

(d) Some regulatory agencies may have concern about the use of peristaltic pumps when sampling for VOCs due to the potential for loss of VOCs during sampling and alteration of other water quality parameters such as pH and alkalinity. Samplers should review the requirements in the project-specific work plan and/or regulatory guidelines prior to performing the work. Explicit approval to use a peristaltic pump for the collection of VOCs may be required by the governing regulatory agency. An option may be to use the “soda straw” method to collect the VOC sample which does not allow the water to go through the pump head:

    (1) After purging the well with the peristaltic pump, collect all fractions <u>except VOCs</u> from the outlet side of the pump (i.e., VOCs will be collected last instead of first).
    (2) Turn the pump off.
    (3) Change into clean gloves.
    (4) Disconnect the tubing coming out of the well from the inlet side of the pump and <u>immediately</u> put a finger over the end of this tubing to prevent water from draining out of the tubing.
    (5) Retrieve tubing from the well, coiling it in one hand as it is being retrieved (maintain finger over end of tubing).
    (6) Open VOC vials. Briefly remove finger from end of tubing to allow water to flow into vial. Replace finger on end of tubing to stop flow. Do this for remaining VOC vials.

(e) In the event that a well cannot be purged and sampled with a pump, the alternative to pumping may be the use of a bottom-filling bailer. The applicable regulatory agency requirements and the Project Manager should be consulted if in doubt about the appropriateness of using a bailer at a site or during a particular sampling event.

(f) During purging and sampling, the tubing should remain filled with water to minimize possible changes in water chemistry due to contact with the atmosphere. All flow-through cells should be shaded from direct sunlight to minimize the potential for off-gassing and temperature fluctuations.

(g) Ensure monitoring instruments (i.e., multi-parameter water quality instrument, turbidity meter, water level measuring device) are maintained in good condition and properly calibrated to ensure accurate readings. Be sure to have appropriate-sized extra batteries on hand.

(h) Adverse weather conditions may present challenges that need to be dealt with on a case-by-case basis. For example, air temperatures below 32°F may cause ice formation in the tubing, flow-through cell, and on the sampling equipment, or heavy rain could cause standing water issues with flush-mounted wells. Heavy rain can also impact electronic sampling equipment; preventative measures should be taken to keep electronic equipment dry.

(i) Observe and avoid any uncontrolled ambient/surrounding air conditions that could affect analytical results (e.g., truck/vehicle exhaust nearby, industrial building vents). Always ensure that vehicles are turned off during sampling to avoid introducing vehicle exhaust into the sample. If uncontrolled ambient/surrounding air conditions cannot be avoided, contact the Project Manager for further instruction; collection of a field blank sample may be warranted in this situation.

(j) Procedures should be established to minimize potential cross-contamination. For example:

- Wrap monitoring and sampling equipment with protective material (e.g., aluminum foil, polyethylene sheeting, Ziploc® bags) after decontamination and between sampling locations to minimize the potential for cross-contamination between well purging events at different locations.
- Use dedicated or disposable sampling equipment or new tubing at each sampling point when appropriate to minimize the need for decontamination.
- Protect sampling equipment and/or the open well head from blowing soil and dust by covering with plastic sheeting as needed.
- If a bailer and rope are used to purge and/or sample the well, then there is the possibility of contamination from the rope used to lower the bailer. New or dedicated rope should be used when appropriate. Alternatively, a decontaminated, Teflon®-coated stainless steel leader can be attached between the rope and the bailer. The leader acts as an extension to the rope and allows for the top of the bailer to enter the water column without immediately placing the rope into the water. It is important to keep the rope clean and not allow contact with the ground surface during bailing.

(k) Disposal of the groundwater collected during purging must be performed in accordance with all applicable regulations and the project-specific work plan.

(l) Clear tape should not be used to cover labels on containers used for certain analyses (e.g., 40-mL vials for VOC analysis) due to potential interference with analytical equipment.

(m) In cases where it is difficult to obtain sufficient sample volume for multiple analytical fractions as well as required quality control (QC) analyses (*e.g.*, field duplicates, matrix spike/matrix spike duplicate [MS/MSD] analyses), discuss this situation with the Project Manager and laboratory prior to sample collection. Laboratories can often "make do" with less volume, especially for inorganic parameters, or increase the reporting limit proportional to the sample volume obtained.

1.7 Personnel Qualifications

Since this SOP will be implemented at sites or in work areas that entail potential exposure to toxic chemicals or hazardous environments, all TRC personnel must be adequately trained. Project- and client-specific training requirements for samplers and other personnel on site should be developed in project planning documents, such as the sampling plan or project-specific work plan. These requirements may include:

- OSHA 40-hour Health and Safety Training for Hazardous Waste Operations and Emergency Response (HAZWOPER) workers
- 8-hour annual HAZWOPER refresher training.

2.0 PROCEDURES

Procedures for collecting groundwater samples from monitoring wells are described below. The project-specific work plan should also be consulted for specific details regarding sampling.

When possible, sampling should always begin at the monitoring well with the least contaminated groundwater and systematically proceed to the well with the most contaminated groundwater, including sites which may have nested wells.

2.1 Pre-sampling Activities

(a) It should be determined if there is the requirement to determine static water level measurements on <u>all</u> wells at the site prior to sampling, regardless if the well is being sampled.

(b) Prior to field activities, review historical groundwater sampling logs (if available) to maintain consistency for the current sampling event (e.g., equipment type, pump intake depth setting, flow rate, etc.)

(c) Organize monitoring, purging, and sampling equipment taking care not to allow cross-contamination. This can be accomplished by laying new polyethylene sheeting near the well or using new buckets, etc.

(d) Calibrate (or perform a calibration check on) all field monitoring equipment on the same day before collecting groundwater samples. Refer to TRC SOPs and manufacturer's equipment calibration instructions. A calibration check may also be required during or at the end of each sampling day. Consult the project-specific work plan.

(e) Unlock the well cover on the well.

(f) Record the sample location, time, and date in the field book and/or on the Groundwater Field Data Record.

(g) On the Groundwater Field Data Record, note the physical condition of the well, including damage, deterioration, and signs of tampering, if any. Collect photographic documentation of serious damage to present to the Project Manager.

(h) Open the well cap and expansion plug, and stay upwind of and not directly over the well. Note any unusual odors, sounds, or difficulties in opening the well and, if required, measure the organic vapor reading at the rim of the well with a suitable organic vapor screening device (e.g., PID or FID), and record the reading in the field book and/or on the Groundwater Field Data Record. If pressure or vacuum is noted or suspected in the well, allow sufficient time for the water level elevation in the well to equilibrate.

(i) Gently lower a clean, decontaminated water level measuring device into the well to determine the static water level. If appropriate for site conditions, check for the presence of LNAPL or DNAPL using an oil/water interface probe (refer to ECR SOP 004, *Water Level and Product Measurements*). If LNAPL or DNAPL is detected, contact the Project Manager before proceeding with purging and sampling activities. Record the information on depth to groundwater to the nearest 0.01 feet, depth to LNAPL or DNAPL, and/or thickness of NAPL in the field book and/or the Groundwater Field Data Record. Refer to ECR SOP 004, *Water Level and Product Measurements*, for proper procedures in performing these measurements.

(j) If required in the project-specific work plan, measure the depth to the bottom of the well to assist in calculating the well volume of the well. If possible, avoid making total well depth measurements on the same day as sampling due to the tendency to disturb sediment during this measurement. If NAPL is suspected, use a decontaminated oil/water interface probe. If the measured depth is less than the constructed depth, this may indicate that the well needs to be redeveloped (see ECR SOP 006, *Well Development*). Consult the project-specific work plan or Project Manager for further instructions.

2.2 Groundwater Purging Activities

Purging is conducted to ensure that representative groundwater is obtained from the water-bearing unit for analysis. The multiple-volume or low-flow purging approach may be used to remove water from the well and monitor the water in order to determine when a well has been adequately purged (i.e., stabilized); at a minimum, the pH, specific conductance and temperature of the groundwater removed during purging should be monitored and recorded in the field notes. Other parameters may be required in some regulatory jurisdictions (e.g., turbidity). Additionally, the purge volume should be monitored and recorded. In some instances, such as when monitoring at solid waste disposal facilities, simply removing an adequate volume of water (e.g., three well volumes) may be suitable for adequate purging, and sampling can commence. Check with the project-specific work plan and appropriate regulatory guidance to determine any specific purging requirements.

If the well has been previously sampled consistent with this SOP, then the prior purging strategy (e.g., method, pump intake depth and the flow rates) should be followed during subsequent sampling events to maintain consistency and minimize potential variability due to the sampling procedure.

2.2.1 Multiple-Volume Purging Approach

The multiple-volume purging approach is typically performed using bailers or submersible or peristaltic pumps. In the multiple-volume purging approach, there are two measurements used to determine adequate purge volume removal prior to sample collection: 1) purge volume and 2) field parameter stabilization. The field parameters should be recorded at regular volumetric intervals. There are no set criteria for establishing how many total sets of measurements are adequate to document stability of parameters. If the calculated purge volume is small, the measurements should be taken frequently enough (e.g., every 3 to 5 minutes) to provide a sufficient number of measurements to evaluate stability. If the purge volume is large, measurements taken every 15 minutes may be sufficient.

Purge Volume

Prior to purging a well, the amount of water inside the well riser and well screen (i.e., water column) should be determined, if possible. To do this, the diameter of the well should be determined and the water level and total depth of the well should be measured and recorded. The specific methodology for obtaining these measurements is included in ECR SOP 004 *Water Level and Product Measurements.*

Once this information is known, the well volume can be calculated using Equation 1:

Well Volume (V) = πr^2 h (cf) **Equation 1**

where:
π = pi (3.14)
r = radius of well in feet (ft)
h = height of the water column in ft. [This may be determined by subtracting the depth to water from the total depth of the well as measured from the same reference point.]
cf = conversion factor in gallons per cubic foot (gal/ft^3) = 7.48 gal/ft^3.

The volume in gallons/linear foot (gal/ft) and liters/linear foot (L/ft) for common-size wells are as follows:

Well Inside Diameter (inches)	Volume (gal/ft)	Volume (L/ft)
1	0.0408	0.1529
2	0.1631	0.6174
3	0.3670	1.3892
4	0.6524	2.4696
6	1.4680	5.5570

If the volumes for the common-size wells above are utilized, Equation 1 is modified as follows:

Well volume = (h)(f) **Equation 2**

where:
h = height of water column (feet)

f = the volume in gal/ft or L/ft

For volumetric purging, an adequate purge is typically achieved when 3 to 5 well volumes have been removed. The field notes should reflect the single-well volume calculations or determinations according to one of the above methods and a reference to the appropriate multiplication of that volume, (i.e., a minimum of 3 well volumes) clearly identified as a purge volume goal.

For volumetric purging, it is suggested that field readings are collected every ½ well/well screen volume after an initial 1 to ½ well volumes are purged. The volume removed between readings can be adjusted as well-specific information is developed.

If removing a specified volume of water (e.g., 3 well volumes) has been determined to be suitable for purging, sampling can commence immediately upon achieving the required purge volume. In other cases, where specified in the project-specific work plan, stabilization of field parameters must be documented prior to sample collection. If, after 3 well volumes have been removed, the field parameters have not stabilized (see discussion in Section 2.2.3), additional well volumes (up to a total of 5 well volumes), should be removed. If the parameters have not stabilized within five well volumes, it is at the discretion of the Project Manager whether or not to collect a sample or to continue purging. If, after 5 well volumes, pH and conductivity have stabilized and the turbidity is still decreasing and approaching an acceptable level, additional purging should be considered to obtain the best sample possible with respect to turbidity. The conditions of sampling should be noted in the field book.

2.2.2 Low-flow Purging Approach

The low-flow purging approach is typically performed using peristaltic pumps or submersible pumps. Low-flow purging (also referred to as low-stress purging, low-volume purging, or Micropurging®) is a method of well purging/sampling that minimizes the volume of water withdrawn from a well in obtaining a representative sample. The term low-flow refers to the low velocity with which water enters the pump intake during purging and sampling. The objective is to draw representative saturated zone water through the well screen to the pump intake while avoiding disturbance of the stagnant water above the well screen through minimizing drawdown of the water column in the well. To achieve this, the flow rate should be adjusted to less than 1 L/min (usually, this will be a rate less than 500 ml/min and may be as low as 100 ml/min). Once drawdown stabilizes, the sampled water is isolated from the stagnant water in the well casing, thus eliminating the need for its removal. This sampling method is based on the principle that water within the screened zone passes through continuously and does not mix with water above the screen. Water entering the pump can be considered representative of water in the formation after drawdown and indicator parameters have stabilized.

When performing low-flow purging and sampling, it is recommended that the pump intake be set in the center of the well screen interval (or center of the water column within the well screen if the water level is below the top of the well screen) to help prevent disturbance of any sediment at the bottom of the well. If known, the pump can be placed adjacent to the areas with the highest hydraulic conductivity or highest level of contaminants. Dedicated pumps can be utilized to minimize disturbance of the water column. Subsequent sampling events should duplicate as closely as possible the pump intake depth and the stabilized flow rate from the previous events.

To begin purging, the pump should be started at the lowest pressure/power flow rate setting (e.g., 100 mL/min) and then slowly increased until water begins discharging. Monitor the water level and slowly adjust the pump speed until there is little or no drawdown or drawdown has stabilized. The pump pressure/power may need to be increased for discharge to occur.

The stabilization of drawdown should be documented. Measure and record the flow rate and water level every 3 to 5 minutes during purging. The flow rate should be reduced if drawdown is greater than 0.3 feet over three consecutive 3 to 5 minute interval readings. Note any flow rate adjustments on the Groundwater Field Data Record. Once an appropriate purge rate has been achieved, record this information, continue purging until water quality indicator parameters have stabilized (see Section 2.2.3), and then sample the well.

Attempts should be made to avoid pumping a well dry. If drawdown cannot be maintained at less than 0.3 feet and the falling water level is approaching the top of the screened interval (or the top of the pump for sampling that began with the water level below the top of the screen), perform the following steps:

1. Reduce the flow rate, or turn the pump off and allow for recovery. (The pump must have a check valve to prevent backflow if it is shut off).
2. Begin pumping again at a lower flow rate.
3. If water draws down to the top of the screened interval again (or the top of the pump for sampling that began with the water level below the top of the screen), turn the pump off and allow for recovery.
4. If two tubing volumes (including volume of water in the pump and flow-through cell) have been removed during purging, sampling can proceed the next time the pump is turned on without waiting for indicator field parameters to stabilize. The project-specific work plan or Project Manager should be consulted for guidance.
5. If this procedure is used, this should be recorded in the field book and/or on the Groundwater Field Data Record.

2.2.3 Field Parameter Stabilization During Purging

Stabilization criteria may depend on project objectives or regulatory-specific requirements. Refer to Appendix A for some of the regulatory-specific requirements for field parameter stabilization. Generally, an adequate purge with respect to the ground water chemistry is achieved when, stability for at least three consecutive measurements is as follows:

- pH ± 0.1 standard unit (SU)
- specific conductance within 3%
- turbidity within 10% for values greater than 5 nephelometric turbidity units (NTUs). If three turbidity readings are less than 5 NTUs, the values are considered as stabilized

Other parameters, such as DO, may also be used as a stabilization parameter. Typical stabilization goals for DO are within 0.2 mg/L or 10% saturation, whichever is greater. DO measurements should be conducted using either a flow-through cell or an over-topping cell to minimize or reduce potential oxygenation of the sample.

Because groundwater temperature is generally not very sensitive in distinguishing between stagnant casing water and formation water and is subject to rapid changes during purging, its usefulness is subject to question for the purpose of determining parameter stability. Even if temperature is not used to determine stability during well purging, it is still advisable to record the sample temperature, along with the other groundwater chemistry parameters, during well purging, as it may be needed to interpret other parameter results.

ORP is not always used as a stabilization parameter since it may also be subject to rapid changes during the purging process; however, it may be measured and recorded during well purging.

2.2.4 Special Considerations During Purging

Wells Purged Dry/Purge Adequacy

For wells with slow groundwater recovery, attempts should be made to avoid purging the well dry. This may be accomplished by slowing the purge rate. As water enters a well that has been purged dry, the water may cascade down the sand pack and/or the well screen, potentially stripping VOCs that may be present and/or potentially mobilizing soil fines into the re-accumulating water column.

However, even with slower purge rates, in some situations, a well may be pumped or bailed dry (evacuated) during the purging process. In these situations, evacuation generally constitutes an adequate purge and the well may be sampled following sufficient recovery (enough volume to allow filling of all sample containers). **It is not necessary that the well be evacuated three times before it is sampled.** Purging parameters should be measured and recorded during sample collection to serve as the measurements of record for the sampling event.

It is particularly important that wells be sampled as soon as possible after purging to maintain sample representativeness. If adequate volume is available upon completion of purging, the well should be sampled immediately. If not, sampling should occur as soon as adequate volume has recovered. If possible, sampling of wells that have a slow recovery should be scheduled so that they can be purged and sampled in the same day after adequate volume has recovered. Wells of this type should, unless it is unavoidable, not be purged at the end of one day and sampled the following day.

Temporary Monitoring Wells

Procedures used to purge temporary groundwater monitoring wells may differ from permanent wells, because temporary wells are installed with different DQOs for immediate sample acquisition. Wells of this type may include standard well screens and risers placed in boreholes created by hand augering, power augering, or by drilling. Alternatively, they may consist of a rigid rod and screen that is pushed, driven, or hammered into place to the desired sampling interval, such as a direct push Wellpoint®, a Geoprobe® Screen Point 15/16 sampler, or a Hydropunch® sampler.

Purging to address stagnant water may not necessarily apply to temporary wells, because stagnant water is not typically present. It is important to note, however, that the longer a temporary well is in place and not sampled, the more stagnant the water column may become, and the more appropriate it may be to apply, to the extent possible, standard permanent monitoring well purging criteria.

In cases where the temporary well is to be sampled immediately after installation, purging is conducted primarily to mitigate the impacts of installation. In most cases, temporary well installation procedures disturb the existing saturated conditions, resulting primarily in increased turbidity. Therefore, the goal of purging, if conducted, may be to reduce the turbidity and remove the volume of water in the area directly impacted by the installation procedure. Low turbidity conditions in these types of wells that are completed within the limit of suction are typically and routinely achieved by the use of low-flow/low-stress purging techniques using variable-speed peristaltic pumps.

2.2.5 Equipment Considerations for Purging

Monitoring well purging is accomplished by using in-place plumbing and dedicated pumps or by using portable pumps/equipment when dedicated systems are not present. The pump of choice is usually a function of the purging approach (e.g., multiple-volume vs. low-flow), well diameter, the DTW, the total depth of the well, the amount of water that is to be removed during purging, the specific analytical testing program for the well, and the equipment previously used during purging and sampling of the well. A peristaltic pump is appropriate for purging whenever the head difference between the sampling location and the water level is less than the limit of suction (approximately 25’ to 30’) and the volume to be removed is reasonably small. For wells where the water level is below the limit of suction, and/or where there is a large volume of water to be purged, the variable-speed electric submersible pump or adjustable-rate bladder pumps would be appropriate. Bailers may also be used for purging in appropriate situations (e.g., shallow wells with small purge volumes); bailers are not suitable for low-flow purging.

The following subsections describe well evacuation devices that are most commonly used. Other devices are available but are not discussed in this SOP due to their limited use. Site-specific operating procedures should be developed in the case that an uncommon purge device is used.

2.2.5.1 Purging with a Suction Pump

There are many different types of suction pumps. They commonly include: centrifugal, peristaltic and diaphragm. Diaphragm pumps can be used for well evacuation at a fast pumping rate and sampling at a low pumping rate. The peristaltic pump is a low-volume pump that incorporates a roller to squeeze flexible tubing, thereby creating suction. This tubing can be dedicated to a well for re-use or discarded. It is recommended that 1/4 inch or 3/8 inch (inner diameter) tubing be used to help ensure that the sample tubing remains filled with water and to prevent water from being aerated as it flows through the tubing. Purging procedures are as follows.

(a) Determine the volume of water to be purged as described in Section 2.2.1 or follow the low-flow approach described in Section 2.2.2 (applicable to peristaltic pumps only).
(b) Take necessary precautions (e.g., laying plastic sheeting around the well) to prevent contamination of pumps, tubing or other purging/sampling equipment with foreign materials.
(c) Assemble the pump, tubing and power source, if necessary, in accordance with manufacturer’s specifications.
(d) Ensure that the pump tubing is set at the pre-determined pump intake depth.
(e) Connect the discharge line from the pump to the flow-through cell for parameter measurements. Use a T-connection or valve prior to the flow-through cell to allow for

collection of water for turbidity measurements. Direct the discharge line from the flow-through cell to a 5-gallon bucket (or equivalent) to contain the purge water for proper disposal. Verify the end of the tubing is not submerged in the purge bucket. Manage purge water as specified in the project-specific work plan.

(f) Do not allow the pump to run dry. If the pumping rate exceeds the well recharge rate, adjust the rate accordingly or, if consistent with the purging and sampling objectives, lower the tubing further into the well and continue pumping.

(g) Using the water quality meter, take an initial reading of the required indicator parameters. All measurements, except turbidity, must be obtained using a transparent flow-through cell unless an unforeseen situation makes this impractical or inadvisable. Initially, turbidity may be elevated. Once turbidity has decreased to a measurable range, begin monitoring indicator parameters at approximately every 3-5 minutes, or as appropriate. Please note that flow-through cell size should be taken into account in conjunction with the flow rate to determine the length of time between water quality parameter readings. At least one flow-through cell volume should be turned over between readings. For example, if the flow through cell size is 500 mL and the flow rate is 100 mL/min, then it would be appropriate to measure water quality parameters every 5 minutes.

(h) Record the readings on the Groundwater Field Data Record. The monitoring probes must be submerged in water at all times. Record the indicator parameters, along with the water level, as described in Step (g) above. If removing a specified volume of water (e.g., 3-5 well volumes) has been determined to be suitable for purging, sampling can commence immediately upon achieving the required purge volume. In other cases, where specified in the project-specific work plan, stabilization of field parameters must be documented prior to sample collection. Stabilization criteria are discussed in Section 2.2.3.

Particulate build-up in the flow-through cell may impact indicator parameters. If the cell must be cleaned during pumping operations, continue pumping and disconnect the cell for cleaning, then reconnect and continue monitoring. Record the start and stop times, and describe the cleaning steps in the field book.

If indicator parameter stabilization is required and parameters have not stabilized after 2-hours of purging (or other pre-determined length of time), one of three options may be taken after consultation with the Project Manager:

1) continue purging until stabilization is achieved;
2) discontinue purging, do not collect any samples, and record in the field book and/or on the Groundwater Field Data Record the stabilization conditions and steps taken to attempt to achieve stabilization; or,
3) discontinue purging, collect samples and document attempts to achieve stabilization.

NOTE: If parameters do not stabilize, or turbidity remains greater than 5 NTU within the project-determined time range (EPA recommends up to 2 hours), contact the Project Manager to develop a modified sampling approach.

(i) Record the volume of water purged on the Groundwater Field Data Record. Record the disposal method used for purge water in the field book.

(j) Once the required volume of water is removed (typically 3 to 5 well volumes) from the well and/or parameters are stabilized to the satisfaction of the project-specific work plan, proceed to Section 2.3, Post-purging Groundwater Sample Collection.

2.2.5.2 Purging with a Submersible Pump

Submersible pumps generally use one of two types of power supplies, either electric or compressed gas. Electric pumps can be powered by a 12-volt DC rechargeable battery, or a 110- or 220-volt AC power supply. Those units powered by compressed gas (e.g., bladder pump) normally use a small electric controller that also needs a 12-volt DC battery or 110-volt AC power. They may also utilize compressed gas from bottles. Pumps differ according to the depth and diameter of the monitoring wells and the height of the potentiometric surface/water table (e.g., pressure head). It is recommended that 1/4-inch or 3/8-inch (inner diameter) tubing be used to help ensure that the sample tubing remains filled with water and to prevent water from being aerated as it flows through the tubing. Purging procedures are as follows.

(a) Determine the volume of water to be purged as described in Section 2.2.1 or follow the low-flow approach described in Section 2.2.2.

(b) Take necessary precautions (e.g., laying plastic sheeting around the well) to prevent contamination of pumps, tubing or other purging/sampling equipment with foreign materials.

(c) Assemble the pump, tubing and power source, if necessary, in accordance with manufacturer’s specifications. If the pump itself is being lowered into the well, ensure a safety line is attached.

(d) Non-dedicated purge/sampling vs. dedicated purge/sampling systems.

Dedicated systems: Pump has already been installed. Refer to historical monitoring well information, and record the depth of the pump intake in the field book and/or on the Groundwater Field Data Record.

Non-dedicated systems: Determine the target depth of the pump intake. Note that this may be a historical intake depth; see well construction data or the project-specific work plan. If there is not an established intake depth, the center of the screened interval should be targeted. If the measured water level is lower than the top of the well screen, position the pump intake at the midpoint of the water column. The intake should be generally 1 to 2 feet above the bottom of the well to minimize potential mobilization of any settled sediment, the risk of the pumping suction being broken, or the entrainment of air in the pump tubing and resulting sample. Slowly lower the pump, safety line, and tubing into the well to the pre-determined pump intake depth. The tubing should be cut to the desired length to assist in installing the pump. Measure the depth of the pump intake while lowering the tubing/pump into location. Record the pump intake depth in the field book and/or on the Groundwater Field Data Record. For deeper wells and large diameter wells, two staff members may be necessary to accomplish this task.

(e) Connect the discharge line from the pump to the flow-through cell for parameter measurements. Use a T-connection or valve prior to the flow-through cell to allow for collection of water for turbidity measurements. Direct the discharge line from the flow-through cell to a 5-gallon bucket (or equivalent) to contain the purge water for proper disposal. Verify the end of the tubing is not submerged in the purge bucket. Manage purge water as specified in the project-specific work plan.

(f) Measure the flow rate of the pump with a graduated container and stop watch. The pump pressure may need to be increased for discharge to occur. Record the volume of water collected for a period of 1 minute and calculate the flow rate as follows.

$$Flowrate\,(mL\,/\,min) = \frac{volume\ collected\ (mL)}{1\,minute}$$

(g) Measure the water level and record the flow rate and the water level. This should be performed every 3 to 5 minutes during purging. For low-flow purging, the flow rate should be adjusted to result in a rate between 100 to 500 mL/min; however, if drawdown of the well is observed, a slower flow rate may be necessary. If using a bladder pump, it is recommended that the pump be set to deliver long pulses of water so that one pulse will fill a 40 mL volatile organic analysis (VOA) vial, if possible.

(h) Prior to recording the water quality indicator parameters, a minimum of one tubing volume should be purged. Note that this includes the volume of the flow-through cell.

(i) Proceed to steps (g) through (j) in Section 2.2.5.1.

2.2.5.3 Purging with a Bailer

(a) Determine the volume of water to be purged as described in Section 2.2.1.

(b) Take necessary precautions (e.g., laying plastic sheeting around the well) to prevent contamination of tubing or other purging/sampling equipment with foreign materials.

(c) Use a well-dedicated bailer (i.e., used exclusively for that well only), a decontaminated bailer or an unused, disposable bailer.

(d) Attach an appropriate length of (a) bailing line, (b) Teflon®-coated bailing wire or (c) rope with Teflon®-coated stainless steel leader to reach the bottom of the well. Secure a knot or series of knots to the top of the bailer. Be sure to have additional length of line to facilitate handling of the bailer at the surface (typically 10 ft).

(e) Lower the bailer gently into the well until it reaches the water column and fills with water from the bottom. Note: It is recommended that the bailer be lowered into the water to a depth that prevents the water from entering the top of the bailer. This is done to prevent excess turbulence caused by filling from the bottom and the top simultaneously. Controlling the line attached to the bailer as it is lowered into the well is also important to prevent degassing of the water as the bailer impacts the water. In shallow wells, controlling the line is not too difficult; however, for wells of greater depths it is common to utilize a hand-over-hand (windmill) approach using both hands to control longer lengths of line and prevent the loops in the line from tangling with one another. This procedure is simple to learn and saves a good deal of time by preventing tangles. Do not allow the bailing line or rope to become contaminated by surface soil.

(f) Once the bailer is full of water, gently withdraw the bailer from the well until it comes out of the top of the well. Be sure to control excess line in your hands to prevent the rope and bailer from touching the ground, and then grasp the bailer as it appears at the top of the well.

(g) Immediately pour the water into a vessel for water quality measurements, and record the measurements in the field book or on the Groundwater Field Data Record (at the project-required frequency). Otherwise, pour water into a 5-gallon bucket or other vessel to track the volume purged. As a general rule, standard 2-inch bailers are able to hold about 1 liter of water when full. This process will have to be repeated several times to complete adequate purging of the well (e.g., three to five well volumes).

(h) Record the volume of water purged on the Groundwater Field Data Record. Record the disposal method used for purge water in the field book.

(i) Once the required volume of water is removed (typically 3 to 5 well volumes) from the well and/or parameters are stabilized to the satisfaction of the project-specific work plan, proceed to Section 2.3, Post-purging Groundwater Sample Collection.

2.3 Post-purging Groundwater Sample Collection

(a) New, disposable gloves should be donned immediately prior to sample collection and should be changed at any point that their cleanliness becomes compromised during sample collection.

(b) If using a submersible or peristaltic pump, maintain the same flow rate as used during purging. Disconnect the pump tubing from the flow-through cell or sample from the T-connector, if used. Samples must be collected directly from the discharge port of the pump tubing prior to passing through the flow-through cell. This is critically important to avoid cross-contamination between wells.

(c) If using bottom-filling bailers,

- Slowly lower the bailer into the well until it is submerged to the point where water does not enter the top (i.e., bottom-filling).
- Retrieve the bailer. The first bailer recovered after well purging must be used for sample collection.

2.3.1 Sample Collection Order

Fractions of the groundwater sample should be collected in the following order (i.e., decreasing volatility) unless otherwise specified in the project-specific work plan:

1. VOCs;
2. Semivolatile organic compounds (SVOCs);
3. Other organic parameters;
4. Unfiltered inorganic constituents (e.g., total metals);
5. Filtered inorganic constituents (e.g., dissolved metals); and
6. Other constituents.

During sample collection, allow the water to flow directly down the side of the sample container without allowing the tubing to touch the inside of the sample container or lid in order to minimize aeration and turbulence and maintain sample integrity. The tubing should remain filled with water.

2.3.2 VOC Sample Collection

<u>Collection of VOCs/Volatile Petroleum Hydrocarbons (VPH):</u> Samples for VOCs will be collected first unless they are being collected by the “straw” method described in Section 1.6.2 (d), and the sample vial must be filled so a meniscus forms over the mouth of the vial. This ensures no air bubbles or headspace will be formed after it has been capped. Ensure the lack of air bubbles and headspace by turning the vial upside down and tapping it lightly. If any bubbles are observed, the vial should be topped off using a minimal amount of sample to re-establish the

meniscus. Care should be taken to not flush any preservative out of the vial when topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected. Note: Extra VOC vials should be obtained prior to the sampling event in case this situation occurs.

Note: When using a bladder pump, it is recommended that the pump be set to deliver long pulses of water so that one pulse will fill a 40 ml VOA vial, if possible.

When acid preservation is used for the collection of VOCs, the acid must be added to the vials before sample collection. However, in most cases 40-ml VOA vials come pre-preserved. If a pre-preserved vial effervesces upon the addition of sample, the acid preservative can be rinsed out of the vial with sample water and then used to collect the sample. The laboratory should be made aware that the affected sample will not be acid-preserved as this may affect the sample holding time. Note effervescence in the field book for future reference.

2.3.3 Non-VOC Sample Collection

Completely fill the remaining sample containers for all non-VOC analyses.

Preserve the non-VOC samples in accordance with method and project-specific requirements following sample collection if the sample containers are not pre-preserved. (**NOTE:** Pre-preserved vials may be supplied by the laboratory, depending on the program).

2.3.4 Field Filtering

Depending upon project requirements, field filtering may be performed for non-VOC analyses. An in-line filter should be fitted at the end of the discharge tubing and the sample should be collected after the filter. Pre-rinse the in-line filter by allowing a minimum of 0.5 to 1 liter of groundwater from the well to pass through the filter prior to sampling. Ensure the filter is free of air bubbles prior to collecting samples. Preserve the filtered water sample immediately or directly fill pre-preserved containers (if provided). Clearly note “filtered” or “dissolved” on sample label and COC document.

2.4 Groundwater Sample Collection Without Purging (Passive Sampling)

Passive sampling can be defined as the free flow of contaminants from the media being sampled to a receiving phase in a sampling device. Depending upon the sampler, the receiving phase can be a solvent (e.g., water), chemical reagent, or porous adsorbent (e.g., activated carbon). While there are many different types of passive samplers, most have a barrier between the medium being sampled and the receiving phase. The barrier determines the sampling rate that contaminants are collected at a given concentration and can be used to selectively permit or restrict various classes of chemicals from entering the receiving phase.

There are three generic forms of passive (no purge) samplers: thief (grab) samplers, diffusion (equilibrium) samplers, and integrating (kinetic) samplers. However, this SOP focuses on the more commonly used diffusion (equilibrium) samplers.

Passive samplers are deployed down a well to the desired depth within the screened interval or open borehole to obtain a discrete sample without using pumping or a purging technique. Most samplers are able to be stacked to obtain samples at multiple depths. Some samplers can also be used to measure contaminants in groundwater as it enters a surface water body.

Diffusion, or equilibrium, samplers are devices that rely on diffusion of the analytes to reach equilibrium between the sampler fluid and the well water. Samples are time-weighted toward conditions at the sampling point during the latter portion of the deployment period. The degree of weighting depends on analyte and device-specific diffusion rates. Typically, conditions during only the last few days of sampler deployment are represented. Depending upon the contaminant of concern, equilibration times range from a few days to several weeks. Diffusion samplers are less versatile than grab samplers as they are not generally effective for all chemical classes.

Both the diffusion and integrating samplers depend upon permeation or diffusion through barriers that hold the receiving phase. This diffusion process is chemical and barrier specific. Diffusion samplers are commonly known as PDBs or rigid porous polyethylene (RPP) samplers. PDBs may be used to sample for VOCs, and RPPs may be used to sample for various organic and inorganic constituents. PDBs must be allowed to remain in the well for a sufficient period of time to allow the deionized water in the sampler to come into equilibrium with the constituents in the ambient groundwater.

Some regulatory agencies allow groundwater samples to be collected without purging the well. This may be accomplished by suspending a passive sampler in the well for a period of time appropriate for the type of passive sampler being used. It is important to confirm that the chosen sampler is compatible with the contaminants of concern including all VOCs of interest at the site.

Diffusion passive samplers are used most commonly and the procedure for their use is as follows:

(a) Passive samplers are deployed at a predetermined depth across the well screen. Typically, the initial sampling event may deploy multiple passive samplers across 5-foot intervals of saturated well screen to observe any potential stratification. Long-term sampling depths typically target a zone of higher concentration, if present.

(b) New passive samplers are attached via PVC cable ties to a tether (a pre-made marine-grade polyethylene rope or stainless steel cable with a weight at the bottom) that is then suspended within the well. There should be sufficient well screen saturation within the well to completely cover the passive sampler. For VOCs, it is recommended that there should be several feet of groundwater above the top of the PDB.

(c) The passive sampler should be allowed to equilibrate with groundwater for an appropriate period of time (e.g., at least 2 weeks for PDB samplers). Longer equilibration times may be necessary in lower permeability formations. Once sufficient time for equilibration has passed, the PDB samplers can be retrieved when convenient.

(d) Raise the passive sampler to the surface using a tether reel. Examine the surface of the passive sampler for evidence of algae, iron, or other coatings, and for tears to the membrane. Note observations in the field book. If tears are present and water is leaking out, the sample is not considered viable. Contact the Project Manager.

(e) Detach the passive sampler from the tether.

(f) Remove excess beaded water from the passive sampler with a clean gloved hand, running top to bottom; this is to minimize the contact of beaded water with water in the passive sampler.

(g) Use a small diameter discharge tube (<0.15 inch diameter to reduce volatilization) and pierce near the bottom, allowing water to smoothly flow into the VOA vial. Tilting the passive sampler will control the flow rate. The VOA vials must be filled within the first several minutes of passive sampler retrieval. (Note that sample vials should be prepared and opened on a stable surface or holding device such as a foam pack. Decanting sample from passive samplers into containers requires techniques that may require some practice and patience.) Refer to Section 2.3.2 for special circumstances regarding the filling of VOA vials.

(h) A small amount of water may remain within the passive sampler after filling the VOA vials and can be used for field parameter measurements if required.

(i) Dispose of the passive sampler after use.

2.5 Post-sampling Activities

(a) Cease pumping and, if system is non-dedicated, disassemble and decontaminate the purging and sampling equipment. Verify the end of the tubing is not submerged in the purge bucket prior to turning off the pump.

(b) Dispose of the bailer (if disposable) and/or rope and/or other disposable equipment in accordance with the project-specific work plan, or store the bailer in a plastic bag for transport to the site decontamination area.

(c) Dispose of the empty passive sampler and/or rope and/or other disposable equipment in accordance with the project-specific work plan, or store the empty passive sampler in a plastic bag for transport to the site decontamination area

(d) Replace the well cap and well cover on the well and lock the outer casing (if present).

(e) Label each sample. If the labels are covered with clear tape, ensure this is not performed for VOA vials.

(f) Place all samples in a cooler with ice.

(g) Ensure samples are delivered to the laboratory well before the required holding time expires.

(h) Consult the project-specific work plan to determine if a calibration check is required at the end of the day for the water quality parameters.

3.0 INVESTIGATION-DERIVED WASTE DISPOSAL

Field personnel should discuss specific documentation and containerization requirements for investigation-derived waste disposal with the Project Manager.

Each project must consider investigation-derived waste disposal methods and have a plan in place prior to performing the field work. Provisions must be in place as to what will be done with investigation-derived waste. If investigation-derived waste cannot be returned to the site, consider material containment, such as a composite drum, proper labeling, on-site storage by the client, testing for disposal approval of the materials, and ultimately the pickup and disposal of the materials by appropriately licensed vendors.

4.0 QUALITY ASSURANCE/QUALITY CONTROL

The collection of QC samples is dependent upon the DQOs. Project-specific work plans should be consulted to determine the required frequency of QC sample collection.

4.1 Field Duplicates

The following procedures should be used for collecting field duplicates of groundwater samples:

(a) For QC purposes, each duplicate sample will be typically submitted to the laboratory as a "blind" duplicate sample, in that a unique sample identification not tied to the primary sample identification will be assigned to the duplicate (e.g., DUP-01). Standard labeling procedures used for groundwater sampling will be employed. However, a sample collection time will not be included on the sample label or the COC form. The actual source of the duplicate sample will be recorded in the field book and/or on the Groundwater Field Data Record.

(b) Each duplicate sample will be collected simultaneously with the actual sample by alternately filling sample and duplicate bottles. Following the order of collection specified for each set of containers (VOCs, SVOCs, other organic parameters, unfiltered inorganic constituents, and filtered inorganic constituents), the duplicate sample containers will be alternately filled with groundwater for each parameter.

(c) All collection and preservation procedures outlined for groundwater sampling will be followed for each duplicate sample.

4.2 Equipment Blanks

Equipment blanks include reagent water that is run through the bailer (if not disposable), rope, leader line, decontaminated pump, a representative section of the pump's tubing, or any other piece of sampling equipment that may have come in contact with the sample. The equipment blanks are collected and preserved in the same sample containers as field samples. If dedicated or disposable systems are used, equipment blanks are not required, although an initial blank could be performed to demonstrate that the dedicated equipment is clean prior to use. If only dedicated tubing is used, the equipment blank will include only the pump in subsequent sampling events. A passive sampler is considered a dedicated device and no equipment blank is required.

Ideally, the reagent water should come from the laboratory and be certified clean. If not certified and/or if not from the laboratory performing the analyses, a separate water blank that has not run through the sampling equipment should be sent to the laboratory for analysis.

4.3 Trip Blanks

Trip blanks will be used to check for potential contamination of VOCs via migration during storage and shipping. Trip blanks typically consist of two to three 40 mL VOA vials filled with analyte-free water and preserved with hydrochloric acid (HCl) to pH <2 SU. Trip blank containers are usually supplied pre-filled by the laboratory. Trip blanks are typically submitted to the laboratory at a frequency of one per cooler for coolers that contain samples for VOC and/or VPH analysis. Trip blanks are analyzed by the laboratory for VOCs and/or VPH, depending on field sample analyses.

4.4 Field Blanks

Field blanks consists of analyte free water exposed to the atmosphere during field sample collection. The water is containerized in an appropriate bottle and preservative for the analytical suite and shipped to the laboratory with the other field samples. The results are used to assess whether or ambient/surrounding air conditions may have influenced analytical results.

4.5 MS/MSDs and MS/Duplicates

MSs are an additional analysis of a sample spiked by the laboratory with a subset or all of the target analytes and are used to demonstrate the accuracy of analytical methods for a given matrix. MSDs are an additional analysis of a sample spiked with a subset or all of the target analytes and are also used to demonstrate the accuracy of analytical methods for a given matrix. MS/MSDs also provide a measure of analytical precision for a given matrix. Duplicates are an additional analysis of a sample and are used to demonstrate the precision of analytical methods for a given matrix.

Triplicate volumes of a field sample must be collected in order for the laboratory to have enough volume to perform the MS/MSD analyses for organic parameters. Duplicate volumes of a field sample must be collected in order for the laboratory to have enough volume to perform MS/Duplicate analyses for inorganic parameters. The sample designated for MS/MSD or MS/Duplicate analyses should be noted in the Comments column of the COC document.

4.6 Temperature Blanks

Temperature blanks consist of a sample container filled with non-preserved water (potable or distilled) and typically are included in all coolers that contain samples that require temperature preservation. These may be added to the coolers by the field team if not provided by the laboratory. Temperature blanks must remain inside the coolers on ice during the sampling process.

5.0 DATA MANAGEMENT AND RECORDS MANAGEMENT

Record the sample location, sample identification, and date and time of collection in the field book and/or the Groundwater Field Data Record. The Groundwater Field Data Record (Attachment B) should be used to record the following information:

- Volume of each sample

- Sample identification number
- Sample location (sketch of the sample point)
- Time and date sample was collected
- Personnel performing the task
- Volume of water removed
- Purging time
- Flow rate during purging and sampling
- Weather conditions during sampling (e.g. temperature, wind)
- Field parameters such as water level, pH, temperature, conductivity, turbidity, ORP, and DO
- Sample collection equipment and method used
- Decontamination procedures
- Analytical parameters
- Preservation method and amount of preservative

All sample numbers must be documented on the COC form that accompanies the samples during shipment. Any deviations from the records management procedures specified in the project-specific work plan must be approved by the Project Manager and documented in the field book.

6.0 REFERENCES

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USEPA, January 19, 2010. *Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells*. USEPA Region 1, Rev. 3.

USEPA. March 6, 2013. *Groundwater Sampling.* SESDPROC-301-R3. USEPA Region 4, Science and Ecosystem Support Division. Athens, Georgia.

USEPA. April 22, 2014. Passive (No Purge) Samples.
http://www.clu-in.org/characterization/technologies/default.focus/sec/Passive_%28no%20purge%29_Samplers/cat/Overview/

7.0 SOP REVISION HISTORY

REVISION NUMBER	REVISION DATE	REASON FOR REVISION
0	AUGUST 2014	NOT APPLICABLE
1	JULY 2016	ADDED ATTACHMENT D TO ACCOMMODATE SOP MODIFICATIONS REQUIRED WHEN SAMPLING FOR PFCS; CHANGED NAMING CONVENTION FOR SOP FROM RMD TO ECR.
2	NOVEMBER 2016	ADDED ADDITIONAL INFORMATION REGARDING PFAS.
3	JANUARY 2020	TRC RE-BRANDING; ADDED FIELD BLANKS TO SECTION 4
4	AUGUST 2020	ADDITIONAL PFAS-SPECIFIC INFORMATION INCLUDED

Attachment A:

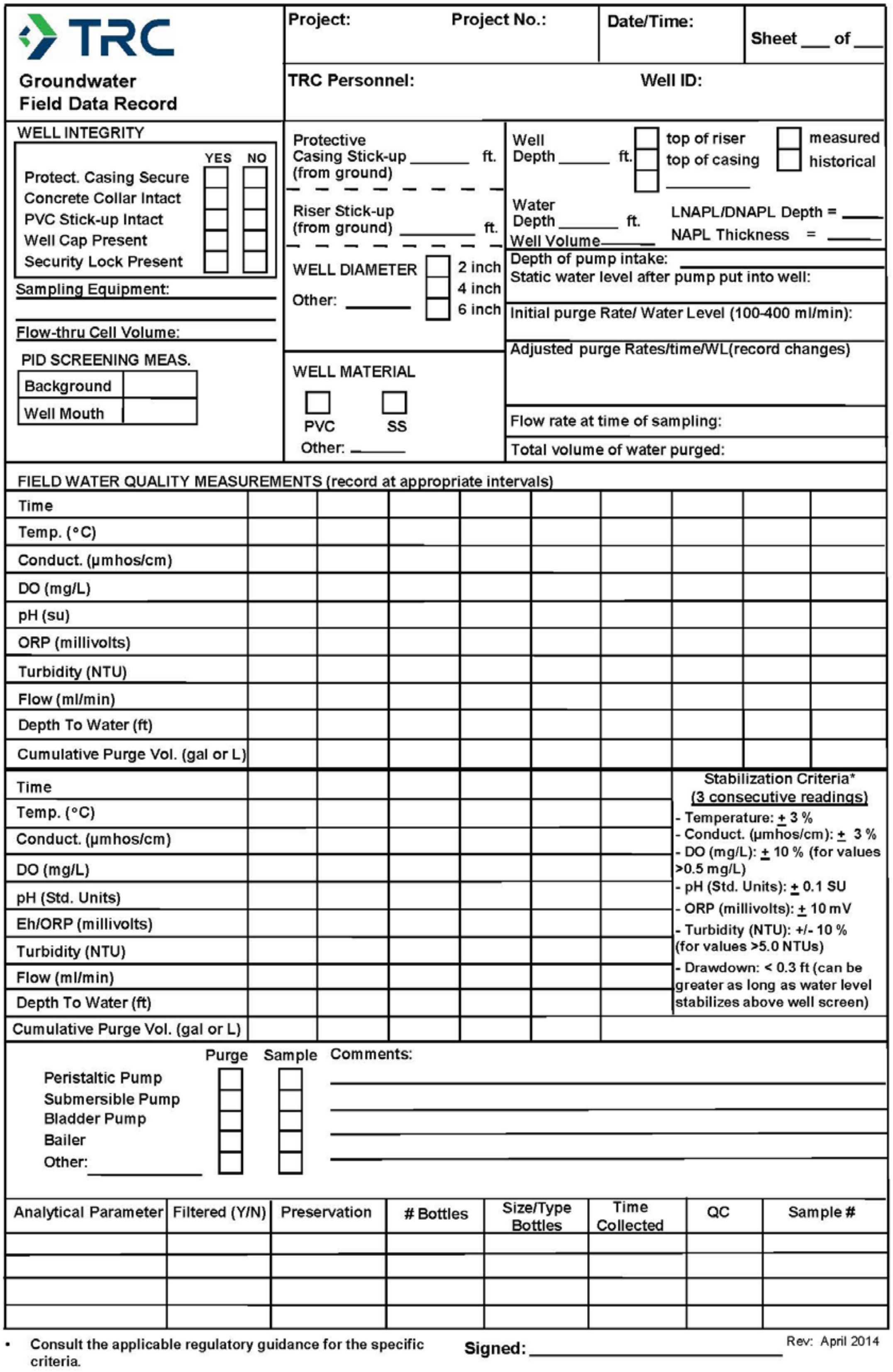
Groundwater Field Parameter Stabilization Criteria for Selected Jurisdictions

Jurisdiction	Information Source	Applicable Stabilization Criteria
USEPA Region 1	Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells; U.S. Environmental Protection Agency Region 1, January 19, 2010. http://www.epa.gov/region1/lab/qa/pdfs/EQASOP-GW001.pdf (for low flow PDF) http://www.epa.gov/region1/lab/qa/qualsys.html (for EPA's Quality System Documents)	pH: ±0.1 unit Specific Conductance: ±3% Temperature: ±3% Turbidity: ±10% if >5 NTUs; if three Turbidity values are <5 NTU, consider the values as stabilized Dissolved Oxygen: ±10% if >0.5 mg/L, if three Dissolved Oxygen values are <0.5 mg/L, consider the values as stabilized Oxidation/Reduction Potential: ±10 millivolts
USEPA Region 2	Groundwater Sampling Procedure: Low Stress (Low Flow) Purging and Sampling, SOP # SST-7, Revision No. 1, November 2010.	Same as above
USEPA Region 4	USEPA Region 4 SOPs: http://www.epa.gov/region4/sesd/fbqstp/index.html See Chemical Parameter Stabilization Criteria (section 3.2.1.1.2 of Groundwater Sampling SOP, revision 3/6/2013: http://www.epa.gov/region4/sesd/fbqstp/Groundwater-Sampling.pdf	pH: ±0.1 unit Specific Conductance: ±5% Temperature: Not used Turbidity: "Stabilized" (no criteria specified) if >10 NTUs ; if three Turbidity values are <10 NTUs, consider the values as stabilized Dissolved Oxygen (optional parameter): ±0.2 mg/L or ±10% of saturation, whichever is greater Oxidation/Reduction Potential: Not used
USEPA Region 5	Ground Water Forum Issue Paper (May 2002, Yeskis and Zavala) http://www.epa.gov/superfund/remedytech/tsp/download/gw_sampling_guide.pdf A minimum set of parameters would include pH, conductivity, and turbidity or DO. Puls and Barcelona, 1996 (pH, specific conductance, ORP, turbidity) Wilde et al., 1998 (pH, turbidity, DO)	pH: ±0.1 unit Specific Conductance: ±3% Temperature: Not used Turbidity: ±10% if >10 NTUs Dissolved Oxygen: ±0.3 mg/L Oxidation/Reduction Potential: ±10 millivolts
USEPA Region 9	See USEPA Region 1 (above)	
USEPA Region 10	See USEPA Region 5 (above)	
Alabama	Alabama Environmental Investigation and Remediation Guidance (section C.3.1) http://www.adem.state.al.us/MoreInfo/pubs/AEIRGInvestigation.pdf	pH: ±0.1 unit Specific Conductance: ±10% Temperature: "Constant" (no criteria specified) Turbidity: Stabilized (no criteria specified), or <10 NTUs Dissolved Oxygen: No criteria specified Oxidation/Reduction Potential: No criteria specified

Jurisdiction	Information Source	Applicable Stabilization Criteria
Indiana	Indiana Department of Environmental Management The Micro-Purge Sampling Option http://www.in.gov/idem/files/remediation_tech_guidance_micro-purge.pdf The parameters normally measured for stability (listed in increasing order of sensitivity) are pH, temperature, specific conductivity, oxidation-reduction potential, DO and turbidity. At least one of the last three listed must be used.	pH: ±0.1 unit Specific Conductance: ±3% Temperature: ±3% Turbidity: ±10% Dissolved Oxygen: ±10% Oxidation/Reduction Potential: ±10 millivolts (document says microvolts, but that may be an error)
Michigan	MDEQ Part 201 Op Memo 2, Attachment 5 http://www.michigan.gov/documents/deq/deq-rrd-OpMemo_2_Attachment5_249853_7.pdf	No specific values to determine stabilization are listed, but the Op Memo lists several other groundwater sampling guidance documents. If a valid reference exists, then it can be used to justify a sampling approach and stabilization parameters.
New Jersey	New Jersey Department of Environmental Protection http://www.state.nj.us/dep/srp/guidance/fspm/	pH: ± 0.1 unit Specific Conductance: ± 3% Temperature: ± 3% Dissolved Oxygen: ± 10% Turbidity: ± 10% for values greater than 1 NTU ORP/Eh: ± 10 millivolts
Ohio	Ohio EPA SOPs: http://www.epa.state.oh.us/portals/30/rules/FSOPs.pdf See Purging Stabilization Criteria (SOP 2.2.4, dated January 2, 2007, review in progress)	pH: ±0.1 unit Specific Conductance: ±3% Temperature: No criteria specified Turbidity: Below 10 NTUs ideal; ±10% if greater than 10 NTUs Dissolved Oxygen: ±0.3 mg/L Oxidation/Reduction Potential: ±10 millivolts
This table was last updated in July 2014.		

Attachment B:

Example Groundwater Field Data Records



Groundwater Field Data Record

Project:	**Project No.:**	**Date/Time:**	**Sheet ___ of ___**
TRC Personnel:		**Well ID:**	

WELL INTEGRITY

	YES	NO
Protect. Casing Secure	☐	☐
Concrete Collar Intact	☐	☐
PVC Stick-up Intact	☐	☐
Well Cap Present	☐	☐
Security Lock Present	☐	☐

Sampling Equipment: __________

Flow-thru Cell Volume: __________

PID SCREENING MEAS.

Background	
Well Mouth	

Protective Casing Stick-up (from ground) _______ ft.

Riser Stick-up (from ground) _________ ft.

WELL DIAMETER ☐ 2 inch ☐ 4 inch ☐ 6 inch

Other: _______

WELL MATERIAL

☐ PVC ☐ SS

Other: ______

Well Depth ______ ft. ☐ top of riser ☐ top of casing ☐ _________ ☐ measured ☐ historical

Water Depth _______ ft. LNAPL/DNAPL Depth = _____

Well Volume_______ NAPL Thickness = ______

Depth of pump intake: __________

Static water level after pump put into well:

Initial purge Rate/ Water Level (100-400 ml/min):

Adjusted purge Rates/time/WL(record changes)

Flow rate at time of sampling:

Total volume of water purged:

FIELD WATER QUALITY MEASUREMENTS (record at appropriate intervals)

Time									
Temp. (°C)									
Conduct. (µmhos/cm)									
DO (mg/L)									
pH (su)									
ORP (millivolts)									
Turbidity (NTU)									
Flow (ml/min)									
Depth To Water (ft)									
Cumulative Purge Vol. (gal or L)									

Time						
Temp. (°C)						
Conduct. (µmhos/cm)						
DO (mg/L)						
pH (Std. Units)						
Eh/ORP (millivolts)						
Turbidity (NTU)						
Flow (ml/min)						
Depth To Water (ft)						
Cumulative Purge Vol. (gal or L)						

Stabilization Criteria*
(3 consecutive readings)
- Temperature: ± 3 %
- Conduct. (µmhos/cm): ± 3 %
- DO (mg/L): ± 10 % (for values >0.5 mg/L)
- pH (Std. Units): ± 0.1 SU
- ORP (millivolts): ± 10 mV
- Turbidity (NTU): +/- 10 % (for values >5.0 NTUs)
- Drawdown: < 0.3 ft (can be greater as long as water level stabilizes above well screen)

	Purge	Sample
Peristaltic Pump	☐	☐
Submersible Pump	☐	☐
Bladder Pump	☐	☐
Bailer	☐	☐
Other: __________	☐	☐

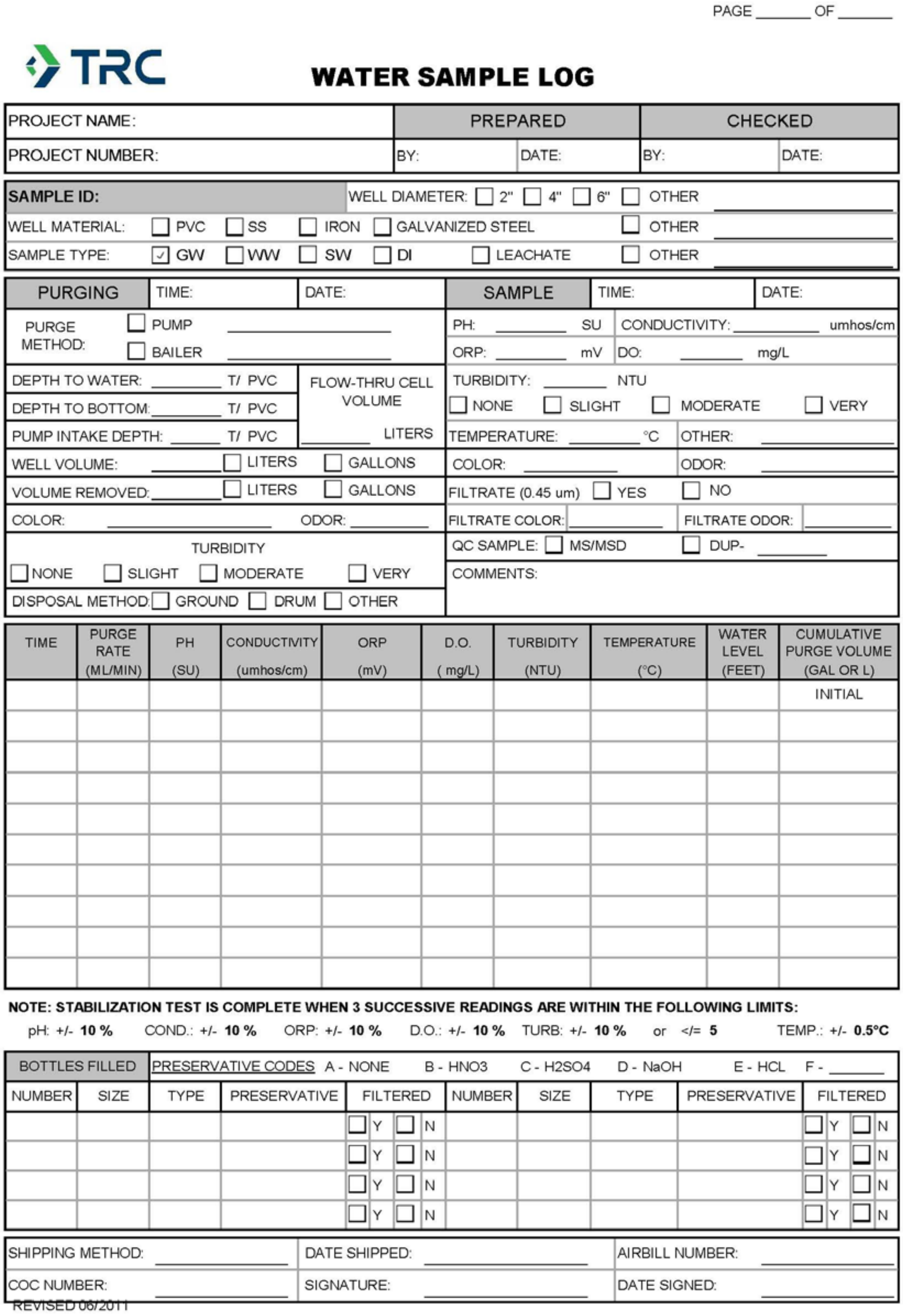
Comments: __________

Analytical Parameter	Filtered (Y/N)	Preservation	# Bottles	Size/Type Bottles	Time Collected	QC	Sample #

- Consult the applicable regulatory guidance for the specific criteria.

Signed: ____________________

Rev: April 2014



PAGE ______ OF ______

WATER SAMPLE LOG

PROJECT NAME:	PREPARED		CHECKED	
PROJECT NUMBER:	BY:	DATE:	BY:	DATE:

SAMPLE ID: WELL DIAMETER: ☐ 2" ☐ 4" ☐ 6" ☐ OTHER ______________
WELL MATERIAL: ☐ PVC ☐ SS ☐ IRON ☐ GALVANIZED STEEL ☐ OTHER ______________
SAMPLE TYPE: ☑ GW ☐ WW ☐ SW ☐ DI ☐ LEACHATE ☐ OTHER ______________

PURGING TIME: DATE:	SAMPLE TIME: DATE:
PURGE METHOD: ☐ PUMP ______________ ☐ BAILER ______________	PH: ________ SU CONDUCTIVITY: ________ umhos/cm ORP: ________ mV DO: ________ mg/L
DEPTH TO WATER: ________ T/ PVC DEPTH TO BOTTOM: ________ T/ PVC PUMP INTAKE DEPTH: ________ T/ PVC FLOW-THRU CELL VOLUME ________ LITERS	TURBIDITY: ________ NTU ☐ NONE ☐ SLIGHT ☐ MODERATE ☐ VERY TEMPERATURE: ________ °C OTHER: ______________
WELL VOLUME: ________ ☐ LITERS ☐ GALLONS	COLOR: ______________ ODOR: ______________
VOLUME REMOVED: ________ ☐ LITERS ☐ GALLONS	FILTRATE (0.45 um) ☐ YES ☐ NO
COLOR: ______________ ODOR: ________	FILTRATE COLOR: ________ FILTRATE ODOR: ________
TURBIDITY ☐ NONE ☐ SLIGHT ☐ MODERATE ☐ VERY	QC SAMPLE: ☐ MS/MSD ☐ DUP- ________
DISPOSAL METHOD: ☐ GROUND ☐ DRUM ☐ OTHER	COMMENTS:

TIME	PURGE RATE (ML/MIN)	PH (SU)	CONDUCTIVITY (umhos/cm)	ORP (mV)	D.O. (mg/L)	TURBIDITY (NTU)	TEMPERATURE (°C)	WATER LEVEL (FEET)	CUMULATIVE PURGE VOLUME (GAL OR L)
									INITIAL

NOTE: STABILIZATION TEST IS COMPLETE WHEN 3 SUCCESSIVE READINGS ARE WITHIN THE FOLLOWING LIMITS:

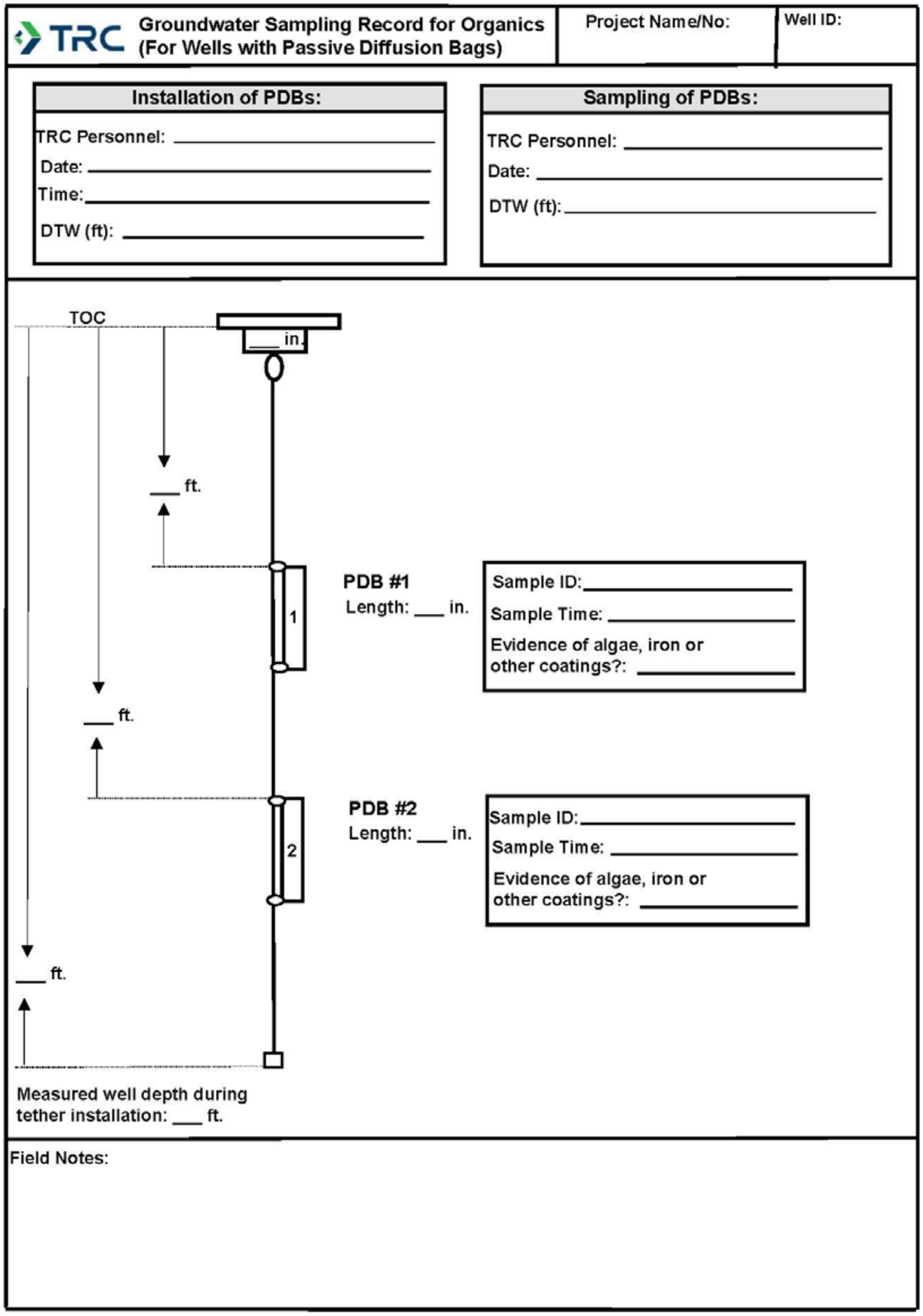
pH: +/- **10 %** COND.: +/- **10 %** ORP: +/- **10 %** D.O.: +/- **10 %** TURB: +/- **10 %** or </= **5** TEMP.: +/- **0.5°C**

BOTTLES FILLED — PRESERVATIVE CODES A - NONE B - HNO3 C - H2SO4 D - NaOH E - HCL F - ________

NUMBER	SIZE	TYPE	PRESERVATIVE	FILTERED	NUMBER	SIZE	TYPE	PRESERVATIVE	FILTERED
				☐ Y ☐ N					☐ Y ☐ N
				☐ Y ☐ N					☐ Y ☐ N
				☐ Y ☐ N					☐ Y ☐ N
				☐ Y ☐ N					☐ Y ☐ N

SHIPPING METHOD: ______________	DATE SHIPPED: ______________	AIRBILL NUMBER: ______________
COC NUMBER: ______________	SIGNATURE: ______________	DATE SIGNED: ______________

REVISED 06/2011



Groundwater Sampling Record for Organics (For Wells with Passive Diffusion Bags)

Project Name/No:

Well ID:

Installation of PDBs:

TRC Personnel: ____________________

Date: ____________________

Time: ____________________

DTW (ft): ____________________

Sampling of PDBs:

TRC Personnel: ____________________

Date: ____________________

DTW (ft): ____________________

PDB #1
Length: ___ in.

Sample ID: ____________________
Sample Time: ____________________
Evidence of algae, iron or other coatings?: __________

PDB #2
Length: ___ in.

Sample ID: ____________________
Sample Time: ____________________
Evidence of algae, iron or other coatings?: __________

Measured well depth during tether installation: ___ ft.

Field Notes:

Rev: April 2014

Attachment C: SOP Fact Sheet

GROUNDWATER SAMPLING

PURPOSE AND OBJECTIVE

The objective of groundwater sampling is to obtain a representative sample of water from a saturated zone or groundwater-bearing unit (i.e., aquifer) with minimal disturbance of groundwater chemistry. This requires that the sample being collected is representative of groundwater within the formation surrounding the well bore as opposed to stagnant water within the well casing or within the filter pack immediately surrounding the well casing.

There are three general approaches to groundwater purging/sampling that can be used to obtain a representative groundwater sample for analysis: 1) the low-flow or micropurge method where the mixing of the stagnant water is minimized using low-flow pumping rates during the collection of the groundwater sample; 2) the multiple well volume removal approach in which the stagnant water is removed from the well and the filter pack prior to sample collection; and 3) the passive sampler procedure where water quality equilibration with the surroundings is achieved through deployment of the passive sampler for a sufficient amount of time prior to sampling. All three approaches are summarized in this document.

WHAT TO BRING

- Site-specific HASP and field book
- Project-specific work plan
- Figure or site map showing well locations and table showing well construction details
- Field data sheets from previous sampling event
- Well wrenches, ratchet set, and turkey baster to remove standing water from flushmount manholes
- Bolt cutters, padlocks and keys
- Water level meter of sufficient length
- Decontaminated pump, control box, power source (i.e., battery, generator, etc.)
- Tubing (Teflon®, Teflon®–lined polyethylene, or HDPE, type dependent upon project objectives)
- Multi-parameter instrument and flow-through cell (typically should include: pH, temperature, conductivity, ORP, and DO)
- Turbidity meter
- Equipment decontamination supplies (refer to ECR SOP 010, *Equipment Decontamination*)
- Appropriate PPE
- Field book
- Sample bottleware, labeled cooler, ice, temperature blank and blank COC forms; may also need field blank bottles and reagent-grade water
- Zip-loc® plastic bags
- Groundwater field data records
- Graduated cylinder and stop-watch
- Rope for tying off pump at desired intake
- Indelible marking pens
- Bubble wrap
- 5-gallon bucket(s)

As Needed:

- Calibrated PID or FID for well mouth readings
- Oil/water interface probe of sufficient length
- Drums for purge water, grease pen and adhesive drum labels; appropriate crescent or socket wrench
- Filtration equipment, if required (0.45 micron filters, or as otherwise required for the project)
- Other non-routine PPE such as Tyvek coveralls or respirators
- Traffic cones
- Field calibration sheets and calibration solutions

OFFICE

- Prepare/update the site-specific HASP; make sure the field team is familiar with the most recent version.
- Review the project-specific work plan with the Project Manager and/or the field team leader. Discuss the following:
  - Communication procedures;
  - Sampling order and designation;
  - Collection and sample method;
  - Analytical parameters, holding times and turn-around times;
  - Laboratory (contact/shipping info, COC, billing references);
  - Purge water management (Drums? Discharge to ground?);
  - QC sample collection; and
  - Decontamination procedures.
- Verify that monitoring wells will be accessible and/or coordinate to have a site contact available to assist.
- Make sure that monitoring well sample designations and QC sample designations/frequency are understood.
- Confirm that all necessary equipment is available in-house or has been ordered. Rental equipment is typically delivered the day before fieldwork is scheduled. Prior to departure or mobilization to site, test equipment and make sure it is in proper working order. Have rental equipment supplier contact information available for use in field.
- Review sample bottle order for accuracy and completeness and damaged bottles.
- Discuss specific documentation and containerization requirements for investigation-derived waste disposal with the Project Manager

ON-SITE

GROUNDWATER SAMPLING

- Review the HASP with all field personnel, sign acknowledgement form and conduct Health & Safety tailgate meeting. Check in security, site contact, or designated person per project-specific work plan or Project Manager.
- Make sure appropriate PPE is worn by all personnel and work area is safe (i.e., utilize traffic cones; minimize interference with on-site activities and pedestrian traffic, etc.)
- Calibrate equipment (if applicable) and record all rental equipment serial numbers in the field book.
- Open wells to allow equilibration and collect full round of water level gauging before sampling is started (unless otherwise noted in project-specific work plan). Record the following:
  - Well mouth PID/FID reading (if necessary);
  - Depth to product and water;
  - Total well depth (not required if free product is measured unless otherwise noted in project-specific work plan); and
  - Condition of wells (i.e., lid broken, pad cracked, rusted lock) and collect photographs if site allows camera use.

SAMPLING PROCEDURES: PRE-PURGE

- Decontaminate pump.
- Take water level measurements prior to pump installation.
- Connect sampling tubing to pump outlet and lower to sample depth; **ALWAYS USE ROPE TO SECURE PUMP TO SURFACE.**
- The pump intake depth(s) for each well should be specified in the project-specific work plan (either specific depth or mid-point of saturated well screen).
- For wells with screened or open borehole intervals greater than 10 feet in length, sampling of multiple intervals may be required.
- If samples are to be collected from multiple depths from an individual well, always collect a sample from the shallowest depth first and leave enough extra tubing coiled at the surface so the pump can be lowered to the next interval; always try to cover excess tubing present at the surface to prevent the air temperature from influencing the measurements and exposure to contaminants on the ground;
- Be careful not to let the pump hit the bottom of the well.
- If using Teflon®-lined tubing, be sure that the lining does not bunch up around the connection. This will restrict water flow and make the pump work harder than it has to.
- Calibrate (or perform a calibration check on) all field monitoring equipment on the same day before collecting groundwater samples. Refer to TRC SOPs and manufacturer's equipment calibration instructions. A calibration check may also be required during or at the end of each sampling day. Consult the project-specific work plan.

SAMPLING PROCEDURES: MULTIPLE-VOLUME PURGING

- The multiple-volume purging approach is typically performed using bailers or submersible or peristaltic pumps. In the multiple-volume purging approach, there are two measurements used to determine adequate purge volume removal prior to sample collection: 1) purge volume and 2) field parameter stabilization.
- The field parameters should be recorded at regular volumetric intervals. There are no set criteria for establishing how many total sets of measurements are adequate to document stability of parameters.
- Prior to purging a well, the amount of water inside the well riser and well screen (i.e., water column) should be determined, if possible. Once this information is known, the well volume can be calculated using the following equation:

  Well Volume (V) = $\pi r2$ h
- For volumetric purging, an adequate purge is typically achieved when 3 to 5 well volumes have been removed.
- For volumetric purging, it is suggested that field readings are collected every ½ well/well screen volume after an initial 1 to ½ well volumes are purged. The volume removed between readings can be adjusted as well-specific information is developed.
- If removing a specified volume of water (e.g., 3 well volumes) has been determined to be suitable for purging, sampling can commence immediately upon achieving the required purge volume.
- In other cases, where specified in the project-specific work plan, stabilization of field parameters must be documented prior to sample collection.
- If, after 3 well volumes have been removed, the field parameters have not stabilized, additional well volumes (up to a total of 5 well volumes), should be removed.
- If the parameters have not stabilized within five well volumes, it is at the discretion of the Project Manager whether or not to collect a sample or to continue purging.

SAMPLING PROCEDURES: LOW-FLOW PURGING

- The low-flow purging approach is typically performed using peristaltic pumps or submersible pumps. Low-flow purging (also referred to as low-stress purging, low-volume purging, or Micropurging®) is a method of well purging/sampling that minimizes the volume of water withdrawn from a well in obtaining a representative sample.
- When performing low-flow purging and sampling, it is recommended that the pump intake be set in the center of the well screen interval to help prevent disturbance of any sediment at the bottom of the well.

GROUNDWATER SAMPLING

- To begin purging, the pump should be started at the lowest pressure/power flow rate setting (e.g., 100 mL/min) and then slowly increased until water begins discharging. Monitor the water level and slowly adjust the pump speed until there is little or no drawdown or drawdown has stabilized. The pump pressure/power may need to be increased for discharge to occur.
- The stabilization of drawdown should be documented. Measure and record the flow rate and water level every 3 to 5 minutes during purging. The flow rate should be reduced if drawdown is greater than 0.3 feet over three consecutive 3 to 5 minute interval readings.
- Attempts should be made to avoid pumping a well dry.

Field Parameter Stabilization During Purging

- ○ Generally, an adequate purge with respect to the groundwater chemistry is achieved when stability for at least three consecutive measurements is achieved. See stability requirements in Appendix A of this SOP.

POST-PURGE GROUNDWATER SAMPLE COLLECTION

- New, disposable gloves should be donned immediately prior to sample collection and should be changed at any point that their cleanliness becomes compromised during sample collection.
- If using a submersible or peristaltic pump, maintain the same flow rate as used during purging. Disconnect the pump tubing from the flow-through cell. Samples must be collected directly from the discharge port of the pump tubing prior to passing through the flow-through cell. This is critically important to avoid cross-contamination between wells.
- If using bottom-filling bailers, slowly lower the bailer into the well until it is submerged to the point where water does not enter the top (i.e., bottom-filling). Retrieve the bailer. The first bailer recovered after well purging must be used for sample collection.
- Collect groundwater samples in the following order:
    - ○ VOCs;
    - ○ SVOCs;
    - ○ Other organic parameters;
    - ○ Unfiltered inorganic constituents; and
    - ○ Filtered inorganic constituents.
- Note that sample vials for VOCs must be filled so a meniscus forms over the mouth of the vial. This ensures no air bubbles or headspace will be formed after it has been capped. Ensure the lack of air bubbles and headspace by turning the vial upside down and tapping it lightly. If any bubbles are observed, see Section 2.3.2 of this SOP.
- Preserve the non-VOC samples in pre-preserved vials supplied by the laboratory or if the sample containers are not pre-preserved, preserve the non-VOC samples in accordance with method and project-specific requirements.
- Depending upon project requirements, filtering may be performed. See procedures listed in Section 2.3.4 of this SOP. Clearly note "filtered" on the sample label and the COC.
- Make sure all sample bottles are appropriately labeled.
- Package the samples with bubble wrap and/or organic absorbent, as necessary. Place into shipping container and cool to 4°C and complete the COC.
- Decontaminate non-disposable sampling equipment between uses.

PASSIVE SAMPLING

- There are three generic forms of passive (no purge) samplers: thief (grab) samplers, diffusion (equilibrium) samplers, and integrating (kinetic) samplers. However, this SOP focuses on the more commonly used diffusion (equilibrium) samplers. Be aware of sample holding times, and arrange for samples to be in the laboratory's possession accordingly.
- Passive samplers are deployed at a predetermined depth across the well screen. Typically, the initial sampling event may deploy multiple passive samplers across 5-foot intervals of saturated well screen to observe any potential stratification. Long-term sampling depths typically target a zone of higher concentration, if present.
- New passive samplers are attached via PVC cable ties to a tether (pre-made marine-grade polyethylene rope or stainless steel cable with a weight at the bottom) that is then suspended within the well.
- The passive sampler should be allowed to equilibrate with groundwater for an appropriate period of time (e.g., at least 2 weeks for PDB samplers).
- Raise the passive sampler to the surface using a tether reel. Examine the surface of the passive sampler for evidence of algae, iron, or other coatings, and for tears to the membrane. Note observations in the field book. If tears are present and water is leaking out, the sample is not considered viable. Contact the Project Manager.
- Detach the passive sampler from the tether.
- Remove excess beaded water from the passive sampler with a clean gloved hand, running top to bottom; this is to minimize the contact of beaded water with water in the passive sampler.

GROUNDWATER SAMPLING

- Use a small diameter discharge tube (<0.15 inch diameter to reduce volatilization) and pierce near the bottom, allowing water to smoothly flow into the VOA vial. The VOA vials must be filled within the first several minutes of passive sampler retrieval.
- A small amount of water may remain within the passive sampler after filling the VOA vials and can be used for field parameter measurements if required.
- Dispose of the passive sampler after use.
- Note that sample vials for VOCs must be filled so a meniscus forms over the mouth of the vial. This ensures no air bubbles or headspace will be formed after it has been capped. Ensure the lack of air bubbles and headspace by turning the vial upside down and tapping it lightly. If any bubbles are observed, see Section 2.3.2 of this SOP.
- Make sure all sample bottles are appropriately labeled.
- Package the samples with bubble wrap and/or organic absorbent, as necessary. Place into shipping container and cool to 4ºC and complete the COC.

DOs AND DO NOTs OF GROUNDWATER PURGING AND SAMPLING

DOs:

- DO have the following items when going into the field: site-specific work plan; site-Specific HASP; appropriate PPE (steel-toed boots, safety glasses, etc.) as required by the Site-Specific HASP; field book and a water-proof ball-point pen; business cards; nitrile gloves; well keys; copies of well installation forms and field data forms from previous sampling event.
- DO make sure that the equipment is set up properly and the bottleware is nearby and ready to be filled. There is little time between taking parameters.
- DO look at the water quality parameters from the previous round of sampling. If there is a large deviation from the previous round's measurements, make sure the meters are properly calibrated and the parameter units are the same. Otherwise, consult the Project Manager or field team leader.
- DO fill sample bottles slowly to make sure that they are not overfilled and that preservative does not become diluted. If collecting filtered samples, fill all non-filtered first, then fill filtered samples - if water is very silty, more than one filter might be required to fill sample bottles.
- DO record the time that purging begins and ends. "Purge Stop" and sample start time are the same.
- DO call your Project Manager or field team leader if unexpected conditions are encountered or at least daily to update them. It is also recommended to call when sampling is winding down for the day to make sure that the project-specific work plan has been fully implemented and there are no additional tasks to complete. Provide shipping tracking numbers to the Project Manager and laboratory contact.
- DO have the numbers for laboratory, vehicle rental and equipment rental providers readily available while in the field.
- DO record sample locations and parameters in the field book and the Groundwater Field Data Records as you purge.
- DO check on the purging setup frequently to make sure proper equipment function is maintained.
- DO bring ice to the site in the morning so that samples are kept cool throughout the entire event. Storing samples in a warm cooler can invalidate sample results and may result in re-sampling on your own time.

DO NOTs:

- DO NOT sign anything in the field. This includes disposal documentation, statements, etc.; call the Project Manager if this is an issue.
- DO NOT allow the pump or sampling equipment to hit the bottom of the well - If the pump hits the bottom of the well, it can stir up mud. Remember, the goal of low-flow sampling is to collect non-turbid samples.
- DO NOT use non-indelible ink to label samples or record field notes – if the field book gets wet, notes become illegible.
- DO NOT leave air bubbles in VOA vials.
- DO NOT pour any extracted water back down into the well.
- DO NOT lean over wells with pens, keys, cell phones, tools, etc. in your pocket.
- DO NOT use clear tape to cover labels on certain analyses (e.g., 40-mL vials for VOC analysis) due to potential interference with analytical equipment.

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Attachment D: SOP Modifications for PFAS

Due to the pervasive nature of PFAS in various substances routinely used during sampling and the need to mitigate potential cross-contamination or sampling bias to ensure representative data are collected, special care should be taken when sampling for PFAS. The following table highlights the required modifications to this SOP when sampling for PFAS.

PFAS Sampling Protocols	
SOP Section Number	**Modifications to SOP**
1.3	• Do not use equipment utilizing Teflon® or low density polyethylene (LDPE)[1] during sample handling or mobilization/demobilization. This includes bailers, tubing, bladders, bailer cord/wire, waterproof/resistant paper products, certain personal protective equipment (PPE) (see below), and Teflon® tape. High density polyethylene (HDPE) or silicone tubing should be used in lieu of Teflon® or Teflon®-lined tubing. • Passive diffusion bags (PDBs) should not be used due to the presence of LDPE material in PDBs. • Blue Ice® (chemical ice packs) must not be used to cool samples or be used in sample coolers. Regular ice in Ziploc® bags can be used. • Do not use LDPE or glass sample containers or containers with Teflon-lined lids. HDPE or polypropylene containers are acceptable for sample storage. HDPE or polypropylene caps are acceptable. • Do not use aluminum foil. • Field notes should be recorded on loose paper field forms maintained in aluminum or Masonite clipboards. Waterproof field books, plastic clipboards and spiral bound notebooks should not be used. • Do not use Post-It Notes during sample handling or mobilization/demobilization. • Refer to TRC's SOP ECR-010 Equipment Decontamination for PFAS-specific decontamination protocols. Ensure that PFAS-free water is used during the decontamination procedure.
1.5	Always consult the Site Specific Health and Safety Plan prior to conducting field work. The following considerations should be made with regards to field preparation during PFAS sampling: • Tyvek® suits should not be worn during PFAS sampling events. Cotton coveralls may be worn. • Boots and other field clothing containing Gore-Tex™ or other waterproof/resistant material should not be worn. This includes rain gear. Boots made with polyurethane and polyvinyl chloride (PVC) are acceptable. • Stain resistant clothing should not be worn. • Food and drink should not be allowed within the exclusion area. Pre-wrapped food or snacks should not be in the possession of sampling personnel during sampling. Bottled water and hydration drinks (e.g., Gatorade®) may be consumed in the staging area only.

PFAS Sampling Protocols	
SOP Section Number	**Modifications to SOP**
	• Personnel involved with sample collection and handling should wear nitrile gloves at all times while collecting and handling samples or sampling equipment. Avoid handling unnecessary items with nitrile gloves. A new pair of gloves must be donned prior to collecting each sample. • Wash hands with Alconox or Liquinox and deionized water after leaving vehicle before setting up to sample a well.
1.6.1	• Avoid wearing clothing laundered with fabric softeners. • Avoid wearing new clothing (recommended 6 washings since purchase). Clothing made of cotton is preferred. • Avoid using cosmetics, moisturizers, hand creams, or other related products as part of cleaning/showering on the day of sampling. • Avoid using sunscreens or insect repellants that are not natural or chemical free.
1.6.2	• Consider collecting samples for total suspended solids which will become important for fate and transport and treatment considerations. Floc accumulates high concentrations of PFAS and specifically some of the longer-chain PFAS; when this floc settles out, concentrations can decrease by an order of magnitude. • Low-flow sampling is preferred for PFAS sampling. Bailers should be avoided due to the potential for PFAS to accumulate at the air/water interface. If bailers need to be used, purging of at least one well volume should be performed to remove static surface conditions. • If sampling for parameters other than PFAS, perform PFAS sampling first. Schedule PFAS sampling at the beginning of the work day to avoid other sources of contamination.
2.0	• Monitoring wells should always be sampled from the lowest contamination to the highest contamination, when possible. In source areas, if deep wells are anticipated to be less contaminated, the deep wells should be sampled prior to sampling the shallow wells to avoid cross-contamination from sampling equipment.
2.2.5	Tubing used to purge and sample groundwater for PFAS must not be LDPE or Teflon®. HDPE and silicone are acceptable.
2.3 and 2.3.3	LDPE and/or glass containers should not be used for sampling. Teflon®-lined caps should also not be used during sample collection. Instead, HDPE or polypropylene containers are acceptable for sample storage. HDPE or polypropylene caps are acceptable.
2.4	Due to LDPE material in PDBs, PDBs cannot be used for PFAS sampling.
2.5 (e)	Avoid using waterproof labels for sample bottles. The use of paper labels covered with clear tape or placed in Ziploc® bags to avoid moisture on the sample label is acceptable.
2.5 (f)	Samples for PFAS analysis must be shipped at <10°C. Standard coolers are acceptable. Keep high-concentration PFAS samples in separate coolers from low-concentration PFAS samples.

Notes:

[1] – PFAS have been used as an additive in the manufacturing of LDPE to smooth rough surfaces and, in the case of LDPE tubing, to allow for less turbulent flow along the surface of the tubing.

Appendix C
Chain-of-Custody Procedures SOP

Title: **Chain-of-Custody Procedures**	Procedure Number: **ECR 002**
	Revision Number: **2**
	Effective Date: **February 2021**

Authorization Signatures			
		Elizabeth Denly	
Technical Review	Date	Environmental Sector Quality Director	Date
Amanda Smith	2/24/21	Elizabeth Denly	2/24/21

TABLE OF CONTENTS

Page No.

FIGURES

ATTACHMENTS

1.0 INTRODUCTION

1.1 Scope & Applicability

This Standard Operating Procedure (SOP) guides TRC personnel in proper Chain-of-Custody (COC) practices.

This SOP was prepared to direct TRC personnel in the sample custody procedure requirements associated with field sample collection. Other state or federal requirements may be above and beyond the scope of this SOP and will be followed, if applicable. Sample custody procedures are an important part of the field investigation program in order to maintain data quality and to be able to document proof of proper handling. Sample custody begins at the time of sample collection and continues until the samples have been analyzed. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files.

Custody is one of several factors that are necessary for the admissibility of environmental data as evidence in a court of law or other evidentiary venue. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. An overriding consideration essential for the validation of environmental measurement data is the necessity to demonstrate that samples have been obtained from the locations stated and that they have reached the laboratory without alteration (i.e., representative of the identified sample media).

1.2 Summary of Method

Evidence of the sample tracking from collection to shipment, laboratory receipt, and laboratory custody must be properly documented.

A sample or evidence file is considered to be in a person's custody if the item is:

- In a person's possession;
- Within sight of the person after they have taken possession;
- Secured and preserved so that no one can tamper with it after having been in a person's possession; and/or
- In a secured area where access is restricted to authorized personnel.

The Field Team Leader or designee is responsible for overseeing and supervising the implementation of proper sample custody procedures in the field and ensuring sample custody until samples have been transferred to a courier or directly to the laboratory. Once received by the laboratory, the samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

1.3 Equipment

The following list is an example of items that may be utilized when implementing sample custody procedures in the field. Project-specific conditions or requirements may warrant the use of additional items or deletion of items from this list. Many of these items may be provided by the selected analytical laboratory for a given project.

- Chain-of-Custody forms
- Sample labels
- Sample tags
- Custody seals
- Computer, tablet or smart device
- Indelible/waterproof ink
- Printer
- Ziploc® bags, or equivalent

2.0 PROCEDURES

Sample custody and transfer procedures are summarized below. These procedures are intended to ensure that the samples will arrive at the laboratory with the COC intact. The COC procedures are initiated in the field immediately following sample collection. The procedures consist of four main components: (1) preparing and attaching a unique sample label to each sample collected, (2) completing the COC form, (3) reviewing the COC form for accuracy, and (4) preparing the samples for shipment and custody transfer. For projects using TRC's Environmental Data Management System (EDMS) the project team's Data Manager can assist in planning sampling events to prepopulate bottle labels and COC forms and log all COC forms generated for the project.

2.1 Specific Chain-of-Custody Procedures

2.1.1 Sample Labels

Field personnel are responsible for uniquely identifying and labeling all samples collected during a field investigation program. All labeling must be completed in indelible/waterproof ink and securely affixed to the sample container. Individual sample containers may be pre-labeled or labeled in the field at the time of collection. Sufficient sample information should be cross-referenced in the field documentation for tracking purposes. A unique sample location may contain multiple sample containers with the same sample identification for the purposes of separate analyses or additional sample volume as required by the laboratory.

Sample labels may contain the following information:

- **Unique sample identification per ECR's Best Practices Document: Environmental Sample Identification and Naming or per a project-specific document (e.g., Quality Assurance Project Plan, Sampling & Analysis Plan)
- Sample location and/or depth/description number, if different from above
- Sample matrix

- Sample container volume
- **Type of analysis to be performed
- **Type of chemical preservation used
- Grab or composite designation
- **Filtered or unfiltered (if submitting both)
- **Sampling date and time using military format (unless blank)
- Sampler's affiliation and initials
- **Site and/or client name

**required

An example of a sample label is provided in Figure 1. TRC's EDMS can produce pre-printed sample labels for regularly scheduled sampling events.

2.1.2 Custody Seals

Custody seals should be secured across the shipping container to ensure content integrity and should be affixed such that the cooler cannot be opened without breaking the seals. The seals contain both the date and the signature of the person affixing them and must be completed in black or blue/black indelible/waterproof ink. Custody seals are attached to the cover seal of the cooler (front and back if cooler opens on both sides) and can be covered with clear plastic tape after being signed and dated by field personnel. An example of a custody seal is shown in Figure 1. The use of custody seals will be determined on a project-specific basis by the Project Manager.

2.1.3 Chain-of-Custody Form

For all analyses, COC forms must be completed and included with each sample set submitted. COC forms are initiated by the samplers in the field. If multiple laboratories are being used, a separate set of COC forms must be completed for each laboratory receiving samples to ensure proper transfer of custody from the time of sample collection to analysis. These forms serve as a record of sample collection, transfer, shipment, and receipt by the laboratory. These forms may contain the following pertinent information:

- Project/site name and/or project number
- Courier or shipping company name, if applicable
- Air bill tracking numbers(s), if known and applicable
- Laboratory name and address
- Sample identifications
- Sample matrices (e.g., soil, water, air, etc.)
- Type of sample (e.g., grab or composite)
- Date/time (military format) sample collected, unless sample is being submitted as a blind duplicate
- Size, type, and number of containers for each sample set
- Preservative(s) used (if any)
- Required analysis or method for each sample set
- Filtered or unfiltered
- Requested turnaround time for sample results
- Names of individuals responsible for sample custody
- Type of deliverables required
- Date shipped or otherwise transferred
- Number of coolers being submitted

Figure 2 provides an example COC form. It should be noted that this is an example format only. Laboratories typically provide their own laboratory-specific COC form. Other COC formats may be used as long as all of the applicable information is included. COC forms will be initiated in the field. TRC's EDMS can produce pre-printed COC forms for regularly scheduled sampling events.

All entries on the COC form must be legible and must be made in blue or black permanent ink. No erasures or obliterations can be made. If an incorrect entry is made, the information must be crossed out with a single strike mark which is signed or initialed and dated by the person recording the information. The correction must be written adjacent to the error. The original entry should still be legible even though crossed out.

2.1.4 Transfer of Custody

Samples will be accompanied by a properly completed COC form during each step of custody transfer and shipment. When physical possession of samples is transferred, both the individual relinquishing the samples and the individual receiving them will sign, date, and record the time of transfer on the COC form.

If at the completion of sampling the samples are not shipped directly from the field or point of collection to the analytical laboratory, the samples will be temporarily stored in an iced cooler at a secure location (e.g., locked vehicle, residence, office). Access to the secure location and transfer of the sample containers for laboratory delivery shall only be provided by a TRC employee and such sample transfer shall be recorded on the COC form.

All samples will be shipped directly to the laboratories by a TRC employee, an overnight commercial courier, or a laboratory-supplied courier service. Occasionally, samples may be relinquished directly to a client for subsequent transfer to the laboratory with proper COC procedures being followed.

In the case of sample shipment by an overnight commercial courier, a package tracking number will serve as an extension of the COC form while the samples are in transit. The COC forms will be sealed inside the sample cooler within a clear plastic bag taped to the inner top of the cooler and the custody seals, if used, will be completed on the outside of the cooler prior to shipment. Commercial couriers are not required to sign off on the custody forms since the forms are sealed inside the cooler prior to shipment; this allows the custody seal to remain intact.

The original COC form will accompany the samples at all times. A copy of all COC forms submitted to the laboratory will be retained by the sampler along with field records/logbooks documenting sample collection and will be placed in the project files. In the case of multiple sample coolers associated with one COC, a copy of the COC should be placed in each cooler and the total number of coolers should be recorded on the COC.

3.0 QUALITY ASSURANCE/QUALITY CONTROL

Following sample collection, all samples will be brought to a location for batching and paperwork checks. At this location, labels and logbook information are cross-checked to ensure there is no error in sample identification or sample collection time and that all samples are accounted for.

The sample information is transferred to the COC form. The samples are packaged to prevent breakage and/or leakage, and the shipping containers are labeled for transport.

The Field Team Leader has the responsibility of maintaining the COC and air bill documentation (if applicable). Individual responsibilities may be delegated to other field staff, as appropriate. Quality control procedures will place emphasis on ensuring that appropriate samples were collected and submitted to the laboratory for the correct analyses. The COC forms will also be reviewed by the Field Team Leader or designee to ensure that all required information is clearly presented.

Many laboratories will provide a sample receipt confirmation via electronic mail upon request. COC forms should be cross-checked with laboratory sample receipt confirmations, if applicable, to ensure that all samples were received and logged in correctly by the laboratory.

4.0 INVESTIGATION-DERIVED WASTE DISPOSAL

Not applicable.

5.0 DATA MANAGEMENT AND RECORDS MANAGEMENT

The Project Manager or Field Team Leader will maintain an inventory of all COC forms completed during the program and will be responsible for ensuring that they are archived in the project files following the completion of the field work.

It is good practice to scan all completed COC forms at the conclusion of field activities and store the resulting electronic PDF files in the project directory.

For projects using TRC's EDMS, the project team's Data Manager can assist in planning sampling events to prepopulate bottle labels and chain of custody forms and log all COC forms generated for the project. The TRC EDMS system has a completeness report that can track the samples collected and the analyses performed as data are received from the laboratory.

6.0 REFERENCES

A Compendium of Superfund Field Operations Methods EPA/540/P-87/001. December 1987.

U.S. Environmental Protection Agency (EPA) Office of Enforcement and Compliance Monitoring – National Enforcement Investigations Center (NEIC) requirements (NEIC, 1986)

7.0 SOP REVISION HISTORY

REVISION NUMBER	REVISION DATE	REASON FOR REVISION
0	MARCH 2013	NOT APPLICABLE
1	JANUARY 2020	TRC RE-BRANDING AND SOP RE-NUMBERING
2	FEBRUARY 2021	GENERAL UPDATES; MOBILE DATA APPLICATIONS ADDED

Figure 1 Example Sample Label and Custody Seal

QEC
Quality Environmental Containers
P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID

SAMPLE DATE

SAMPLE TIME

SAMPLED BY

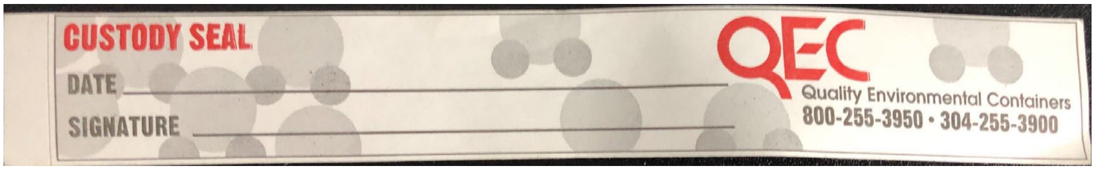
PRESERVATIVE

ANALYSIS REQUESTED

____ GRAB

____ COMPOSITE

Example Sample Label



CUSTODY SEAL

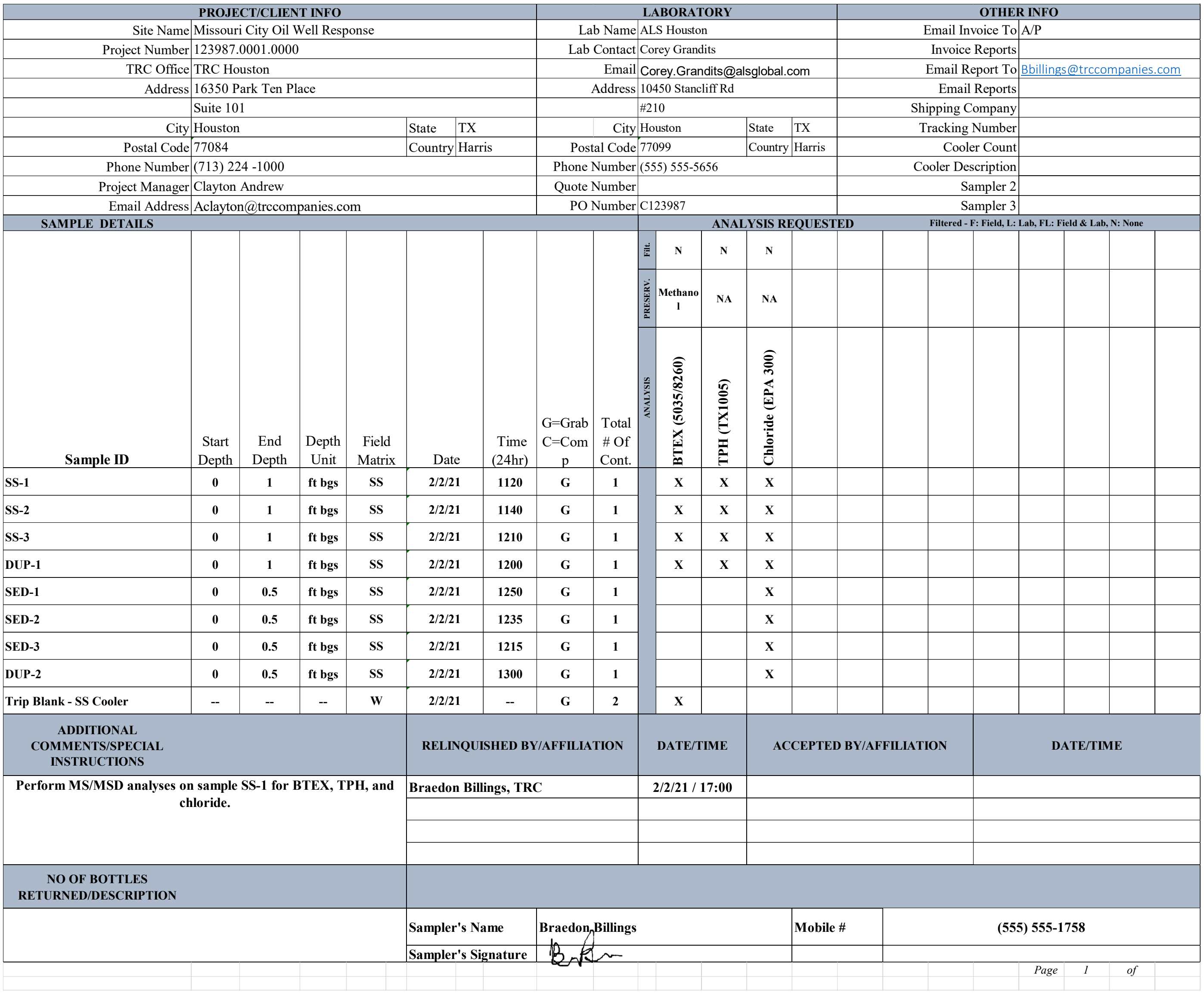
DATE ____________________

SIGNATURE ____________________

QEC
Quality Environmental Containers
800-255-3950 • 304-255-3900

Example Custody Seal

Figure 2 Example Chain-of-Custody Form



PROJECT/CLIENT INFO				LABORATORY				OTHER INFO	
Site Name	Missouri City Oil Well Response			Lab Name	ALS Houston			Email Invoice To	A/P
Project Number	123987.0001.0000			Lab Contact	Corey Grandits			Invoice Reports	
TRC Office	TRC Houston			Email	Corey.Grandits@alsglobal.com			Email Report To	Bbillings@trccompanies.com
Address	16350 Park Ten Place			Address	10450 Stancliff Rd			Email Reports	
	Suite 101				#210			Shipping Company	
City	Houston	State	TX	City	Houston	State	TX	Tracking Number	
Postal Code	77084	Country	Harris	Postal Code	77099	Country	Harris	Cooler Count	
Phone Number	(713) 224 -1000			Phone Number	(555) 555-5656			Cooler Description	
Project Manager	Clayton Andrew			Quote Number				Sampler 2	
Email Address	Aclayton@trccompanies.com			PO Number	C123987			Sampler 3	

SAMPLE DETAILS | **ANALYSIS REQUESTED** | Filtered - F: Field, L: Lab, FL: Field & Lab, N: None

									Filt.	N	N	N
									PRESERV.	Methanol	NA	NA
Sample ID	Start Depth	End Depth	Depth Unit	Field Matrix	Date	Time (24hr)	G=Grab C=Comp	Total # Of Cont.	ANALYSIS	BTEX (5035/8260)	TPH (TX1005)	Chloride (EPA 300)
SS-1	0	1	ft bgs	SS	2/2/21	1120	G	1		X	X	X
SS-2	0	1	ft bgs	SS	2/2/21	1140	G	1		X	X	X
SS-3	0	1	ft bgs	SS	2/2/21	1210	G	1		X	X	X
DUP-1	0	1	ft bgs	SS	2/2/21	1200	G	1		X	X	X
SED-1	0	0.5	ft bgs	SS	2/2/21	1250	G	1				X
SED-2	0	0.5	ft bgs	SS	2/2/21	1235	G	1				X
SED-3	0	0.5	ft bgs	SS	2/2/21	1215	G	1				X
DUP-2	0	0.5	ft bgs	SS	2/2/21	1300	G	1				X
Trip Blank - SS Cooler	--	--	--	W	2/2/21	--	G	2		X		

ADDITIONAL COMMENTS/SPECIAL INSTRUCTIONS	RELINQUISHED BY/AFFILIATION	DATE/TIME	ACCEPTED BY/AFFILIATION	DATE/TIME
Perform MS/MSD analyses on sample SS-1 for BTEX, TPH, and chloride.	Braedon Billings, TRC	2/2/21 / 17:00		

NO OF BOTTLES RETURNED/DESCRIPTION				
	Sampler's Name	Braedon Billings	Mobile #	(555) 555-1758
	Sampler's Signature			

Page 1 of

Attachment A: SOP Fact Sheet

PURPOSE AND OBJECTIVE

Chain-of-Custody procedures have been developed to direct TRC personnel in the sample custody procedure requirements associated with field sample collection. Other state or federal requirements may be above and beyond the scope of this SOP and should be followed, if applicable. Sample custody procedures are an important part of the field investigation program to maintain data quality and to be able to document proof of proper handling. Sample custody begins at the collection of the samples and continues until the samples have been analyzed. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files.

WHAT TO BRING

- Chain-of-Custody (COC) forms
- Sample Labels
- Custody Seals (if required)
- Indelible/waterproof ink

ON-SITE

- Complete all sample labels with indelible/waterproof ink.
- At a minimum, sample labels should include: site name; unique sample identification; analysis to be performed; preservation method; indication of filtering, if performed; sample date and time.
- COC forms must be completed for each sample set and must be initiated in the field by the sampler.
- COC forms must be completed in blue or black permanent ink.
- At a minimum, the COC forms should include: site name; sample identification; sample matrix; type of preservative; type of analysis; sampling date; and sampler’s name.
- Once sampling activity is completed and the COC form is filled out, place samples in sample coolers.
- Package samples to prevent breakage and/or leakage.
- The COC forms will be reviewed by the Field Team Leader or designee prior to relinquishing the samples.
- The original COC form must accompany samples to the laboratory.
- When samples are transferred from one person to another, both the relinquisher and the person receiving the samples should sign, date and record the date of transfer on the COC form.
- If samples are not sent directly to laboratory, samples need to remain on ice and be stored in a secure location.

Appendix D
Laboratory QA/QC Plans

The testing protocol at the Trail Street Laboratory includes the following elements as general QA/QC requirements that have to be met for each analytical method before results data are validated and reported. Any exceptions to these requirements are noted in the case narrative, and the affected results are flagged accordingly.

Metals by EPA 6020B

QC Element	Control Limits (%)
Calibration Verification	90-110
LCS	80-120
LRB	< RL
Duplicate RPD	0-20
MS Recovery	75-125
MS/MSD RPD	0-20

Mercury by EPA 7470A

QC Element	Control Limits (%)
Calibration Verification	90-110
LCS	85-115
LRB	< RL
Duplicate RPD	0-20
MS Recovery	80-120
MS/MSD RPD	0-20

Inorganic Anions by EPA 300.0

QC Element	Control Limits (%)
Calibration Verification	90-110
LCS	90-110
LRB	< RL
Duplicate RPD	0-20
MS Recovery	80-120
MS/MSD RPD	0-20

Total Dissolved Solid (TDS) by SM 2540C

QC Element	Control Limits (%)
LCS	ERA Certificate of Analysis
LRB	< RL
Duplicate RPD	0-5 of average
MS Recovery	N/A
MS/MSD RPD	N/A

The analysis for each test method is performed based on the following SOPs:

- Metals; CHEM-2.5.10 (Rev 5) - Metals Analysis by Inductively Coupled Plasma - Mass Spectrometry
- Mercury; CHEM-2.5.03 (Rev 4) - Mercury Analysis in Water Samples by Cold Vapor AAS
- Anions; CHEM-2.5.40 (Rev 5) - Anions Analysis by Ion Chromatography
- TDS; CHEM-2.5.55 (Rev 0) - Filterable (TDS) Residue

CONSUMERS
ENERGY

Chemistry Department

Standard Analytical Procedure

PROC CHEM-2.5.03
PAGE 1 OF 13
REVISION 4

TITLE: MERCURY ANALYSIS IN WATER SAMPLES BY COLD VAPOR AAS

Written or Revised by ______________________ Date 06/18/2020
Level I or Above

Technical Review/Approval by ______________________ Date 06/18/20
Level III (not author)

Administrative Approval by ______________________ Date 06/18/20
Department Head

TITLE: MERCURY ANALYSIS IN WATER SAMPLES BY COLD VAPOR AAS

1.0 SCOPE

1.1 This procedure is used as the digestion method of aqueous samples for mercury and the analysis by cold vapor atomic absorption spectroscopy (CVAAS). Mobility-procedure extracts, aqueous wastes, surface and groundwater, brackish waters, industrial and domestic wastewaters are all considered applicable matrices for analysis by this SAP.

1.2 This SAP follows the guidelines of EPA Method 7470A.

1.3 A typical Method Detection Limit (MDL) for mercury with this procedure is 0.05µg/L. The practical quantitation level (PQL) or minimum level (ML) is 0.10µg/L.

2.0 APPLICABLE DOCUMENTS AND REFERENCES

2.1 EPA SW-846, Test Methods for Evaluation Solid Waste, Physical/Chemical Methods, Method 7470A, Mercury in Liquid Waste.

2.2 EPA Method 245.1, Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry.

2.3 Chemistry Standard Operating Procedures, as applicable.

2.4 Laboratory Services Quality Assurance Procedures, as applicable.

2.5 Manufacturer’s Instruction Manual for Hot Block and Nippon Mercury Analyzer.

3.0 DEFINITIONS

3.1 Sample Batch – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed, and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

3.2 Laboratory Control Sample (LCS) – A known matrix spiked with compound(s) representative of the target analytes. The control sample should be carried through the complete sample preparation and analytical procedure. This is used to document laboratory performance.

3.3 Laboratory Reagent Blank (LRB) or Method Blank (MB) – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process. For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern should not be higher than the highest of either:

(1) 2.2 x MDL (method detection limit), or
(2) Five percent of the regulatory limit for that analyte, or
(3) Ten percent of the measured concentration in the sample.

3.4 Matrix Duplicate (DUP) – An intra-laboratory split sample undergoing all preparation and analysis steps as the parent sample and which is used to document the precision of the method. The precision (or repeatability) is expressed as % RPD (Relative Percent Difference).

3.5 Matrix Spike (MS) – An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

3.6 Matrix Spike Duplicates (MSD) – Intra-laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

3.7 Method Detection Limit (MDL) – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte.

3.8 ERA – An optional secondary standard, similar or in addition to the ICV (Initial Calibration Verification), purchased from Environmental Resources Associates and provided with a certificate of analysis and acceptance criteria limits by the vendor, per each lot. When a specific ICV standard is not used, the ERA serves as the following:

- Quality Control Sample or QCS, referred in EPA Method 245.1
- Initial Calibration Verification or ICV, referred in SW-846 methods

4.0 SUMMARY OF METHOD

4.1 A known portion of a water sample is transferred to a digestion vessel, digested in a mixture of Sulfuric and Nitric Acid, and oxidized at 95°C in a hot block for 2 hours with potassium permanganate/potassium persulfate.

4.2 After the preparation is complete, the Mercury in the digested sample is reduced with stannous chloride to elemental mercury and measured by conventional cold vapor atomic absorption spectrometry (CVAAS).

4.2 The typical range for mercury with this standard analytical procedure (SAP) is 0.1-10 µg/L.

5.0 PREREQUISITES

5.1 MEASURING AND TEST EQUIPMENT

5.1.1 Hot Block – A heating device capable of maintaining a temperature of 95°C.

5.1.2 Digestion polypropylene vessels, 50mL.

5.1.3 Automatic Eppendorf pipettes with adjustable volumes, 10-100 µL (yellow tips); and 100 - 1,000 µL (blue tips).

5.1.4 CVAAS Mercury Analyzers – Nippon Instruments Corporation, Model RA-3 equipped with an auto sampler, SC-3, reagent dispenser, RD-3, and computerized software control, RA3Win, version 1.1.7. Nippon Instruments Corporation, Model RA-3A equipped with an auto sampler, SC-3, reagent dispenser, RD-3, and computerized software control, RA3000AWin, version 1.1.6.

5.2 REAGENTS

5.2.1 Reagent Water – All reagent water shall be analyte free reagent water. All references to water in this procedure refer to reagent water unless otherwise specified.

5.2.2 Potassium permanganate, $KMnO_4$, 5% solution (w/v) – Dissolve 100 g of potassium permanganate in 2 liters of reagent water.

5.2.3 Sulfuric acid, H_2SO_4, concentrated – Redistilled or Trace Metal grade of low mercury content.

5.2.4 Nitric acid, HNO_3, concentrated – Redistilled or Trace Metal grade of low mercury content.

5.2.5 Potassium persulfate, 5% solution (w/v) – Dissolve 100 g of potassium persulfate in 2 liters of reagent water.

5.2.6 Sodium Chloride-Hydroxylamine Sulfate solution – Dissolve 60 g of sodium chloride and 60 g of hydroxylamine sulfate in reagent water and dilute to 500mL. Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate, preparation of Hydroxylamine hydrochloride remains the same.

5.2.7 Stannous Chloride, 20% w/v – Dissolve 10 g of stannous chloride in 15 ml of concentrated HCl; add 35 ml of reagent water.

5.2.8 Mercury Stock Solution, 1000 ppm – Purchased commercially from manufacturers like High Purity Standards, Inorganic Ventures , or SPEX Industries.

5.2.9 Mercury Working Standards – Add 100µL of the 1,000ppm Hg Stock (5.2.8), 1mL HCl and 1ml of HNO_3 to a 100mL Class A volumetric flask. Bring to volume with reagent water to make a 1 ppm Hg solution. Add 10 mL of the 1 ppm Hg working standard, 1mL HCl and 1ml HNO_3 to a 100mL Class A volumetric flask. Bring to volume with reagent water to make a 0.1 ppm Hg solution. These working standards are prepared fresh, each day when mercury is analyzed.

5.2.10 Solution volumes stated may be adjusted to fit sample load, but solution concentrations must remain as stated.

5.3 CALIBRATION REQUIREMENTS

5.3.1 Dispense 1.5 ml of H_2SO_4 and 0.75 mL of HNO_3 in preparation vessel containing 30 ml of reagent water. For first standard of 0.2 ppb, add 60 µL of 0.1 ppm working standard (Section 5.2.9). Follow the steps in 9.4 – 9.9 to finish digestion of standards along with samples.

5.3.2 To prepare the next set of standards, follow Steps in 5.3.1 using additions of 150 µL, 300 µL, 600 µL of 0.1 ppm working standard, and 150 µL, 300 µL of 1.0 ppm working standard to produce standards of 0.5, 1.0, 2.0, 5.0, and 10 ppb, respectively. A calibration blank is prepared along with the standards in a similar manner, except for the addition of the working standard. Follow steps 9.4 – 9.9 to finish digestion of standards along with samples.

5.3.3 The calibration curve described above would consist of a minimum of 5 standard levels (0, 0.2, 0.5, 1.0, 2.0, and 10.0 µg/L) and must achieve a minimum correlation coefficient of 0.995 in order to be considered valid. The 5.0 ppb standard is used as a mid-point continuing calibration verification standard.

5.3.4 The mid points of the calibration curve cannot be dropped. If a low point is dropped the reporting limit must be increased to the level of the lowest standard included in the curve. If a high point is dropped samples must be diluted within the concentration range of highest point. When permissible changes (as described above) to the calibration curve are necessary, the date and initials of the person making the adjustment, and the reason for the adjustment are to be documented. The change must be approved by the Technical Director (or delegate) before the curve is used to generate results for analytical samples.

5.4 QUALITY CONTROL DOCUMENTS AND RECORDS

5.4.1 The preparation of the samples is recorded in a laboratory notebook, along with the lot numbers and/or solution numbers of all reagents and standards used during digestion. A digestion worksheet (or form similar in intent) can be used as well for this purpose and must have the initials and date of the person completing the preparation on every page. An example of the digestion worksheet is found on the Company network, at “K:\CHEM\Mercury\Mercury”.

5.4.2 Raw data hardcopy printouts.

5.4.3 After the analysis has been completed the results are summarized onto an Excel Reporting Form, which also includes the QA/QC summary for the analytical run, and traceability information for the calibration standards.

5.4.4 When the information in section 5.4.2 is entered directly in LIMS, the Excel Reporting Form is considered optional and its completion is left at the analyst’s discretion.

5.5 PERSONNEL REQUIREMENTS

5.5.1 All tests and data reporting shall be performed by certified persons of Level I or above, in the appropriate discipline. The project report shall be issued and reviewed by a certified person of Level II or above, in the appropriate discipline. The project report if so, indicated on the work request (or form similar in intent), may require approval from a certified person of Level III, in the appropriate discipline.

5.5.2 For projects where reporting under the 2009 TNI Standard has been requested, all tests and data reporting shall be performed by analysts with a completed initial, or an on-going Demonstration of Capability (IDOC/DOC), as applicable.

5.6 ENVIRONMENTAL CONDITIONS

5.6.1 Digestion and the addition of reagents must take place in a fume hood.

5.6.2 Samples must be prepared and analyzed within 28 days of sample collection date, in accordance with CHEM-1.2.02 – Sample Preservation, Hold Time and Containers.

6.0 PRECAUTIONS

6.1 Observe normal safety practices as specified in the latest online revision of the Accident Prevention Manual and the Consumers Energy Chemical Hygiene Plan.

6.2 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Every effort should be made to minimize the generation of excess waste in the preparation of standards and reagents related to this procedure.

6.3 For guidance on proper disposal of unused samples, stock chemicals and reagents refer to SOP CHEM-1.2.08 Handling and Disposal of Lab Testing Waste.

7.0 LIMITATIONS AND ACTIONS

7.1 Sulfide is a possible interference with mercury determination and is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/Kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury.

7.2 Copper has also been known to interfere; however, copper at concentrations as high as 10 mg/Kg has had no effect on recovery of mercury from spiked samples.

7.3 Samples high in chlorides require additional permanganate to eliminate the production of free chlorine from chloride, which interferes with the analysis wavelength of 253.7 nm. Free chlorine is removed by the addition of excess hydroxylamine sulfate.

7.4 Organic compounds which have broadband UV absorbance at/near 253.7 are interferences. When a sample is known to be a solvent, glycol, or thin oil, and is to be analyzed by this method, verify the matrix effect by employing a matrix spike for the suspect sample, in addition to the regular sample aliquot.

7.5 This digestion procedure is not intended for sample preparation and digestion for low-level mercury (below 0.1 ppb).

7.6 Certain volatile organic materials that absorb at the 253.7 nm wavelength may cause interference. A preliminary run at the instrument, without reagents should determine if this type of interference is present.

8.0 ACCEPTANCE CRITERIA

8.1 The frequency of the quality control items, their acceptance criteria and associated corrective action for this method SAP is listed in the table below:

Quality Control Items, Frequency and Corrective Action

QC Item	Frequency	Acceptance Criteria	Corrective Action
ICV	After initial instrument calibration.	90-110% (EPA 7470A) 95-105%(EPA 245.1)	Reanalyze to confirm. If confirmed, reprep and reanalyze batch.
ICB	Immediately following ICV	<2.2 x MDL or <10% of analyte in samples	Reanalyze to confirm. If confirmed, reprep and reanalyze batch.
ERA	Once per analysis.	As the range provided on the certificate of analysis	Reanalyze to confirm. If confirmed, reprep and reanalyze batch. Replace calibration standard(s) if necessary.
LCS	Every batch of 20 samples.	85-115%	Reanalyze to confirm. If confirmed, reprep and reanalyze batch.
LRB	Every batch of 20 samples.	<2.2 x MDL or <10% of analyte in samples	Reanalyze to confirm. If confirmed, reprep and reanalyze batch.
CCV	Before and after each batch. Every 10 samples.	90-110%	All samples following the last acceptable CCV must be reanalyzed after the problem has been identified and corrected.
CCB	Before and after each batch. Every 10 samples.	<2.2 x MDL or <10% of analyte in samples	All samples following the last acceptable CCB must be reanalyzed after the problem has been identified and corrected.
DUP	Every batch of 20 samples.	RPD 0-20%	Reanalyze to confirm. If confirmed, reprep and reanalyze batch.
MS	Every 20 samples. (EPA 7470A) Every 10 samples. (EPA 245.1)	80-120% (EPA 7470A) 70-130% (EPA 245.1)	(EPA 7470A) Reanalyze to confirm. If confirmed, reprep and reanalyze batch. (EPA 245.1) If LCS and CCV's are within limits data is still acceptable. The issue is judged to be matrix related not system related. Otherwise follow instructions for EPA 7470A.
MSD	Same as MS.	Same as MS with RPD 0-20%	Same as MS.

8.2 Method detection limit (MDL) - A mercury MDL must be established using an LRB solution fortified at a concentration of two to three times the estimated detection limit. Refer to the most current EPA Method to determine the MDL, referenced in 40 CFR Part 136. When an MDL is not established, all results are to be evaluated for reporting at the quantitation level, as defined in the applicable TNI standard, typically at the lowest standard level included in the calibration curve.

9.0 PROCEDURE

9.1 Transfer 30 mL sample, or an aliquot diluted to 30 mL with reagent water, by pouring from the sample bottle or by using a pipette, to a 50mL digestion vessel.

NOTE: Reduced or increased volumes or semi-automated versions of this method that use the same reagents and molar ratios are acceptable.

CAUTION: All reagent additions should take place in a fume hood.

9.2 Add 1.5 mL H_2SO_4, conc., and swirl to mix.

9.3 Add 0.75 mL HNO_3, conc., and swirl to mix.

9.4 Add 4.5 mL $KMnO_4$ solution to all samples, in the hood. Swirl to mix and wait 15 minutes before the next step to ensure that the purple color persists. Add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 minutes (record the volume of excess potassium permanganate on the digestion worksheet).

9.5 Add 2.4 mL potassium persulfate and swirl to mix.

9.6 Cap loosely and heat for 2 hours in a hot block at 95°C ± 3°C.

9.6.1 A capped digestion vial, containing about 30mL of reagent water, is randomly placed in a different location within the hot block with each batch during digestion. This capped blank uses a calibrated temperature measuring device to monitor the internal sample temperature. To prevent the sample from boiling, take caution to not exceed internal temperature of 100°C. The vertical columns of the hot block are labeled with ‘A’– ‘F’ and the horizontal rows are labeled with ‘1’– ‘9’. The position used (ex. C10), time, and temperature at the beginning and end of digestion is recorded on the digestion worksheet. The temperature of the hot block is set to 112°C to allow for the internal temperature to be 95°C ± 3°C.

9.7 Remove the vessels and cool.

9.8 Cautionary note: although the strength of the oxidizing agent is verified in section 9.4 above, it’s best when samples have a purple hue after removal from the hot block and before addition of sodium chloride-hydroxylamine sulfate, or hydroxylamine hydrochloride. When a sample does not maintain a purple hue, it could be an indication of an incomplete oxidation. The sample should be considered suspect for matrix effect; re-digestion starting with Section 9.1 might be needed. A diluted aliquot and/or increased potassium permanganate should be considered if the sample is to be re-digested. Record all dilutions, re-digestions, and excess solution added on the digestion worksheet.

9.9 Add 1.8 mL sodium chloride-hydroxylamine sulfate, or hydroxylamine hydrochloride, to reduce excess permanganate. Additional sodium chloride-hydroxylamine sulfate, or hydroxylamine hydrochloride, solution may be required if additional permanganate solution was added (record the volume of any excess solution added).

9.10 Samples are now ready for analysis.

9.11 Standards and Quality Assurance.

9.11.1 Prepare the following, alongside the samples, using Sections 9.1 through 9.9:

- Laboratory Reagent Blank, one per batch of 20 samples.
- Laboratory Control Sample using reagents and mercury standard for a final concentration of 2 µg/L, ppb (or other designated concentration), one per batch of 20 samples.
- Duplicate sample, one per batch of 20 samples if reporting method 7470A or one for every 10 samples if reporting method 245.1.
- Matrix spike and a matrix spike duplicate using the mercury standard for a final added concentration of 2 µg/L, ppb (or other designated concentration). The MS/MSD should be analyzed at a frequency of one per batch of 20 samples if reporting method 7470A, or one for every 10 samples if reporting method 245.1.

  NOTE: The MS/MSD is prepared following the steps in 9.1 through 9.9 above. The sample volume used for MS/MSD shall be equal to the volume used for the parent sample. If only a limited sample volume is available, the duplicate analysis above is optional, and the repeatability is to be evaluated by calculating the % RPD for the MS/MSD pair.

9.12 Calibration and use of Nippon Instruments Reducing Vaporization Mercury Analyzer MERCURY/RA-3.

9.12.1 Turn on power switches. It will take approximately 20 minutes for instrument to warm up.

9.12.2 Prepare the Stannous Chloride (5.2.7) in designated bottle located in holder on the instrument. Prepare 3% v/v HCl (97 mL reagent water and 3 mL HCL) in the designated bottle located in holder on the instrument. Fill Rinse bottle with fresh reagent water. Attach appropriate pump tubing to each bottle.

9.12.3 Once the instrument has warmed up, the main screen will appear on RA-3, use touch screen to select <SC/RD >.

9.12.4 Use arrow button to highlight “1. Measurement Preparation” and press the Enter button. Make sure tubing is in place, press Start/Stop button. Reagents will be injected.

9.12.5 When the injections have finished, press the ESC button to return to the main screen.

- Note: Program/Instrument will not start if not on main screen.

9.12.6 Open RA3Win program on computer.

9.12.7 Enter appropriate Calibration concentrations, starting with blanks and ending with highest concentration, and volume amounts in the STD tab.

9.12.8 Pipette 5 mL of each standard into a clean, dry sample tube and place in the corresponding positions indicated in the program. Check boxes for all standard positions in use, click green Start button in upper left of program.

9.12.9 Once Calibration Curve is acceptable, enter sample information under SMP tab. Pipette 5 mL of each sample into a clean, dry sample tube and place in the corresponding positions indicated in the program. Check boxes for all positions in use, click green start to begin run.

9.12.10 At the end of the run, save the run using a specific name (Ex. AB20-0117), print the calibration and raw data, then select <SC/RD> on the touch screen of RA-3.

9.12.11 Use arrow button to highlight “2. Finishing Measurement”. Press Enter. Follow instructions on screen to rinse and drain reagents from pump tubing.

- Note: There are multiple steps to draining and rinsing pump tubing.

9.12.12 Turn off power switches on rear of RA-3 and SC-3.

9.13 Calibration and use of Nippon Instruments Reducing Vaporization Mercury Analyzer MERCURY/RA-3A.

9.13.1 Turn on power switches on rear of RA-3 and SC-3. It will take approximately 20 minutes for instrument to warm up.

9.13.2 Prepare the Stannous Chloride (5.2.7) in designated bottle located in holder on the instrument. Prepare 3% v/v HCl (97 mL reagent water and 3 mL HCL) in the designated bottle located in holder on the instrument. Fill Rinse bottle with fresh reagent water. Attach appropriate pump tubing to each bottle.

9.13.3 Once the instrument has warmed up, the main screen will appear on RA-3A. Open RA3000AWin program on computer. Click the ‘Run’ tab at the top left of the program. Scroll down to ‘Reagent Operation’ and click on ‘Reagent Filling’. Follow the steps to introducing the reagents prior to analyzing samples.

9.13.4 Follow Section 9.12.7 – 9.12.9 within this procedure to analyze samples.

9.13.5 At the end of the run, save the run using a specific name (Ex. AB20-0117), print the calibration and raw data, then select the ‘Run’ tab at the top left of the program. Scroll down to ‘Reagent Operation’ and click on ‘Reagent Discharge’. Follow the steps to discharge all reagents from the tubing.

9.13.6 Turn off power switches on rear of RA-3 and SC-3.

10.0 CALCULATIONS

10.1 MDL - Calculate the MDL as follows:

$$MDL = t \times S$$

where,
t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t= 3.14 for seven replicates]
S = standard deviation of the replicate analyses

10.2 Percent Recovery – Calculate percent recovery using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

TITLE: MERCURY ANALYSIS IN WATER SAMPLES BY COLD VAPOR AAS

where,
R = percent recovery
C_s = fortified sample concentration
C = sample background concentration
s = concentration equivalent of analyte added to sample

10.3 Relative Percent Difference - Calculate RPD as follows:

$$RPD = \frac{|C_1 - C_2|}{\frac{C_1 + C_2}{2}} \times 100$$

where,
RPD = relative percent difference,
C_1 = first analyte concentration,
C_2 = second analyte concentration.

11.0 DATA REPORTING

11.1 Create an analysis batch in LIMS.

11.2 Generate a .csv file and place it in the parser folder for Mercury, to be transferred electronically into LIMS. Data can also be manually entered into LIMS.

11.3 Print the instrument raw data for the analysis run and forward it along with the sample preparation and QA/QC summary to the person responsible for data review & validation.

CONSUMERS
ENERGY

Chemistry Department

Standard Analytical Procedure

PROC CHEM-2.5.10
PAGE 1 OF 32
REVISION 5

TITLE: METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

Written or Revised by ______________________ Date 06/18/2020
Level I or Above

Technical Review / Approval by ______________________ Date 06/18/20
Level III (not author)

Administrative Approval by ______________________ Date 06/18/20
Department Head

1.0 SCOPE

1.1 Inductively coupled plasma-mass spectrometry[1-2] (ICP-MS) is applicable to the determination of sub-µg/L concentrations of a large number of elements in water samples and in soil, solids, and waste extracts or digests. When dissolved constituents are required, samples are filtered through 0.45 µm membrane filters and acid-preserved prior to analysis. No digestion is needed prior to analysis for dissolved elements in water samples; however, it is recommended that dissolved solids be lower than 0.2% (w/v), in order to reduce potential interferences. Acid digestion prior to filtration and analysis is performed for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are needed. This SAP (Standard Analytical Procedure) follows the guidelines of both, EPA Method 200.8 and SW-846 Method 6020B.

1.2 The elements that can be determined using this SAP are listed below. Typical MDL and reporting limits are listed in Table 1 (Attachment A).

Metal	Symbol	CAS No	Metal	Symbol	CAS No
Aluminum	Al	7429-90-5	Magnesium	Mg	7439-95-4
Antimony	Sb	7440-36-0	Manganese	Mn	7439-96-5
Arsenic	As	7440-38-2	Molybdenum	Mo	7439-95-4
Barium	Ba	7440-39-3	Nickel	Ni	7440-02-0
Beryllium	Be	7440-41-7	Potassium	K	7440-09-7
Boron	B	7440-42-8	Selenium	Se	7782-49-2
Cadmium	Cd	7440-43-9	Silver	Ag	7440-22-4
Calcium	Ca	7440-70-2	Sodium	Na	7440-23-5
Chromium	Cr	7440-47-3	Strontium	Sr	7440-24-6
Cobalt	Co	7440-48-4	Tin	Sn	7440-31-5
Copper	Cu	7440-50-8	Titanium	Ti	7440-32-6
Iron	Fe	7439-89-6	Thallium	Tl	7440-28-0
Lead	Pb	7439-92-1	Vanadium	V	7440-62-2
Lithium	Li	7439-93-2	Zinc	Zn	7440-66-6

1.3 If this procedure is used to determine any analyte not listed in the table above, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the samples to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

1.4 Users of the method data should state the data-quality objectives prior to analysis. Users of the method must document and have on file the required initial demonstration performance data described in Section 11.2 prior to using the method for analysis.

1.5 With the exception of silver, where this method is approved for the determination of certain metal and metalloid contaminants in drinking water, samples may be analyzed directly by pneumatic nebulization without acid digestion if the samples have been properly preserved with acid and have turbidity of <1 NTU at the time of analysis. This total recoverable determination procedure is referred to as "direct analysis".

2.0 APPLICABLE DOCUMENTS AND REFERENCES

2.1 Laboratory Services Quality Assurance Procedures, as applicable.

2.2 Chemistry Department Standard Operating Procedures, as applicable.

2.3 Applicable Metals Sample Preparation Procedure(s).

2.4 Horlick, G., et al., *Spectrochim. Acta 40B,* 1555 (1985).

2.5 Gray, A. L., *Spectrochim. Acta 40B,* 1525 (1985); 41B, 151 (1986).

2.6 Tan, S. H., and Horlick, G., *Appl. Spectrosc. 40,* 445 (1986).

2.7 Vaughn, M. A., and Horlick, G., *Appl. Spectrosc. 40*, 434 (1986).

2.8 Holden, N. E., "Table of the Isotopes," in Lide, D. R., Ed., *CRC Handbook of Chemistry and Physics,* 74th Ed., CRC press, Boca Raton, FL, 1993.

2.9 Hinners, T. A., Heithmar, E., Rissmann, E., and Smith, D., *Winter Conference on Plasma Spectrochemistry,* Abstract THP18; p. 237, San Diego, CA (1994).

2.10 Lichte, F. E., et al., *Anal. Chem. 59,* 1150 (1987).

2.11 Evans, E. H., and Ebdon, L., *J. Anal. At. Spectrom. 4,* 299 (1989).

2.12 Beauchemin, D., et al., *Spectrochim. Acta 42B,* 467 (1987).

2.13 Houk, R. S., *Anal. Chem. 58,* 97A (1986).

2.14 Thompson, J. J., and Houk, R. S., *Appl. Spectrosc. 41,* 801 (1987).

2.15 SW-846, Method 6020A, Revision 1, February 2007.

2.16 EPA Method 200.8, Revision 5.5, 1998.

3.0 DEFINITIONS

3.1 Accuracy – The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

3.2 Batch – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

3.3 Bias – The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike). Thus, the bias (B) due to matrix effects based on a matrix spike is calculated as:

$$B = (x_s - x_u) – K$$

Where:
x_s = measured value for spiked sample
x_u = measured value for unspiked sample
K = known value of the spike in the sample

Using the following equation yields the percent recovery (%R):

$$\%R = 100\,(x_s - x_u)/K$$

3.4 Blank – See Equipment Rinsate, Field Blank, Laboratory Reagent Blank and Method Blank.

3.5 Calibration Blank – A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to calibrate the ICP instrument.

3.6 Calibration Curve or Standard Curve – A plot of concentrations of known analyte standards versus the instrument response to the analyte.

3.7 Calibration Standard – A solution used to calibrate the instrument response with respect to the analyte concentration. Calibration standards are prepared by successively diluting a stock standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section, using the same type of acid or solvent and at the same concentration as the samples to be analyzed, following sample preparation.

3.8 Continuing Calibration Verification (CCV) – A mid-point standard prepared from the same stock solution as the calibration standards, used to check the performance of the instrument and verify that the instrument is properly calibrated on a continuing basis. It is also referred to as the Instrument Check Standard. The CCV standard should be prepared so as to contain metal concentrations equal, or nearly equivalent to the midpoint concentration of the calibration curve.

3.9 Control Sample – A QC sample introduced into a process to monitor the performance of the system.

3.10 Dissolved Analyte – The concentration of analyte in an aqueous sample that will pass through a 0.45 µm membrane filter assembly prior to sample acidification.

3.11 Duplicate (Dup) – See Matrix Duplicate, Field Duplicate and Matrix Spike Duplicate.

3.12 Equipment Blank – See Equipment Rinsate.

3.13 Equipment Rinsate – A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

3.14 Field Blank – An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory or in the field, and treated as a sample in all respects, including shipment to the sampling site, exposure to the sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if method analytes or other interferences are present in the field environment.

3.15 Field Duplicates – Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

3.16 Initial Calibration Verification (ICV) – A standard made from a stock solution obtained from a source different than the one used for the calibration standards, used to verify the validity of the calibration curve. The ICV standard should be prepared so as to contain metals concentrations that are near, but not equal to the midpoint concentration level of the calibration curve.

3.17 Instrument Detection Limit (IDL) – The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 7-10 replicate measurements of the calibration blank signal at the selected analytical masses. The smallest concentration detectable on a specific instrument.

3.18 Internal Standard (IS) – Pure analytes added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

3.19 Laboratory Control Sample (LCS) – A known matrix, solid or liquid, spiked with compound(s) representative of the target analytes. This is used to document laboratory performance. The LCS is analyzed exactly like a sample, carried through all steps of the sample preparation, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. If the matrix used is aqueous, the LCS is equivalent to the Laboratory Fortified Blank (LFB) described below.

3.20 Laboratory Fortified Blank (LFB) – An aliquot of LRB to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

3.21 Laboratory Reagent Blank (LRB) or Method Blank or Preparation Blank – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process. Based on the needs of the project, the concentration in the blank of any analyte of concern should not be higher than either of:

- The method detection limit times 2.2
- Half of the lower limit of quantitation (LLOQ)
- Ten percent of the measured concentration in the sample.

3.22 Linear Dynamic Range (LDR) – The concentration range over which the instrument response to an analyte is linear. The Upper Linear Dynamic Range limit is defined as the concentration where the measured value is within 10% of the actual prepared value of the linear range verification (LRV) standard. An LRV standard is analyzed at the beginning of every analytical run. If a linear verification standard is not employed, all sample results above the highest calibration standard are to be diluted in the working range of the analyzer. Typical analytes included in the LRV standard and their expected concentrations are listed in Table 1 (Attachment A).

3.23 Lower Limit of Quantitation (LLOQ) – A low concentration level determined through the analysis of at least 7 replicate samples spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. The mean recovery should be within 35% of the true value and the RSD should be < 20%.

3.24 Lower Limit of Quantitation (LLOQ) Check Standard – A low concentration level standard (at LLOQ) used to check the performance of the instrument at the LLOQ level. It is also referred to as the low level read-back standard or verification, or blank spike (BS) standard. Typical analytes and their expected concentration at the LLOQ are listed in Table 1 (Attachment A).

3.25 Matrix – The component or substrate (i.e., surface water, drinking water, soil) which contains the analyte of interest.

3.26 Matrix Duplicate (Dup) – An intra-laboratory split sample which is used to document the precision of a method in a given sample matrix.

3.27 Matrix Spike (MS) – An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

3.28 Matrix Spike Duplicates (MSD) – Intra-laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

3.29 Method Detection Limit (MDL) – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte.

NOTE: The MDL is to be performed by following the EPA guidelines approved at CFR 40 Part 1136.

3.30 Precision – The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses. These samples should contain concentrations of analyte above the MDL and may involve the use of matrix spikes. The most commonly used estimates of precision are the relative standard deviation (RSD) or the coefficient of variation (CV), and the relative percent difference (RPD) when only two samples are available.

$$RSD = CV = 100\frac{s}{\overline{x}}$$

Where *x* = the arithmetic mean of the x_i measurements, and S = variance.

$$RPD = 100\left[(x_1 - x_2)/\{(x_1 + x_2)/2\}\right]$$

3.31 Quality Assurance – An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific data collection activity.

3.32 Quality Control Sample (QCS) – A solution of method analytes of known concentrations which is used to fortify an aliquot of Reagent Water and acidified to the same acid type and concentration as present in calibration standards and analytical samples. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is the equivalent of the ICV standard.

3.33 Reagent Blank – See Method Blank, Laboratory Reagent Blank.

3.34 Reagent Grade – Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

3.35 Reagent Water – Water that has been generated by any method which would achieve the performance specifications for ASTM Type 1 water (ASTM 1193D).

3.36 Reference Material – A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

3.37 Rinse Blank – A solution consisting of 2% Nitric Acid in reagent grade water, used to flush the instrument for a predetermined amount of time, between each measurement of standards and/or samples, during the instrument’s “wash” cycle. The purpose of the wash is to ensure that all traces of the analytes from the previous measurement are rinsed out before moving to the next measurement.

3.38 SOP – Standard Operating Procedure, a written document that details the process for an operation, analysis, or action, with thoroughly prescribed techniques and steps, and that is officially approved as the method for performing certain routine or repetitive tasks. Used mainly for administrative procedures.

3.39 SAP – Standard Analytical Procedure, a written document that details the process for an operation, analysis, or action, with thoroughly prescribed techniques and steps, and that is officially approved as the method for performing certain routine or repetitive tasks. Used mainly for analytical procedures.

3.40 Split Samples – Aliquots of sample taken from the same container and analyzed independently. In cases where aliquots of samples are impossible to obtain, field duplicate samples should be taken for the matrix duplicate analysis. These are usually taken after mixing or composting and are used to document intra- or inter-laboratory precision.

3.41 Standard Addition – The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

3.42 Stock Standard Solution – A concentrated solution containing one or more method analytes, purchased from a reputable commercial source, and accompanied by a Certificate of Analysis which provides NIST traceability information.

3.43 Total Recoverable Analyte – The concentration of analyte determined by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion with hot mineral acid(s) as specified in the sample preparation methods.

3.44 Tuning Solution – A solution which is used to determine acceptable instrument performance prior to calibration and sample analyses, based on the specifications of the method and/or manufacturer.

4.0 SUMMARY OF METHOD

4.1 An aliquot of a well-mixed, homogeneous aqueous or solid sample is accurately weighed or measured for sample processing. For total recoverable (“acid-leachable”) analysis of a solid or an aqueous sample containing undissolved material, analytes are first solubilized through the use of appropriate sample digestion procedures (EPA SW-846, Method 3005 – Method 3052, SM 3030E). For the determination of dissolved analytes in a filtered aqueous sample aliquot, or for the “direct analysis” total recoverable determination of analytes in drinking water where sample turbidity is <1 NTU, the sample is made ready for analysis by the appropriate addition of nitric acid, Internal Standard, and dilution to a predetermined volume. The samples should be mixed well before analysis.

4.2 During analysis, sample material in solution is introduced by pneumatic nebulization into radio-frequency plasma where energy transfer processes cause desolvation, atomization and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer having a minimum resolution capability of 0.90 amu peak width at 10% peak height. The ions transmitted through the quadrupole are detected by an electron multiplier or Faraday detector and the ion information processed by a data handling system. Interferences relating to the technique (Section 7.1) must be recognized and corrected. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas reagents or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by the sample matrix must be corrected for by the use of internal standards.

5.0 PREREQUISITES

5.1 MEASURING AND TEST EQUIPMENT

5.1.1 Inductively coupled plasma-mass spectrometers manufactured by Perkin Elmer; **Elan DRC II Standard Mode and NexION** ICP-MS models**.**

5.1.1.1 The Elan is a freestanding mobile bench-mounted ICP mass spectrometer designed for routine trace element analysis. The vacuum system utilizes a 360 L/sec and a 50 L/sec side-mounted turbomolecular pump (only one turbo-pump for Elan DRC II), backed by two conventional roughing pumps. All required electronics, turbomolecular, and roughing pumps are located within the bench housing, which is fitted with doors on all four sides to provide easy access for maintenance and service.

5.1.1.2 A compact, fully automated free-running RF generator powers the ICP at 40 MHz, which provides highly efficient power coupling to the plasma with rapid and responsive electronic autotuning. The operating power and autotuning of the generator is under full computer control and provides an operating range of 600 to1600 watts.

5.1.1.3 A unique PlasmaLok® interface eliminates secondary discharges and stabilizes ion energies, ensuring that optimum performance is maintained across a wide range of samples types and operating conditions.

5.1.1.4 The sample introduction system consists of a cross flow nebulizer with, a quartz chamber inert to most mineral acids and organic solvents, and a fully-demountable torch assembly on a quick-change bayonet mount, with 1.2 mm Injector Tube.

5.1.1.5 The AutoLens ion optics system enables on-the-fly computer controlled lens optimization to generate maximum analyte ion signal with minimum interference.

5.1.1.6 The quadrupole of the ICP-MS systems, driven by a high-frequency 2.5 MHz power supply, was carefully designed to produce a perfect hyperbolic field for optimum resolving power and ion transmission efficiency, producing an abundance sensitivity of greater than 10^6 that helps minimize spectral interferences. The combination of the thermally stabilized quadrupole power supply and the state-of-the-art binary, gold-metallized ceramic roads offers exceptional mass calibration stability.

5.1.1.7 A dual-stage discrete dynode electron multiplier provides detection over a full eight orders of magnitude of dynamic range.

5.1.1.8 The instruments are controlled by the Elan and NexION software. The software, in conjunction with the System Controller, continuously monitors over 70 test points of the instrument to ensure that if a failure should occur, the system will alert the operator or, if necessary, shut the instrument down safely.

5.1.2 S-10 Auto-samplers.

5.1.3 PolyScience, Model 6105PE Cooling Units.

5.1.4 Argon gas supply: 160 L Dewar of high purity liquid Argon.

5.1.5 Digital bottle top dispenser capable of dispensing volumes of 0.2 mL in 0.01 mL increments.

5.1.6 Automatic pipette with disposable 2.50 mL combitips capable of dispensing volumes from 100 to 500 µL.

5.1.7 Automatic pipettors with adjustable volumes, 10-100 µL (yellow tips); 100-1,000 µL (blue tips).

5.1.8 Polypropylene vessels, 50 mL.

5.1.9 Volumetric flasks of various volumes, 10 mL to 1 L.

5.2 REAGENTS

5.2.1 Nitric Acid (HNO_3), concentrated, Trace Metal Grade. Acids used in the preparation of standards and for sample processing must be of high purity. Trace metal grade (also known as re-distilled) acids are recommended because of the high sensitivity of ICP-MS. Nitric acid at 2% (v/v) or less in the solution to be analyzed is required for the ICP-MS, in order to minimize damage to the interface.

5.2.2 Hydrochloric Acid (HCl), concentrated, Trace Metal Grade. Several polyatomic ion interferences result when HCl is used. However, its use is recommended to maintain stability in solutions containing high concentrations of antimony and silver. When used, corrections for the chloride polyatomic ion interference must be applied to all data. At the same time, the standards used for calibration and/or calibration verification should contain the same percentage of HCl as the samples to be analyzed.

5.2.3 Reagent Water (Deionized Water). All references to reagent water in the method refer to ASTM Type I water (ASTM D1193), unless otherwise specified.

5.2.4 Internal Standard Stock Solutions:

- Lithium 6, 1,000 µg/mL stock solution
- Scandium, 1,000 µg/mL stock solution

- Germanium, 1,000 µg/mL stock solution
- Rhodium, 1,000 µg/mL stock solution
- Indium, 1,000 µg/mL stock solution
- Rhenium, 1,000 µg/mL stock solution

5.2.5 Internal Standard Working Solution (IS-WS) – From the above stock solutions, 2.0 mL of each is transferred to a 1000 mL polyethylene container, along with 20 mL of concentrated HNO_3 and brought to a final volume with 970 mL of reagent water. The concentration in the flask will be 2.0 mg/L. This represents the internal standards working solution from which 0.5 mL (for a 50 mL final volume) will be added to all calibration standards and blanks. This will provide a 20 µg/L internal standard concentration in all calibration standards, similar to the concentration of internal standards in analytical samples. The appropriate volume of the IS-WS is to be added to samples during sample preparation or dilution, in order to have the same concentration of internal standard present in samples to be analyzed.

5.2.6 SPEX Industries (S) (or like) – Multielement standard solution containing 100 µg/mL each of Al, Sb, As, Ba, Be, Bi, B, Cd, Cr, Co, Cu, Fe, Pb, Li, Mn, Mo, Ni, Se, Ag, Sr, Sn, Ti, Tl, V, Zn. This solution is used for the preparation of the initial calibration verification (ICV) standard. Pertaining information is recorded in the Standard Preparation Log (see Attachment B, as example only; the most current form is maintained in a dedicated folder on the Company network).

5.2.7 SPEX Industries (SM) (or like) – Multielement standard solution containing 1,000 µg/L each of Ca, Mg, K, and Na. This solution is used for the preparation of the initial calibration verification (ICV) standard. Pertaining information is recorded in the Standard Preparation Log (Attachment B, as example only; the most current form is maintained in a dedicated folder on the Company network).

5.2.8 High Purity Standards – Solution B (HB) – 1,000 µg/mL each of Ca, Mg, K, Na. This solution is used for the preparation of the calibration and for the continuing calibration verification (CCV) standards. Pertaining information is recorded in the Standards Preparation Log (see Attachment B). The solution is also used for the preparation of the Laboratory Control Sample (LCS), and for spiking of the MS/MSD samples, during sample preparation. Pertaining information is recorded in the Sample Preparation Log (see Attachment C, as example only; the most current form is maintained in a dedicated folder on the Company network).

5.2.9 High Purity Standards – Solution A (HA) – 100 µg/mL each of Al, Sb, As, Ba, Be, Bi, B, Cd, Cr, Co, Cu, Fe, Pb, Li, Mn, Mo, Ni, Se, Ag, Sr, Sn, Ti, Tl, V, Zn. This solution is used for the preparation of the calibration standards and for the continuing calibration verification (CCV) standard. Pertaining information is recorded in the Standards Preparation Log (see Attachment B). The solution is also used for the preparation of the Laboratory Control Sample (LCS), and for spiking of the MS/MSD samples, during sample preparation. Pertaining information is recorded in the Sample Preparation Log (see Attachment C, as example only; the most current form is maintained in a dedicated folder on the Company network).

5.2.9.1 High Purity Standards – Working Solution (HA-WS) – 10 µg/mL each of the elements listed in Section 5.2.9. Prepare this solution, as needed, by transferring 5.0 mL of the HA stock solution to a 50 mL polypropylene tube, add 1 mL of HNO_3 and dilute to volume with reagent water in the hood. The solution is used for the preparation of standards and/or daily spiking of LCS/MS/MSD samples, as needed. Record the preparation information in the ICP-MS Metal Standards Preparation Log and label the working solution vial with the ID of the standard, preparation date, expiration date and initials of the preparer.

5.2.10 High Purity Standards – LLOQ stock standard solution – 2.0 µg/mL each of B, Fe; 1.0 µg/mL each of Al, Zn; 0.1 µg/mL each of Sb, As, Ba, Be, Bi, Cr, Co, Cu, Pb, Li, Mn, Mo, Ni, Se, Ag, Sr, Sn, Ti, Tl, V; 0.02 µg/mL each of Cd, and Ag. This solution is used for the preparation of the lower level of quantitation standard (LLOQ).

5.2.11 High Purity Standards – LRV stock standard solution – 1000 µg/mL each of As, Ba, B, Cr, Co, Cu, Fe, Pb, Mn, Mo, Ni, Sr, Zn. This solution is used for the preparation of the liner range verification standard.

NOTE: The stock solutions are NIST traceable and provided with a certificate of analyses and MSDS sheets by the vendor. A receipt inspection is performed once the standards are received at the Laboratory, following the steps defined in the administrative procedures and LSQA-05, Procurement Control.

NOTE: Exercise caution when analytes, that have low concentrations in most samples, cause the instrument to switch into analog mode due to high signals or currents. Be aware that a fresh dual detector calibration may be needed to ensure linearity.

5.3 CALIBRATION REQUIREMENTS

5.3.1 Multielement calibration standard solutions are prepared by diluting the stock standard solutions to levels in the linear range for the instrument in a solvent consisting of 2% (v/v) HNO_3 in reagent water. The calibration standard solutions must contain a suitable concentration of an appropriate internal standard for each analyte. The calibration standards are kept in polypropylene vessels (50 mL) and prepared as needed. They must be verified by the analysis of a quality control standard (ICV). Tables 2, 3, and 4 (Attachment A) can be used as guidance, when preparing standards.

- A calibration curve must be analyzed daily. The instrument may be calibrated using a single point standard and a calibration blank, or a multipoint calibration curve.

- For a multilevel calibration curve, a minimum correlation coefficient of 0.995 is required in order for the curve to be considered valid. Generally, a 0.9999 correlation coefficient is achieved for the majority of elements. The lowest standard point must be at or below the LLOQ.

- The mid points of the calibration curve cannot be dropped. If a low point is dropped the reporting limit must be increased to the level of the lowest standard included in the curve. If a high point is dropped samples must be diluted within the concentration range of highest point. When permissible changes (as described above) to the calibration curve are necessary, the date and initials of the person making the adjustment, and the reason for the adjustment are to be documented. The change must be approved by the Technical Director (or delegate) before the curve is used to generate results for analytical samples.

- After the initial calibration is completed, it is verified through the analysis of an initial calibration verification standard (ICV) and an initial calibration blank (ICB). A low-level readback or verification is used to validate the quantitation level. For a multi-point calibration, the low level standard (included as the lowest point in the calibration curve) should quantitate to within 80-120% of the true value. For a single point calibration, a standard from the same source as the calibration standard, and at/or below the LLOQ is analyzed and should recover within 80-120% of the true value.

5.3.2 Blanks – Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank (LRB) is used to monitor possible contamination resulting from the sample preparation procedure. The rinse blank (also called optional rinse or autosampler wash) is used to flush the system between all samples and standards.

5.3.2.1 The calibration blank and the continuing calibration blank (CCB) consists of the same concentration(s) of the same acid(s) used to prepare the calibration standards, along with the appropriate concentration of internal standard.

5.3.2.2 The preparation (or reagent) blank (LRB) must be carried through the complete preparation procedure and contain the same volumes of reagents as the sample solutions.

5.3.2.3 The rinse blank consists of reagent water only, and is used to flush the system between standards and samples.

5.3.3 The interference check solution (ICS) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. Chloride in the ICS provides a means to evaluate software corrections for chloride-related interference such as $^{35}Cl^{16}O^+$ on $^{51}V^+$ and $^{40}Ar^{35}Cl^+$ on $^{75}As^+$. Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits.

5.3.3.1 Interference check **stock** solution A, containing 1,000 µg/mL each of Al, Mg, P, K, S, 2,000 µg/mL of C, 2,500 µg/mL each of Fe and Na, 3,000 µg/mL of Ca, 21,215 µg/mL of Cl, and 20.0 µg/mL each of Mo and Ti. The ICS-A solution is prepared by transferring 5 mL of the stock solution in a 50 mL polypropylene vessel, followed by the addition of 0.5 mL IS-WS, 1 mL HNO_3, and dilution to 50 mL with reagent water.

5.3.3.2 Interference check **stock** solution B, containing 20.0 µg/mL each of Cr, Co, Cu, Mn, Ni, and V, 10.0 µg/mL each of As, Cd, Se, Zn and 5.00 µg/mL of Ag.

5.3.3.3 The interference check working solution ICS-AB is prepared by transferring 5 mL of the ICS A stock solution into a 50 mL polypropylene vessel, 0.5 mL of the ICS B stock solution, followed by the addition of 0.5 mL IS-WS, 1 mL HNO_3, and dilution to 50 mL with reagent water. The final concentration of the elements in ICS-A and ICS-AB, after the appropriate dilution from the stock, is listed in Attachment A, Table 6. The concentrated stock solution A and B are generally purchased form commercial vendors like Inorganic Ventures, Inc. and come with an expiration date of 1 year. Within the 1 year expiration interval, the diluted solutions A, B and AB are prepared as needed.

5.3.4 The quality control standard is the initial calibration verification solution (ICV), which must be prepared in the same acid matrix as the calibration standards. This solution must be an independent standard near (but not equal to) the midpoint of the calibration curve, at a concentration other than that used for instrument calibration. An independent standard is defined as a standard from a source different from those used in the standards for instrument calibration (see Sections 5.2.6 and 5.2.7).

5.3.5 Mass spectrometer tuning solution – A solution containing elements representing all of the mass regions of interest must be prepared to verify that the resolution and mass calibration of the instrument are within the required specifications (see Section 9.4). This solution is also used to verify that the instrument has reached thermal stability (see Section 9.5).

5.3.5.1 Tuning solution, 10 ppb – In a 1 L volumetric flask, add approximately 200 mL of reagent water, then pipette 10 µL of the 1,000 ppm stock solutions for Indium, Cerium, Rhodium, HPS Solution B, and 100 µL of the 100 ppm HPS Solution A. Add 20 mL of concentrated Nitric Acid, in the hood, and dilute to the 1 L mark. This solution is to be used for daily verifications of the performance of the instrument and mass calibration, and periodical calibration of the static lens and auto-lens of the Elan system.

5.3.5.2 Tuning Solution, 200 ppb – In a 1 L volumetric flask, add approximately 200 mL of reagent water, then pipette 2 mL of the 100 ppm HPS Solution A, and 0.2 mL of HPS Solution B. Add 20 mL of concentrated Nitric Acid, in the hood, and dilute to the 1 L mark. This solution is to be used for the calibration of the Dual Detector, every time the voltage on the detector (analog and/or pulse) has been changed, or when a new detector is installed.

5.4 QUALITY CONTROL DOCUMENTS AND RECORDS

5.4.1 **Documents and Records**

5.4.1.1 Keep hard copies and electronic copies of raw data in accordance with LS (Laboratory Services) record retention policy.

5.4.1.2 Back up all files associated with a given run, either on CD/DVDs, external hard drive or on the Company network, as appropriate.

5.4.1.3 Keep electronic records of mass calibration and daily performance and generate hard copies to aid in the peer data review.

5.4.1.4 The printouts of the mass calibration, daily performance results, Sample/Batch Report and Data Set Report are submitted for secondary review. They do not need to become part of the project folder, since the original electronic versions exist. However, it is good practice to scan and archive all of the original hard copy data, including forms that might not necessarily become part of the project folder.

5.4.1.5 Record all maintenance performed on the instrument in the instrument maintenance book, or electronically, in either a spreadsheet summary, or in the comments section available when performing daily instrument performance verifications/optimizations.

5.4.2 **Tables and Forms**

5.4.2.1 Attachment A

- Table 1 List of elements analyzed.
- Table 2 Standard preparation for all elements except Fe, Ca, Mg, K, Na.

- Table 3 Standard preparation for Fe.
- Table 4 Standard preparation for Ca, Mg, K, Na.
- Table 5 Instrument operating parameters.
- Table 6 ICS components and concentration.
- Table 7 Recommended analytical isotopes and additional masses to be monitored.
- Table 8 Quality control items, frequency, and corrective action.

5.4.2.2 Attachment B – Metal Standards Preparation Log (as example only; the most current form is maintained in a dedicated folder on the Company network).

5.4.2.3 Attachment C – Samples Preparation Log (as example only; the most current form is maintained in a dedicated folder on the Company network).

5.4.2.4 Attachment D – Peak Width Relationship at 5% and 10% Height

5.5 PERSONNEL REQUIREMENTS

5.5.1 The use of this procedure is restricted to those analysts experienced in the use of Inductively Coupled Plasma Mass-Spectrometry and who are knowledgeable in the recognition and in the correction of spectral, chemical and physical interferences in ICP-MS. A minimum of six months experience with commercial instrumentation is recommended.

5.5.2 For projects where reporting under the 2009 TNI Standard has been requested, all tests and data reporting shall be performed by analysts with a completed initial, or an on-going Demonstration of Capability (iDOC/DOC), as applicable.

5.6 ENVIRONMENTAL CONDITIONS

5.6.1 The instrument shall be vented to a fume hood or appropriate device.

6.0 PRECAUTIONS

6.1 Observe normal safety practices as specified in the latest online revision of the Environmental and Laboratory Services Accident Prevention Manual and the Consumers Energy Chemical Hygiene Plan.

6.2 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Every effort should be made to minimize the generation of excess waste in the preparation of standards and reagents related to this procedure.

6.3 For guidance on proper disposal of unused samples, stock chemicals and reagents, refer to SOP CHEM-1.2.08 Handling and Disposal of Lab Testing Waste.

7.0 LIMITATIONS AND ACTIONS

7.1 Interferences – There are three fundamentally different sources of interference in ICP-MS: spectroscopic interferences, physical, and memory interferences.

7.1.1 Spectroscopic Interferences – Interferences caused by the presence of compounds or elements entering the mass spectrometer that have the same nominal mass to charge (m/z ratio) as the analyte elements. They can be isobaric elemental and isobaric molecular interferences (polyatomic, refractory oxide, and doubly charged ions).

7.1.2 Isobaric Elemental Interferences in ICP-MS – Caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/Z) as the analyte element. These can be managed by the selection of an alternate isotope for analysis or by the use of elemental interference equations. These equations use the naturally occurring isotope ratios of most elements to estimate and allow for the subtraction of isobaric interferences. An example of an elemental isobaric interference is ^{40}Ar on ^{40}Ca. In this case, the use of ^{43}Ca or ^{44}Ca is recommended. The appropriate elemental interference equations are incorporated in the methods (or parameter) used for calibration and data acquisition.

7.1.3 Isobaric Molecular and Doubly Charged Ion Interferences in ICP-MS – Caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that affect ICP-MS determinations have been identified[3-4]. Examples include $ArCl^+$ ions on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundance[5], the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1%) counting statistics.

7.1.3.1 Example for As – Because the ^{35}Cl natural abundance of 75.77% is 3.13 times the ^{37}Cl abundance of 24.23%, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06% of the $^{40}Ar^{35}Cl^+$ signal): Corrected arsenic signal (using natural isotopes abundance for coefficient approximations) = m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal), (where the final term adjusts for any selenium contribution at 77 m/z).

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (eg, $^{81}BrH^+$ from bromine wastes[6]).

NOTE: When Selenium concentrations are high, the correction listed above is not always accurate, and it can also lead to a high Arsenic bias. The effect is expected to be even more pronounced in matrices where Chloride is present at low concentrations. The analyst must always compare corrected versus uncorrected Arsenic results to identify if a bias is present. When a high bias occurs, the lower concentration result from the uncorrected Arsenic is to be used for data reporting, provided all other method required QA/QC is found acceptable.

7.1.3.2 Example of Cd – Corrected cadmium signal (using natural isotopes abundance for coefficient approximations) = (m/z 114 signal) - (0.027)(m/z 118 signal) - (1.63)(m/z 108 signal), (where last 2 terms adjust for any Sn or MoO^+ contributions at m/z 114).

NOTES: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108, but use of m/z 111 for Cd is even subject to direct ($^{94}ZrOH^+$) and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

Since there is a certain degree of uncertainty as to which equation is better to use, and in what cases, it is up to the analyst to determine how the interference will be corrected, upon evaluation of data. It is suggested that the elemental isobaric interference equations be included in all methods (parameters) at all times, but potential polyatomic species (masses) that could interfere by only monitored. The Elan Data System allows the user to display both type of results, corrected for interference and uncorrected, on a single Quantitative Analysis Summary Report. Corrected elements should be flagged, to distinguish them from the uncorrected ones. Generally, an interference is easy to spot when multiple isotopes of an element show significantly different results. Since the interference is additive, the use of the isotope with the lowest result is suggested for data reporting, providing that all other QC criteria are met.

7.1.4 Abundance Sensitivity – A property defining the degree to which the wings of a mass peak contribute to adjacent masses. Wing overlap interference may occur when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized, and the spectrometer resolution adjusted to minimize them. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require matrix separation, or analysis using another verified and documented isotope.

7.1.5 Physical Interferences – Associated with the physical processes, which govern the transport of sample into the plasma, sample conversion process within the plasma and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the samples and calibration standards.[9] Physical interferences may occur in the transfer of solution to the nebulizer (eg, viscosity effects), at the point of aerosol formation and transport to the plasma (eg, surface tension effects), during the atomization and ionization process within the plasma itself, or during the transfer of ions through the interface and mass spectrometer (space charge effects). To minimize some of these effects, acid composition and concentration should be matched for all standards, blanks, and samples. Total solid levels below 0.2% (2,000 mg/L) have been currently recommended[10] to minimize solid deposition. Internal standardization may be effectively used [11] to compensate for many physical interference effects. Internal standards should ideally display similar analytical behavior to the elements being determined. Generally, an internal standard should be no more than 50 amu removed from the analyte. However, instances were observed when high levels of easily ionized elements (like Na) in the samples lead to abnormal suppression of the low mass internal standard (ie, ^{45}Sc). As a result, the elements close in mass to 45 amu, which have Scandium assigned as the internal standard would be biased high. A heavier element like ^{103}Rh should be used in those cases as the internal standard. Recommended internal standards include ^{6}Li, ^{45}Sc, ^{70}Ge, ^{103}Rh, ^{115}In, ^{159}Tb, ^{169}Ho, ^{185}Re, and ^{209}Bi.

7.1.6 Memory Interferences – Result when elements in a previous sample contribute to signals measured in a subsequent sample. Memory effects can result from the deposition of sample on various components of the sample introduction system, including sample and peristaltic pump tubing, spray chamber, torch, and interface cones. The site(s) where deposition may occur is dependent on the sample and may need to be minimized through the use of a rinse blank between samples. Routine maintenance (cleaning and/or replacement) of sample introduction components is necessary for long-term minimization of memory effects. The possibility of memory interferences within an analytical run should be recognized and suitable rinse times should be used to reduce them. Memory effects are evaluated by using a minimum of three replicate integrations for data acquisition. High relative standard deviation (% RSD) of the three replicates caused by a consecutive drop in signal intensity is indicative of carryover from the previous sample. If memory interference is suspected, the sample should be reanalyzed after analysis of a blank, which indicates that the carryover has been eliminated.

8.0 ACCEPTANCE CRITERIA

8.1 The initial demonstration of performance is used to characterize instrument performance (determination of linear calibration ranges) and laboratory performance (determination of method detection limits) prior to analyses conducted by this method.

8.2 Linear calibration ranges – Linear calibration ranges are primarily detector limited. The upper limit of the linear calibration range should be established by determining the signal responses from different concentration standards, one of which is close to the upper limit of the linear range. Care should be taken to avoid potential damage to the detector during this process. The linear calibration range, which may be used for the analysis of samples, should be judged by the analyst from the resulting data. The upper LDR is defined as the maximum concentration for which the measured concentration is within ±10% of the true value. Sample analyte concentrations that are greater than the upper LDR limit must be diluted and reanalyzed. The LDR should be verified whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be re-determined.

NOTE: The linear range establishes the highest concentration that may be reported without diluting the sample. A liner range standard is analyzed at the beginning of every analytical run, following the calibration. If a linear range standard is not analyzed for any specific element, or if analyzed but fails to be recovered within 90-110% of the true value, the highest concentration standard in the calibration becomes the linear range. For analytes with concentration above the linear range, the samples shall be diluted in the working range of the instrument.

NOTE: When the liner range standard is not analyzed for a particular analyte, an alternative approach that verifies the linear range is to dilute the actual sample with measured concentrations above the highest calibration standard. If the analyte concentration of a sample diluted to read below the highest calibration standard is within 10% of the initial, undiluted result, the response of the instrument is verified to be linear up the concentration of the undiluted sample.

8.3 Method detection limits (MDL) must be established for all analytes, using reagent water (blank) fortified at a concentration of 2 to 10 times the estimated detection limit. To determine MDL values, follow the EPA procedure listed at 40 CFR Part 136. MDL’s should be evaluated annually, when a new operator begins work, or whenever, in the judgment of the analyst, a change in the instrument hardware or operating conditions would dictate they be redetermined.

8.4 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantification or the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferant itself, but that a molecular species may be monitored to indicate the presence of the interferant. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon, and sulfur molecular ions could impact the analytes of interest. If an interference source is present, and cannot be corrected, the sample elements impacted must be flagged. When correction equations are used, all QC criteria must also be met.

8.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard falls outside limits as compared with the calibration blank, the following procedure is followed: the sample must be diluted at least fivefold (1 + 4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal-standard intensities fall within the prescribed window. The intensity levels of the internal standards for the ICV/ICB, CCV/CCB, LCS/LRB must also be within the specified acceptance limits (refer to Section 8.11.1.3 and 8.11.2.5 for limits). If they are not within limits, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.6 Check the instrument calibration by analyzing appropriate quality control solutions as follows:

8.6.1 Check instrument calibration by analyzing the initial calibration verification solution (ICV), initial calibration blank (ICB), and the LLOQ verification standard.

8.6.2 Verify calibration at a frequency of every 10 analytical samples with the CCV standard, and the continuing calibration blank (CCB). These solutions must also be analyzed for each analyte at the beginning of the sample batch and after the last sample.

8.6.3 The results of the ICV and CCV must agree within ±10%, and the LLOQ standard must agree within ±20% of the expected value. If the ICV or LLOQ are outside limits, terminate the analysis, correct the problem, and recalibrate the instrument. If the CCV’s are outside the limits, address the problem according to the requirements of the method to be reported (200.8 or 6020). See Section 8.11.1.1 and 8.11.2.1 or Attachment A, Table 8 for the appropriate corrective action.

8.6.4 The results of the ICB and CCBs must be less than 2.2 times the current MDL for each element or less than the reporting limits for water samples, whichever is greater. With the exception of Ca, Mg, K, Na, Fe, B, Al, Cu, Ni, and Zn, which have fairly high reporting limits, for most of the analytes, a blank level of ≤1 ppb is acceptable, even though higher than 2.2 x MDL in some cases. An exception is Cd and Ag, where the blank level should be < 0.2 ppb, at a minimum. Care must be used when evaluating the blank so that sample results are not impacted by a high blank level. It is also important to take into account the dilution factor associated with sample analysis, which could further exacerbate the problem. If the limit for ICB/CCBs is exceeded, the reason for the out-of-control condition must be found and corrected, and the affected samples must be reanalyzed.

8.7 A Laboratory Control Sample (LCS) must be analyzed for each analyte using the same sample preparations, analytical methods, and QA/QC procedures employed for the test samples. One LCS must be prepared and analyzed for each sample batch at a frequency of one LCS for each 20 samples or less. The recovery limits for the LCS are 85-115% for Method 200.8 and 80-120% for Method 6020, of the true value (stated in the sequence log or calculated from the volume and concentration of stock solution recorded on the digestion worksheet). If the limits are exceeded, the samples in the associated analytical batch must be prepared again and re-analyzed. If the analysis must continue under the out-of-control condition, the results shall be flagged in the data report.

8.8 The laboratory must analyze at least one LRB with each batch of 20 samples. LRB data are used to assess contamination from the laboratory environment and to characterize spectral background from the reagents used in sample processing. LRB values that exceed the MDL indicate that laboratory or reagent contamination should be suspected. When LRB values are above ½ LLOQ, constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL, whichever is greater, the source of contamination must be corrected and samples in the associated analytical batch must be prepared again and re-analyzed. If the analysis must continue under the out-of-control condition, the results shall be flagged in the data report.

8.9 Analyze matrix spike (MS) samples with every batch. For majority of the elements, the aqueous samples are spiked at levels similar to the LCS (50 ppb in the analysis solution for all elements except Ca, Mg, K, Na, which are spiked usually with 1 ppm in the analysis solution). Soils or solid samples can have spike concentrations that vary proportional to the expected analyte level in the samples. Calculate the percent recovery of each analyte, corrected for background concentrations appropriate to the matrix, using the following equation:

$$R = \frac{C_s - C}{S} x100$$

Where: R = percent recovery
C_s = spiked sample concentration
C = sample background concentration
S = concentration equivalent of analyte added to fortify the sample

8.10 Analyze matrix duplicate (Dup) samples with every batch. A matrix spike duplicate (MSD) can be used instead or in addition to the matrix duplicate. The decision on whether to prepare and analyze duplicate samples or an MS/MSD pair must be based on knowledge of the sample in the analysis batch. If samples are expected to contain target analytes above the LLOQ, a duplicate sample could be used. If samples are not expected to contain target analytes above the LLOQ, an MS/MSD pair should be used, to avoid meaningless RPD values. A control limit of 20% RPD should not be exceeded for analyte values measured above LLOQ. If this limit is exceeded and laboratory performance for that analyte is shown to be in control (ICV/ICB, CCV/CCB, and LCS/LRB within the limits), the problem encountered is judged to be matrix related. Reanalyze at the instrument the sample and its duplicate (or MSD) in order to confirm the out-of-control result. Based on project specific requirements, the samples are to be redigested and reanalyzed, or the data user is to be informed that the result for that analyte is suspect due to the heterogeneous nature of the sample. If the performance of the laboratory is not in control (ICV/ICB, CCV/CCB, and/or LCS/LRB outside the limits), the reason for the out-of-control situation must be found and corrected, and any samples analyzed during the out-of-control condition for that analyte must be reanalyzed.

The relative percent difference (RPD) between duplicate (or MSD) determinations must be calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} x100$$

Where: RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (duplicate)

8.11 The quality control requirements and limits vary slightly, based upon the method referenced in the analytical report (i.e., 200.8 vs 6020). For both methods, the calibration is verified through the analysis of ICV/ICB and CCV/CCB. Recalibration is required when either one falls outside the limits. The performance of the method is evaluated by the analysis of the LCS/LRB, and MS/MSD/Dup samples for every batch.

8.11.1 Method 200.8 Specific Requirements

8.11.1.1 When the recovery for ICV/CCV falls outside ±10% terminate the analysis and recalibrate the instrument. If the last CCV was within 15% of the true concentration, the results for the samples are still acceptable. If this is not the case, the only acceptable results are the ones corresponding to samples analyzed before the last CCV that was within 15% of the true concentration. All other samples are to be analyzed again, after recalibration of the instrument.

8.11.1.2 Matrix Spike samples are to be analyzed for every 10 samples or two sets for a batch of 20 samples. The recovery limits for MS samples are 70-130%. If the recovery of any analyte falls outside the designated range and the laboratory performance is shown to be in control (ICV/ICB, CCV/CCB, and LCS/LRB within the limits), the recovery problem encountered with the spiked sample is judged to be matrix related, not system related. The data user must be informed that the result for the analyte in the unspiked sample is suspect due to an uncorrected matrix effect. Recovery is not required if the concentration of the analyte added is less than 30% of the concentration of the analyte in the original sample.

8.11.1.3 The absolute response of any one internal standard must not deviate by more than 60 to 125% of the original response in the calibration blank. If deviations greater than these are observed flush the instrument with rinse blank, than analyze a CCB. If the responses of the internal standards are now within the limit proceed with sample dilution as described in Section 8.4. If the responses of the internal standards are not within the limit, terminate the analysis, recalibrate the instrument, and reanalyze the samples from the last CCB with acceptable internal standard recoveries.

8.11.2 Method 6020 Specific Requirements

8.11.2.1 When the recovery for ICV/CCV falls outside ±10% or LLOQ falls outside ±20% of the true value terminate the analysis and recalibrate the instrument. The samples from the last CCV that was within limits are to be reanalyzed, after recalibration of the instrument.

8.11.2.2 Post-Digestion Spike Addition – An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125% of the known value. The spike addition should be based on the indigenous concentration of each element of interest in the sample. If the spike is not recovered within the specified limits, the original sample must be diluted to compensate for the matrix effect, and reanalyzed, after a new post-digestion spike is added. The same recovery limits apply to the spiked dilution.

8.11.2.3 Digested matrix spike (MS); if the digested spike is not recovered within 75-125%, either because of matrix effects or because the sample background for the analyte is too high, follow 8.11.2.2 above and analyze a post-digested spike (also referred to as “know addition” / KA).

8.11.2.4 Matrix spike duplicate, MSD, for either the Post-Digestion Spike Addition, or the digested spike duplicate; the evaluation of the MSD is similar to the evaluation of the duplicate analysis described in Section 8.10 except that for soil samples the RPD calculation should be performed on the percent recoveries for both, the MS and MSD, in order to account for the different dilution factor resulting from the sample preparation.

8.11.2.5 When the intensity of any internal standard in the sample falls outside 30-130% of the intensity of that internal standard in the calibration blank, follow the procedure described in Section 8.5. The intensity levels of the internal standards for the ICV/ICB and CCV/CCB must agree with 60-125% of the intensity level of the calibration blank. If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.11.2.6 Dilution Test – If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 25 x LLOQ), an analysis of a fivefold (1 + 4) dilution must agree within ±20% of the original determination. If a high concentration sample is not available for performing the dilution test, a post digestion spike could be used instead. The matrix spike is often a good choice of sample for the dilution test, since reasonable concentrations of most analytes are present. One dilution test must be included with every batch of 20 samples. Elements that failed the dilution test are to be flagged.

8.11.2.7 Verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours of continuing sample analysis, whichever is more frequent. Do this by analyzing the interference check solutions ICS-A and ICS-AB. Results for the unspiked elements in the ICS-A should be less than 2 x LLOQ. The recovery of the spiked/elements of interest in ISC-AB (listed in Attachment A, Table 6) should be between 70-130%.

8.12 Summary of the QC requirements and performance acceptance limits are shown in the following table:

	Method 6020		**Method 200.8**	
QC Type	**Limit %**	**Frequency**	**Limit (%)**	**Frequency**
ICV	90-110	After initial calibration	90-110	After initial calibration
ICB	< ½ x LLOQ	After initial calibration	< ½ x LLOQ	After initial calibration
LLOQ/BS	80-120	After initial calibration*	70-130	After initial calibration*
ERA‡	Variable per lot	After initial calibration	Variable per lot	After initial calibration
CCV	90-110	Every 10 samples	90-110**	Every 10 samples
CCB	< ½ x LLOQ	Following CCV	< ½ x LLOQ	Following CCV
LCS	80-120	Every batch or 20 samples	85-115	Every batch or 20 samples
LRB	< ½ x LLOQ or <10%***	Every batch or 20 samples	< ½ x LLOQ or <10%	Every batch or 20 samples
Dup	0-20	Every batch or 20 samples	0-20	Every 10 samples
MS	75-125	Every batch or 20 samples	70-130	Every 10 samples
MSD	0-20	Every batch or 20 samples	0-20	Every 10 samples
ICS A	< ½ x LLOQ	Beginning of run or 12 hr	NA	NA
ICS AB	70-130	Beginning of run or 12 hr	NA	NA
Int. Std.	60-125 for CCV/CCB 30-135 for samples	Every analysis	60-125 for all	Every analysis

NOTE:
* Although not required by the method, running the LLOQ also at the end of the run may be beneficial.
** Sample results are still acceptable if the last CCV is recovered between 85-115%.
*** Percent (<10%) of the analyte found in samples.
‡ Optional 3rd source verification standard.

9.0 PROCEDURE

9.1 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

9.1.1 All samples are collected in appropriate containers. Water samples are collected in HNO_3 pre-preserved plastic container and are acidified to pH of <2. Filtration is required through a 0.45 µm filter before preservation, for dissolved analysis.

9.1.2 If for some reason such as high alkalinity the sample pH is verified to be > 2, more acid must be added, pH verified to be < 2, and the sample held for 16 hours before analysis.

9.1.3 Soil samples are collected without preservation, usually in glass containers with Teflon lined caps. Non-aqueous samples should be refrigerated upon receipt.

9.1.4 Holding times for metals are 6 months from the date of sampling.

9.2 Solubilization and digestion procedures are presented in the applicable Sample Preparation SAPs or Digestion Methods 3015-3051 in SW-846 method compendium, or SM 3030E in Standards Methods for the Examination of Water and Wastewater, 22nd Edition. Samples for dissolved analysis are not digested. Instead, they are only acidified to a 2% (v/v) HNO_3 matrix. Generally, a 2 mL aliquot of a well-mixed sample is taken and brought to a 10 mL final volume, after the addition of 0.2 mL HNO_3 (in the hood), 100 µL of **IS-WS** (2 ppm stock) to all samples, and 50 µL of the **HA-WS** (10 ppm stock) to LCS/MS/MSD.

9.3 INITIATE APPROPRIATE OPERATING CONFIGURATION OF THE INSTRUMENT

9.3.1 Launch Elan software.

9.3.2 Set up the instrument with the proper operating parameters according to the instrument manufacturer’s instructions (Attachment A, Table 5).

9.3.3 Ignite plasma and allow a warm-up of 15 - 30 minutes. The tuning procedures may be carried out during warm-up.

9.4 Open the Tuning Workspace and conduct mass calibration and resolution checks in the mass regions of interest by analyzing the 10 ppb tuning solution (Section 5.3.5.1). Required elements for tuning are Li, Be, Mg, Co, Rh, In, Tl and Pb. The mass calibration and resolution parameters are required criteria that must be met prior to any samples being analyzed. If mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be within 0.6-0.8 amu full width at 10% peak height. The Elan system cannot perform resolution checks at 5% peak height, but it was shown by Perkin Elmer that 0.65 amu at 10% peak height would be the equivalent of 0.70 amu at 5% peak height (see Attachment D, Peak Width Relationship at 5% and 10% Peak Height). When Method 200.8 needs to be reported, a resolution of approximately 0.65-0.70 amu at 10% peak height would satisfy the recommendation of having a resolution of about 0.75 amu at 5% peak height.

9.5 Open the Daily Performance workspace and verify that the instrument has reached thermal stability by analyzing the 10 ppb tuning solution. An RSD of 5% or lower for five consecutive replicate measurements is required for the elements specified above. The analysis of the tune solution also provides information about the sensitivity level of the instrument and the level of oxide and doubly charged ions formed in the plasma. At a minimum, the following specifications need to be met:

• Sensitivity	– Mg	> 30,000 cps
	– Rh	> 250,000 cps
	– Pb	> 100,000 cps
• Oxide Ratio	– Ce/CeO	≤ 3.5%
• Doubly Charged Ratio	– Ba^+/Ba^{++}	≤ 3%

9.6 When the performance of the instrument is not at the specified level, the Auto Lens needs to be recalibrated and/or the cones need to be cleaned. Calibration of the Auto Lens is performed from the Auto Lens Calibration workspace. The elements selected for calibration, according to the manufacturer instructions are Be, Co and In. Load the workspace, select the Auto Lens tab in the Optimization window, clear the old calibration, load the elements from the method, and calibrate the lens. If the cones need to be cleaned to further improve the performance, record the cleaning or any other maintenance procedure in the instrument maintenance log (the most current form is maintained in a dedicated folder on the Company network).

9.7 If the performance is still not satisfactory, the voltage on the Electron Multiplier Detector might need to be readjusted. Manually change the pulse and analog voltage in steps of 25 V until the desired level of sensitivity is obtained. Adjustments of the voltage, or detector replacement is to be followed with the calibration of the dual detector mode, by the analysis of the 200 ppb tuning solution, from the Dual Detector Calibration workspace. Make sure that the method has all the analytes that are expected to be high, and switch to the analog mode during sample analysis.

9.8 When the performance of the instrument is satisfactory set up the sequence of samples to be analyzed, enter all the information needed for each sample (i.e., sample amount/volume, preparation volume, dilutions at the instrument) and establish a method file with the elements to be measured. Define a new Data Set and calibrate the instrument for the analytes of interest, using the calibration blank and three initial calibration standards according to the instrument manufacturer's procedure. Attachment A, Tables 2, 3, and 4 provide information as to what calibration standards to use. Flush the system with the rinse blank between each standard solution. Use the average of at least three integrations for both calibration and sample analyses.

NOTE: For projects reported under the TNI scope of accreditation all dilution factors shall be entered in LIMS (instead of the analyzer sequence table) in order to allow a consistent evaluation of the effect sample dilution has on PQL and project specific reporting limits.

NOTE: When the quantitation level for all required analytes is confirmed through the analysis of the LLOQ standard to be low, such that the diluting the sample during preparation does not impact the established reporting limits, all calculations can still be performed at the instrument, and only final results transferred into the LIMS.

9.9 All masses that could affect data quality should be monitored to determine potential effects from matrix components on the analyte peaks. The recommended isotopes to be monitored are listed in Attachment A, Table 7. If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes.

9.10 Immediately after the calibration has been established, the calibration must be verified and documented for every analyte by the analysis of both the calibration verification solution and low-level calibration verification solution. When measurements exceed ±10% of the accepted value (CCV)the analyses must be terminated, the problem corrected, the instrument re-calibrated, and the new calibration verified. During the course of an analytical run, the instrument may be "re-sloped" or re-calibrated to correct for instrument drift. A re-calibration must then be followed immediately by a new analysis of a CCV and CCB before any further samples may be analyzed. Corrective actions for specific out-of-control situations are summarized in Attachment A, Table 8.

9.1.1 Flush the system with the rinse blank solution until the signal levels return to the method's levels of quantitation (usually about 30 seconds) before the analysis of each sample. Nebulize each sample until a steady-state signal is achieved (usually about 20 seconds) prior to collecting data. Analyze the calibration verification solution

(CCV), and the continuing calibration blank (CCB) at a frequency of at least once every 10 analytical samples.

9.1.1 Dilute and reanalyze samples that are more concentrated than the linear dynamic range of the instrument (LDR Section 8.2) for an analyte or measure an alternate less-abundant isotope. The linearity at the alternate mass must be confirmed by appropriate calibration.

NOTE: Precautions must be taken to protect the channel electron multiplier from high ion currents. The channel electron multiplier suffers from fatigue after being exposed to high ion currents. This fatigue can last from several seconds to hours depending on the extent of exposure. During this time period, response factors are constantly changing, which invalidates the calibration curve, causes instability, and invalidates sample analyses.

10.0 CALCULATIONS

10.1 The quantitative values shall be reported in appropriate units, such as micrograms or milligrams per liter (µg/L or mg/L) for aqueous samples and micrograms or milligrams per kilogram (µg/Kg or mg/Kg) for solid samples.

10.1.1 Calculations performed by the data system include appropriate interference corrections, internal-standard normalization, and the summation of signals at 206, 207 and 208 m/z for lead (to compensate for any differences in the abundances of these isotopes between samples and standards).

10.1.2 The appropriate initial sample weight or volume and the preparation volume resulted from sample preparation is entered in the data system for each sample at the time of programming the sequence to be analyzed. If additional dilutions are performed at the instrument, the appropriate “aliquot” and “diluted to volume” must be entered in the data system as well.

NOTE: For projects reported under the TNI scope of accreditation all dilution factors shall be entered in LIMS (instead of the analyzer sequence table) in order to allow a consistent evaluation of the effect sample dilution has on PQL and project specific reporting limits.

NOTE: When the quantitation level for all required analytes is confirmed through the analysis of the LLOQ standard to be low, such that the diluting the sample during preparation does not impact the established reporting limits, all calculations can still be performed at the instrument, and only final results transferred into the LIMS.

11.0 DATA REPORTING

11.1 After the analysis has been completed generate an electronic .csv file of the raw data. Upload the .csv file in the designated Microsoft Access database for processing and print a results summary with the analytes requested by the client along with a representative QC package for the analytical batch to be placed in the project folder.

11.2 Use the saved export template in the Access database to generate an Excel file for the electronic transfer into the LIMS.

11.3 Open the file and sort all records by sample ID and Analyte Name. This sorting is required before results can be imported into the LIMS. If special letter codes are present following the space next to the aliquot name in Column B, like the F-Dup, F-MS, F-MSD designators for the field QC samples, ensure that those designators are removed before saving the file for importation.

11.4 Copy the file to the metals parser folder to be imported into the LIMS. The folder is located at *\\LIMS Server\Titan Files\ImportDrop\Lims.Result\07-Metals2DRC\07-Metals2DRCMapper\07-Metals2DRCParser*.

11.5 Open the corresponding analytical batch or project/work order in LIMS and validate that all results got transferred, for all samples and all analytes. When all results rows are populated, the status of the analytical batch in LIMS will automatically transition from “created” to “entered”.

11.6 Enter the ID of the instrument run file(s) corresponding to that particular analytical batch, add qualifying statements if necessary, in the “comments” section and save the batch.

11.7 For projects reported under the TNI scope of accreditation, follow to steps below to evaluate the effect of sample dilution on final project reporting limits.

11.7.1 Ensure all sample dilution and/or preparation factors are entered in LIMS, consistent with how they appear on the preparation notebook page (or bench form).

11.7.2 All quantitation limits in LIMS are automatically adjusted (increased) by the dilution factor during the raw data import.

11.7.3 Final results are listed in the “Reported Column” in LIMS. Results found below quantitation are displayed as “< (PQL x Dilution)”.

11.7.4 Compare the “less than (<)” value with project specific reporting limits and flag data appropriately for the cases where the (PQL x Dilution) limit ends up higher that the project required reporting limit for the given analyte.

NOTE: When the quantitation level for all required analytes is confirmed through the analysis of the LLOQ standard to be low, such that the diluting the sample during preparation does not impact the established reporting limits, all calculations can still be performed at the instrument, and only final results transferred into the LIMS. In those cases, ensure the dilution column in LIMS is set to “1”.

11.8 When all data review in LIMS is complete, highlight all rows and transition the analytical batch to “Validated”.

11.9 Place a copy of any notebook pages involving the project (i.e. sample preparation log) in the Project Folder.

TITLE: METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

Table 1. List of elements analyzed, Typical MDL and Reporting Levels/PQL's.

Element	Symbol	CAS#	Typical MDL* (µg/L)	Reporting Level** (µg/L)	LLOQ Level (µg/L)	LRV*** Level (µg/L)
Aluminum	(Al)	7429-90-5	0.98	10	2	Cal Std
Antimony	(Sb)	7440-36-0	0.05	2	0.2	Cal Std
Arsenic	(As)	7440-38-2	0.07	1	0.2	2000
Barium	(Ba)	7440-39-3	0.05	10	0.2	2000
Beryllium	(Be)	7440-39-3	0.27	1	0.2	Cal Std
Boron	(B)	7440-42-8	0.50	20	4	2000
Cadmium	(Cd)	7440-43-9	0.04	0.2	0.04	Cal Std
Calcium	(Ca)	7440-70-2	1.6	1000	50	10000
Chromium	(Cr)	7440-47-3	0.07	5	0.2	2000
Cobalt	(Co)	7440-48-4	0.13	5	0.2	2000
Copper	(Cu)	7440-50-8	0.09	10	0.2	2000
Iron	(Fe)	7439-89-6	2.38	20	4	2000
Lead	(Pb)	7439-92-1	0.04	3	0.2	2000
Lithium	(Li)	7439-93-2	0.26	10	0.2	Cal Std
Magnesium	(Mg)	7439-95-4	2.5	1000	50	10000
Manganese	(Mn)	7439-96-5	0.08	5	0.2	2000
Molybdenum	(Mo)	7439-98-7	0.07	5	0.2	2000
Nickel	(Ni)	7440-02-0	0.12	5	0.2	2000
Potassium	(K)	7440-09-7	1.8	100	50	10000
Selenium	(Se)	7782-49-2	0.16	5	0.2	Cal Std
Silver	(Ag)	7440-22-4	0.03	0.2	0.04	Cal Std
Sodium	(Na)	7440-23-5	3.2	1000	50	10000
Strontium	(Sr)	7440-24-6	0.06	5	0.2	2000
Tin	(Sn)	7440-31-5	0.04	10	0.2	Cal Std
Titanium	(Ti)	7440-32-6	0.21	5	0.2	Cal Std
Thallium	(Tl)	7440-28-0	0.06	2	0.2	Cal Std
Vanadium	(V)	7440-62-2	0.08	10	0.2	Cal Std
Zinc	(Zn)	7440-66-6	0.17	10	2	2000

NOTE: * Represents an MDL obtained with the Elan 6000 during the initial demonstration of performance. Current MDL might be slightly different.

** Typical reporting level for clean water samples, it can change for other applications/matrices.

*** Cal Std represents the value of the highest calibration standard.

TITLE: METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

Table 2. Standard preparation for all elements except Fe, Ca, Mg, K, Na.

Standard type	Concentration	Volume of **S** 100 ppm stock	Volume of **HA-WS** 10 ppm stock	Volume of **HNO_3**	Volume of **IS-WS**	Final volume
	(µg/L)	(µL)	(µL)	(mL)	(mL)	(mL)
Blank	0	-	-	1	0.5	50
Standard1: *HA***100**-*mmddyy*	100	-	500	1	0.5	50
ICV: *S***40**-*mmddyy*	40	20	-	1	0.5	50
BS: *HA***.0.***04-mmddyy*	0.04	-	20uL of HA100	1	0.5	50
BS: HA0.2-mmddyy	0.2	-	100 uL of HA100	1	0.5	50
BS: HA4.0-mmddyy	4.0	-	2mL of HA100	1	0.5	50
CCV: *HA***50**-*mmddyy*	50	-	250	1	0.5	50

Table 3. Standard preparation for Fe.

Standard type	Concentration	Volume of **S** 100 ppm stock	Volume of **H-WS** 10 ppm stock	Volume of **HNO_3**	Volume of **IS-WS**	Final volume
	(µg/L)	(µL)	(µL)	(mL)	(mL)	(mL)
Blank	0	-	-	1	0.5	50
Standard1: *HA***200**-*mmddyy*	200	-	1000	1	0.5	50
ICV: *S***80**-*mmddyy*	80	40	-	1	0.5	50
BS: *HA***4.0**-*mmddyy*	4.0	-	2 mL of HA100	1	0.5	50
CCV: *HA***100**-*mmddyy*	100	-	500	1	0.5	50

Table 4. Standard preparation for Ca, Mg, K, Na from a 1000 ppm stock solution.

Standard type	Concentration	Volume of **S**	Volume of **HB**	Volume of **HNO_3**	Volume of **IS-WS**	Final volume
	(mg/L)	(µL)	(µL)	(mL)	(mL)	(mL)
Blank	0	-	-	1	0.5	50
Standard3: *HB***2.0**-*mmddyy*	2.0	-	100	1	0.5	50
ICV: *S***0.8**-*mmddyy*	1.0	50	-	1	0.5	50
BS: *HB***0.01**-*mmddyy*	0.01	-	500uLof HB2.0	1	0.5	50
BS: HB0.05-mmddyy	0.05	-	2.5mL of HB2.0	1	0.5	50
CCV: *HB***1.0**-*mmddyy*	1.0	-	50	1	0.5	50

Table 5. Instrument operating parameters.

Instrument Parameter	Operating Condition
Plasma forward power	1300 W
Plasma Argon flow rate	16.0 L/min
Auxiliary Argon flow rate	1.0 L/min
Nebulizer Argon flow rate	~0.9 L/min
Spray chamber temperature	2°-25°C
Analyzer vacuum	3×10^{-5} – 6×10^{-4} Torr

Table 6. Interference Check Solutions Components and Concentration.

Solution Component	ICS-A (ppm)	ICS-AB (ppm)
Al	100.0	100.0
Ca	300.0	300.0
Fe	250.0	250.0
Mg	100.0	100.0
Na	250.0	250.0
P	100.0	100.0
K	100.0	100.0
S	100.0	100.0
C	200.0	200.0
Cl	2121.5	2121.5
Mo	2.0	2.0
Ti	2.0	2.0
As	0.0	0.100
Cd	0.0	0.100
Cr	0.0	0.200
Co	0.0	0.200
Cu	0.0	0.200
Mn	0.0	0.200
Ni	0.0	0.200
Se	0.0	0.100
Ag	0.0	0.050
V	0.0	0.200
Zn	0.0	0.100

TITLE: METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

Table 7. Recommended analytical isotopes (underlined) and additional masses to be monitored.

MASS	ELEMENT	I.S. USED	ELEMENTAL CORRECTION	POTENTIAL INTERFERENCES
6	Li	**I.S.**	$-(0.0813)(^{7}C)$	
$\underline{7}$	Li	^{6}Li or Sc		
$\underline{9}$	Be	^{6}Li or Sc		
10	B	^{6}Li or Sc		
$\underline{11}$	B	^{6}Li or Sc		
$\underline{19}$	K	Sc or Rh		
$\underline{23}$	Na	Sc or Rh		
$\underline{24}$	Mg	Sc or Rh		
$\underline{27}$	Al	^{6}Li or Sc		
43	Ca	Sc or Rh		
$\underline{44}$	Ca	Sc or Rh	$(-0.0271)(^{88}C)$	Sr^{++}
45	Sc	**I.S.**		CO_2H^+
47	Ti	^{6}Li or Sc		
$\underline{49}$	Ti	^{6}Li or Sc		
$\underline{51}$	V	^{6}Li or Sc	$-(3.127)(^{53}C)+(0.352)(^{52}C)$	$^{35}ClO^+$, $^{34}SOH^+$
$\underline{52}$	Cr	Sc, Ge or Rh		ArC^+, ArO^+, $^{35}ClHO^+$
$\underline{53}$	Cr	Sc, Ge or Rh		$^{37}ClHO^+$
$\underline{54}$	Fe	Sc, Ge or Rh	$-(0.0284)(^{52}C)$	
$\underline{55}$	Mn	Sc, Ge or Rh		$ArNH^+$
$\underline{57}$	Fe	Sc, Ge or Rh		
$\underline{59}$	Co	Sc, Ge or Rh		
$\underline{60}$	Ni	Sc, Ge or Rh		
62	Ni	Sc, Ge or Rh		TiO
$\underline{63}$	Cu	Sc, Ge or Rh		$^{31}PO_2^+$, $^{40}ArNa^+$, TiO
$\underline{65}$	Cu	Sc, Ge or Rh		TiO
$\underline{66}$	Zn	Sc, Ge or Rh		TiO
68	Zn	Sc, Ge or Rh		
$\underline{74}$	Ge	**I.S.**		
$\underline{75}$	As	Ge or Rh	$-(3.132)(^{77}C)+(2.736)(^{83}C)$	$^{40}Ar^{35}Cl^+$
76	$^{40}Ar^{36}Ar^+$	Ge or Rh		$^{40}Ar^{37}Cl^+$
77	Se	Ge or Rh		
78	Se	Ge or Rh	$-(0.1869)(^{76}C)^{\ddagger}$	$^{40}Ar^{38}Ar^+$
$\underline{82}$	Se	Ge or Rh		$^{81}BrH^+$
83	Kr	Ge or Rh		
$\underline{88}$	Sr	Ge or Rh		
90	Zr	Ge or Rh		
$\underline{95}$	Mo	Ge or Rh		$^{79}BrO^+$
98	Mo	Ge or Rh	$-(0.146)(^{99}C)$	$^{79}BrHO^+$
103	Rh	**I.S.**		
105	Pd	Rh		
106	Pd, Cd	Rh		ZrO,
$\underline{107}$	Ag	Rh		ZrO
108	MoO	Rh		ZrO, MoO
109	Ag	Rh		ZrO, MoO
$\underline{111}$	Cd	Rh		ZrO, MoO
112	Cd	Rh	$-(0.040)(^{118}C)$	ZrO, MoO
$\underline{114}$	Cd	Rh	$-(0.0269)(^{118}C)$	MoO
$\underline{118}$	Sn	Rh		
119	Sn	Rh		
120	Sn	Rh	$-(0.0127)(^{125}C)$	
$\underline{121}$	Sb	Rh	$-(0.124)(^{125}C)$	$^{40}Ar^{81}Br^+$
123	Sb	Rh		
$\underline{137}$	Ba	Rh		
138	Ba	Rh	$-(8.91E-04)(^{139}C)-(2.82E-04)(^{140}C)$	
140	Ce	Rh		
185	Re	**I.S.**		
$\underline{203}$	Tl	Re		
205	Tl	Re		
206	Pb	Re		
207	Pb	Re		
$\underline{208}$	Pb	Re	$+(1.0)(^{206}C)+(1.0)(^{2.07}C)$	

NOTES:
1) C = Counts at specified mass.
2) When the concentration of Na in the samples is high, the ionization of Sc is suppressed leading to positive bias of the results, therefore Rh or Ge should be used as the internal standard, even if more than 50 amu removed from the element of interest.

TITLE: METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

Table 8. Quality Control Items, Frequency, and Corrective Action.

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tuning	After warm-up. Every 12 hours.	Manufacturer specifications	Check operating parameters, clean cones, replace malfunctioning components if necessary. Re-evaluate the tuning.
ICV	After initial calibration.	90-110%	Verify that method parameters are valid, check calibration tables, replace calibration standards if necessary, and recalibrate the instrument.
ICB	Following ICV.	< 2.2 x MDL, or < ½ LLOQ, or < RL for water samples	Prepare fresh calibration blank and/or increase the rinse time between analyses; reanalyze ICB; if within limits, continue the run; if still outside limits, determine the source of the problem, make the necessary corrections, and start from the beginning with a new calibration.
LLOQ	After initial calibration.	80-120% if above RL	Verify that method parameters are valid, check calibration tables, replace calibration standards if necessary, prepare a fresh calibration blank, and recalibrate the instrument.
CCV	Before and after each batch. Every 10 sample. After re-calibration.	90-110%	Recalibrate the instrument. Follow method specific requirements (6020 or 200.8) as to what data prior to the CCV can be used.
CCB	Following CCV.	< 2.2 x MDL, or < ½ LLOQ, or < RL for water samples	Prepare fresh calibration blank; reanalyze CCB; if within limits, continue the run; if still outside limits, eliminate the source of the contamination, clean the sample introduction system if necessary, and recalibrate the instrument. Reanalyze all samples from the last good CCB.
LCS	Every batch of 20 samples.	80-120%for 6020 85-115% for 200.8	Stop analysis, re-prep samples and reanalyze. Look for any project specific requirements.
LRB	Every batch of 20 samples.	< 2.2 x MDL, or < ½ LLOQ, or < RL for water samples < 10% of analyte in samples	Stop analysis, re-prep samples and reanalyze if it adversely impacts project specific data or requirements.
Dup	Every 20 samples (6020) Every 10 samples (200.8)	0-20%	If all other QC is acceptable continue the run; sample result should be flagged; otherwise recalibrate instrument and reanalyze samples.
MS	Every 20 samples (6020) Every 10 samples (200.8)	70-130% with 200.8 75-125% with 6020	For 200.8 flag data if all other QC met; otherwise recalibrate instrument and reanalyze affected samples. For 6020 dilute original sample, re-spike dilution, and reanalyze until within limits.
MSD†	Every 20 samples (6020) Every 10 samples (200.8)	0-20%	Same as for duplicate.
Dil‡	Every batch of 20 samples.	0-20%	If concentration analyzed >100 x MDL, flag data for possible matrix interference.
ICS-A‡	Begging and every 12 hours.	< 2.2 x MDL, or < ½ LLOQ, or < RL for water samples < 10% of analyte in samples	Reevaluate the equations used for corrections, make the necessary adjustments, and recalibrate the instrument. If changes in the optimization parameters do not improve instrument response, add explanatory note in the report case narrative.
ICS-AB‡	Begging and every 12 hours.	75-125	Re-evaluate the equations used for corrections, make the necessary adjustments, and recalibrate the instrument.
IS	With every analysis.	60-125% with 200.8 30-135% samples with 6020 80-125% for CCB with 6020	For samples, dilute 4+1 and reanalyze until in control. For CCV/CCB's recalibrate the instrument and reanalyze the affected samples.

NOTE:
- RL = Reporting Limit.
- Dil = Dilution Test.
- † MSD optional instead of duplicate sample.
- ‡ When Method 6020 referenced in the analytical report.

TITLE: METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

METALS STANDARDS PREPARATION LOG

Pipettor ID: LS021386 ☼ 10-100 µL
LS021384 ♣ 100-1000 µL
LS021385 ♯ 0.5-5 mL

Perkin Elmer: Elan / NexION
HNO_3 Lot # ____________
Internal Std ID # ____________

DATE	ANALYST	WORKING STANDARD ID CONC.* (PPM, BOLD)	SOURCE	LOT NUMBER	EXP. DATE	REMARKS
		HA-WS**10**-	HPS-A	1913438	05/31/2020	

DATE	ANALYST	STANDARD ID AND CONC.* (µg/L, BOLD)	SOURCE	LOT NO OR SOURCE ID	EXP. DATE	REMARKS
		HPS **LLOQ**-	HPS	1913533	05/31/2020	
		HA**50**-	HA-WS	HA-WS**10**-	05/31/2020	
		HA**100**-	HA-WS	HA-WS**10**-	05/31/2020	
		HA**200**-	HA-WS	HA-WS**10**-	05/31/2020	
		HPS **LRV**-	HPS	1913504	05/31/2020	
		S**40**-	SPEX	52-021CR	05/30/2020	
		S**80**-	SPEX	52-021CR	05/30/2020	
		ERA-	ERA	P282-500	10/31/2020	
		A-	IV	N2-MEB671037	04/29/2020	
		AB-	IV	N2-MEB664996	04/29/2020	
		HB**0.01**-	HPS-B	1913435	05/31/2020	
		HB**0.05**-	HPS-B	1913435	05/31/2020	
		HB**1.0**-	HPS-B	1913435	05/31/2020	
		HB**2.0**-	HPS-B	1913435	05/31/2020	
		SM**0.8**-	SPEX	52-022CR	05/30/2020	
		ERA-M	ERA	P269-506-507	09/30/2020	
		IVSi**0.05**-	IV-Si	P2-SI676242	09/11/2020	
		IVSi**1.0**-	IV-Si	P2-SI676242	09/11/2020	
		IVSi**2.0**-	IV-Si	P2-SI676242	09/11/2020	
		SSi**0.8**-	SPEX-Si	24-93SIX	09/30/2020	

DATE	ANALYST	INTERNAL STANDARD (IS) ID AND PPM CONC.	SOURCE	LOT NUMBER	EXP. DATE	REMARKS
		IS**2.0**-	IV-Li^6	P2-LI677020	09/11/2020	
			IV-Sc	P2-SC677026	09/11/2020	
			IV-Ge	P2-GE677722	09/11/2020	
			IV-Rh	N2-RH673649	09/11/2020	
			IV-Re	M2-RE656694	09/11/2020	

NOTES: 1) The following abbreviations were used:

- HPS = High Purity Standards, Inc.
- SPEX = SPEX CertiPrep, Inc.
- WS = Working Standard, 10 ppm solution, a 10/1 dilution of the HPS-A stock solution.
- A, IV = Interference Check Solution, SIC A, from Inorganic Ventures, Inc.
- AB, IV = Interference Check Solution, SIC AB, from Inorganic Ventures, Inc.

TITLE: METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

2) Stock solutions used, source, concentration and the elements present:

- HA-WS = HPS, 10 ppm each of Al, Sb, As, Ba, Be, Bi, B, Cd, Cr, Co, Cu, Fe, Pb, Li, Mn, Mo, Ni, Se, Ag, Sr, Sn, Tl, Ti, V, Zn.
- S = SPEX, 100 ppm each of Al, Sb, As, Ba, Be, Bi, B, Cd, Cr, Co, Cu, Fe, Pb, Li, Mn, Mo, Ni, Se, Ag, Sr, Sn, Tl, Ti, V, Zn.
- A = IV, 1000 μg/ml each of Al, Mg, P, K, S, 2000 mg/mL of C, 2500 mg/mL each of Fe and Na, 3000 mg/mL of Ca, 21215 mg/mL of Cl, and 20.0 mg/mL each of Mo and Ti.
- AB = IV, 20.0 mg/mL each of Cr, Co, Cu, Mn, Ni, and V, 10.0 mg/mL each of As, Cd, Se, Zn and 5.00 mg/mL of Ag.
- HB = HPS solution B, 1000 ppm each of Ca, Mg, Na, and K.
- SM = SPEX, 1000 ppm each of Ca, Mg, Na, and K.
- HPSLLOQ = HPS, 2 ppm each of Fe, B; 1 ppm each of Al, Li, Zn; 0.2 ppm each of Sb, As, Ba, Be, Bi, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Sr, Sn, Tl, Ti, V; 0.02 ppm each of Cd, Ag.
- HPSLRV = HPS, 1000 ppm each of Al, Sb, As, Ba, Be, Bi, B, Cd, Cr, Co, Cu, Fe, Pb, Li, Mn, Mo, Ni, Se, Ag, Sr, Sn, Tl, Ti, V, Zn.

3) * Working solution, HB/SM and Silicon stock standards concentrations are listed in ppm.

PREPARATION GUIDE

Standard		**Pipettor**	**HA**	**HPS**	**SPEX**	**ERA**	**A**	**AB**	**HB**	**SM**	**Si****	**IS*****	**HNO_3**	**IS**	**Final volume**
			(μL)	(μL)	(μL)	(μL)	(μL)	(μL)	(μL)	(μL)	(μL)	(μL)	(mL)	(mL)	(mL)
Cal Blank	0 ppb		-	-	-	-	-	-	-	-	-	-	1	0.5	50
HPS-**LLOQ**	LL ppb	☼	-	100	-	-	-	-	-	-	-	-	1	0.5	50
HA**50**	50 ppb	♣	250	-	-	-	-	-	-	-	-	-	1	0.5	50
HA**100**	100 ppb	♣	500	-	-	-	-	-	-	-	-	-	1	0.5	50
HA**200**	200 ppb	♣	1000	-	-	-	-	-	-	-	-	-	1	0.5	50
HPS-**LRV**	2000 ppb	☼	-	100	-	-	-	-	-	-	-	-	1	0.5	50
S**40**	40 ppb	☼	-	-	20	-	-	-	-	-	-	-	1	0.5	50
S**80**	80 ppb	☼	-	-	40	-	-	-	-	-	-	-	1	0.5	50
ERA	varies	☼	-	-	-	20	-	-	-	-	-	-	1	0.5	50
A-	varies	♯	-	-	-	-	5000	-	-	-	-	-	1	0.5	50
AB-	varies	♯,♣	-	-	-	-	5000	500	-	-	-	-	1	0.5	50
HB**0.01**	0.01 ppm	♣	-	-	-	-	-	-	250*	-	-	-	1	0.5	50
HB**0.05**	0.05 ppm	♣	-	-	-	-	-	-	1250*	-	-	-	1	0.5	50
HB**1.0**	1.0 ppm	☼	-	-	-	-	-	-	50	-	-	-	1	0.5	50
HB**2.0**	2.0 ppm	☼	-	-	-	-	-	-	100	-	-	-	1	0.5	50
SM**0.8**	0.8 ppm	☼	-	-		-	-	-	-	40	-	-	1	0.5	50
ERA-M	varies	♣	-	-	-	1000	-	-	-	-	-	-	1	0.5	50
HSi**0.05**	0.05 ppm	☼	-	-	-	-	-	-	-	50	1250*	-	1	0.5	50
HSi**1.0**	1.0 ppm	☼	-	-	-	-	-	-	-	-	50	-	1	0.5	50
HSi**2.0**	2.0 ppm	☼	-	-	-	-	-	-	-	-	100	-	1	0.5	50
SSi**0.8**	0.8 ppm	☼	-	-	-	-	-	-	-	-	40	-	1	0.5	50
IS**2.0**	2.0 ppm	♣	-	-	-	-	-	-	-	-	-	2000	20	-	1000

* The volume stated for the HB and Si low level check standard is taken from the 2.0 ppm calibration standard (serial dilution).

** The volume stated applies to both HPS and SPEX Si sources.

*** The volume stated applies to all sources used as analytes for the Internal Standard (IS) mix.

TITLE: METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

DIGESTION WORKSHEET

LCS/MS/MSD - added 75uL of 10ppm of HPS-A 1913438, Procurement #19050680

added 15uL of HPS-B 1913435, Procurement #19050680

Pipette M&TE#/ Range/ Cal: LS021380/ 0.5-5mL/ 03-2020 LS021486/20-200uL/03-202 LS021488/100-1000uL/03-2020

LS021486/5-50uL/03-2020

HNO_3 - Lot# 18417996, Item# 63, Exp. Date: 09/12/23, Procurement: 19050760

15 mL Tube: Lot# 1812114

Hot Block M&TE#: 024242

Start Time/Temp: __________________ **Thermocouple M&TE#:** LS011969 (10/09/20) **Probe M&TE#:** CP3464

End Time/Temp: _____________________

Position: ____________

Sample ID	Volume (mL)	Final Vol (mL)	Remarks
LCS	3	15	
LRB	3	15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	
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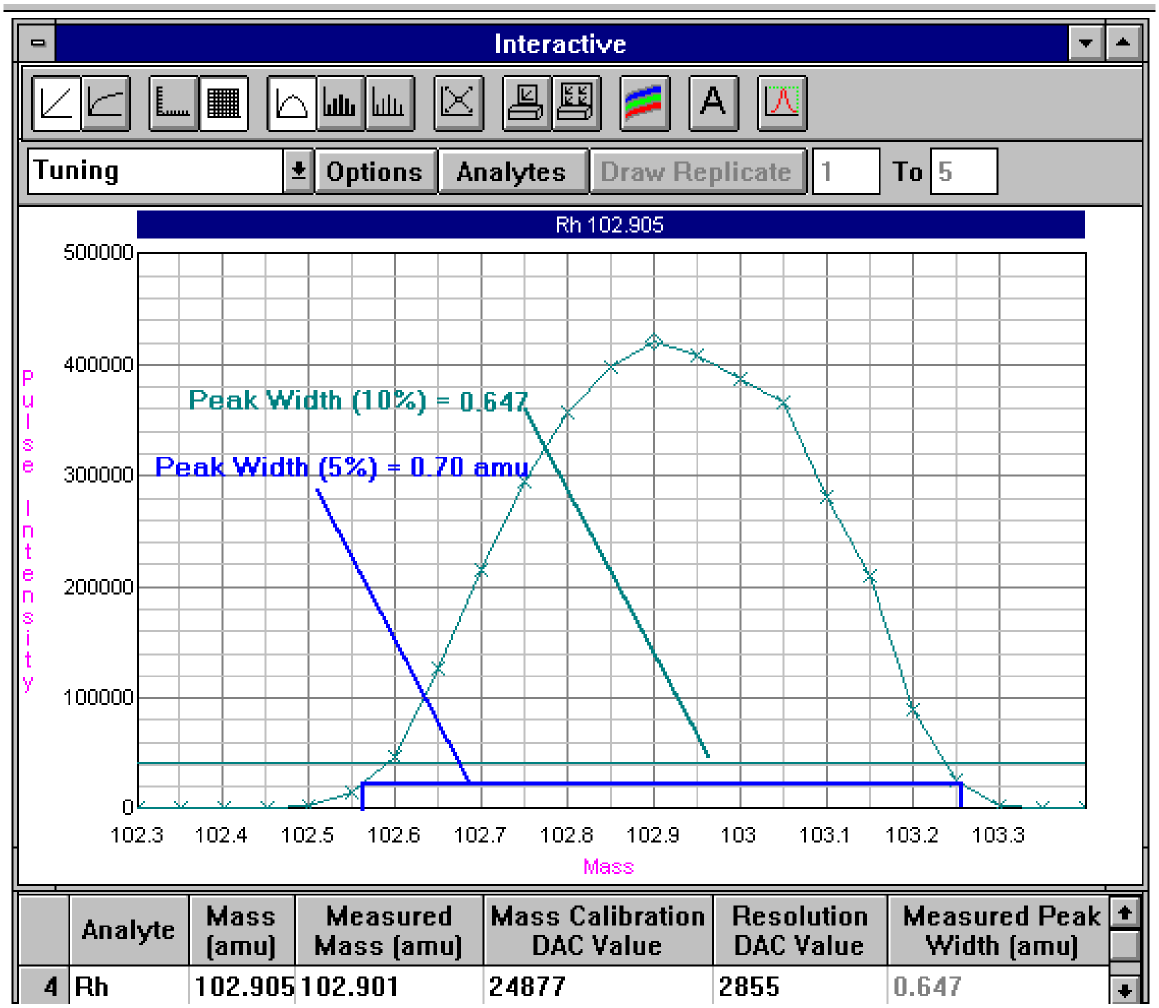
		15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	
		15	

Analyst: ________________________ Date: ________________________

Peak Width Relationship at 5% and 10% Peak Height



	Analyte	Mass (amu)	Measured Mass (amu)	Mass Calibration DAC Value	Resolution DAC Value	Measured Peak Width (amu)
4	Rh	102.905	102.901	24877	2855	0.647

CONSUMERS ENERGY	Chemistry Department Standard Analytical Procedure	PROC CHEM-2.5.40 PAGE 1 OF 16 REVISION 5

TITLE: ANIONS ANALYSIS BY ION CHROMATOGRAPHY

Written or Revised by Dawn Williams Date 6.18.20
Level I or Above

Technical Review / Approval by ________________ Date 06/18/20
Level III (not author)

Administrative Approval by ________________ Date 06/18/20
Department Head

TITLE: ANIONS ANALYSIS BY ION CHROMATOGRAPHY

1.0 SCOPE

1.1 The purpose of this procedure is to set forth a method by which the Chemistry Department of Laboratory Services will determine following inorganic anions:

- Bromide
- Nitrite
- Chloride
- Ortho-Phosphate-P
- Fluoride
- Sulfate
- Nitrate

1.2 The method is applicable to the analysis of drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after the extraction step described in section 9.1), and leachates (when no acetic acid is used)

1.3 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a fortified sample matrix covering the anions of interest.

1.4 This SAP follows the guidelines of USEPA Method 300.0 “Determination of Inorganic Anions by Ion Chromatography” Revision 2.1 August 1993.

1.5 Typical Method Detection Limits (MDL’s) and practical quantitation levels (PQL’s) for anions determined with this procedure are listed in the following table:

Fluoride	9.9	100
Chloride	8.4	100
Nitrite as N	5.6	50
Bromide	19.6	100
Nitrate as N	4.7	50
Sulfate	4.7	50
Phosphate (-ortho)	16.0	500

Current MDL’s and PQL’s are listed on the Company Network, at K:\CHEM\MDLs_Current.

2.0 APPLICABLE DOCUMENTS AND REFERENCES

2.1 Chemistry Department Standard Operating Procedures, as applicable

2.2 Laboratory Services Quality Assurance (LSQA) Procedures, as applicable

2.3 Pfaff, John D. USEPA Method 300.0 Determination of Inorganic Anions by Ion Chromatography Revision 2.1 August 1993

2.4 Instrument Manual, as applicable

2.5 USEPA Method 26A “Determination of Hydrogen Halide and Halogen Emissions from Stationary Sources – Isokenetic Method.”

3.0 DEFINITIONS

3.1 Continuing Calibration Blank (CCB) -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

3.2 Calibration Standard (CAL) -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.3 Field Duplicates (FD) -- Two separate samples collected at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

3.4 Continuing Calibration Verification (CCV) -- A solution of one or more method analytes used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.5 Matrix Spike (MS) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

3.6 Laboratory Reagent Blank (LRB) or Method Blank (MB) -- An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment and reagents used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

NOTE: The CCB's, LRB's or MB's are all reagent blank water for this method.

3.7 Linear Calibration Range (LCR) -- The concentration range over which the instrument response is linear.

3.8 Safety Data Sheet (SDS) -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.9 Method Detection Limit (MDL) -- The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

3.10 Proficiency Testing Sample (PT) -- A solution of method analytes distributed by various PT providers to multiple laboratories for analysis. A volume of the solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used by the PT provider to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst. Analyte true values are unknown to the analyst.

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3.11 Laboratory Control Sample (LCS) -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The LCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.12 Stock Standard Solution (SSS) -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

3.13 Sample Batch -- A set of 20 samples validated by at least an LCS, LRB and a matrix spike.

3.14 ERA – An optional secondary standard, in addition to the LCS, purchased from Environmental Resources Associates and provided with a certificate of analysis and acceptance criteria limits by the vendor, per each lot.

4.0 SUMMARY OF METHOD

4.1 A small volume of sample is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

4.2 An extraction procedure must be performed to use this method for solids (See Section 9.1).

4.3 Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in Section 8.0, Acceptance Criteria.

5.0 PREREQUISITES

5.1 MEASURING AND TEST EQUIPMENT

5.1.1 Balance -- Analytical, capable of accurately weighing to the nearest 0.000l g.

5.1.2 Ion chromatograph – Dionex ICS-5000 analytical system complete with ion chromatograph and all required accessories including analytical columns and detectors.

5.1.2.1 Anion guard column: Dionex IonPac AG22 (4mm) or equivalent.

5.1.2.2 Anion analytical column: Dionex IonPac AS22 (4mm) or equivalent.

5.1.2.3 Anion suppressor device: Dionex ASRS 300 4mm Self-regenerating or equivalent.

5.1.2.4 Detector: Conductivity cell, Dionex ICS-5000+ Electrochemical detector or equivalent.

5.1.2.5 Autosampler: Dionex ASDV Autosampler or equivalent.

5.1.2.6 Software: Dionex Chromeleon Version 7.0 or equivalent.

5.1.3 Vials and Caps: Dionex Polyvial 5mL Vials and Filter Caps or equivalent.

5.2 REAGENTS

5.2.1 Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

Eluent Solution: 4.5 mM Sodium Carbonate/1.4mM Sodium Bicarbonate from AS22 Eluent Concentrate (ICS-5000 system).

5.2.2 Individual stock standard solutions, l000mg/L (1mg/mL): Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105°C for 30 minutes) as listed below.

5.2.2.1 Bromide 1000mg/L: Dissolve 1.2876g sodium bromide (NaBr, CASRN 7647-15-6) in reagent water and dilute to 1L.

5.2.2.2 Chloride l000mg/L: Dissolve 1.6485g sodium chloride (NaCl, CASRN 7647-l4-5) in reagent water and dilute to 1L.

5.2.2.3 Fluoride 1000mg/L: Dissolve 2.2100g sodium fluoride (NaF, CASRN 7681-49-4) in reagent water and dilute to 1L.

5.2.2.4 Nitrate 1000mg/L: Dissolve 6.0679g sodium nitrate ($NaNO_3$, CASRN 7631-99-4) in reagent water and dilute to 1L.

5.2.2.5 Nitrite 1000mg/L: Dissolve 4.9257g sodium nitrite ($NaNO_2$, CASRN 7632-00-0) in reagent water and dilute to 1L.

5.2.2.6 Phosphate 1000mg/L: Dissolve 4.3937g potassium phosphate (KH_2PO_4, CASRN 7778-77-0) in reagent water and dilute to 1L.

5.2.2.7 Sulfate 1000mg/L: Dissolve 1.8141g potassium sulfate (K_2SO_4, CASRN 7778-80-5) in reagent water and dilute to 1L.

5.2.3 Seven Anion Stock Solution (alternative to 5.2.2): Contains fluoride, chloride, nitrite, bromide, nitrate, phosphate, and sulfate. Dionex Seven Anion Standard II (part #57590) or equivalent.

5.2.4 Seven Anion Check Standard: Contains all anions listed in 5.2.4, but procured from a separate source. (SPEX Certiprep)

5.2.5 Chloride 10,000 mg/L for soil preparation, such as Inorganic Ventures part # ICCL10-125ML.

5.2.6 Sulfate 10,000 mg/L for soil preparation, such as Inorganic Ventures part # ICSO410-125ML.

5.3 CALIBRATION REQUIREMENTS

5.3.1 The IC is calibrated prior to use with standards.

5.3.2 The calibration curve is composed of a sufficient number of standards to ensure that the resulting curve is linear, at a minimum three concentration levels and a blank. The calibration curve should be of sufficient range to bracket the expected concentration of the sample analytes of interest. Each standard level is prepared by pipetting the corresponding volume of the stock standard and diluent (method dependent) into the autosampler vial. The exact volumes and the resulting calibration standard concentrations are listed in the Dionex folder on the Company network.

5.3.3 The calibration curve for each analyte should achieve a minimum correlation coefficient of 0.995 in order to be considered valid.

5.3.4 The mid points of the calibration curve cannot be dropped. If a low point is dropped the reporting limit must be increased to the level of the lowest standard included in the curve. If a high point is dropped samples must be diluted within the concentration range of highest point. When permissible changes (as described above) to the calibration curve are necessary, the date and initials of the person making the adjustment, and the reason for the adjustment are to be documented. The change must be approved by the Technical Director (or delegate) before the curve is used to generate results for analytical samples.

5.3.5 The calibration curve should be determined with every new lot of standards, when the CCV results show signs of shifting, are not within the required limits, and if a significant change in instrument response is observed or expected.

5.3.6 See section 10.2 of USEPA Method 26A for requirements.

5.4 QUALITY CONTROL DOCUMENTS AND RECORDS

5.4.1 Chromatograms associated with samples are printed and provided to the project lead along with the data summary, including all recovery calculations for QC elements (i.e. CCV/LCS/Duplicates/MS/MSD). They are stored in the project folder and scanned with the completed project report.

5.5 PERSONNEL REQUIREMENTS

5.5.1 All tests and data reporting shall be performed by certified persons of Level I or above, in the appropriate discipline. The project report shall be issued and reviewed by a certified person of Level II or above, in the appropriate discipline. The project report, if so indicated on the work request (or form similar in intent,) may require approval from a certified person of Level III, in the appropriate discipline.

5.5.2 This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

5.5.3 For projects where reporting under the 2009 TNI Standard has been requested, all tests and data reporting shall be performed by analysts with a completed initial, or an on-going Demonstration of Capability (IDOC/DOC), as applicable.

5.6 ENVIRONMENTAL CONDITIONS

5.6.1 Aqueous samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

5.6.2 Sample preservation and holding times for the anions that can be determined by this method are as follows:

Analyte	**Preservation**	**Holding Time**
Bromide	None	28 Days
Chloride	None	28 Days
Fluoride	None	28 Days
Nitrate-N	Cool to 4˚C	48 Hours
Combined (Nitrate/Nitrite)	Conc. H_2SO_4 to a pH < 2	28 Days
Nitrite-N	Cool to 4˚C	48 Hours
o-Phosphate-P	Cool to 4˚C	48 Hours
Sulfate	Cool to 4˚C	28 Days

5.6.3 The method of preservation and the holding time for samples analyzed by this method are determined by the anions of interest. In a given sample, the anion that requires the most preservation treatment and the shortest holding time will determine the preservation treatment. It is recommended that all samples be cooled to 4°C and held for no longer than 28 days.

5.6.4 Soil samples are collected without preservation, usually in glass containers with Teflon lined caps. Non-aqueous samples should be stored at 4˚C upon receiving.

5.6.5 Sample preservation, containers and hold times are also detailed in Standard Operating Procedure CHEM-1.2.02.

6.0 PRECAUTIONS

6.1 Observe normal practices as specified in the latest online revision of the Consumers Energy Accident Prevention Manual and the Consumers Energy Chemical Hygiene Plan.

6.2 Safety glasses should be worn at all times, because the ion chromatograph is a pressurized system.

6.3 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Every effort should be made to minimize the generation of excess waste in the preparation of standards and reagents related to this procedure.

6.4 For guidance on proper disposal of unused samples, stock chemicals and reagents refer to SOP CHEM-1.2.08 Handling and Disposal of Lab Testing Waste.

7.0 LIMITATIONS AND ACTIONS

7.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

7.2 The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of the equivalent of 1mL of concentrated eluent (5.2.2 100X) to l00mL of each standard and sample.

7.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

7.4 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

7.5 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known co-elution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

7.6 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

7.7 The quantitation of un-retained peaks should be avoided, such as low molecular weight organic acids (formate, acetate, propionate etc.) which are conductive and co-elute with or near fluoride and would bias the fluoride quantitation in some drinking and most waste waters.

8.0 ACCEPTANCE CRITERIA

8.1 Initial Demonstration of Performance

8.1.1 The initial demonstration of performance is used to characterize instrument performance (determination of linear calibrated range or LCR and analysis of laboratory control sample or LCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

8.1.2 The linear calibrated range or LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to ensure that the resulting curve is linear. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

8.1.3 When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a LCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

8.1.4 Method detection limits or MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of reagent water fortified with 1-5 times the estimated detection concentration for the analyte and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. To calculate the MDL see Section 10.1. MDLs should be determined annually or whenever there is a significant change in the background or instrument response.

8.2 Assessing Laboratory Performance

8.2.1 Analyze a laboratory reagent blank or LRB for every batch of 20 samples. The data produced is used to assess contamination from the laboratory environment. Values that exceed the 2.2 x MDL or are more than 10% of the background measured in the analytical samples indicate contamination and corrective actions must be taken before continuing the analysis (re-prep and reanalyzed affected sample batch).

8.2.2 Analyze a laboratory control sample (LCS) for every batch of 20 samples. The LCS is purchased from a source independent of the laboratory and different from the source of calibration standards. If its determined concentrations are not within ±10% of the stated values then analysis must be suspended until the source of the problem is identified and corrected.

8.2.3 For all determinations the laboratory must analyze the continuing calibration verification or CCV (a mid-range check standard) and a continuing calibration blank or CCB following the calibration, after every tenth analytical sample (or more frequently, if required) and at the end of the sample run. Analysis of the CCV and CCB following the calibration must verify that the instrument is within ±10% of true value for the standard. Subsequent analyses of the CCV must verify the calibration is still within ±10%. If the calibration cannot be verified within the specified limits, reanalyze the CCV. If the second analysis of the CCV confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable CCV must be reanalyzed once the problem has been corrected. The analysis data of the CCB and CCV must be kept on file with the sample analyses data.

8.2.4 Analyze an ERA sample with each analytical batch, and evaluate the results using the acceptance criteria limits provided on the certificate of analysis from the vendor, per each lot.

8.2.5 See section 11.1.3 of USEPA Method 26A for requirements.

8.3 Assessing Analyte Recovery and Data Quality

8.3.1 The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples. Analyze one matrix spike (MS) and one matrix spike duplicate (MSD) for every 10 samples. Calculate accuracy as percent recovery (Section 10.2). In each case the MS aliquot must be a duplicate of the aliquot used for sample analysis. The analyte concentration must be high enough to be detected above the original sample and should not be less than four times the MDL. If the recovery of any analyte falls outside the required control limits of 80-120%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses. If the relative percent difference (%RPD) between the MS/MSD pair falls outside the required 0-20% limits the analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses. The affected samples batches must be re-prepped and reanalyzed.

8.3.1.1 If the concentration of a spike is less than 25% of the background concentration of the matrix the matrix recovery should not be calculated.

8.3.1.1 If the recovery of any analyte falls outside the designated MS recovery range and the laboratory performance for that analyte is shown to be in control (Section 8.2), the recovery problem encountered with the MS is judged to be either matrix or solution related, not system related.

8.3.2 In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of different columns and/or eluents, to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the initial demonstration of performance procedure in Section 8.1.

8.3.3 It is recommended that other lab performance procedures be adopted when applicable, depending on the nature of the samples. Some productive quality assurance practices to utilize are: analyzing field duplicates, participating in performance evaluation sample studies, dilution tests, and analyzing other certified check standards.

8.3.4 Matrix spikes are not required for Method 26A.

8.3.5 The acceptance criteria, frequency of control items and appropriate corrective action is listed in the table below:

Quality Control Items, Frequency and Corrective Action

QC Item	Frequency	Acceptance Criteria	Corrective Action
LRB	Every batch of 20 samples.	2.2 x MDL	Reanalyze to confirm. If confirmed, re-prep and reanalyze batch.
LCS	Every batch of 20 samples.	90-110%	Reanalyze to confirm. If confirmed, re-prep and reanalyze batch.
CCV	Before and after each batch. Every 10 samples.	90-110%	All samples following the last acceptable CCV must be reanalyzed.
CCB	Every 10 samples.	2.2 x MDL	All samples following the last acceptable CCB must be reanalyzed
MS	Every 10 samples.	80-120%	See Section 8.3.1
MSD	Every 10 samples.	80-120%, RPD 0-20%	Same as MS. RPD - See Section 8.3.1
ERA	Once per analysis. Applies to Chloride only.	As per Certificate of Analysis for given lot for PT acceptance.	Reanalyze to confirm. If confirmed, investigate and address the problem, recalibrate if needed. Re-prep and reanalyze batch once issue is resolved.
Soil/Solid LCS	Every batch of 20 extracted samples.	See In-House criteria.	Re-prep batch, reanalyze.
Soil/Solid MS/MSD	Every batch of 20 extracted samples.	See In-House criteria.	Re-prep batch, reanalyze.

9.0 PROCEDURE

9.1 The following extraction should be used for solid materials prior to analyzing (all other samples start at 9.2):

9.1.1 In a 15mL plastic centrifuge tube, weigh out approximately 1g of sample. Add an amount of reagent water equal to 10 times the weight of solid material taken as a sample. Different amounts of sample may be used as needed. Maintain same ratio of reagent water to sample.

9.1.2 Mix the slurry for 10 minutes using a tumbler or a mechanical shaker.

9.1.3 Filter the resulting slurry before injecting using a 0.45µ membrane type filter. This can be done using vacuum filtration or filter and syringe set up. For turbid samples, centrifuging may be necessary before filtration.

9.1.4 Care should be taken to show that good recovery and identification of peaks is obtained with the matrix through the use of spiked samples (Section 5.2).

9.2 Launch Chromeleon software.

9.3. Confirm that the instrument is set to the proper operating parameters according to the manufacturer's instructions.

9.4 Enter start-of-run batch QC. A typical opening QC consists of:

- Blank
- CCV-(date of run)
- CCB
- LCS-(date of run)
- LRB
- ERA-(date of run)

9.5 Add standards, at their appropriate dilution, to clean vials and cap.

9.6 Place vials into autosampler.

9.7 Ready check the software and start the batch through Chromeleon.

9.8 After analysis refer to the spectra and data table that is generated by the software. Assign and adjust peaks as needed (including manual integration for consistent baseline linearity). Confirm that the laboratory standards and other quality control are within the appropriate range.

9.9 Repeat Section 9.2 through 9.6 with unknown samples, analyzing appropriate QC (CCV, CCB, MS, and MSD) as frequently as previously stated in Section 8.0.

9.10 If peak response exceeds the calibration range, dilute the sample and reanalyze.

10.0 CALCULATIONS

10.1 MDL - Calculate the MDL as follows:

$$MDL = t \times S$$

where,
t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t= 3.14 for seven replicates]
S = standard deviation of the replicate analyses

10.2 Percent Recovery – Calculate percent recovery using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where,
R = percent recovery
C_s = fortified sample concentration
C = sample background concentration
s = concentration equivalent of analyte added to sample

10.3 Relative Percent Difference - Calculate RPD as follows:

$$RPD = \frac{\left|C_1 - C_2\right|}{\frac{C_1 + C_2}{2}} \times 100$$

where,
RPD = relative percent difference,
C_1 = first analyte concentration,
C_2 = second analyte concentration.

11.0 DATA REPORTING

11.1 The data is formatted, evaluated and summarized in a table that provides the sampling site, date and time of the analysis, method, sample description, analyst, dilution factor, all applicable QC evaluations and the result. Each requested ion is reported on a separate table and all tables are attached to the printed sample ion chromatogram(s) for review. An example of a data table can be found on the Company network, in the Archived Chromeleon Data Folder.

11.2 All data results are to be entered into the Laboratory Information Management System (LIMS), with hard copies of the data to be retained in project folder, as referenced in Section 5.4.1.

11.3 Chromeleon raw data archiving should be performed quarterly, or as often as necessary to prevent data loss due to computer malfunction. This is done by exporting the sequences as .cmbx files onto an external hard drive or Company network. In the Data tab of Chromeleon, perform the following steps to export the sequence:

- Click on “Audit Trails”
- Highlight the sequence(s) to save
- Click **File** and select **Send To** from the drop-down box; a Send To window will open
- Click **Start;** a Save As window will open
- **Save** to the external hard drive or Company network as .cmbx file (Chromeleon Back up)

CONSUMERS
ENERGY

Chemistry Department

Standard Analytical Procedure

PROC CHEM-2.5.55
PAGE 1 OF 8
REVISION 0

TITLE: FILTERABLE (TDS) RESIDUE

Written or Revised by Deanne Terrell
Level I or Above
Date 6/18/2020

Technical Review / Approval by
Level III (not author)
Date 06/18/20

Administrative Approval by
Department Head
Date 06/18/20

TITLE: FILTERABLE (TDS) RESIDUE

1.0 SCOPE

1.1 The purpose of this procedure is to set forth a method by which the Chemistry Department of Laboratory Services will analyze environmental and processed waters for filterable residue (TDS).

1.2 This SAP follows the guidelines of SM 2540C, Total Dissolved Solids Dried at 180ºC.

2.0 APPLICABLE DOCUMENTS AND REFERENCES

2.1 Chemistry Department Standard Operating Procedures, as applicable

2.2 Standard Methods for the Examination of Water and Wastewater, 23rd Edition.

3.0 DEFINITIONS

3.1 Total Residue – The sum of the homogenous suspended and dissolved materials in a sample.

3.2 Filterable (Dissolved) Residue – Solids capable of passing through a glass fiber filter and dried to constant weight at 180°C. Total Dissolved Solids (TDS) – The portion of sample that passes through a filter.

4.0 SUMMARY OF METHOD

4.1 Total Dissolved Solids – A well-mixed sample is filtered through a standard glass fiber filter and dried to constant weight at 180°C.

5.0 PREREQUISITES

5.1 MEASURING AND TEST EQUIPMENT

5.1.1 Glass Fiber Filter Discs – 4.7 cm without organic binder as listed below:

- TDS – Environmental Express # F92447MM1.5 micron, binderless Borosilicate Glass Fiber or equivalent.

5.1.2 StableWeigh 6-Place Filling Station or Suction Flask, Filter Holder, and Membrane Filter Funnel.

5.1.3 Modular oven rack and weighing bracket.

5.1.4 StableWeigh TDS Sample Vessels (sample bags) or 100 mL Gooch Crucibles.

5.1.5 Heater – 24-Position, StableWeigh TDS HotBlock or a controlled electric hot plate, heat lamp, or steam bath for maintaining temperature of evaporating sample near boiling point.

5.1.6 Drying Oven – 180°C ± 2°C

5.1.7 Desiccator.

5.1.8 Analytical Balance – Capable of weighing to 0.1 mg along with static diffuser such as Mettler Toledo Balance Deionizer & Power Supply, Item No.: EE1133696.

5.1.9 100 mL Class A Graduated Cylinder.

5.2 REAGENTS

5.2.1 Water – Distilled water shall be ASTM D1193, Type III, or better

5.2.2 Laboratory Control Sample – ERA Cat# 506 or equivalent.

5.3 CALIBRATION REQUIREMENTS

5.3.1 The balance must be calibrated according to CHEM-1.2.7, Balance Calibration, Verification and Usage.

5.4 QUALITY CONTROL DOCUMENTS AND RECORDS

5.4.1 The Quality Control elements required for this SOP are listed in Tables 2020: I and 2020: II of the Standards Methods referenced in Section 2.2.

5.4.2 Due to the nature of the analysis and consistent with the requirements of the approved analytical method for NPDES (SM2540C), some of the twelve essential quality control elements published at 40 CFR Part 136.7 are not applicable for Total Dissolved Solids determination. They are the following:

- Method Detection Limit (MDL)
- Matrix Spike and Matrix Spike Duplicate (MS/MSD)
- Internal Standards or Surrogate Standards
- Calibration, initial and continuing (ICV/CCV)

5.4.3 The precision and bias of the method is evaluated through the analysis of duplicate samples, along with a Laboratory Reagent Blank (MB or LRB) and a Laboratory Control Sample (LCS) with each analytical batch. The frequency requirements and acceptance criteria are listed in Section 8.

5.5 PERSONNEL REQUIREMENTS

All tests and data reporting shall be performed by certified persons of Level I or above, in the appropriate discipline. The project report shall be issued and reviewed by a certified person of Level II or above, in the appropriate discipline. The project report, if indicated on the work request (or form similar in intent), may require approval from a certified person of Level III, in the appropriate discipline.

For projects where reporting under the 2009 TNI Standard has been requested, all tests and data reporting shall be performed by analysts with a completed initial, or an on-going Demonstration of Capability (IDOC/DOC), as applicable.

5.6 ENVIRONMENTAL CONDITIONS

5.6.1 Containers – Generally, samples are collected in plastic containers.

5.6.2 Preservation – Upon collection, samples are refrigerated at ≤ 6°C to minimize microbiological decomposition of solids. Bring samples to room temperature before analysis.

5.6.3 Maximum holding time for refrigerated sample is 7 days from sample collection. Begin analysis as soon as possible because of the impracticality of preserving samples.

6.0 PRECAUTIONS

Observe normal safety practices as specified in the latest online revision of the Consumers Energy Chemical Hygiene Plan.

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. For guidance on proper disposal of unused samples, stock chemicals and reagents refer to SOP CHEM-1.2.08 “Handling and Disposal of Lab Testing Waste”.

7.0 LIMITATIONS AND ACTIONS

7.1 INTERFERENCES

7.1.1 For all residue determinations – Excessive residue is sample vessel will crust over and entrap water that will not be driven off during drying. Total residue should be limited to approximately 200 mg (allowance for the limit to be within 10% for brine samples).

7.2 CORRECTIVE ACTION

Initiate corrective action to identify and correct the issues whenever results for LCS, LRB and duplicate sample analysis or are outside the limits summarized in Section 8, below.

8.0 ACCEPTANCE CRITERIA

Quality Control Items, Frequency and Corrective Action

QC Item	Frequency	Acceptance Criteria	Corrective Action
LCS (ERA)	Every batch of 20 samples.	As stated on the certificate of analysis or in-house derived limits	Re-prep and reanalyze batch.
LRB	Every batch of 20 samples.	< 2.5 mg/L	Re-prep and reanalyze batch.
Duplicate	Every batch and every 10 samples.	RPD of 0-5% of average weight	Re-prep and reanalyze batch.
Drying cycle	Every sample	4% of previous weight or 0.5 mg, whichever is less	Repeat drying cycle until within limits

9.0 PROCEDURE (Using 6-Place Filling Station)

9.1 PREPARATION OF EQUIPMENT

9.1.1 Pre-heat TDS Hot Block.

9.1.2 Insert pre-weighed sample vessel into support holder of the 6-place filling station.

9.1.3 Insert filter into filtration apparatus and replace filter funnel. Apply vacuum and wash filter with three successive 20 mL rinses of reagent water. Continue suction to remove all traces of water. Discard washings. If using washed and dried filters, there will be no need to rinse the filter.

9.2 SAMPLE ANALYSIS

9.2.1 Remove samples from refrigerator and allow them to equilibrate to room temperature, ±2°C. Measure and record room temperature and the min/max sample temperature prior to analysis on the worksheet presented as an example in attachment A, bench book, electronic spreadsheet, or equivalent form in intent.

9.2.2 Transfer 50-100 ml of well mixed sample using a 100 mL graduated cylinder to a filter funnel with applied vacuum.

9.2.3 Wash with three successive volumes of approximately 10 mL of reagent water. Then rinse sides of filtering funnel. This will ensure complete transfer of sample. Continue suction until all visible water has been removed from the filter.

9.2.4 Place sample vessel in TDS Hot Block until evaporated to dryness.

9.2.5 Place sample vessel in drying rack and dry evaporated sample for at least 1 hour in an oven at 180 ± 2ºC, cool in a desiccator to balance temperature, and weigh. Repeat drying cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% of previous weight or 0.5 mg, whichever is less. Record all weights and reweighs on the worksheet presented as an example in Attachment A, bench book, electronic spreadsheet, or equivalent form in intent.

9.3 Analyze an LCS (reference standard), LRB and a sample duplicate through all procedure steps in section 9.2 above, as applicable for TDS.

10.0 PROCEDURE (Using Suction Flask, Filter Holder, and Membrane Filter Funnel)

10.1 PREPARATION OF EQUIPMENT

10.1.1 Insert filter with wrinkled side up into filtration apparatus. Apply vacuum and wash filter with three successive 20 mL rinses of reagent water. Continue suction to remove all traces of water. Discard washings.

10.1.2 Preparation of evaporating dishes: Heat a clean dish to 180 ±2°C for one hour. Cool in desiccator and store until needed. Weigh immediately before use.

NOTE: Record all weights to nearest 0.1 mg

10.2 SAMPLE ANALYSIS

10.2.1 Remove samples from refrigerator and allow them to equilibrate to room temperature, ±2°C. Measure and record room temperature and the min/max sample temperature prior to analysis on the worksheet presented as an example in attachment A, bench book, electronic spreadsheet, or equivalent form in intent.

10.2.2 Transfer 50-100 ml of well mixed sample using a 100 mL graduated cylinder to a glass fiber filter with applied vacuum.

10.2.3 Wash with three successive 10 ml volumes of reagent water and continue suction for about three minutes after filtration is complete.

10.2.4 Transfer filtrate and washings to a weighed evaporating dish and evaporate to dryness using heat lamp. If necessary, add additional portions of sample to the same dish after evaporation.

10.2.5 Dry evaporated sample for at least 1 hour in an oven at 180 ± 2ºC, cool in a desiccator to balance temperature, and weigh. Repeat drying cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% of previous weight or 0.5 mg, whichever is less. Record all weights and reweighs on the worksheet presented as an example in Attachment A, bench book, electronic spreadsheet, or equivalent form in intent.

10.3 Analyze an LCS (reference standard), LRB and a sample duplicate through all procedure steps in section 9.2 and 9.3 above.

11.0 CALCULATIONS

11.1 SAMPLE CONCENTRATION DETERMINATION

11.1.1 TDS concentration in mg/L = $\frac{(A-B)\ x\ 1000}{C}$

Where: A = Weight of sample vessel + dried residue, mg
B = Weight of sample vessel, mg
C = Sample volume, mL

12.0 DATA REPORTING

12.1 Report the results on the worksheet presented as an example in Attachment A. An electronic spreadsheet or an equivalent form in intent can be used as well. If a spreadsheet is used for calculation and/or reporting, it shall be validated before use, with the cells used for calculation protected. Save the spreadsheet on the company network, at K:\CHEM\Total Suspended or Dissolved Solids.

12.2 Place the completed Attachment A (or equivalent reporting form) into the project folder and scan it as a PDF document into the corresponding analysis batch in LIMS.

Filterable (TDS) Residue Worksheet

SM2540C

Analysis Batch: ____________________
Analysis Date: ____________________
Analysis By: ____________________
Reviewed By: ____________________
Review Date: ____________________
Vessel Lot No.: ____________________
Vessel Item No.: ____________________

Analytical Balance M&TE No: ____________________
Balance Calibration Due Date: ____________________
LCS Lot No. /Proc. No./ Exp. Date: ____________________
Measured Concentration: ____________________
Acceptable Range Concentration: ____________________
Min/Max Sample Temperature: ____________________
IR M&TE No/Exp. Date: ____________________
Room Temperature: ____________________
Thermocouple M&TE#: ____________________

	In / Time	In / Temp (°C)	Out / Time	Out / Temp (°C)
1st Weighing				
2nd Weighing				

Oven M&TE No: ____________ Thermocouple M&TE No: ____________ Probe M&TE No: ____________

ANALYSIS RESULTS NOTE: Limit load to 2.5 - 200 mg NOTE: Record all weights to nearest 0.1mg

Sample Vessel ID	Sample Aliquot ID	Sample Volume (mL) **(C)**	1st Vessel and dry sample (mg) **(A_1)**	2nd Vessel and dry sample (mg) **(A_2)**	Dry Sample Differenc (mg) **(A_1-A_2)**	Vessel Mass (mg) **(B)**	Sample Load (mg) **(A_2-B)**	Final Concentration (mg/L) $\frac{(A_2 - B)x1000}{C}$

Appendix E
Statistical Evaluation Methods

Groundwater Statistical Evaluation Plan

JH Campbell Power Plant
Dry Ash Landfill
West Olive, Michigan

October 2017, Revised November 2021

Prepared For:

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TABLE OF CONTENTS

FIGURES

1.0 Introduction

1.1 Regulatory Framework

Pursuant to the Coal Combustion Residuals (CCR) under the Resource Conservation and Recovery Act (RCRA) (the CCR Rule) and the State of Michigan enacted Public Act No. 640 of 2018 (PA 640) to amend the Natural Resources and Environmental Protection Act, also known as Part 115 of PA 451 of 1994, as amended (a.k.a., Michigan Part 115 Solid Waste Management), the owner or operator of a CCR unit must develop the groundwater sampling and analysis program to include selection and certification of the statistical procedures to be used for evaluating groundwater in accordance with §257.93 and R 299.4908 of the Part 115 Solid Waste Management Rules. This certification must include a narrative description of the statistical method that will be used for evaluating groundwater monitoring data.

TRC prepared this Groundwater Statistical Evaluation Plan (Statistical Plan) for the JH Campbell (JHC) Dry Ash Landfill on behalf of Consumers Energy. This Statistical Plan was prepared in accordance with the requirements of §257.93 and R 299.4908 and describes how data collected from the groundwater monitoring system will be evaluated. As part of the evaluation, the data collected during detection monitoring events are evaluated to identify statistically significant increases (SSIs) in detection monitoring constituents (Section 11511a.(3)(c) of PA 640) to determine if concentrations in detection monitoring well samples exceed background levels. Data collected from assessment monitoring events are evaluated for statistically significant exceedances of an established groundwater protection standard (GWPS) for constituents in Sections 11511a(c)(3) and 11519b(2) of PA 640 to evaluate the risk associated with those constituents.

The CCR Rule and Part 115 are not prescriptive with regards to the actual means and methods to be used for statistically evaluating groundwater data, and there is flexibility in the method selection, as long as specific performance metrics are met. Statistical methods that meet the performance objectives of the CCR Rule and Part 115 are described in USEPA's *Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities, Unified Guidance* (*Unified Guidance,* USEPA, 2009).

2.0 Groundwater Monitoring System

2.1 Groundwater Monitoring System

A groundwater monitoring system has been established for the JHC Dry Ash Landfill, which established the following locations for detection monitoring. The locations are shown on Figure 1.

Background:

- JHC MW-15023
- JHC MW-15024
- JHC MW-15025
- JHC MW-15026
- JHC MW-15027
- JHC MW-15028

Downgradient:

- JHC MW-15017
- JHC MW-15018
- JHC MW-15031
- JHC MW-15035
- JHC MW-15036
- JHC MW-15037
- MW-B3 REPLACEMENT
- MW-B4 REPLACEMENT

2.2 Constituents for Detection Monitoring

R 299.4440 describes the requirement for detection monitoring. The detection monitoring parameters are identified in Section 11511a(3)(c) of PA 640 and consist of the following:

- Boron
- Calcium
- Chloride
- Fluoride
- Iron
- pH
- Sulfate
- Total Dissolved Solids (TDS)

2.3 Constituents for Assessment Monitoring

Assessment monitoring per R 299.4441 is required when a SSI over background has been detected for one or more of the detection monitoring constituents identified in Section 11511a(3)(c). As required in Section 11519b(2) the following assessment monitoring constituents will be monitored:

- Antimony
- Arsenic
- Barium
- Beryllium
- Cadmium
- Chromium
- Cobalt
- Copper
- Fluoride
- Lead
- Lithium
- Mercury
- Molybdenum
- Nickel
- Selenium
- Silver
- Thallium
- Vanadium
- Zinc
- Radium 226 and 228 (combined)

3.0 Statistical Analysis

Groundwater sampling and analytical requirements are described in R 299.4908. The owner or operator of the CCR unit must select a statistical method specified in R 299.4908(1) to be used in evaluating groundwater monitoring data. The test shall meet the performance standards outlined in R 299.4908(2). The goal of the statistical evaluation plan is to provide a means to formulate an opinion or judgement as to whether the CCR unit has released contaminants into groundwater. This plan describes the statistical procedures to be used to determine if a statistically significant increase (SSI) or in the case of pH, a statistically significant difference (SSD), indicating that data is from a different population than background. This plan was developed using applicable guidance, including the Unified Guidance. In addition to using applicable guidance documents, commercially available statistical evaluation tools will be applied to the JHC Dry Ash Landfill groundwater data to develop statistically derived limits so that detection monitoring results can be compared to background.

The CCR Rule and Part 115 allow a variety of methods for conducting statistical evaluations. The specific procedure for a given data set depends on several factors including the proportion of the data set with detected values and the distribution of the data. It is generally anticipated, that the tolerance or prediction interval procedure will be the preferred method of conducting detection monitoring data evaluation to the extent that the data support the use of that method. For assessment monitoring, the preferred statistical evaluation method for comparisons to a fixed standard will be confidence limits as detailed in Section 5.0. This statistical procedure is described below in this section of the plan and in detail in the Unified Guidance.

3.1 Establishing Background

Background groundwater monitoring was conducted for constituents in Appendix III and Appendix IV of the CCR Rule from December 2015 through August 2017 using the background monitoring wells in accordance with the JH Campbell Monitoring Program Sample and Analysis Plan (SAP) (ARCADIS, 2016). Background will be established for the Section 11511a(3)(c) constituents not already included in the CCR Rule Appendix III (i.e., iron) throughout eight sampling events. Per R 299.4907(7), the owner or operator of the CCR unit must establish background groundwater quality in hydraulically upgradient or background well(s). The development of a groundwater statistical evaluation program for detection monitoring involves the proper collection of background samples, regardless of whether an inter-well or intra-well monitoring strategy is implemented. Background may be established at wells that are not located hydraulically upgradient from the unit if it meets the requirement of R 299.4906(1)(a). A determination of background quality may include sampling of wells that are not hydraulically upgradient of the CCR management area where:

1. Hydrogeologic conditions do not allow the owner or operator of the CCR unit to determine what wells are hydraulically upgradient; or
2. Sampling at other wells will provide an indication of background groundwater quality that is as representative as or more representative than that provided by the upgradient wells.

The purpose of obtaining adequate background groundwater data is to approximate, as accurately as possible, the true range of ambient concentrations of targeted constituents. Background groundwater data should eliminate, to the extent possible, statistically significant concentration increases not attributable to the CCR unit. Specifically, the owner or operator of a CCR unit must install a groundwater monitoring system that consists of a sufficient number of wells, installed at appropriate locations and depths, to yield groundwater samples from the uppermost aquifer that accurately represent the quality of background groundwater that has not been affected by leakage from a CCR unit. The sampling frequency should be selected so that the samples are physically independent. These background groundwater parameters can be adequately qualified by doing the following:

- Collecting the minimum number of samples that satisfy the requirements of the statistical methods that are used (*i.e.,* that result in adequate statistical power);
- Incorporating seasonal and/or temporal variability into the background data set; and
- Incorporating the spatial component of variability into the background data set (*i.e.,* the variability that comes with obtaining samples from different locations within the same groundwater zone).

The initial background/baseline sampling period is at least eight independent events. This provides a minimal background data set to initiate statistical comparisons. Over time, the short baseline period may result in a high risk of false positive statistical results. The facility may periodically update background data to account for variability in background conditions. The *Unified Guidance* recommends that background data be updated every 4 to 8 measurements (i.e., every two to four years if samples are collected semi-annually, or one to two years if samples are collected quarterly). The background data will be reviewed for trends or changes that may necessitate discontinuation of earlier portions of the background data set. Updates to the background statistical limits will be submitted to the Michigan Department of Environment, Great Lakes, and Energy (EGLE) for approval.

3.2 Data Evaluation and Data Distributions

Consumers Energy will evaluate the groundwater data for each constituent included in the groundwater monitoring program using inter-well tolerance or prediction limits. The tolerance or prediction interval statistical procedure establishes an interval that bounds the ranges of expected concentrations representative of unaffected groundwater using the distribution of background data. The upper tolerance or prediction limit of that interval is then used for comparison to the concentration level of each constituent in each compliance well. Development of the tolerance or prediction limits used for comparison during detection monitoring will be conducted in accordance with the Unified Guidance. The following is a summary of descriptive statistics and tolerance or prediction limit choices.

3.2.1 Background Determination

Statistical limits will be calculated after the collection of a minimum of eight independent samples. The analytical results from the eight “background” samples will be used to determine the statistical limits for each individual parameter. For inter-well comparisons, background data should be “pooled” creating a single, combined background dataset from the background monitoring wells.

The background dataset (and hence the prediction limits) will be updated as appropriate (as discussed above in Section 3.1) to maintain necessary statistical sensitivity. New data will be compared to the existing background data set to determine if there are outlier values, and whether the data are statistically similar. If there are no outliers and the data are statistically similar, the new data will be added to the existing background data set.

3.2.2 Outlier Evaluation

Outliers and anomalies are inconsistently large or small values that can occur as a result of sampling, analytical, or transcription errors; laboratory or field contamination; or shelf-life exceedance; or extreme, but accurately detected environmental conditions (*e.g.*, spills). Data will be reviewed graphically using tools such as time concentration trend plots, box and whisker plots and/or probability plots to illustrate and identify outliers, trends, or otherwise unusual observations at each monitoring location. This will be accomplished prior to further in-depth review of the data sets to identify any obvious field or laboratory anomalies. Data points that are determined to be non-representative will be ‘flagged’ for further detailed evaluation prior to removing from the background data or designating as an outlier.

3.2.3 Testing for Normality

Statistical tests often assume that data are normally distributed or that data can be normalized by various standard methods. The assumption of normality can be tested in various ways. Formal normality testing such as utilizing the Shapiro-Wilk test (for $n<50$) or the Shapiro-Francia Test (for $n>50$) or calculation of a coefficient of skewness may be utilized in accordance with the *Unified Guidance*. Alternatively, graphing data on a probability plot can also be used to test for normality. If the data appear to be non-normal, mathematical transformations of the data may be utilized such that the transformed data follow a normal distribution (e.g., lognormal distributions). Alternatively, non-parametric tests may be utilized when data cannot be normalized.

The following are guidelines for decision making during normality testing:

1. If the original data show that the data are not normally distributed, then apply a natural log-transformation to the data and test for normality using the above methods.
2. If the original or the natural log-transformed data confirm that the data are normally distributed, then apply a normal distribution test.
3. If neither the original nor the natural log-transformed data fit a normal distribution, then apply a distribution-free test.

3.2.4 Evaluation of Non-Detects

Background concentrations that are reported as less than the practical quantitation limit (PQL) (herein referred to as non-detects) will be evaluated differently, depending upon the percentage of non-detects to the reported concentrations for a given parameter at a given monitoring well.

The evaluation of non-detects was as follows:

Less Than 15% Non-detects

For data that was normally or lognormally distributed and less than 15% non-detects, one-half the value of the method detection limit will be used to calculate the prediction limit. If normally or lognormally cannot be met using one-half of the method detection limit, and if the method detection limits were equal, alternating zero with the value of the method detection limit will be considered in order to determine the normality of the data set.

15% to 50% Non-detects

If more than 15% but less than 50% of the overall data are less than the detection limit, either Aitchison’s adjustment, or Cohen’s adjustment, or the Kaplan Meijer adjustment will be used to determine the statistical limits in accordance with the *Unified Guidance*.

51% to 100% Non-detects

For data sets that contain greater than 50% non-detects, the non-parametric statistical limits will be utilized as described below.

3.3 Parametric Tolerance or Prediction Limits

Tolerance and prediction intervals are similar approaches to establish statistical ranges constructed from background or baseline data. However, tolerance limits define the range of data that fall within a specified percentage with a specified level of confidence (where a proportion of the population is expected to lie), whereas prediction limits involve predicting the upper limit of possible future values based on a background or baseline data set and comparing that predicted limit to compliance well data.

Inter-well tolerance or prediction limits are calculated using the pooled background data set. The tolerance or prediction limit will be calculated in accordance with the Unified Guidance. If the data set is log-normally distributed, the tolerance or prediction limits will be calculated using the log-normally transformed data, and subsequently un-transformed to normal units.

R 299.4908(2)(b) states that for multiple comparisons, each testing period should have a Type I error rate no less than 0.05 while maintaining an individual well Type I error rate of no less than 0.01. Per R 299.4908(2)(d), these Type I limits do not apply directly to tolerance intervals or prediction intervals; however, the levels of confidence for the tolerance or prediction limit approach must be at least as effective as any other approach based on consideration of the number of samples, distribution, and range of concentration values in the background data set for each constituent.

3.4 Non-Parametric Tolerance or Prediction Limits

Parameters that consist of mainly non-detect data usually violate the assumptions needed for normal based parametric tolerance or prediction intervals. Therefore, as recommended in the Unified Guidance, the non-parametric tolerance or prediction limit method will be chosen.

A non-parametric upper tolerance or prediction limit is constructed by setting the limit as a large order statistic selected from background (*e.g.*, the maximum background value). This method has lower statistical power than parametric methods; therefore, it is important to control outliers within the dataset to maintain adequate statistical power that this method can provide. Due to the lack of statistical power of this method, it will only be used when other methods are not available.

3.5 Double Quantification Rule

The double quantification rule is discussed in Section 6.2.2 of the Unified Guidance. In the cases where the background dataset for a given well is 100% non-detect, a confirmed exceedance is registered if any well-constituent pair exhibits quantified measurements (i.e., at or above the reporting limit) in two consecutive sample and resample events. This method will be used for non-detect data sets.

3.6 Verification Resampling

In order to achieve the site wide false positive rates (SWFPR) recommended in the Unified Guidance, a verification resampling program is necessary. Without verification resampling, the SWFPR cannot be reasonably met, and much larger statistical limits would be required to achieve a SWFPR of 5% or less. Furthermore, the resulting false negative rate would be greatly increased. Under these circumstances, if there is an exceedance of a tolerance limit or prediction limit for one or more of the parameters, the well(s) of concern will be resampled within 30 days of the completion of the initial statistical analysis. Only constituents that initially exceed their statistical limit (i.e., have no previously recorded SSIs) will be analyzed for verification purposes. This verification sampling must be performed within the same compliance period as the event being verified. If the verification sample remains statistically significant, then statistical significance will be considered. If the verification sample is not statistically significant, then no SSI will be recorded for the monitoring event.

4.0 Evaluation of Detection Monitoring Data

4.1 Statistical Evaluation during Detection Monitoring

According to R 299.4440(8), if the facility determines, pursuant to R 299.4908(5), that there is a statistically significant increase (SSI) over background levels for one or more of the detection monitoring constituents, the facility will, within 14 days of the determination of a SSI, place a notice in the operating record that indicates which constituents show a SSI and notify EGLE. Within 45 days of detecting a SSI, the facility will prepare an assessment monitoring plan **<or>** demonstrate that:

- A source other than the CCR unit caused the SSI, or
- The SSI resulted from error in sampling, analysis, statistical evaluation, or natural variation in groundwater quality.

The owner or operator must complete a written demonstration (i.e., Alternative Source Demonstration, ASD), of the above within 30 days of confirming the SSI and submit the ASD to EGLE as required by R 299.4440(9). If a successful ASD is completed, a certification from a qualified professional engineer is required, and the CCR unit may continue with detection monitoring. If a successful ASD is made, the facility must determine if the constituents in groundwater render the unit unmonitorable in accordance with R 299.4440(9)(b).

If a successful ASD is not completed within the 30-day period, within 15 days of notification from EGLE that the demonstration is unsuccessful, the owner or operator of the CCR unit must prepare an assessment monitoring plan as required in R 299.4441 and submit a response action plan as required in R 299.4442. The facility will initiate an assessment monitoring program within 60 days of the submittal of the assessment monitoring plan as required in R 299.4441 and within 90 days of detecting a SSI, described further in Section 5.

5.0 Assessment Monitoring

As discussed in Section 4, the facility must begin assessment monitoring for the CCR unit if a SSI is identified, and the SSI cannot be attributed to an ASD. Per R 299.4441, assessment monitoring must begin within 60 days of submitting an assessment monitoring plan. Per the CCR Rule, assessment monitoring must begin within 90 days of identification of a SSI that is not attributed to an alternative source. Wells included in the groundwater monitoring system will be sampled for assessment monitoring constituents included in Section 11519b(2) of PA 640. Within 14 days of receiving sample results, the owner or operator will place a notice of the detected assessment monitoring parameters in the operating record and notify EGLE as required under R 299.4441(4)(a). Within 90 days of obtaining the results from the first assessment monitoring event, all of the wells will be sampled for detection monitoring constituents and the detected assessment monitoring constituents in the initial assessment monitoring event. Background will be established for the Section 11519b(2) constituents not already included in the CCR Rule Appendix IV (i.e., copper, nickel, silver, vanadium, and zinc) throughout eight sampling events in accordance with R 299.4441(4)(c).

If assessment monitoring is triggered pursuant to R 299.4440(8), data are compared to Groundwater Protection Standards (GWPSs) or background groundwater quality. The CCR Rule [§257.95(h)] and the Part 115 Rules [R 299.4441(4)(d)] require GWPSs to be established for assessment monitoring constituents (Section 11519b(2)) that have been detected during baseline sampling, per Part 115 this includes establishing GWPSs for all detected detection monitoring constituents listed in Section 11511a(3(c)). The GWPS will be set at the lowest of the EPA maximum contaminant level (MCL), the EPA Regional Screening Level (RSL), or the lowest applicable Michigan Part 201 residential criteria (Part 201 RC), or a value based on background data. The lowest of the MCLs or RSLs or applicable Part 201 RC will be the GWPSs unless the background concentration is greater than the MCL or RSL or applicable Part 201 RC, in which case, the statistically-determined background value becomes the GWPS. For GWPSs that are established using background, tolerance limits are anticipated to be used to calculate the GWPS. It is anticipated that the background will be updated every two years, along with the resulting GWPS, consistent with the *Unified Guidance*. If additional assessment monitoring constituents become detected during the assessment monitoring, GWPSs will be developed for those constituents in the same manner as the initial parameters.

Consistent with the *Unified Guidance*, the preferred method for comparisons to a fixed standard will be confidence limits. An exceedance of the standard occurs when the 99 percent lower confidence level of the downgradient data exceeds the GWPS. Confidence intervals will be established in a manner appropriate to the data set being evaluated (proportion of non-detect data, distribution, etc.). If the statistical tests conclude that an exceedance of the GWPS or background has occurred, verification resampling may be conducted by the facility. Once the resampling data are available, the comparison to the GWPS or background will be evaluated.

Additionally, it is noted in R 299.4441(5) that if the concentrations of all assessment monitoring constituents are shown to be at or below background values using statistical procedures in R 299.4908 for two consecutive sampling events, the owner or operator may return to detection monitoring of the CCR unit. A notification must be prepared stating that the detection monitoring is resuming for the CCR unit.

If the statistical tests and verification resampling conclude that an exceedance of the GWPS has occurred, the facility will conduct an assessment of corrective measures, select a remedy for affected groundwater, and implement a remedial action plan in accordance with the requirements and schedules outlined in R 299.4443, R 299.4444, and R 299.4445.

6.0 References

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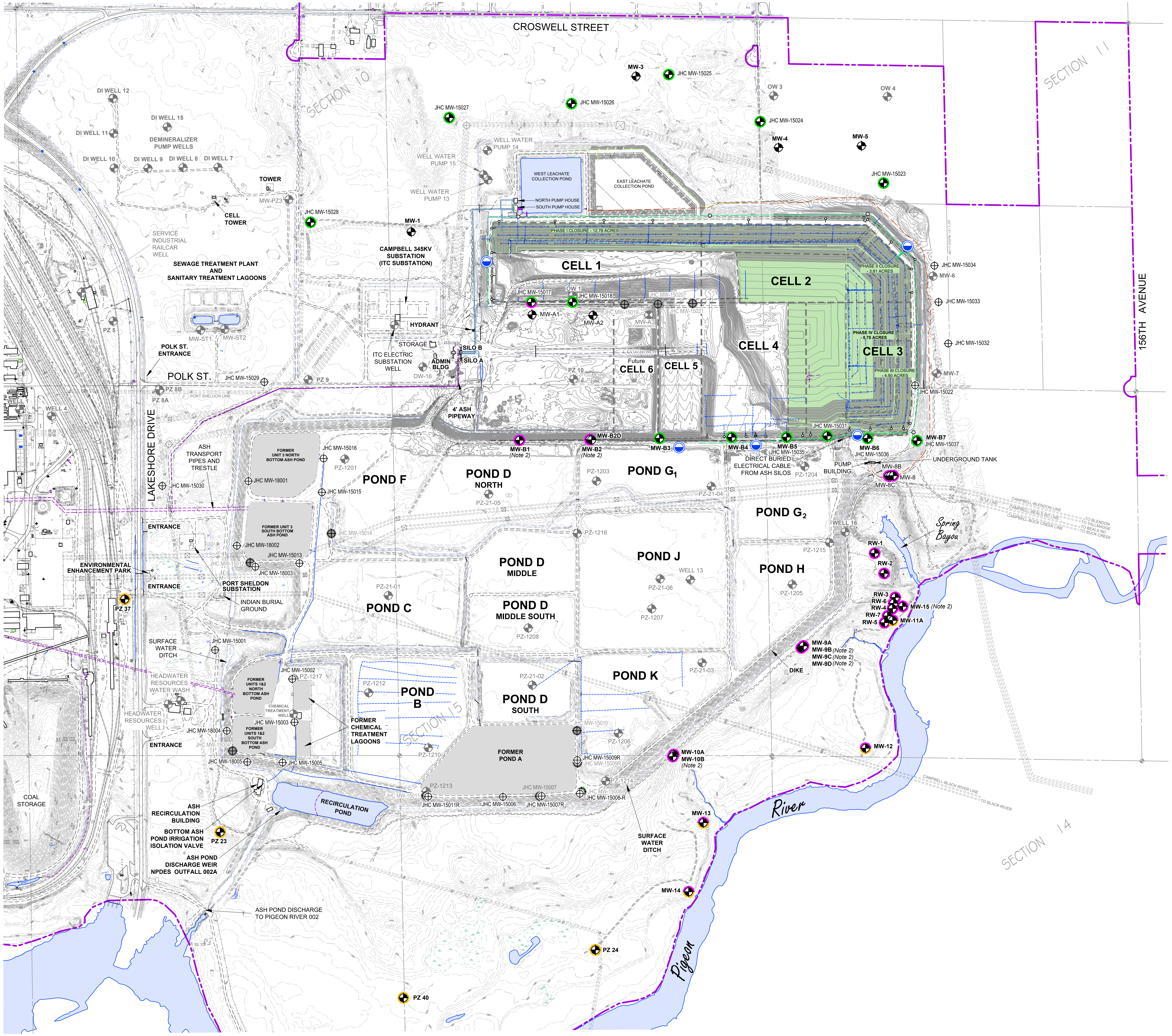
Figures



LEGEND

— - - —	APPROXIMATE PROPERTY BOUNDARY
—— - - ——	SECTION LINE
┼┼┼┼┼	RAILROAD TRACKS
■■■■■■	CELL BOUNDARY
—x——x——x—	FENCELINE
— · · — · · —	DITCH
═══════	UNPAVED ROAD OR DRIVE
▤▤▤▤▤▤	OVERHEAD POWER LINES
——E——	ELECTRIC LINE
— — S— — —	SANITARY PIPE
——U——	UTILITY
— — — — —	LEACHATE PIPE
———————	ASH PIPE
- - - - - - -	PIPELINE
- - - - - - -	IRRIGATION PIPE
🟦	WATER
🟩	FACILITY COVER
	WETLAND
∅	POLE
MW-1 ⊕	MONITORING WELL
MW-7 ⊕	WELL
JHC MW-15034 ⊕	RCRA MONITORING PROGRAM WELL
JHC MW-15004 ⊕	DECOMMISSIONED RCRA MONITORING PROGRAM WELL
MW-5 ⊕	DRY ASH LANDFILL HMP MONITORING WELL
PZ-23 ⊕	CELLS B-K HMP WELL
RW-1 ⊕	RAP MONITORING WELL
◒	APPROXIMATE SUMP SAMPLING LOCATION

NOTES

1. BASEMAP DEVELOPED FROM CONSUMERS ENERGY, "CAMPBASE.DWG", DATED 02/10/2014 AND NEDERVELD "CAMPBELL PLANT MONITORING WELLS-CCR MONITORING" , DATED 11/25/2015. PROVIDED BY CONSUMERS ENERGY.
2. RAP SENTINEL WELL

0 360' 720' 1080' 1440'
APPROXIMATE SCALE IN FEET

PROJECT:
CONSUMERS ENERGY
JH CAMPBELL POWER PLANT
WEST OLIVE, MICHIGAN

TITLE:
SITE FEATURES MAP WITH TOPOGRAPHIC CONTOURS

DRAWN BY:	D.STEHLE	PROJ. NO.:	367390.0001.01.03
CHECKED BY:	K.LOWERY	FIGURE 1	
APPROVED BY:	S.HOLMSTROM		
DATE:	NOVEMBER 2021		

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FILE NO.: 367390.0001.01.03.02.dwg

22x34 — USER: ADragan — ATTACHED XREFS: CAMPBASE; Contours Misc; Contours Topo 2020-07-22 wo 7-8-9 bdrs — ATTACHED IMAGES:
DRAWING NAME: \\annarbor-fp2\CADD\PJT\00_TRC\Consumers\JH Campbell\367390\0001\01\03\ 367390.0001.01.03.02.dwg — PLOT DATE: November 04, 2021 - 2:33PM — LAYOUT: FIG02B Site Features

Version: 2017-10-21

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