Phase 2 Remedial Investigation Work Plan Addendum, Revision 1

Niacet Site
400 47th Street
Niagara Falls, New York
Site No. C932172

Prepared For:

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Certification

I, Colin Wasteneys, certify that I am currently a Qualified Environmental Professional as defined in 6 NYCRR Part 375 and that this Site Characterization/ Remedial Investigation Work Plan was prepared in accordance with all applicable statutes and regulations and in substantial conformance with the DER Technical Guidance for Site Investigation and Remediation (DER-10).

Respectfully submitted,

Colin Wasteneys

Colin Wasteneys, P.G.

February 20, 2023

Date

February 2023, Final

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1. Introduction

This Remedial Investigation Work Plan (RIWP) Addendum (Addendum) summarizes the work elements for the additional field investigations and activities to be conducted as part of the Remedial Investigation (RI) at the Niacet Site (the Site) in Niagara Falls, Niagara County, New York (Figure 1-1). This Addendum must be used in conjunction with the RIWP for the Site prepared by AECOM in January 2020 (AECOM, 2020a) and the following supporting documents: Quality Assurance Project Plan (QAPP) dated January 2020 (Appendix F of the RIWP) (AECOM, 2020b) and the Site-specific Health and Safety Plan (HASP) that will be updated prior to commencement of field activities.

1.1 Background

The Site was accepted into the New York State Brownfield Cleanup Program (BCP) pursuant to the Brownfield Cleanup Agreement (BCA) between New York State Department of Environmental Conservation (NYSDEC) and Union Carbide Corporation (Participant), dated February 11, 2019 (NYSDEC, 2019). The RIWP was approved in a letter from NYSDEC dated March 3, 2020. During June and July 2020, AECOM completed the Phase 1 RI field activities consisting of the following primary investigation tasks:

- Completed 18 soil borings and collected 15 surface and 37 subsurface soil samples;
- Performed radiological screening of all drill cuttings and soil samples generated during soil boring activities;
- Installed and developed three groundwater monitoring wells;
- Conducted hydraulic conductivity testing of one existing and three new monitoring wells;
- Collected groundwater samples from one existing and three new monitoring wells;
- Collected one co-located surface water and sediment sample from the concrete-lined cooling water pond; and
- Surveyed all new sampling locations.

Details and results of the Phase 1 RI field activities are presented in the Phase 1 RI Report (RIR) prepared by AECOM and dated May 2021 (AECOM, 2021). Recommendations for additional investigation activities presented in the Phase 1 RIR included the following:

- Investigation of groundwater quality in bedrock through installation of monitoring wells installed to target water bearing zones in the upper 10 to 20 feet of bedrock. The number and locations of bedrock monitoring wells to be installed and sampled will be determined during preparation of the RIWP Addendum.
- Investigation of mercury impacts to overburden soils near off-site surface soil sample BS-02 located to the west across 47th Street. The number and locations of off-site soil sampling locations will be determined during preparation of the RIWP Addendum.
- Additional soil and groundwater investigation to delineate on-site areas where elevated concentrations of Site constituents of potential concern have been identified during Phase 1 RI activities. The number and locations of sampling locations will be determined during preparation of the RIWP Addendum.
- Further evaluation of the 47th Street sewer tunnel and Pike Creek stormwater pipe to evaluate safe collection of sediment and water samples.

In a letter dated March 22, 2022, NYSDEC requested the following RI field activities in addition to the Phase 1 RIR recommendations listed above:

- As discussed, the use of the alternate soil cleanup objective (SCO) for mercury salts will be considered, however, additional sampling and speciation will be required to determine the applicability of the alternate SCO as it pertains to the remedy;
- In addition to the installation of bedrock monitoring wells, additional overburden wells will be installed and sampled in the eastern and southern portions of the Site to provide additional data points and provide a better understanding of groundwater flow. Monitoring wells MW-09 and MW-10 will also be re-sampled during the next sampling event;
- Based on detections of chlorinated volatile organic compounds (CVOCs) in groundwater and the volatility of mercury, a soil vapor intrusion (SVI) evaluation will be completed in buildings occupied by Niacet personnel. A table will be submitted in the addendum RIWP identifying on-site buildings and their current use (i.e. office, lab, warehouse, process building, etc.) to evaluate the need for an SVI evaluation. The use of engineering controls (air exchangers, ventilations systems, etc.) in on-site buildings will be considered when determining the need for inclusion in the SVI evaluation.

Project Objectives

Additional work elements needed to achieve the RI objectives have been identified based upon data gathered from the Site to date and on NYSDEC and New York State Department of Health (NYSDOH) review comments. Additional recommended work tasks are summarized below:

1. Delineation of mercury impacts to overburden soils at the off-site surface soil sample BS-02 located to the west across 47th Street.
2. Delineation of elevated chromium impacted soil at boring SB-118.
3. Investigation of groundwater quality in bedrock through installation of five monitoring wells installed to target water bearing zones in the upper 10 to 20 feet of bedrock.
4. Installation and sampling of four additional overburden monitoring wells in the central, eastern and southern portions of the Site to provide additional data points and provide a better understanding of groundwater flow.
5. Completion of SVI evaluation in select buildings occupied by Niacet personnel.
6. Further evaluation of the 47th Street sewer tunnel and Pike Creek stormwater pipe to evaluate safe collection of sediment and water samples.
7. Additional mercury speciation sampling.

2. Phase 2 Remedial Investigation

Details of the additional RI field activities to be completed during the Phase 2 RI are presented in the following subsections. As stated in Section 1, the Phase 2 activities described below will be completed in accordance with the 2020 RIWP, 2020 QAPP, and the current Site-specific HASP. During the Phase 2 RI field activities, the following supporting tasks which are detailed in the 2020 RIWP but are not detailed in this RIWP Addendum will be completed:

- Prior to commencing any intrusive activities, all locations will be cleared for underground utilities in accordance with Section 3.3.2 of the 2020 RIWP;
- Radiological screening with field instruments of soil cuttings and samples will be performed during all intrusive activities as described in Section 3.3.7 of the 2020 RIWP;
- Air monitoring will be performed in accordance with the Community Air Monitoring Plan procedures outlined in Section 9.0 and Appendix E of the 2020 RIWP. In addition to the Department and DOH being notified of CAMP exceedances, notification will be provided within 24-hours of an exceedance of the site CAMP action limit of 0.125 microgram per cubic meter (µg/m3) for mercury vapor;
- All new investigation locations will be surveyed in accordance with Section 3.3.4 of the 2020 RIWP; and
- Decontamination procedures will be completed in accordance with Section 8.0 of the 2020 RIWP.

2.1 Off-Site Soil Sampling

Site investigation activities completed from 2002 to 2004 included off-site surface soil sampling at four locations – BS-01 through BS-04 (Figure 2-1). Analytical results for one sample, BS-02 with a mercury concentration of 13.4 milligrams per kilogram (mg/kg), exceeded the NYSDEC New York Codes, Rules, and Regulations (NYCRR) Part 375 Industrial SCOs of 5.7 mg/kg (NYSDEC, 2006). This task includes completing four additional surface soil samples near BS-02 to delineate mercury concentrations.

Four shallow soil borings, SB-128 to SB-131 (Figure 2-1) will be completed approximately 5 feet to the north, south, east, and west of the original BS-02 location. Table 2-1 summarizes the Phase 2 RI sampling locations and rationale. Borings will be completed using hand auger techniques and soil samples will be collected at two depth intervals: surface soil (0 to 2-inches below the vegetative cover or surface) and subsurface (2 feet below ground surface (ft bgs)). A total of eight soil samples will be analyzed for total mercury by United States Environmental Protection Agency (USEPA) method 7471B. Sample collection and analyses will be completed in accordance with procedures described in the 2020 RIWP.

2.2 Delineation of Chromium-Impacted Soil at SB-118

During the Phase 1 RI, total chromium was detected at a concentration of 40,900 mg/kg in the soil sample collected from dark silty/soft sludge material at a depth of 9.6-10.6 feet bgs in boring SB-118. The NYSDEC NYCRR Part 375 Industrial Use SCO for total chromium is 6,800 mg/kg. Chromium concentrations of two shallower soil samples from the same boring were 186 mg/kg (1-2 feet bgs) and 35.4 mg/kg (4-5 feet bgs).

Boring SB-118 is located within the footprint of a former “spray pond” similar to the pond located directly north of this location. The southern pond was filled in at an unknown date. This task includes conducting additional soil investigation in the vicinity of boring SB-118 to further delineate the extent of elevated chromium concentrations. Four soil borings, SB-132 to SB-135 (Figure 2-1) will be completed approximately 10 feet to the north, south, east, and west of the original boring SB-118 location. Table 2-1 summarizes the Phase 2 RI sampling locations and rationale. Borings will be advanced manually (during pre-clearing) and by direct-push technology (DPT) methods to the top of bedrock estimated to be approximately 10-11 feet bgs. Soils will be continuously logged during subsurface sampling and soil cuttings will be screened for organic vapors using a 10.6 electron-volt (eV) photoionization detector (PID) and for chromium and mercury using an x-ray fluorescent (XRF) analyzer. One subsurface soil sample for laboratory analysis will be collected from each new soil boring or monitoring well installation from the interval exhibiting greatest indication of contamination (e.g., elevated XRF reading, staining) above the water table. If no visible sign of contamination is present above the water table, the interval just above the water table will be sampled. An additional soil sample for laboratory analysis will be collected from the dark silty/soft sludge material or from just above bedrock/refusal if the material is not present.

A minimum of four and up to eight soil samples will be analyzed for total metals by USEPA methods 6010C and 7471B as well as for hexavalent chromium by USEPA method 7196A. Sample collection and analyses will be completed in accordance with procedures described in the 2020 RIWP.

2.3 Additional Overburden Groundwater Investigation

In their review comments on the Phase I RI Report, NYSDEC requested installation of additional overburden groundwater monitoring wells in the eastern and southern portions of the Site to provide additional data points and provide a better understanding of groundwater flow. Phase 2 RI Task 2 includes the installation of four additional overburden groundwater monitoring wells at the locations shown on Figure 2-1. Well SB-125/MW-11 is located immediately north of Building 5A and is downgradient of the VIS-4 IRM excavation area. Monitoring wells SB-126/MW-12 and SB-127/MW-13 are located in the southern portion of the Site to evaluate groundwater quality and flow in that area. Monitoring well SB-135/MW-14 will be located near the SB-118 location to evaluate possible groundwater impacts by elevated chromium concentration in soil at that location.

Soil borings/monitoring wells will be drilled to the top of bedrock using HSA drilling methods in accordance with procedures detailed in Section 5.0 of the RIWP. At each boring, soil will be continuously sampled from ground surface to the top of bedrock using a split-spoon sampler. Surface soil and select subsurface soil samples will be retained for analytical testing for TCL SVOCs, pesticides, PCBs, 1,4-dioxane, PFAS, and TAL metals in accordance with the RIWP. Table 2-2 summarizes the laboratory analyses to be completed during the Phase 2 RI. Additional information regarding analytical parameters is discussed in the 2020 QAPP.

A minimum of 24 hours following well completion, the newly installed monitoring wells will be developed to remove fine grained soils introduced during the drilling process and to improve hydraulic connection between the formation and the well screen. Well development procedures are described in Section 4.4 of the 2020 RIWP.

2.4 Bedrock Monitoring Well Installation

Phase 2 RI Task 3 includes the installation of five shallow bedrock groundwater monitoring wells at the locations shown on Figure 2-1. As discussed in Section 2.5.2 of the 2020 RIWP, the Site is underlain by fill/soil overburden ranging in thickness from two feet in the north to approximately 11 feet in the central part of the Site near Pike Creek. The overburden materials are underlain by dolomite bedrock of the Lockport Formation which underlies the city of Niagara Falls and extends from the Niagara Escarpment southward to Grand Island. Groundwater within the Lockport formation is fracture controlled, as groundwater is present in wells that intercept fractures within the bedrock.

Bedrock monitoring wells will be located to assess possible mercury impacts to bedrock water bearing zones from areas currently or formerly exhibiting mercury-impacted overburden soil. Three monitoring wells will be installed to evaluate bedrock groundwater in the areas north of Pike Creek, and two monitoring wells will be installed to evaluate bedrock groundwater in the area south of Pike Creek. Table 2-1 summarizes the rationale for the selected bedrock well locations. Bedrock monitoring well BR-MW-01 will be installed downgradient of the former elemental mercury-impacted soil areas in the northern portion of the Site (Figure 2-1). Well BR-MW-02 will be installed near existing overburden monitoring well MW-09 located south of Pike Creek. Bedrock well BR-MW-03 will be installed as a well cluster pair with overburden well MW-11 near the center of the Site downgradient of known former and current mercury-impacted areas. BR-MW-04 will be installed near existing overburden monitoring well MW-01 to complete an overburden/bedrock well cluster. BR-MW-05 will be installed in the central portion of the area south of Pike Creek.

Bedrock wells will be installed in accordance with procedures described in Section 4.3.2 of the 2020 RIWP. As described there, the wells are expected to be completed to depths of approximately 10 feet into the top of bedrock to intersect water bearing fractures anticipated to be present in that interval from historical regional data.

2.5 Groundwater Sampling

Following completion of the well development, the wells will be allowed to stabilize for at least 72 hours, prior to sampling. Groundwater samples will be collected from all newly installed overburden and bedrock monitoring wells and from existing overburden wells MW-09 and MW-10 using low-flow sampling techniques to the extent practicable. If there is insufficient water in a well for low-flow sampling or the well goes dry during purging, a grab sample may be obtained after the well recharges. Proposed groundwater sample analyses are summarized on Table 2-2. Detailed groundwater sampling procedures are discussed in Section 4.5 of the 2020 RIWP.

2.6 Soil Vapor Intrusion Evaluation

Niacet building locations are identified by number on Figure 2-2 and listed on Table 2-3 including their current uses, typical number of occupants, and whether or not they are equipped with active ventilation systems. Two buildings, 10 and 101, are equipped with air handling/ventilation. These buildings also have the highest number of regular occupants at 12 and 18, respectively. The following subsections discuss the SVI evaluation approaches for VOCs and mercury vapor, respectively. SVI sampling rationale discussed below is primarily based on building locations in relation to documented impacted soil and/or groundwater areas and whether or not buildings are regularly occupied.

2.6.1 VOC SVI Assessment

As discussed in the 2020 RIWP, groundwater occurrence in overburden materials is limited to approximately the southern two thirds of the Site as the depth to bedrock is too shallow in the northern third of the Site. Groundwater analytical results from the 2020 Phase 1 RI sampling activities showed limited detections of VOCs in groundwater only in wells MW-09 and MW-10. VOCs detected included 1,2-dichloroethane, 1,2-dichloropropane, acetone, chloroform, and methylene chloride (MC). Of these, the only compound included in the NYSDOH SVI guidance decision matrices is MC which was detected at a concentration of 8 micrograms/liter in well MW-09. Well MW-09 is located immediately north and upgradient of Building 105.

Groundwater flow in the vicinity of well MW-09 is toward the south-southwest. AECOM proposes to perform SVI sampling for VOCs in two buildings located downgradient of well MW-09 – Buildings 105 and 101. Building 105 is located immediately downgradient of monitoring well MW-09 and is used for production purposes. Building 101 is located further downgradient of well MW-09 and is used as office and laboratory space with 18 regular occupants.

VOC SVI Sampling Program

AECOM will perform the following SVI sampling program to evaluate the presence of potential Site-related VOCs in buildings 101 and 105:

- Two sub-slab soil vapor samples will be collected beneath the lowest level of each building. The sample locations within each building will be field verified prior to collection of samples. The sub-slab samples will be tested for an adequate surface seal before and after testing using the helium tracer gas test procedures outlined in Section 2.2 of the SVI Field Sampling Plan (Appendix A).

- Two indoor air samples will be collected inside each building. The samples will be co-located with the sub-slab vapor samples.
- One outdoor air sample will be collected from an area located upwind of Building 105. Actual placement will be determined based on the wind direction at the start of sampling.
- One field duplicate indoor air sample will be collected from the office space.

The samples will be submitted for VOC analysis using modified USEPA Method TO-15, as further discussed below.

<u>VOC SVI Field Methodology</u>

Collection of SVI samples will be conducted in accordance with the Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York (NYSDOH, dated October 2006, and updates through May 2017) and the SVI Field Sampling Plan (FSP) included as Appendix A to this Phase 2 RIWP Addendum. A pre-sampling survey will be performed in accordance with the NYSDEC Structure Sampling Questionnaire and Building Inventory form. The pre-sampling survey will include use of a PID capable of detecting parts per billion by volume (ppbv) levels to identify the presence of potential indoor VOC sources prior to collection of indoor air samples. This will help to ensure that potential indoor sources are documented and removed prior to collection of indoor air samples. If indoor sources are found, they will be removed and one day (i.e., approximately 24 hours) will be allowed to pass before performing indoor air sampling. The NYSDEC Structure Sampling Questionnaire and Building Inventory form (Appendix A) will be completed and subsequently provided in the Final RIR.

At each location, sample collection will be performed using a 6.0-liter stainless steel, laboratory-provided Summa® canister equipped with an 8-hour regulator. AECOM will request standard 10-day turn-around time for analytical samples. The field scientist will record the sample identification, canister and regulator identification, date and time of sample collection, sample location and height, and the sampling method and device on a field log sheet (Appendix A). In addition, the purge volume, sample volume, canister vacuum pre- and post-sampling, and sampler name will be recorded.

Weather conditions (i.e., precipitation, outdoor temperature, barometric pressure, wind speed and direction) will be noted for the 24- to 48-hour period prior to sampling. Weather condition data from the period prior to sampling will be downloaded from the Internet at the following web site: http://www.wunderground.com/weatherstation/WXDailyHistory.asp?ID=KNYLANCA3. Pertinent field observations (i.e., odors or readings from field instrumentation, personal materials or other items of concern in the area of the sample [e.g., work bench, aerosols, etc.]) will be noted during sampling.

<u>Laboratory Analyses</u>

The SVI samples will be shipped under proper chain-of-custody protocol to Eurofins Laboratories, Inc. located in Knoxville, Tennessee, a NYSDOH Environmental Laboratory Approval Program (ELAP)-certified laboratory. Samples will be analyzed for VOCs using modified USEPA Method TO-15, as follows:

- Sub-slab vapor and indoor/outdoor air sample reporting limits will meet NYSDOH requirements of 1 $\mu g/m^3$ for all VOCs.
- In accordance with May 2017 NYSDOH Soil Vapor/Indoor Air Matrix A and C, indoor and outdoor air samples for the following VOCs will meet 0.2 $\mu g/m^3$ reporting limits: trichloroethene, cis-1,2-dichloroethene, 1,1-dichloroethene, carbon tetrachloride, and vinyl chloride.
- In order to achieve reporting levels to allow for comparison to the screening levels in the May 2017 NYSDOH update, sub-slab vapor samples will be analyzed using modified USEPA Method TO-15 and indoor/outdoor air samples will be analyzed using modified USEPA Method TO-15 low level.

- The laboratory will provide a Category B data report. The data deliverable package will include the following elements: analytical report; quality assurance/quality control summary; chain of custody; method blank; laboratory control samples – control limits; reporting limits; and surrogate recoveries for gas chromatography–mass spectrometry analysis with control limits.

2.6.2 Mercury Vapor SVI Assessment

NYSDEC has requested the evaluation of select buildings due to the presence of elemental mercury in soil in areas of the Site. The following mercury vapor sampling program will be completed to address this request:

Mercury Vapor SVI Sampling Program

AECOM will perform the following SVI sampling program to evaluate the presence of potential mercury vapors in onsite buildings:

- Two sub-slab soil vapor samples will be collected beneath the lowest level of buildings 10 and 17. Building 10 has been selected for sub-slab sampling due to its proximity to the former elemental mercury areas identified as SB-72/74 & SB-75. Building 17 will be sampled due to historic reports of elemental mercury being identified beneath the building. The sample locations within each building will be field verified prior to collection of samples.
- Two indoor air samples will be collected inside buildings 10 and 17 concurrently with the sub-slab samples. The samples will be co-located with the sub-slab vapor samples.
- One outdoor ambient air sample will be collected from an upwind location.
- In addition, AECOM will collect one indoor air sample only from all other regularly occupied buildings to evaluate the potential presence of mercury vapor. Indoor air samples will be collected from the following buildings: 15, 101, 102, 103, 104, 105, 106, 107, 108, 114, and 116.

The samples will be submitted for analysis using modified National Institute for Occupational Safety & Health (NIOSH) Method 6009 (NIOSH, 1994), as further discussed below.

In order to assess SVI for mercury in Buildings 10 and 17, the sub-slab vapor and indoor air samples will be sampled. Similar to the VOC SVI sampling, the mercury sub-slab vapor samples will be collected concurrently with indoor air samples collected at the same location in the building. The indoor air samples will be collected in the breathing zone height for commercial workers. The results of the sampling will be utilized to evaluate the vapor intrusion risk from mercury in the buildings at this Site.

USEPA has developed a Vapor Intrusion Screening Level (VISL) calculator for determining screening levels in sub slab vapor and indoor air for residential and commercial settings. The Occupational Safety and Health Administration (OSHA) has also published work place permissible exposure limits (PELs) for mercury based on an 8-hour Time-Weighted Average exposure. The OSHA PELs are legally enforceable and found in the 29 CFR 1910.1000, Table Z-2. NIOSH and American Conference of Governmental Industrial Hygienists (ACGIH) have also published recommended exposure limits (RELs) and Threshold Limit Values (TLVs), respectively.

The VISLs, OSHA PEL, and recommended exposure limits (NIOSH and ACGIH) are listed in the Table 2-4, below:

Table 2-4, Vapor Intrusion Screening Levels and Workplace Exposure Limits for Mercury

Source	Mercury Concentration, Units	Notes
USEPA VISL- Sub-Slab Vapor	4.38E+01 µg/m^3	Screening Level – Sub Slab Vapor
USEPA VISL- Commercial Worker (Indoor Air)	1.31 µg/m^3	Screening Level – Indoor Air for Commercial Worker
OSHA PEL	0.1 mg/m^3(100 µg/m^3)	Legally enforceable workplace exposure limit based on 8-hr workday
NIOSH REL	0.05 mg/m^3(50 µg/m^3) 0.1 mg/m^3 (Ceiling Limit)	Recommended exposure limit based on 10-hr work day
ACGIH TLV	0.025 mg/m^3(25 µg/m^3)	Recommended exposure limit based on 8 hour work day

Notes:
µg/m^3= micrograms per cubic meter; mg/m^3= milligrams per cubic meter;
USEPA Vapor Intrusion Screening Level (VISL) Calculator Results (8/2/22) based on Total Cancer Risk (TCR) of 1 E-06 and a Hazard Quotient (HQ) of 1.0.

Sampling Methodology

A sub-slab soil vapor sample will be collected at two locations each in Buildings 10 and 17 (Figure 2-2). A modified OSHA ID-140 or NIOSH 6009 will be utilized for collection of indoor air and sub slab vapor. The method utilizes a Hopcalite or Hydrar sampling tube through which air is drawn. The ends of the sampling tube are broken off prior to connecting to a portable sampling pump with flexible tubing. For the indoor air sample, the pump and sample tube assembly will be placed in a fixed position at the approximate breathing height of a worker. For the sub slab vapor sample, the inlet end of the tube will be connected to the sub slab vapor pin using a small piece of flexible tubing. The pump will be pre-calibrated to a flow rate of 0.15 to 0.25 liters per minute (LPM). Once the samples of indoor air or sub slab vapor have been collected, the ends of the sorbent tube will be capped and any mercury fixed on the media is stable for a period of 30 days @ 25 °C. The sampling pumps will be post-calibrated and the actual flow rate will be recorded on a field sampling record along with a calculation of the sample volume in liters. The samples will be labeled with a unique sample identification and entered on a sample Chain of Custody form along with the date of sampling, the sample volume, and analysis request. The samples will be shipped under COC procedures to a certified testing laboratory for analysis by NIOSH 6009. The analytical method includes desorption of the mercury from the sorbent media utilizing concentrated acid nitric followed by hydrochloric acid. The desorbed sample is diluted with water and then analyzed for mercury utilizing a cold-vapor atomic absorption spectrophotometer. NIOSH 6009 has a limit of detection (LOD) of approximately 0.03 µg/sample tube. A copy of the NIOSH Method 6009 is attached for reference in Appendix B. An approximate sampling period of 8-hours (approximately 100 liters sample size) will be utilized for sample collection for the sub slab and indoor air sampling. This may be adjusted based on field conditions. One unopened sealed sorbent tube will also be submitted to the analytical laboratory for analysis as a media blank for quality assurance.

2.7 Sewer Tunnel Evaluation and Sampling

AECOM will evaluate options for collecting sediment and water samples from the 47th Street sewer tunnel and the Pike Creek culvert using non-entry inspection and sampling methods. AECOM will perform a non-entry reconnaissance of the sewer manholes at three locations along the 47th Street tunnel and two manhole locations along the Pike Creek culvert. The 47th Street manhole located in the intersection of Niagara Falls Boulevard and 47th Street will not be accessed for inspection at this time due to the risks associated with working in a roadway.

The main purpose of the inspection will be to assess downhole conditions at each accessible manhole and determine if sediment exists at each location in quantities that allow sample collection. Depending on the results of reconnaissance (e.g., quantity and consistency of sediment) and on available sampling equipment, AECOM may attempt to collect sediment and water samples at that time. If AECOM determines that sampling is not possible/practical at the time, the information obtained during these inspections will allow for planning of future sampling efforts either by additional non-entry methods or confined space entry methods. Sewer inspection work will be closely coordinated with the Niagara Falls Water Board and will be planned for low flow conditions. Water levels in the 47th Street Sewer tunnels, the Pike Creek Culvert, and other accessible on-site stormwater sewers will be measured from a surveyed point(s) to evaluate the potential for groundwater infiltration into the sewers.

Water and sediment samples will be submitted for laboratory analysis in accordance with the sampling matrix presented in Table 3-2 of the 2020 RIWP. In addition to analyses listed in Table 3-2 of the 2020 RIWP, water and sediment samples collected from Pike Creek will also be analyzed for hexavalent chromium by USEPA method 7196A (Phase 2 RIWP Addendum, Table 2-2).

2.8 Mercury Speciation Sampling

The NYSDEC Part 375 Industrial SCO for mercury of 5.7 mg/kg is based on an adult worker's inhalation exposure for elemental mercury. This SCO had been considered the presumptive SCO for the Niacet Site since adoption by the NYSDEC of Part 375 SCOs in 2006. The New York State Brownfield Cleanup Program, Development of Soil Cleanup Objectives Technical Support Document (TSD) (NYSDEC and NYSDOH, 2006) presents exposure pathway specific SCOs for various land uses. Table 5.3.6-1(e) of the TSD presents the exposure pathway specific Industrial SCOs for mercury (inorganic salts) of 260 mg/kg for an adult worker and 220 mg/kg for an adolescent trespasser.

In December 2016, AECOM completed a sampling program to sample for the presence of inorganic mercury salts in areas where elemental mercury had not been previously identified to evaluate if the inorganic mercury salts SCO should be applied to those areas. Sampling activities consisted of completing test pits and collecting shallow soil samples from eight areas of the Site where no elemental mercury had been previously identified. Samples were submitted to Brooks Applied Labs of Bothell, WA, for mercury speciation analyses using selective sequential extraction (SSE) techniques to evaluate mercury salts content relative to elemental mercury. Speciation data conclusively indicated that there is no elemental mercury present in these samples. Results of this evaluation were presented in a Technical Memorandum prepared by AECOM and submitted to NYSDEC in May 2017 (AECOM, 2017). The memorandum recommended applying the mercury salts SCO of 220 mg/kg to areas where no visible/elemental mercury has been identified.

In their review comments on the Phase I RIR (NYSDEC, 2021), NYSDEC stated that the use of the alternate SCO for mercury salts discussed in the 2017 Technical Memorandum will be considered by NYSDEC/NYSDOH; however, additional sampling and speciation will be required to determine the applicability of the alternate SCO as it pertains to the remedy.

Additional soil sampling and mercury speciation analyses will be performed during the Phase 2 RI field mobilization. The objective of this sampling is to collect additional soil samples from areas not containing elemental mercury and perform mercury speciation analysis to further support evaluation of using the mercury salts SCO as part of the overall remedial strategy. Samples will be collected from the 12 locations from areas where visible/elemental mercury has not been identified previously. Proposed sample locations are shown on Figure 2-3.

Similar to sampling performed in 2016, samples will be collected using a hand auger to advance to a depth of 1 foot bgs. Soil from the 0-1 ft interval will be composited and collected into three laboratory provided 4-ounce glass sample jars equipped with Teflon-lined lids. Samples will further be sealed with custody tape and placed into a cooler on ice for transportation to the laboratory. All sampling equipment will be decontaminated prior to commencing sampling and between each sample.

Samples will be shipped via FedEx under chain-of-custody protocol to Brooks Applied Labs in Bothell, Washington. Samples will be analyzed for the following parameters using a selective sequential extraction procedure:

- Total Mercury by USEPA Method 1631
- Volatile Mercury by Thermal Desorption/ USEPA Method 1631E

- Extractable Mercury Hg(II) by IP-CV-ICP-MS

Results of additional mercury speciation analysis will be incorporated into an updated Technical Memorandum discussing the sampling activities performed, analytical results, and recommendations for selecting an appropriate SCO for areas of the Site not impacted by elemental mercury.

3. Project Schedule and Deliverables

3.1 Schedule

RI activities commenced in late-November 2022 and are anticipated to proceed through the spring 2023 with preparation and submittal of a RIR following in summer. Minor changes to the schedule may occur due to the NYSDEC review and approval process, unforeseen weather delays, etc.

3.2 Final Remedial Investigation Report

Upon completion of the field activities, a Final RIR will be prepared to document the findings of the investigations performed at the Site. The report will be consistent with the requirements presented in DER-10 [NYSDEC, 2010] and Section 13.0 of the 2020 RIWP.

4. References

AECOM, 2017. Technical Memorandum, Evaluation of Soil Cleanup Objectives for the Niacet Site, 47th Street, Niagara Falls, New York. May.

AECOM, 2020a. Remedial Investigation Work Plan, Niacet Site, 400 47th Street, Niagara Falls, New York, Site No. C932172. January.

AECOM 2020b. Quality Assurance Project Plan, Remedial Investigation, Niacet Site, 400 47th Street, Niagara Falls, New York, Site No. C932172. January.

AECOM 2021. Phase 1 Remedial Investigation Report, Niacet Site, 400 47th Street, Niagara Falls, New York, Site No. C932172. May.

National Institute for Occupational Safety and Health (NIOSH), 1994. NIOSH Manual of Analytical Methods (NMAM), Fourth Edition, 8/15/94. August, 1994.

NYSDEC, 2006. 6 NYCRR 375-6.8 Soil Cleanup Objectives. Effective December 14, 2006.

NYSDEC and NYSDOH. 2006. New York State Brownfield Cleanup Program Development of Soil Cleanup Objectives Technical Support Document. September.

NYSDEC, 2010. DER-10 Technical Guidance for Site Investigation and Remediation. Issued May 3, 2010.

New York State Department of Environmental Conservation (NYSDEC), 2019. Brownfield Site Cleanup Agreement, Index No. C932172-11-18. February.

NYSDOH, 2017. Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York. Issued October 2006, and updates through May 2017.

Figures

Legend

Site Boundary

Source: © 2013 National Geographic Society, i-cubed;
1:24:000-scale USGS Topographic Quadrangles:
Niagara Falls, 1980; Tonawanda West, 1980

2,000 0 2,000 Feet

AECOM

UNION CARBIDE CORPORATION NIACET SITE
SITE LOCATION MAP

FIGURE 1-1

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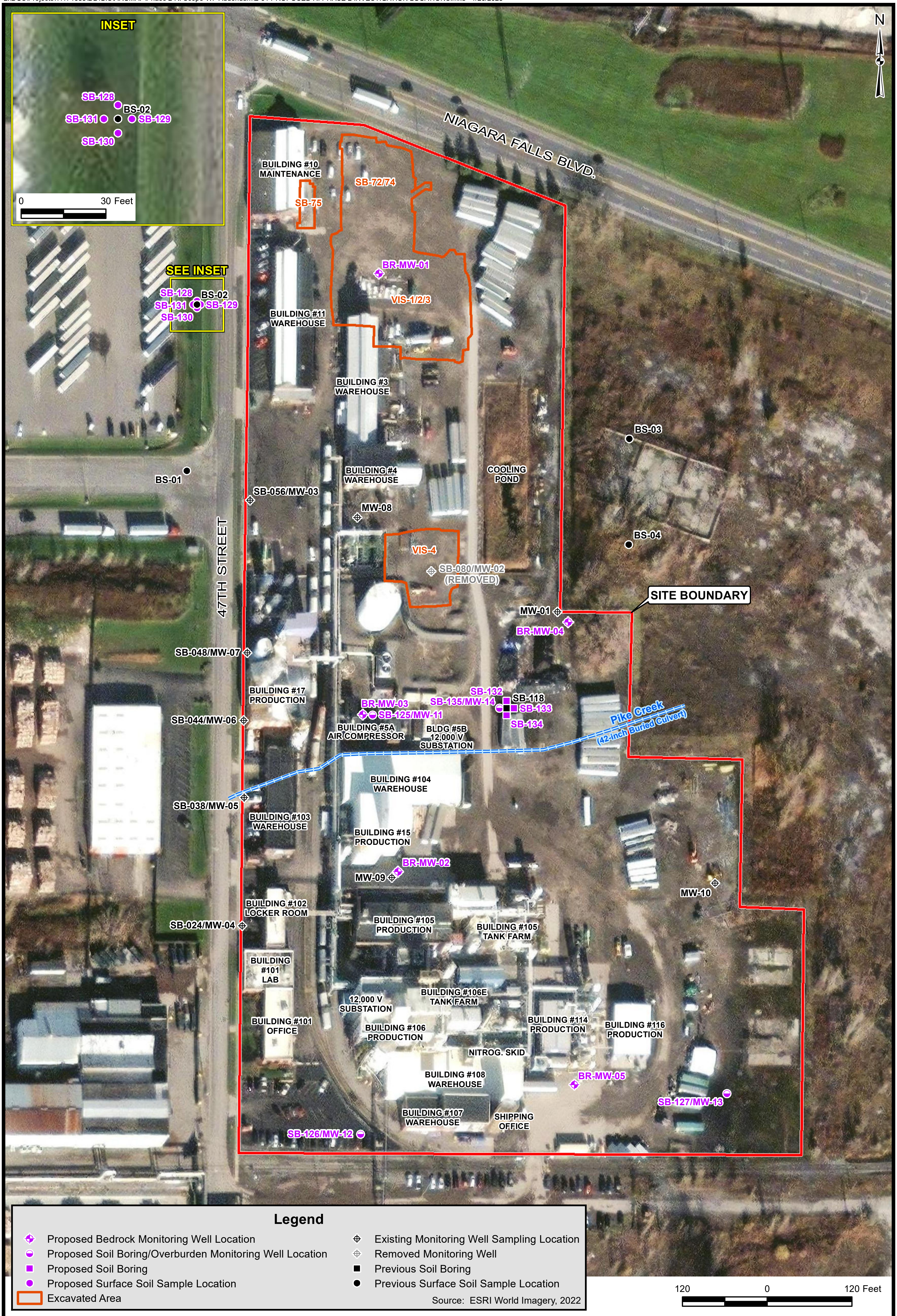
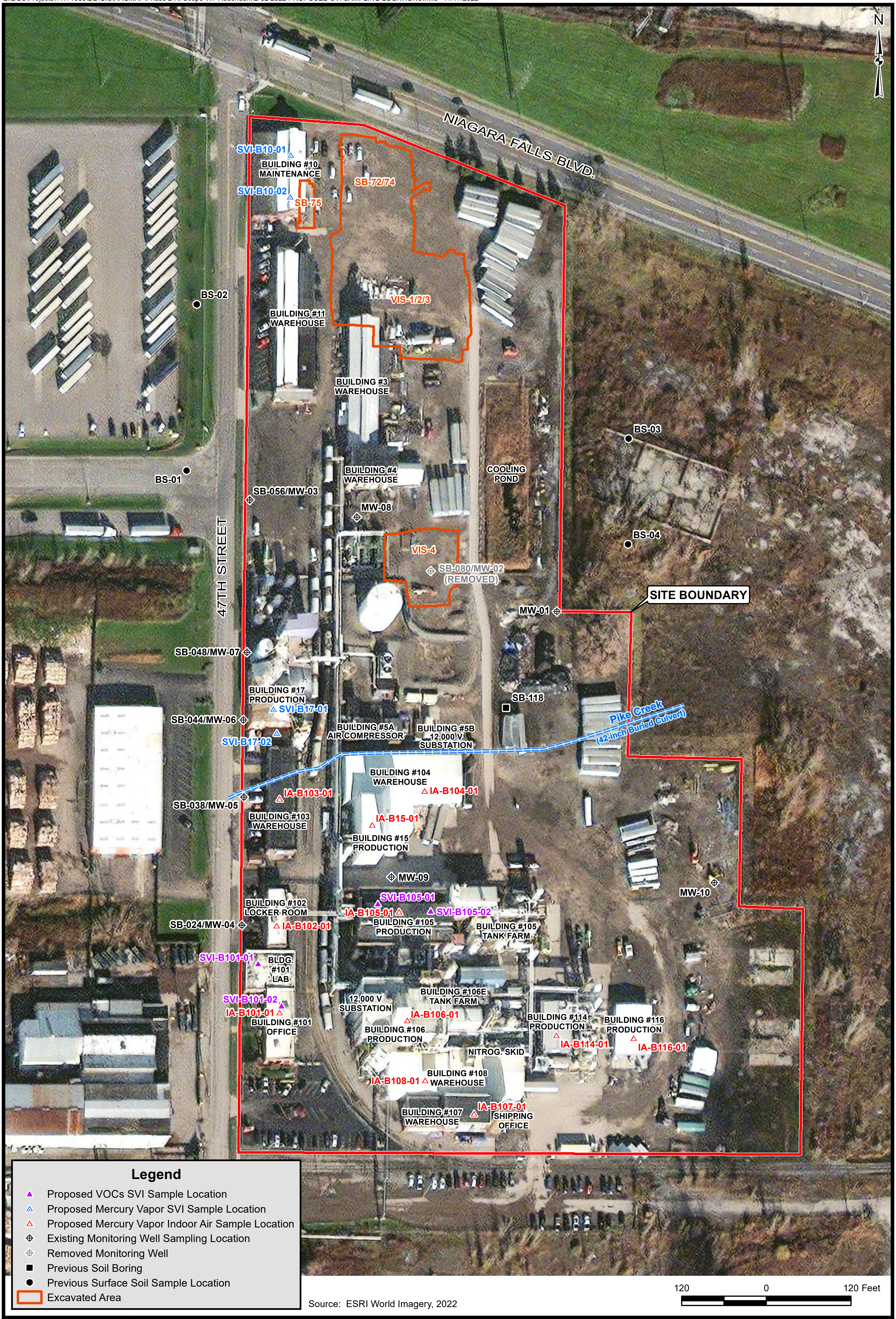


AECOM

UNION CARBIDE CORPORATION NIACET SITE
PROPOSED RI PHASE 2 INVESTIGATION LOCATIONS

FIGURE 2-1

L:\DCS\Projects\11171085\DB\GIS\ARCMAP\Phase 2 RI Scope-WP Addendum\2-02 2022 PROPOSED SVI SAMPLING LOCATIONS.mxd 11/17/2022



AECOM

UNION CARBIDE CORPORATION NIACET SITE
2023 PROPOSED SVI SAMPLING LOCATIONS

FIGURE 2-2

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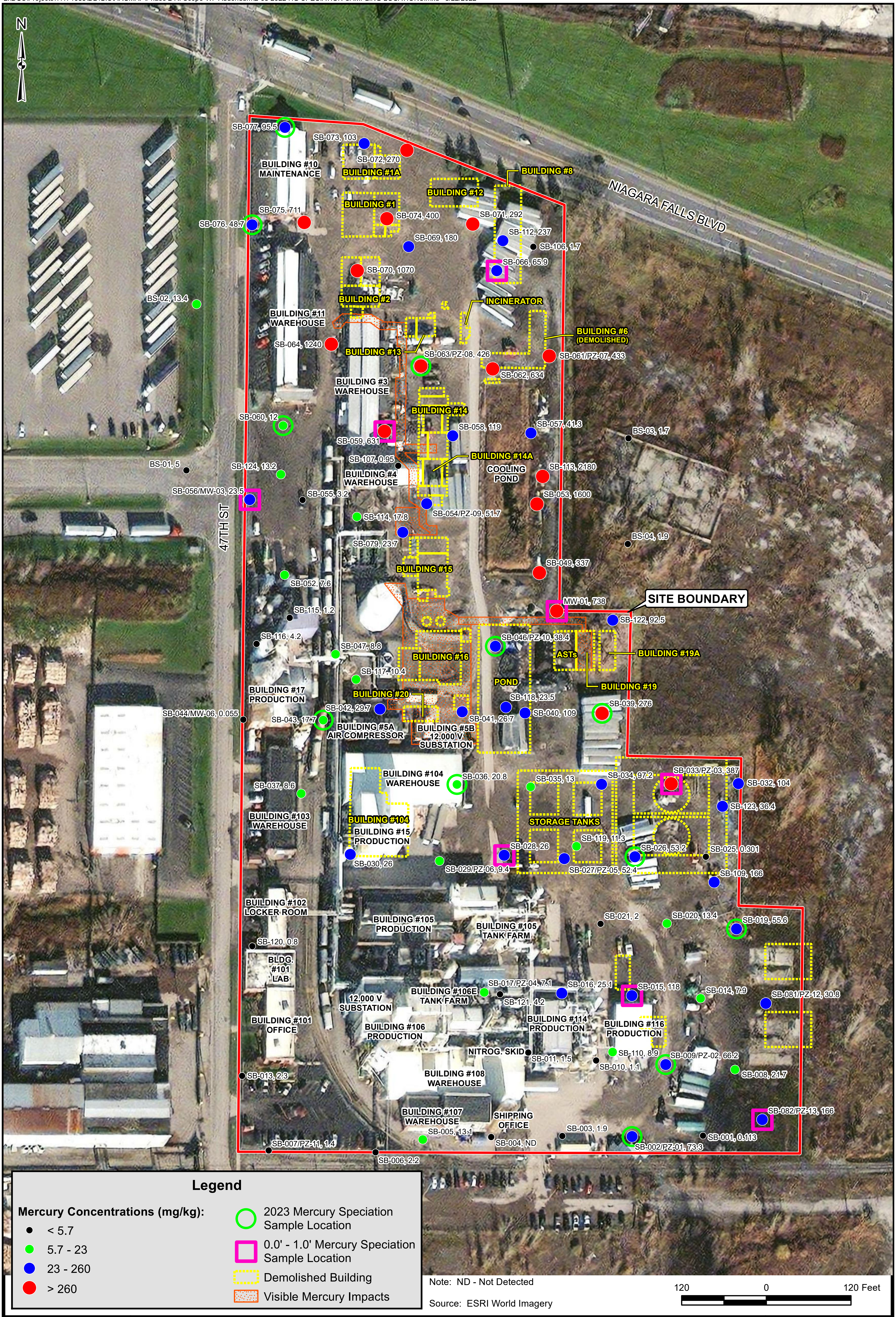

AECOM

UNION CARBIDE CORPORATION NIACET SITE
2023 MERCURY SPECIATION SAMPLING

FIGURE 2-3

Tables

Table 2-1

Proposed Phase 2 RI Soil Boring and Monitoring Well Locations and Rationale
Niacet Facility RI Work Plan Addendum
Niagara Falls, NY

Location/Sample ID	Type of Samples to be Collected	Area of Sample	Rationale/Comments
BR-MW-01	groundwater	North of Building #3	Bedrock well located within VIS-1/2/3 IRM excavation area. No overburden soil samples will be collected due to the well being located in the previously excavated area that was backfilled with crushed stone from a commercial quarry.
BR-MW-02	groundwater	Near existing well MW-09	Bedrock well located south of Pike Creek. No overburden soil samples will be collected as the well will be co-located with previously completed boring SB-108/MW-09.
BR-MW-03	groundwater	East of Building #17 & North of Building #5A	Bedrock well located downgradient of IRM excavation area VIS-4 in a cluster with overburden well MW-11. No overburden soil samples will be collected as the well will be co-located with boring SB-125/MW-11.
BR-MW-04	surface soil/subsurface soil/groundwater	East Side of Site & North of Pike Creek	Bedrock well located upgradient of IRM excavation areas and colocated with existing overburden well MW-01.
BR-MW-05	surface soil/subsurface soil/groundwater	East of Building 105 Tank Farm	Bedrock well centrally located in area south of Pike Creek.
SB-125/MW-11	surface soil/subsurface soil/groundwater	East of Building #17 & North of Building #5A	Overburden well colocated with bedrock well BR-MW-3
SB-126/MW-12	surface soil/subsurface soil/groundwater	Southwest Corner of Site & West of Building #107	Overburden well located in southwest portion of site
SB-127/MW-13	surface soil/subsurface soil/groundwater	Southeast Corner of Site	Overburden well located in southeast portion of site
SB-128	surface soil (0-0.2 ft bgs)/ subsurface soil (2 ft bgs)	North of BS-02 & West of 47th Street	Located approximately 5 feet north of BS-02 and in the grassy area west of 47th Street
SB-129	surface soil (0-0.2 ft bgs)/ subsurface soil (2 ft bgs)	East of BS-02 & West of 47th Street	Located approximately 5 feet east of BS-02 and in the grassy area west of 47th Street
SB-130	surface soil (0-0.2 ft bgs)/ subsurface soil (2 ft bgs)	South of BS-02 & West of 47th Street	Located approximately 5 feet south of BS-02 and in the grassy area west of 47th Street
SB-131	surface soil (0-0.2 ft bgs)/ subsurface soil (2 ft bgs)	South of BS-02 & West of 47th Street	Located approximately 5 feet west of BS-02 and in the grassy area west of 47th Street
SB-132	subsurface soil	North of SB-118	Located approximately 10 feet north of SB-118 to delineate chromium impacted soil
SB-133	subsurface soil	East of SB-118	Located approximately 10 feet east of SB-118 to delineate chromium impacted soil
SB-134	subsurface soil	South of SB-118	Located approximately 10 feet south of SB-118 to delineate chromium impacted soil
SB-135/MW-14	subsurface soil/groundwater	West of SB-118	Located approximately 10 feet west of SB-118 to delineate chromium impacted soil

SB - Soil Boring
MW - Monitoring Well

Table 2-2

Proposed Phase 2 RI Sampling Matrix
Niacet Facility RI Work Plan Addendum
Niagara Falls, NY

	Parameters																						
	Soils								Groundwater									Pike Creek SW/SED	SVI Samples (sub-slab & indoor air)		Mercury Speciation		
Sample ID	TCL VOCs[4] (8260C)	SVOCs (8270D)	PFAS (537)	1,4-Dioxane (8270D)	PCBs (8082A)	Pesticides (8081A)	TAL Metals (6010C & 7471B)	Hexavalent Chromium (7196A)	TCL VOCs (8260C)	SVOCs (8270D)	PFAS (537)	1,4-Dioxane (8270D SIM)	PCBs (8082A)	Pesticides (8081A)	TAL Metals (6010C & 7470A)	Dissolved Metals (6010C & 7470A)	Hexavalent Chromium (7196A)	Hexavalent Chromium (7196A)	VOCs (TO-15)	Mercury Vapor (NIOSH 9006)	Total Mercury (1631)	Volatile Mercury by Thermal Desorption (1631E)	Extractable Mercury Hg(II) (IP-CV-ICP-MS)
MW-09									1	1	1	1	1	1	1	1							
MW-10									1	1	1	1	1	1	1	1							
BR-MW-01									1	1	1	1	1	1	1	1							
BR-MW-02									1	1	1	1	1	1	1	1							
BR-MW-03									1	1	1	1	1	1	1	1							
BR-MW-04	1	2	1	1	2	2	2		1	1	1	1	1	1	1	1							
BR-MW-05	1	2	1	1	2	2	2		1	1	1	1	1	1	1	1							
SB-125/MW-11	2	3	1	1	3	3	3		1	1	1	1	1	1	1	1							
SB-126/MW-12	2	3			3	3	3		1	1	1	1	1	1	1	1							
SB-127/MW-13	2	3			3	3	3		1	1	1	1	1	1	1	1							
SB-128 (1)							2																
SB-129 (1)							2																
SB-130 (1)							2																
SB-131 (1)							2																
SB-132							2	2															
SB-133							2	2															
SB-134							2	2															
SB-135/MW-14							2	2	1	1	1	1	1	1	1	1	1						
SVI-B10-01																				2			
SVI-B10-02																				2			
SVI-B17-01																				2			
SVI-B17-02																				2			
SVI-B101-01																			2				
SVI-B101-02																			2				
SVI-B105-01																			2				
SVI-B105-02																			2				
SVI-OA-01																			1	6			
IA-B15-01 (5)																				1			
IA-B101-01 (5)																				1			
IA-B102-01 (5)																				1			
IA-B103-01 (5)																				1			
IA-B104-01 (5)																				1			
IA-B105-01 (5)																				1			
IA-B106-01 (5)																				1			
IA-B107-01 (5)																				1			
IA-B108-01 (5)																				1			
IA-B114-01 (5)																				1			
IA-B116-01 (5)																				1			
SB-002-22																					1	1	1
SB-009-22																					1	1	1
SB-019-22																					1	1	1
SB-026-22																					1	1	1
SB-036-22																					1	1	1
SB-039-22																					1	1	1
SB-043-22																					1	1	1
SB-046-22																					1	1	1
SB-060-22																					1	1	1
SB-063-22																					1	1	1
SB-076-22																					1	1	1
SB-077-22																					1	1	1
SW/SED-02_PCMH-2																		2					
SW/SED-03_PCMH-5																		2					
Monitoring Subtotal	**8**	**13**	**3**	**3**	**13**	**13**	**29**	**8**	**11**	**11**	**11**	**11**	**11**	**11**	**11**	**11**	**1**	**4**	**9**	**25**	**12**	**12**	**12**
QA/QC																							
Duplicates (2)	1	2	1	1	2	2	3	1	2	2	2	2	2	2	2	2	1	1	1	3	1	1	1
Matrix Spike (2)	1	1	1	1	1	1	2	1	1	1	1	1	1	1	1	1	1	1					
Matrix Spike Duplicate (2)	1	1	1	1	1	1	2	1	1	1	1	1	1	1	1	1	1	1					
Trip Blanks	4	0	0	0	0	0	0	0	2	0	0	2	0	0	0	0							
Rinse Blank (3)	2	2	2	2	2	2	2	2	1	1	1	1	1	1	1	1	1	1					
Total Per Sampling Event	**17**	**19**	**8**	**8**	**19**	**19**	**38**	**13**	**18**	**16**	**16**	**18**	**16**	**16**	**16**	**16**	**5**	**8**	**10**	**28**	**13**	**13**	**13**

Notes:
(1) Two samples each to analyzed only for total mercury by USEPA method 7471B
(2) Duplicates will be collected at a rate of 1 per 10 samples. Matrix Spike and Matrix Spike Duplicate samples will be collected at a rate of 1 per 20 samples.
(3) Rinse Blanks and Ambient Blanks (for PFAS only) will be taken on a basis of 1 per week per media.
(4) Surface soil samples will only be analzyed for VOCs if visual, olfactory, or other field screening methods identify potential impacts.
(5) Only indoor air samples will be collected at these locations.

MW - Monitoring Well
SB - Soil Boring
SW - Surface Water
SED - Sediment
TAL - Target Analyte List

TCL VOC - target compount list volatile organic compound
SVOC - semi-volatile organic compound
PFAS - per-and polyfluoroalkyl substances
PCB - polychlorinated biphenyls
TOC - total organic carbon
QA/QC - quality assurance/quality control

Table 2-3

Niacet Buildings Summary
Niacet Phase 2 RI Work Plan Addendum
Niagara Falls, NY

Building	Building Use	Regular Occupants	Air Exchangers/Ventilation
3	Warehouse	0	None
4	Warehouse	0	None
5A	Air Compressor	0	None
5B	12,000 V Substation	0	None
10	Maintenance	12	Yes
11	Warehouse	0	None
15	Production	2	None
17	Production	4	None
101	Office & Lab	18	Yes
102	Locker Room	2	None
103	Warehouse	1	None
104	Warehouse	2	None
105	Production	2	None
106	Production	4	None
107	Warehouse	1	None
108	Warehouse	2	None
114	Production	2	None
116	Production	2	None

Appendices

Appendix A

VOCs Soil Vapor Intrusion Field Sampling Plan

APPENDIX A

VOCs SOIL VAPOR INTRUSION FIELD SAMPLING PLAN

NIACET SITE

400 47TH STREET

NIAGARA FALLS, NY

Prepared For

UNION CARBIDE CORPORATION

Prepared By

AECOM

ONE JOHN JAMES AUDUBON PARKWAY

AMHERST, NEW YORK 14228

AUGUST 2022

TABLE OF CONTENTS

APPENDICES

1.0 INTRODUCTION

This Field Sampling Plan (FSP) is designed to provide detailed step-by-step procedures for the field activities associated with soil vapor intrusion (SVI) investigation activities at the Niacet Site. It will serve as the field procedures manual to be strictly followed by all AECOM Technical Services, Inc. (AECOM) personnel. Adherence to these procedures will ensure the quality and defensibility of the field data collected. In addition to the field procedures outlined in this document, all personnel performing field activities must do so in compliance with: (1) the appropriate Health and Safety guidelines found in the existing Site-specific Health and Safety Plan (HASP); (3) the scope of work outlined in the RIWP Addendum prepared in August 2022; and (4) the proposed time schedule outlined in the Work Plan.

2.0 SOIL VAPOR INTRUSION INVESTIGATION

This SVI investigation will include the following tasks: 1) conducting occupant interviews and a brief survey of chemicals present in each building and evaluating their potential to affect air sample results; 2) collecting two indoor air samples per building from the breathing zones of the first floor area; (3) collecting two soil vapor samples per building from beneath the concrete slab, and; (4) collecting one outdoor ambient air sample from near the sampling area.

2.1 Indoor Air Quality Survey and Questionnaire

NYSDEC Structure Sampling Questionnaire and Building Inventory form (Appendix A) will be used. Once the questionnaires have been completed, a brief inventory of chemicals stored in the buildings will be prepared. The inventory will consist of a general description of areas where chemicals are stored and the types and approximate numbers of chemicals present. During this inventory, a ppbRAE photo ionization detector (PID) will be used to measure the presence of volatile organic compounds (VOCs) in those areas where the chemicals are found. Also during the inventory, a handout will be provided to the occupants that list activities that should be avoided prior to and during the air sampling. This handout is provided in Attachment A. The general procedures to be followed during the surveys are summarized below:

- Identify all areas on the floor to be sampled that may be used for storage of chemical containers. Also record the general types and approximate quantities of chemicals stored and VOCs present in the atmosphere in the areas of chemical storage.
- Other potential sources that may influence air quality testing that should be noted and scanned with the PID include new construction/remodeling/painting, new carpeting, and freshly dry-cleaned clothing.

2.2 Sub-Slab Air Sampling Procedures

Two sub-slab air samples will be collected at each building. The sub-slab samples will be tested for an adequate surface seal before and after testing using the helium tracer gas test procedures outlined below.

The sub-slab air sampling procedures are summarized below:

1. Select and prepare the sub-slab sample collection point by observing the condition of the building floor slab for apparent penetrations such as concrete floor cracks, floor drains, or sump holes. The floor conditions will be noted and a potential location of a subsurface probe will be selected. The location will ideally be central to the building, and away from the foundation walls, apparent penetrations and buried pipes. **Review all locations with the Occupant prior to drilling any hole!** Photograph and document all sample locations.

2. In locations where bare concrete is available, drill a 5/8-inch diameter hole about one-inch (1") into the concrete using an electric hammer drill. Extend the hole through the remaining thickness of the slab using a ½ -inch drill bit. Lengthen the hole about three inches (3") beyond the sub-slab using the drill bit.

3. Remove the concrete dust within the 5/8-inch drilled hole and around the hole using wire brushes and a brush and dust pan, then dabbing the surface with Sculpey brand clay (see below).

4. Insert a 5/8-inch outside diameter (OD) by ¼-inch inside diameter (ID) rubber stopper onto and three-inches beyond the end of a 1/4-inch OD by 1/8-inch ID Teflon tube. Insert the Teflon tube into the 5/8-inch hole so the stopper is seated into the top of the ½-inch drilled hole.

5. Seal the annular space between the 5/8-inch hole and the Teflon tubing with white Sculpey Brand modeling clay (or equivalent). Bring the clay above the floor's surface and around the tubing in a volcano-like shape.

6. In locations where only linoleum tile is available, **AND THE OWNER AGREES**, drill a small (1/4-inch best) hole through the tile and slab. Place the sample tubing into the hole and seal to the floor with clay.

7. Purge the sampling tube by connecting the Teflon tubing to the inlet of an air-sampling pump (GilAir 300 or 500) with 3/8-inch OD silicone tubing, and connecting a 1 liter (L) Tedlar bag to the outlet of the pump with silicone tubing. Purge approximately one liter (1L) of gas from the subsurface probe into the Tedlar bag, using the air-sampling pump. Analyze the 1L Tedlar bag containing the sub-slab purged air with a gas detector that records the concentrations of CH4, CO2 and O2. Record the purge times (start and stop) and the gas concentrations on a Summa Canister Sampling Field Data Sheet (Appendix A). Purging flow rates must not exceed 0.2 L/min.

8. Assign sample identification to the Summa canister identification tag and record on chain of custody (COC), and the Summa Canister Sampling Field Data Sheet. Also record the Summa® canister's serial number on the Summa Canister Sampling Field Data Sheet.

9. Remove brass plug from canister fitting.

10. Attach a pre-calibrated/certified 8-hour flow controller, and particulate filter to the Summa® canister. Record the regulator serial number on the Summa Canister Sampling Field Data Sheet.

11. Attach the sample tube to the Summa® canister using a ¼-inch Swagelok® nut with appropriate ferrules, via the flow controller/particulate filter assembly.

12. Open canister valve to initiate sample collection and record sample start time, date and initial vacuum on the canister identification tag and on the Summa Canister Sampling Field Data Sheet. If the canister does not show sufficient vacuum (generally less than 25" Hg), do not use. Take a digital photograph of canister setup and surrounding area. Include in the photograph a dry erase board or similar display which presents sample ID and date.

13. After 8 hours, record sample end time and canister pressure on the Summa Canister Sampling Field Data Sheet, and close valve.

14. Disconnect the Teflon tubing and remove flow controller/particulate filter assembly from canister. Seal canister with brass plug.

15. Seal the hole in the slab with hydraulic cement patch.

16. Ship samples with COCs, overnight, to the selected laboratory, for modified United States Environmental Protection Agency (EPA) Method TO-15 analysis as described in the work plan.

Using Helium Tracer Gas to Test Floor Seals.

1. Drill the concrete floor and attach and seal the Teflon sample tubing to the floor as described above.

2. Place a 2-quart (or similar size) bucket over the floor seal after threading the Teflon sample tube through a hole in the top of the bucket. Seal the tube to the bucket with clay.

3. The bucket should also have a hole in the top for the injection of helium gas. An additional hole should be present in the side, near the bottom, to measure the concentration of helium

gas in the bucket.

4. Connect helium (99.999%) cylinder tubing to the top port of bucket enclosure and seal with clay or other sealing material. Insert a helium detector probe to the bottom port of the bucket.

5. Release enough helium to displace any ambient air in the bucket until the concentration of helium reaches a minimum of 90%. Maintain this minimum concentration by testing with a helium detector. The helium cylinder should be open during the purge time to cause a slight positive pressure within the enclosure.

6. Connect the sample tubing to a GilAir vacuum pump or equivalent using 3/8-inch O.D. silicone tubing. Connect a 1-liter Tedlar bag to the outlet of the pump using silicone tubing and collect a 1-liter sample. Analyze the Tedlar bag for helium using a helium detector, and record the results on the Summa Canister Sampling Field Data Sheet. Also analyze the Tedlar bag for the presence of methane, H2S, CO2 and O2 and record the result on the Summa Canister Sampling Field Data Sheet. A concentration of helium 10% or greater indicates a poor seal of the sample tubing to the floor. The tubing must be resealed to the floor and another helium test conducted.

7. Purging flow rates must not exceed 0.2 L/min.

8. After purging, remove the bucket enclosure and assign sample identification to the Summa® canister identification tag and record on the COC, and the Summa Canister Sampling Field Data Sheet. Also record the Summa® canister's serial number on the Summa Canister Sampling Field Data Sheet.

9. Connect the 1/4-inch Teflon OD sample tubing to the Summa® canister regulator inlet using a ¼-inch Swagelok® nut with appropriate ferrules. Open the canister valve to initiate sample collection and record the start time and date and beginning vacuum on the canister identification tag and on the Summa Canister Sampling Field Data Sheet. If the canister does not show sufficient vacuum (generally less than 25" Hg), do not use.

10. After 8 hours, record sample end time and final vacuum on the Summa Canister Sampling Field Data Sheet and close the valve.

11. Disconnect the Teflon tubing from the Summa® canister and remove the flow controller/particulate-filter assembly from canister. Seal canister with brass plug.

12. Remove the sample tubing, stopper and clay from the hole in the slab and seal with hydraulic cement patch.

13. Ship the samples, with COCs, overnight to the selected lab, for TO-15 analysis.

2.3 Indoor Air and Outdoor Air Sampling Procedure

Sampling procedures for the indoor samples and outdoor air sample are summarized below:

1. Place the indoor air Summa® canisters at breathing height in a high traffic location. The breathing height is defined as approximately three feet above the floor. Place the outdoor air sample at least two to three feet above the ground.

2. Record the canister's serial number on the Summa Canister Sampling Field Data Sheet.

3. Assign sample identification to the canister identification tag (see Section 2.5 below) and record on the COC and the Summa Canister Sampling Field Data Sheet.

4. Remove brass plug from canister fitting.

5. Attach a pre-calibrated/certified 8-hour flow controller and particulate filter to the Summa® canister, open valve completely to initiate sampling, and record the sample start time and date, and beginning vacuum reading on the canister identification tag and the Summa Canister Sampling Field Data Sheet. Also record the regulator serial number on the Summa Canister Sampling Field Data Sheet. If the canister does not show sufficient vacuum (generally less than 25" Hg), do not use.

6. Take a digital photograph of canister setup and surrounding area. Include a dry erase board or similar display which presents sample ID and date.

7. After 24 hours, record end time and pressure on the Summa Canister Sampling Field Data Sheet, and close valve.

8. Disconnect flow controller/particulate filter assembly from canister.

9. Seal canister with brass plug.

10. Ship canister standard overnight, with COC, to the selected laboratory, for TO-15 analysis.

2.4 Quality Control

A field duplicate for the indoor air sample will be collected by attaching the T-fitting supplied by the laboratory to two Summa® canisters with attached regulators. The inlet for the T-fitting will then be attached to the sub-slab sample tubing. Tubing will not be required unless needed to the raise the sampling point to the breathing zone. For sampling, both Summa® canister valves are opened and closed simultaneously.

2.5 Field Documentation

Field notebooks will be used during all on-site work. A dedicated field notebook will be maintained by the field technician overseeing the site activities. In addition to the notebook, any and all original sampling forms, purge forms and notebooks used during field activities will be submitted to the NYSDEC as part of the final report. Indoor air sampling procedures should be photo-documented.

The field sampling team will maintain sampling records that include the following data:

1. Sample Identification
2. Date and time of sample collection
3. Identity of samplers
4. Sampling methods and devices
5. Purge volumes (soil vapor)
6. Volume of soil vapor sample extracted
7. The Summa® canister vacuum before and after samples collected
8. Chain of Custody and shipping information

The proper completion of the following forms/logs will be considered correct procedure for documentation during the indoor air-sampling program:

1. Field Log Book - weather-proof hand-bound field book
2. Questionnaire and Building Inventory Form (Appendix A)
3. Summa Canister Sampling Field Data Sheet (Appendix A)
4. Chain of Custody Form (Appendix A)

<u>Reference:</u> NYSDOH *Guidance for Evaluating Soil Vapor Intrusion in the State of New York*

3.0 SAMPLE SHIPPING

Summary: Proper documentation of sample collection and the methods used to control these documents are referred to as chain-of-custody procedures. Chain-of-custody procedures are essential for presentation of sample analytical chemistry results as evidence in litigation or at administrative hearings held by regulatory agencies. Chain-of-custody procedures also serve to minimize loss or misidentification of samples and to ensure that unauthorized persons do not tamper with collected samples.

The procedures used in this FSP follow the chain-of-custody guidelines outlined in NEIC Policies and Procedures, prepared by the National Enforcement Investigations Center (NEIC) of the U.S. Environmental Protection Agency Office of Enforcement.

Procedure:

1. The chain-of-custody (COC) record (Appendix A) should be completely filled out, with all relevant information.
2. The original COC goes with the samples. It should be placed in a Ziploc bag and taped inside the shipping container. The sampler should retain a copy of the COC.
3. Summa® canisters are shipped in the same boxes the laboratory used for shipping.
4. Place the lab address on top of box. For out-of-town laboratory, add the following: Put "This side up" labels on all four sides and "Fragile" labels on at least two sides. Affix numbered custody seals on front right and left of cooler. Cover seals with wide, clear tape.
5. Ship samples via overnight carrier the same day that they are collected.

APPENDIX A

FIELD ACTIVITY FORMS

Structure Sampling Questionnaire and Building Inventory

New York State Department of Environmental Conservation

Site Name: ______ Site Code: ______ Operable Unit: ______

Building Code: ______ Building Name: ______

Address: ______ Apt/Suite No: ______

City: ______ State: ______ Zip: ______ County: ______

Contact Information

Preparer's Name: ______ Phone No: ______

Preparer's Affiliation: ______ Company Code: ______

Purpose of Investigation: ______ Date of Inspection: ______

Contact Name: ______ Affiliation: ______

Phone No: ______ Alt. Phone No: ______ Email: ______

Number of Occupants (total): ______ Number of Children: ______

☐ Occupant Interviewed? ☐ Owner Occupied? ☐ Owner Interviewed?

Owner Name (if different): ______ Owner Phone: ______

Owner Mailing Address: ______

Building Details

Bldg Type (Res/Com/Ind/Mixed): ______ Bldg Size (S/M/L): ______

If Commercial or Industrial Facility, Select Operations: ______

If Residential Select Structure Type: ______

Number of Floors: ______ Approx. Year Construction: ______ ☐ Building Insulated? ☐ Attached Garage?

Describe Overall Building 'Tightness' and Airflows(e.g., results of smoke tests):

Foundation Description

Foundation Type: ______ Foundation Depth (bgs): ______ Unit: FEET

Foundation Floor Material: ______ Foundation Floor Thickness: ______

Foundation Wall Material: ______ Foundation Wall Thickness: ______ Unit: INCHES

☐ Floor penetrations? Describe Floor Penetrations: ______

☐ Wall penetrations? Describe Wall Penetrations: ______

Basement is: ______ Basement is: ______ ☐ Sumps/Drains? Water In Sump?: ______

Describe Foundation Condition (cracks, seepage, etc.) : ______

☐ Radon Mitigation System Installed? ☐ VOC Mitigation System Installed? ☐ Mitigation System On?

Heating / Cooling / Ventilation Systems

Heating System: ______ Heat Fuel Type: ______ ☐ Central A/C Present?

Vented Appliances

Water Heater Fuel Type: ______ Clothes Dryer Fuel Type: ______

Water Htr Vent Location: ______ Dryer Vent Location: ______

Structure Sampling Questionnaire and Building Inventory

New York State Department of Environmental Conservation

PRODUCT INVENTORY

Building Name: ______________________ Bldg Code: ______________ Date: ______________

Bldg Address: ______________________________________ Apt/Suite No: ______________

Bldg City/State/Zip: ____

Make and Model of PID: ______________________________ Date of Calibration: ______________

Location	Product Name/Description	Size (oz)	Condition *	Chemical Ingredients	PID Reading	COC Y/N?
						☐
						☐
						☐
						☐
						☐
						☐
						☐
						☐
						☐
						☐
						☐
						☐
						☐
						☐
						☐
						☐

* Describe the condition of the product containers as **Unopened (UO)**, **Used (U)**, or **Deteriorated (D)**

** Photographs of the **front and back** of product containers can replace the handwritten list of chemical ingredients. However, the photographs must be of good quality and ingredient labels must be legible.

Product Inventory Complete? ☐ Were there any elevated PID readings taken on site? ☐ ☐ Products with COC?

Structure Sampling Questionnaire and Building Inventory

New York State Department of Environmental Conservation

Site Name: ____________________ Site Code: __________ Operable Unit: __________

Building Code: ____________________ Building Name: ____________________

Address: ____________________ Apt/Suite No: __________

City: ____________________ State: ______ Zip: ______ County: __________

Factors Affecting Indoor Air Quailty

Frequency Basement/Lowest Level is Occupied?: [ ] Floor Material: [ ]

☐ Inhabited? ☐ HVAC System On? ☐ Bathroom Exhaust Fan? ☐ Kitchen Exhaust Fan?

Alternate Heat Source: [ ] ☐ Is there smoking in the building?

☐ Air Fresheners? Description/Location of Air Freshener: ____________________

☐ Cleaning Products Used Recently?: Description of Cleaning Products: ____________________

☐ Cosmetic Products Used Recently?: Description of Cosmetic Products: ____________________

☐ New Carpet or Furniture? Location of New Carpet/Furniture: ____________________

☐ Recent Dry Cleaning? Location of Recently Dry Cleaned Fabrics: ____________________

☐ Recent Painting/Staining? Location of New Painting: ____________________

☐ Solvent or Chemical Odors? Describe Odors (if any): ____________________

☐ Do Any Occupants Use Solvents At Work? If So, List Solvents Used: ____________________

☐ Recent Pesticide/Rodenticide? Description of Last Use: ____________________

Describe Any Household Activities (chemical use,/storage, unvented appliances, hobbies, etc.) That May Affect Indoor Air Quality:

☐ Any Prior Testing For Radon? If So, When?: ____________________

☐ Any Prior Testing For VOCs? If So, When?: ____________________

Sampling Conditions

Weather Conditions: [ ] Outdoor Temperature: [ ] °F

Current Building Use: [ ] Barometric Pressure: [ ] in(hg)

Product Inventory Complete? [ ] ☐ Building Questionnaire Completed?

Structure Sampling Questionnaire and Building Inventory

New York State Department of Environmental Conservation

Building Code: ______________________ Address: ______________________

Sampling Information

Sampler Name(s): ______________________ Sampler Company Code: ______________________

Sample Collection Date: ______________________ Date Samples Sent To Lab: ______________________

Sample Chain of Custody Number: ______________________ Outdoor Air Sample Location ID: ______________________

SUMMA Canister Information

Sample ID:					
Location Code:					
Location Type:					
Canister ID:					
Regulator ID:					
Matrix:					
Sampling Method:					

Sampling Area Info

Slab Thickness (inches):					
Sub-Slab Material:					
Sub-Slab Moisture:					
Seal Type:					
Seal Adequate?:	☐	☐	☐	☐	☐

Sample Times and Vacuum Readings

Sample Start Date/Time:					
Vacuum Gauge Start:					
Sample End Date/Time:					
Vacuum Gauge End:					
Sample Duration (hrs):					
Vacuum Gauge Unit:					

Sample QA / QC Readings

Vapor Port Purge:	☐	☐	☐	☐	☐
Purge PID Reading:					
Purge PID Unit:					
Tracer Test Pass:	☐	☐	☐	☐	☐

Sample start and end times should be entered using the following format: MM/DD/YYYY HH:MM

Structure Sampling Questionnaire and Building Inventory

New York State Department of Environmental Conservation

LOWEST BUILDING LEVEL LAYOUT SKETCH

Please click the box with the blue border below to upload a sketch of the lowest building level .
The sketch should be in a standard image format (.jpg, .png, .tiff)

Clear Image

Design Sketch

Design Sketch Guidelines and Recommended Symbology

- Identify and label the locations of all sub-slab, indoor air, and outdoor air samples on the layout sketch.
- Measure the distance of all sample locations from identifiable features, and include on the layout sketch.
- Identify room use (bedroom, living room, den, kitchen, etc.) on the layout sketc
- Identify the locations of the following features on the layout sketch, using the appropriate symbols:

B or F	Boiler or Furnace	o	Other floor or wall penetrations (label appropriately)
HW	Hot Water Heater	**xxxxxxx**	Perimeter Drains (draw inside or outside outer walls as appropriate)
FP	Fireplaces	**######**	Areas of broken-up concrete
WS	Wood Stoves	● SS-1	Location & label of sub-slab samples
W/D	Washer / Dryer	● IA-1	Location & label of indoor air samples
S	Sumps	● OA-1	Location & label of outdoor air samples
@	Floor Drains	● PFET-1	Location and label of any pressure field test holes.

Structure Sampling Questionnaire and Building Inventory

New York State Department of Environmental Conservation

FIRST FLOOR BUILDING LAYOUT SKETCH

Please click the box with the blue border below to upload a sketch of the first floor of the building.
The sketch should be in a standard image format (.jpg, .png, .tiff)

Clear Image

Design Sketch

Design Sketch Guidelines and Recommended Symbology

- Identify and label the locations of all sub-slab, indoor air, and outdoor air samples on the layout sketch.
- Measure the distance of all sample locations from identifiable features, and include on the layout sketch.
- Identify room use (bedroom, living room, den, kitchen, etc.) on the layout sketc
- Identify the locations of the following features on the layout sketch, using the appropriate symbols:

B or **F**	Boiler or Furnace	o	Other floor or wall penetrations (label appropriately)
HW	Hot Water Heater	**xxxxxxx**	Perimeter Drains (draw inside or outside outer walls as appropriate)
FP	Fireplaces	**######**	Areas of broken-up concrete
WS	Wood Stoves	● SS-1	Location & label of sub-slab samples
W/D	Washer / Dryer	● IA-1	Location & label of indoor air samples
S	Sumps	● OA-1	Location & label of outdoor air samples
@	Floor Drains	● PFET-1	Location and label of any pressure field test holes.

Structure Sampling Questionnaire and Building Inventory

New York State Department of Environmental Conservation

OUTDOOR PLOT LAYOUT SKETCH

Please click the box with the blue border below to upload a sketch of the outdoor plot of the building as well as the surrounding area. The sketch should be in a standard image format (.jpg, .png, .tiff)

Clear Image

Design Sketch

Design Sketch Guidelines and Recommended Symbology

- Identify and label the locations of all sub-slab, indoor air, and outdoor air samples on the layout sketch.
- Measure the distance of all sample locations from identifiable features, and include on the layout sketch.
- Identify room use (bedroom, living room, den, kitchen, etc.) on the layout sketc
- Identify the locations of the following features on the layout sketch, using the appropriate symbols:

B or **F**	Boiler or Furnace	o	Other floor or wall penetrations (label appropriately)
HW	Hot Water Heater	**xxxxxxx**	Perimeter Drains (draw inside or outside outer walls as appropriate)
FP	Fireplaces	**######**	Areas of broken-up concrete
WS	Wood Stoves	● SS-1	Location & label of sub-slab samples
W/D	Washer / Dryer	● IA-1	Location & label of indoor air samples
S	Sumps	● OA-1	Location & label of outdoor air samples
@	Floor Drains	● PFET-1	Location and label of any pressure field test holes.

INDOOR AIR QUALITY INVESTIGATION

Instructions for Occupants

(To be followed starting at least 24 hours prior to and during the sampling event)

- Do not open windows, fireplace openings or vents.
- Do not keep doors open.
- Do not operate ventilation fans or air conditioners.
- Do not smoke in the building.
- Do not use wood stoves, fireplaces or auxiliary heating equipment (e.g., kerosene heaters).
- Do not paint or varnish.
- Do not use cleaning products (e.g., bathroom cleaners, furniture polish, appliance cleaners, all-purpose cleaners, floor cleaners or other cleaners with petroleum or oil-based products).
- Do not use cosmetics, including hair spray, nail polish, nail polish remover, perfume/cologne, etc.
- Do not use air fresheners, scented candles or odor eliminators.
- Do not partake in indoor activities that use solvents or other volatile chemicals.
- Do not apply pesticides.
- Do not store containers of gasoline, oil or petroleum-based or other solvents within the building (except for fuel oil).
- Do not operate or store automobiles in an attached garage.
- Do not operate lawn mowers, snow blowers or pave with asphalt.
- Do not bring home items that have been dry-cleaned.

Summa Canister Sampling Field Data Sheet

Site:

Samplers:

Date:

Sample #					
Location					
Summa Canister ID					
Flow Controller ID					
Additional Tubing Added	NO/ YES - How much	NO/ YES - How much	NO/ YES - How much	NO/ YES - How much	NO/ YES - How much
Purge Time (Start)					
Purge Time (Stop)					
Total Purge Time (min)					
Purge Volume					
Tracer Gas Results					
PID (ppb)					
Pressure Gauge - before sampling					
Sample Time (Start)					
Sample Time (Stop)					
Total Sample Time (min)					
Pressure Gauge - after sampling					
Sample Volume					
Canister Pressure Went To Ambient Pressure?	YES / NO	YES / NO	YES / NO	YES / NO	YES / NO
Weather 24 hours before and during sampling					
General Comments:					

Appendix B

NIOSH Method 6009

MERCURY 6009

Hg MW: 200.59 CAS: 7439-97-6 RTECS: OV4550000

METHOD: 6009, Issue 2 **EVALUATION: PARTIAL** **Issue 1: 15 May 1989**
Issue 2: 15 August 1994

OSHA : C 0.1 mg/m^3 (skin)
NIOSH: 0.05 mg/m^3 (skin)
ACGIH: 0.025 mg/m^3 (skin)

PROPERTIES: liquid; d 13.55 g/mL @ 20 °C; BP 356 °C; HP -39 °C; VP 0.16 Pa (0.0012 mm Hg; 13.2 mg/m^3) @ 20 °C; Vapor Density (air=1) 7.0

SYNONYMS: quicksilver

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (Hopcalite in single section, 200 mg)	**TECHNIQUE:**	ATOMIC ABSORPTION, COLD VAPOR
FLOW RATE:	0.15 to 0.25 L/min	**ANALYTE:**	elemental mercury
VOL-MIN:	2 L @ 0.5 mg/m^3	**DESORPTION:**	conc. HNO_3/HCl @ 25 °C, dilute to 50 mL
-MAX:	100 L	**WAVELENGTH:**	253.7 nm
SHIPMENT:	routine	**CALIBRATION:**	standard solutions of Hg^{2+} in 1% HNO_3
SAMPLE STABILITY:	30 days @ 25 °C [1]	**RANGE:**	0.1 to 1.2 µg per sample
FIELD BLANKS:	2 to 10 field blanks per set	**ESTIMATED LOD:**	0.03 µg per sample
MEDIA BLANKS:	at least 3 per set	**PRECISION ($\bar{S}_r$):**	0.042 @ 0.9 to 3 µg per sample [4]

ACCURACY

RANGE STUDIED:	0.002 to 0.8 mg/m^3 [2] (10-L samples)
BIAS:	not significant
OVERALL PRECISION ($\hat{S}_{rT}$):	not determined
ACCURACY:	not determined

APPLICABILITY: The working range us 0.01 to 0.5 mg/m^3 for a 10-L air sample. The sorbent material irreversibly collects elemental mercury. A prefilter can be used to exclude particulate mercury species from the sample. The prefilter can be analyzed by similar methodology. The method has been used in numerous field surveys [3].

INTERFERENCES: Inorganic and organic mercury compounds may cause a positive interference. Oxidizing gases, including chlorine, do not interfere.

OTHER METHODS: This replaces method 6000 and its predecessors, which required a specialized desorption apparatus [4,5,6]. This method is based on the method of Rathje and Marcero [7] and is similar to the OSHA method ID 145H [2].

REAGENTS:

1. Water, organics-free, deionized.
2. Hydrochloric acid (HCl), conc.
3. Nitric acid (HNO_3), conc.
4. Mercuric oxide, reagent grade, dry.
5. Calibration stock solution, Hg^{2+}, 1000 µg/mL. Commercially available or dissolve 1.0798 g of dry mercuric oxide (HgO) in 50 mL of 1:1 hydrochloric acid, then dilute to 1 L with deionized water.
6. Intermediate mercury standard, 1 µg/mL. Place 0.1 mL 1000 µg/mL stock into a 100 mL volumetric containing 10 mL deionized water and 1 mL hydrochloric acid. Dilute to volume with deionized water. Prepare fresh daily.
7. Stannous chloride, reagent grade, 10% in 1:1 HCl. Dissolve 20 g stannous chloride in 100 mL conc. HCl. Slowly add this solution to 100 mL deionized water and mix well. Prepare fresh daily.
8. Nitric acid, 1% (w/v). Dilute 14 mL conc. HNO_3 to 1 L with deionized water.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame sealed ends with plastic caps, containing one section of 200 mg Hopcalite held in place by glass wool plugs (SKC, Inc., Cat. #226-17-1A, or equivalent).
   NOTE: A 37-mm, cellulose ester membrane filter in a cassette preceding the sorbent may be used if particulate mercury is to be determined separately.
2. Personal sampling pump, 0.15 to 0.25 L/min, with flexible connecting tubing.
3. Atomic absorption spectrophotometer with cold vapor generation system (see Appendix) or cold vapor mercury analysis system.*
4. Strip chart recorder, or integrator.
5. Flasks, volumetric, 50-mL, and 100-mL.
6. Pipet, 5-mL, 20-mL, others as needed.
7. Micropipet, 10- to 1000-µL.
8. Bottles, biological oxygen demand (BOD), 300-mL.

* See SPECIAL PRECAUTIONS

SPECIAL PRECAUTIONS: Mercury is readily absorbed by inhalation and contact with the skin. Operate the mercury system in a hood, or bubble vented mercury through a mercury scrubber.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break ends of sampler immediately prior to sampling. Attach sampler to pump with flexible tubing.
3. Sample at an accurately known rate of 0.15 to 0.25 L/min for a total sample size between 2 and 100 L.
   NOTE: Include a minimum of three unopened sampling tubes from the same lot as the samples for use as media blanks.
4. Cap sampler and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the Hopcalite sorbent and the front glass wool plug from each sampler in separate 50-mL volumetric flasks.
6. Add 2.5 mL conc. HNO_3 followed by 2.5 mL conc. HCl.
   NOTE: The mercury must be in the oxidized state to avoid loss. For this reason, the nitric acid must be added first.
7. Allow the sample to stand for 1 h or until the black Hopcalite sorbent is dissolved. The solution will turn dark brown and may contain undissolved material.
8. Carefully dilute to 50 mL with deionized water. (Final solution is blue to blue-green).
9. Using a volumetric pipet, transfer 20 mL of the sample to a BOD bottle containing 80 mL of deionized water. If the amount of mercury in the sample is expected to exceed the standards, a smaller aliquot may be taken, and the volume of acid adjusted accordingly. The final volume in

the BOD bottle must be 100 mL. To prevent possible loss of mercury during transfer, place the pipet tip below the surface of the liquid in the BOD bottle.

CALIBRATION AND QUALITY CONTROL:

10. Prepare a minimum of two series (six levels each) of working standards covering the range 0.01 to 0.5 µg Hg per aliquot by adding known amounts of the intermediate standard to BOD bottles containing enough 1% nitric acid to bring the final volume to 100 mL.
11. Analyze the working standards together with the samples and blanks (steps 13 through 16). Analyze full set of standards at the beginning of the run, and a second set at the end of the run. Additional standards may be run intermediately during the analysis to confirm instrument response.
12. Prepare calibration graph (peak height vs. solution concentration, µg/sample).

MEASUREMENT:

13. Zero the spectrophotometer by removing the bubbler from the BOD bottle, allowing the baseline on the recorder to stabilize.
14. Place the bubbler in a BOD bottle containing 0.5 µg mercury in 100 mL 1% nitric acid. Adjust the spectrophotometer so that it will give a 75% to full-scale deflection of the recorder.
15. Vent the mercury vapor from the system.
16. Analyze standards, samples and blanks (including media blanks).
    a. Remove the bubbler from the BOD bottle.
    b. Rinse the bubbler with deionized water.
    c. Allow the recorder tracing to establish a stable baseline.
    d. Remove the stopper from the BOD bottle containing the next sample to be analyzed. Gently swirl the BOD bottle.
    e. Quickly add 5 mL 10% stannous chloride solution.
    f. Quickly place the bubbler into the BOD bottle.
    g. Allow the spectrophotometer to attain maximum absorbance.
    h. Vent the mercury vapor from the system.
    i. Place the bubbler into an empty BOD bottle. Continue venting the mercury until a stable baseline is obtained.
    j. Close the mercury vent.

CALCULATIONS:

17. Calculate the amount of mercury in the sample aliquot (W, µg) from the calibration graph.
18. Calculate the concentration C (mg/m^3), of mercury in the air volume sampled, V (L):

$$C = \frac{W \cdot \frac{V_s}{V_a} - B}{V}.$$

Where: Vs = original sample volume (step 8; normally 50 mL)
Va = aliquot volume (step 9; normally 20 mL)
B = average amount of mercury present in the media blanks

EVALUATION OF METHOD:

Rathje and Marcero originally used Hopcalite (MSA, Inc.) as the sorbent material [7]. Later, Hopcalite was shown superior to other methods for the determination of mercury vapor [8]. Atmospheres of mercury vapor for the study were dynamically generated in the range 0.05 to 0.2 mg/m^3 and an adsorbent tube loading of 1 to 7 µg was used. The Hydrar material sometimes used is similar to Hopcalite. No significant difference in the laboratory analysis of mercury collected on the two sorbent materials was observed [9]. OSHA also validated a method for mercury using Hydrar [2]. An average 99% recovery, with $\bar{S}_r$ = 0.042, was seen for 18 samples with known amounts (0.9 to 3 µg) of mercury added (as $Hg(NO_3)_2$) [10]. No change in recovery was seen for samples stored up to 3 weeks at room temperature or up to 3 months at -15 °C; longer storage times were not investigated [10].

REFERENCES:

[1] Evaluation of Mercury Solid Sorbent Passive Dosimeter, Backup Data Report. Inorganic Section, OSHA Analytical Laboratory, Salt Lake City, Utah, 1985.
[2] Mercury in Workplace Atmospheres (Hydrar Tubes). Method ID 145H, Inorganic Section, OSHA Analytical Laboratory, Salt Lake City, UT, 1987.
[3] NIOSH/MRSB. Reports for analytical Sequence Nos. 5854, 5900, 6219, and 6311, NIOSH (Unpublished, 1987-1988).
[4] NIOSH Manual of Analytical Methods, 3rd. ed., Method 6000. (1984).
[5] NIOSH Manual of Analytical Methods. 2nd. ed., V. 4, S199, U.S. Dept. of Health. Education, and Welfare Publ. (NIOSH) 79-141 (1979).
[6] Ibid., V. 5, P&CAM 175, Publ. (NIOSH) 79-141 (1979).
[7] Rathje, A.O., Marcero, D.H. Improved hopcalite procedure for the determination of mercury in air by flameless atomic absorption, Am. Ind. Hyg. Assoc. J. 37, 311-314 (1976).
[8] McCammon, C.S., Edwards, S.L., Hull, R.D., Woodfin, W.J., A comparison of four personal sampling methods for the determination of mercury vapor, Am. Ind. Hyg. Assoc. J., 41, 528-531 (1980).
[9] Internal Methods Development Research, DataChem Laboratories, Inc., Salt Lake City, UT (1982).
[10] Eller, P.M., NIOSH, unpublished data (1987-88).

METHOD WRITTEN BY:

Keith R. Nicholson and Michael R. Steele, DataChem Laboratories, Inc., Salt Lake City, Utah, under NIOSH contract No. 200-87-2533.

APPENDIX: COLD VAPOR MERCURY ANALYSIS SYSTEM

1. The valve should direct the vented vapors to a hood or to a mercury scrubber system.
2. When the valve is opened to "Vent" the peristaltic pump should draw room air. Place a Hopcalite tube in the air intake to eliminate any mercury that may be present.
3. Adjust the peristaltic pump to a flow that will create a steady stream of bubbles in the BOD bottle, but not so great that solution droplets enter the tubing to the quartz cell.
4. If water vapor condenses in the quartz cell, heat the cell slightly above room temperature by wrapping it with a heating coil and attaching a variable transformer.
5. The bubbler consists of a glass tube with a bulb at the bottom, slightly above the bottom of the BOD bottle. The bulb contains several perforations to allow air to escape into the solution (in a stream of small bubbles). A second tube is provided to allow the exit of the vapor. The open end of the second tube is well above the surface of the liquid in the bottle. The two tubes are fixed into a stoppering device (preferably ground glass) which fits into the top of the bottle. A coarse glass frit can be used in place of the bulb on the first tube. However, it is more difficult to prevent contamination when a frit is used.
6. Replace the flexible tubing (Tygon or equivalent) used to connect the bubbler, cell, and pump periodically to prevent contamination from adsorbed mercury.

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