



IN-SITU CHEMICAL OXIDATION PILOT TEST WORK PLAN

FORMER AIR FORCE PLANT 51
4777 Dewey Avenue
Greece, New York
NYSDEC Site #828156

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1.0 INTRODUCTION

Groundwater & Environmental Services, Inc. (GES), on behalf the New York State Department of Environmental Conservation (NYSDEC), has prepared this pilot test workplan which outlines proposed pilot testing activities using chemical oxidation methods for Operable Unit 1 (OU1) which includes Area of Concern 1 (AOC1, former plating pond and lagoon) and AOC2 (lagoon and storm water outfall) at the Former Air Force Plant 51 facility, located at 4777 Dewey Avenue, Greece, New York (the “Site”). The proposed pilot testing activities are based on a review of historical documentation provided to GES which includes a “Pilot Phase Remedial Design Plan” prepared by Day Environmental Inc. (issued June 25, 2008) and a letter prepared by Day Environmental outlining pilot test well installation details (dated March 11, 2009). A generalized site map indicating pertinent site features is presented as **Figure 1**.

This workplan was prepared with the purpose of evaluating the feasibility of applying in-situ chemical oxidation (ISCO) technologies in order to address the chlorinated hydrocarbon compounds identified in subsurface soils and groundwater at OU1. Two liquid oxidants, hydrogen peroxide and sodium permanganate, have been selected for the ISCO testing. The hydrogen peroxide will be applied using GES’ patented HypeAir® technology. The following sections in this work plan outline methodologies for design and implementation of the ISCO pilot testing work scope at OU1. The proposed activities will be conducted in accordance with all applicable federal, state and local rules and regulations.

1.1 In-Situ Chemical Oxidation Technology Overview

ISCO has been proven to be an effective remedial technology for the oxidation of volatile organic compounds (VOCs) in subsurface soils and groundwater. Strong oxidizers such as hydrogen peroxide can be injected into the subsurface to chemically oxidize a wide variety of VOCs, including chlorinated compounds such as tetrachloroethylene (PCE) and trichloroethylene (TCE), converting them into carbon dioxide and water.

When introduced into groundwater, hydrogen peroxide is unstable, and will readily react with organic contaminants and other subsurface organic materials. Injecting concentrations of hydrogen peroxide as low as 100 milligrams per liter (mg/L) can also cause oxygen concentrations in groundwater to exceed the solubility limit of oxygen in groundwater (typically 9-10 mg/L at atmospheric pressures and typical groundwater temperatures). When this occurs, oxygen gas is formed and is lost in the form of bubbles that rise through the saturated zone to the water table and into the unsaturated zone. Hydrogen peroxide alone is not capable of oxidizing VOCs; it typically requires a suitable catalyst in order to generate reactive hydroxyl radicals. The catalyst can be ozone, a metal catalyst such as ferrous iron, permanganate, or ultraviolet light. Hydrogen peroxide is particularly effective when it reacts with ferrous iron to produce Fenton's reagent. Ferrous iron may be naturally present in the subsurface soils and/or groundwater or can be enhanced through the addition of reagents such as ethylene diamine tetra acetic acid (EDTA) or iron salts such as ferric sulfate.

Catalyzed hydrogen peroxide (CHP) is a mixture of hydrogen peroxide (H₂O₂) and an iron catalyst such as iron-EDTA or acidified ferrous iron that can potentially oxidize a wide range of VOCs. The simplified reaction is presented below:



If iron is naturally occurring in groundwater or present in soil, then it may be possible to achieve the similar reactivity with H₂O₂ and EDTA, since the EDTA will chelate the iron and help keep it in solution.



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Catalyzed hydrogen peroxide (CHP) is a mixture of hydrogen peroxide (H₂O₂) and an iron catalyst such as iron-EDTA or acidified ferrous iron that can potentially oxidize a wide range of VOCs. The simplified reaction is presented below:



If iron is naturally occurring in groundwater or present in soil, then it may be possible to achieve the similar reactivity with H₂O₂ and EDTA, since the EDTA will chelate the iron and help keep it in solution.



Iron can precipitate as $\text{Fe}(\text{OH})_3$ at high pH and the solid iron will catalytically decompose peroxide. Anecdotal evidence indicates that this occurs frequently during ISCO with H_2O_2 and iron. Chelating agents, such as EDTA, minimize the precipitation of iron and create favorable conditions for the catalytic production of hydroxyl radicals.

CHP is non-selective and may react not only with the target compounds, but also with soil particles and natural organic matter. In addition, the system is catalytic so it is not possible to write a stoichiometric reaction for the oxidation of a specific compound or to determine the amount of H_2O_2 and iron that will be needed to convert a specific contaminant to carbon dioxide and water.

Like all oxidants, CHP may potentially have long term or short term secondary affects such as oxidation of soil-bound metals or mobilization of metals due to the presence of a chelating agent or changes in pH. The magnitude and duration of any such changes are site-specific and may or may not be significant.

Chlorinated VOCs may also be oxidized via permanganate. Permanganate is commercially available as both potassium and sodium salts. Potassium permanganate is solid purple crystal that is typically applied as a 1 to 7% solution. Sodium permanganate is a dark purple solution that can be applied at concentrations ranging from 10% to 40%. Permanganate is a milder oxidant in comparison to peroxide, and can rapidly convert a wide range of chlorinated VOCs to carbon dioxide, water, and chloride ions. The permanganate is reduced to insoluble manganese dioxide during the reaction. Permanganate oxidation involves a direct electron transfer unlike other oxidants, such as persulfate and hydrogen peroxide, which use a free radical process. Permanganate has a unique affinity for oxidizing organic compounds containing carbon-carbon double bonds, aldehyde groups or hydroxyl groups. As an electrophile, the permanganate ion is strongly attracted to the electrons in carbon-carbon double bonds found in chlorinated alkenes, borrowing electron density from these bonds to form a bridged, unstable oxygen compound known as the cyclic hypomanganate ester. This intermediate product further reacts by a number of mechanisms including hydroxylation, hydrolysis or cleavage. Under most naturally occurring subsurface pH and temperature conditions, the carbon-carbon double bond of alkenes is broken spontaneously and the unstable intermediates are converted to carbon dioxide through either hydrolysis or further oxidation by the permanganate ion.

1.2 The GES HypeAir® Process

Hydroxyl radicals are unstable compounds and can react with organic compounds in order to achieve a more stable chemical configuration. Carbon dioxide gas and water are typically the ultimate end products that are formed. These reactions can occur rapidly, so the hydroxyl radicals may not have sufficient time to come into contact with other organic compounds. GES has developed a patented process called “HypeAir®” which can overcome this limitation. The HypeAir® technology can direct various chemical oxidants and air into injection points and site monitoring wells to remediate both adsorbed-phase and dissolved-phase organic compounds.

The distribution of the oxidant solution throughout the formation is facilitated through the application of compressed air at the injection wells. The injection of pressurized air disperses the liquid oxidizer laterally (through the saturated soil zones), and to some extent, vertically (into the vadose zone). Organic compounds that are adsorbed to organic material in the soil may effectively be protected from typical ISCO systems, particularly for areas with significant layers or lenses of low permeability. The HypeAir® System helps to promote desorption of organic compounds off the soil by a pseudo-scrubbing action. The increased groundwater temperature and the high air sparge velocities essentially create a low pressure region along soil particles that enhances desorption of chemical constituents. An additional benefit of



ISCO is the temporary increase of oxygen levels in and around the treatment area. The increased oxygen concentrations can stimulate the growth of naturally occurring micro-organisms, resulting in aerobic biodegradation processes and leading to further site remediation.

The major components of the HypeAir® system include a holding tank, transfer pump, a soil vapor extraction (SVE) system, and an air compressor. All equipment is mounted within a mobile box truck platform. Electrical power to the various system components is provided by an on-board generator. The holding tank is used to mix and contain the oxidant solution, which can be pumped into three injection wells simultaneously. Flow rates used for the injection process typically range between 0.5 to 5 gallons per minute (gpm). Compressed air may also be sparged into injection wells at a total flow rate of up to 60 standard cubic feet per minute (scfm). The SVE recovery system can operate at a maximum vacuum of 24 inches of mercury (inHg) and 60 scfm air flow. A process and instrumentation diagram (P&ID) depicting the HypeAir® equipment is included as **Figure 2**. A P&ID legend is included as **Figure 3**.

2.0 PILOT TEST WELLS

In order to complete the proposed pilot testing, GES plans to install 8 new wells. These wells will include:

- 2 – GES Patented HypeAir injection wells
- 2 – Permanganate injection wells
- 2 – SVE wells
- 2 – Monitoring points

Prior to advancing any of these new wells, GES will contact the underground facilities protection organization (UFPO) in order to verify that the proposed locations are clear of any public subsurface utilities. Each of the locations will be pre-cleared to 5 ftbg using soft dig technologies to percent damage to potentially unknown subsurface structures and utilities.

During the pre-clearing and well installation GES will monitor the breathing zone in the immediate work area for the presence of VOCs and particulates. Additional air monitoring details will be outlined in the site specific health and safety plan (HASP) to be submitted to the NYSDEC prior drilling mobilization.

All of the proposed wells will completed with a protective well vault, mounted in concrete and flush with grade. The wells will also be developed by hand surging and pumping to remove sediment from the well and the surrounding sand pack.

All soil and water waste materials generated during the drilling activities will be containerized and staged onsite for future disposal. Additional detail of waste handling and disposal will be submitted to the NYSDEC in the form of a waste management plan prior to drilling mobilization.

2.1 Pilot Test Well Installation

GES proposes the installation of two pairs of nested HypeAir® injection wells (IP-1, IP-2) at OU1 within AOC1 (pilot test area #1), and two single injection points (IP-3, IP-4) in the area downgradient of AOC1, located to the east of the drainage swale (pilot test area #2) as shown in **Figure 4**. Pilot testing in test areas #1 and #2 will be conducted using GES' patented HypeAir® injection process, and the sodium permanganate injection process, respectively. Additionally, two proposed SVE wells (SVE-1, SVE-2) will also be installed in pilot test area #1 for the purposes of SVE testing and groundwater monitoring during the proposed HypeAir® pilot testing activities. Two monitoring points (MP-1, MP-2) will be also



installed in pilot test area #2 to collect data to evaluate the effectiveness of the sodium permanganate testing.

Soil sampling will be conducted continuously with the advancement of split spoon samplers ahead of the advancement of the augers. Soil samples will be classified in the field and screened for the presence of ionizable VOCs using a photo-ionization detector (PID). It is anticipated that the total depths of the wells will not exceed 30ftbg. The final injection well installation depths and screen intervals will be based on the results of this field analysis.

2.2 Soil and Groundwater Sample Collection

During the advancement of the proposed wells, one soil sample will be collected from each location and submitted for laboratory analysis of the chemicals of concern (COCs) which will include vinyl chloride (VC), 1,1-dichloroethene (1,1-DCE), trans-1,2 dichloroethene (t-1,2-DCE), cis-1,2 dichloroethene (c-1,2-DCE), trichloroethene (TCE), 1,1 dichloroethane (1,1-DCA), 1,1,1 trichloroethane (1,1,1-TCA), and 1,1,2 trichloroethane (1,1,2-TCA). Samples will also be analyzed for EPA target analyte listed (TAL) metals and for total organic carbon (TOC).

Initial baseline groundwater samples will also be collected from the proposed SVE and monitoring wells. These samples will be submitted for laboratory analysis of the COCs specified above, along with total and dissolved TAL metals and total alkalinity. The proposed soil and groundwater sampling and analysis plan is presented on **Table 1**.

During the collection of the initial groundwater samples, field instrumentation will be used to screen the groundwater for dissolved oxygen (DO), oxidation-reduction potential (ORP), pH, temperature, conductivity and other parameters. The full list of parameters and instrumentation is provided on **Table 1**.

The field screening and laboratory analytical data will be used as baseline data to be evaluated in conjunction with the results of the pilot study and post-injection sampling data. This evaluation will assist in the design and implementation of a full-scale remedial plan.

2.3 HypeAir Injection Point Construction

The proposed HypeAir® injection points (IP-1, IP-2) will be specially constructed in accordance with GES patented MaxOx well design specifications as outlined below in order to allow for an efficient method of dispersing hydrogen peroxide solution, ozone and air in the subsurface. This construction specification allows for an increased radius of influence and greater formation of hydroxyl radicals. The HypeAir® injection point screens will be prefabricated by GES and will include a nested set of both a hydrogen peroxide and an air/ozone injection point installed to a maximum depth of approximately 30 feet below grade in a single borehole. The final installation depth will be dependent on field screening results as described in **section 2.1**.

The air/ozone portion of the nested points will be installed to the full depth of the boring and constructed of ¾ inch diameter inch stainless steel (SS) components. The screen section will between 3 to 5-foot in length and set in sand pack which will extend 6 inches below and above the screen. A 1.5 to 2 foot thick bentonite/grout seal will be installed on top of this sand pack to separate the air/ozone screen from the peroxide injection portions of the nested point. The top of the casing will extend to 0.5 ftbg and finished with a threaded fitting to allow the connection of GES' MaxOx injection assembly.



The peroxide portion of the nested point will be installed to a maximum depth of approximately 30 inches above the top of the air/ozone injection point screen interval and constructed of a combination of 2 inch diameter polyvinyl chloride (PVC) and SS components. . The injection screens will be 5-foot long, PVC wire-wrapped/continuous slot set in sand pack which will extend 6 inches above and below the screen. A 1.5 foot thick bentonite seal will be installed on top of the sand pack, followed by grout to approximately 1 ftbg. PVC casing will be used to extend the well from the top of screen to 3.5 ftbg, at which point the well components will transition to SS. This will allow for the safe application of pressurized oxidant solution into the injection wells. The top of the well will be finished at 0.5 ftbg with a SS threaded fitting to allow the connection of the MaxOx injection assembly.

2.4 Permanganate Injection Point Construction

The proposed sodium permanganate injection wells (IP-3, IP-4) will be constructed similar to the peroxide portion of the nested HypeAir injection points. The injection screens will be 5-foot long, PVC wire-wrapped/continuous slot set in sand pack which will extend 6 inches above and below the screen. A 1.5 foot thick bentonite seal will be installed on top of the sand pack, followed by grout to approximately 1 ftbg. PVC casing will be used to extend the well from the top of screen to 3.5 ftbg, at which point the well components will transition to SS. This will allow for the safe application of pressurized oxidant solution into the injection wells. The top of the well will be finished at 0.5 ftbg with a SS threaded fitting to allow the connection of the MaxOx injection assembly. The final installation depth will be dependent on field screening results as described in **section 2.1**, above.

2.5 Monitoring Point Construction

Monitoring points MP-1 and MP-2 will be installed in pilot test area #2 to monitor various parameters that will be used to assess the effectiveness and possible limitations of the sodium permanganate application. Each monitoring point will be installed as a schedule 40 PVC well utilizing 2-inch diameter screens and risers. Each well screen will be approximately 20-feet long, and will be installed from approximately 10 to 30 feet below grade. Sand pack will be placed around each well screen, to a minimum depth of approximately 6 inches above the top and bottom extent of the well screens. A bentonite seal (minimum of 12 inches thick) will be placed above the sand pack. The monitoring points will be cased between 6 inches to 10 feet below grade.

2.6 Soil Vapor Extraction Well Construction

Soil vapor extraction wells SVE-1 and SVE-2 will be installed in pilot test area #1. These wells will be utilized to for SVE and the collection of groundwater field parameters during the proposed hydrogen peroxide injection activities in this area. Each SVE well will be installed as a schedule 40 PVC well utilizing 4-inch diameter screens and risers. Each well screen will be approximately 25-feet long, and will be installed from approximately 5 to 30 feet below grade. Sand pack will be placed around each well screen, to a minimum depth of approximately 6 inches above the top and bottom extent of the well screens. A bentonite seal (minimum of 18 inches thick) will be placed above the sand pack. The SVE wells will be cased between 6 inches to 5 feet below grade.

2.7 Well Development

Following installation of the above proposed pilot test wells, each will be developed by hand in order to ensure proper communication between the well and the aquifer material. Prior to conducting the pilot test, the previously installed injection wells will be gauged to ensure silt has not entered the wells. If



sediments have entered the wells and a significant amount of screen is covered, the injection wells will be re-developed on the day of the injection. Purge water generated during well development activities will be containerized and staged onsite for transport to a NYSDEC approved disposal facility at a later date.

3.0 PILOT TEST METHODOLOGY

GES proposes to conduct up to four days of pilot testing following the installation, sampling and development of the proposed pilot test wells described above. Additionally, the existing groundwater monitoring wells at OU1 will be used for monitoring groundwater quality parameters as outlined below and on **Table 1**. Equipment and materials used during the testing will be staged near the northern portion of the on-site building.

Hydrogen peroxide will be delivered to the Site in 300-gallon plastic totes at concentrations ranging between 10 to 15%. The totes will be stored within a portable containment berm that will provide a minimum capacity of 110% of the total liquid oxidant volume. A separate 300-gallon tote will be used for storage of water that will be used for rinsing equipment. Sodium permanganate solution will be delivered to the Site via a tanker truck at a pre-mixed concentration of 10%. The truck will be staged within a containment berm that will provide a minimum capacity of 110% of the total liquid oxidant volume. Material Safety Data Sheets (MSDS) will be available on-site for all the chemicals that are used during the testing. Specifications for the containment berms to be used for the hydrogen peroxide totes and sodium permanganate solution in the tanker truck are provided in **Appendix A**.

3.1 Permitting

As per the requirements of the EPA's underground injection control (UIC) program, GES will submit a letter of notification, and an injection well inventory form (EPA form number 7520-16) to the EPA's UIC branch at least 30 days prior to conducting the proposed pilot testing activities.

Under the UIC program, there are five classes of injection wells (Types I through V). Injection wells used for environmental remediation purposes fall under the generalized Type V classification. The inventory form will include the Site information and will designate the four proposed injection wells (IP-1, IP-2, IP-3 and IP-4) as Active Type V injection wells.

GES will also submit a notification letter to the local fire department at least one week in advance of the proposed pilot testing activities. This letter will include the general scope of work and the quantity and types of chemicals that will be on-site.

3.2 Health and Safety Considerations

Due to the nature of the chemical oxidation reactions, potential safety risks may develop. The exothermic chemical oxidation reactions, if not monitored and controlled properly, can raise the temperature of groundwater, produce steam, and generate significant pressures in the application area. Additionally, preferential flow paths to subsurface utility conduits, porous and/or non-native soils, and fill material may develop during the injection process. These pathways may serve as conduits to potential vapor receptors in the area, including subsurface utilities. To manage these potential risks, GES will conduct the following activities during each event:



- A photo-ionization detector (PID) and a combustible gas indicator (CGI) will be used to monitor for total VOCs, percent lower explosion limit (LEL) and percent oxygen, respectively in the breathing zone, the space within the injection point vault, and in any utility vault and or storm drain in the vicinity of the injection area.
- During peroxide injection activities in pilot test area #1, a SVE system will be operated to ensure that significant levels of oxygen and organic vapors do not develop in the subsurface. The proposed soil vapor extraction wells (SVE-1, SVE-2) will be utilized for this purpose. Additional monitoring wells in the vicinity of the application may also be used for SVE if deemed necessary.
- A detailed account of specific health and safety precautions taken during implementation of this technology will be summarized in the site specific HASP, the Job Safety Analysis worksheets (JSAs), the Spill Contingency Plan, and the site-specific Contingency Plan, all of which will be presented to the NYSDEC prior to and available on-site during the execution of the proposed scope of work.
- Downwell transducers will be installed in up to 4 nearby monitoring wells to collect depth to water and pressure/vacuum data continuously. The data will be monitored to determine if excessive groundwater mounding and pressures are present during the injection procedure. Depth to water reading in other monitoring wells may also be collected manually using a Solinst or similar water level meter.

3.3 Water Quality Parameter Monitoring

Prior to initiating injection activities, GES will collect baseline parameters including DO, pH, ORP, temperature, and conductivity from the proposed monitoring wells MP-1 and MP-2, from proposed SVE wells SVE-1 and SVE-2, and from existing monitoring wells MW1-4, MW1-5, MW1-6, MW1-7, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-16 and MW1-22. A YSI 556 multi-parameter water quality meter/probe or equivalent, will be used to collect these parameters. The probe will be decontaminated in between each measurement by rinsing it thoroughly with a solution of Alconox, followed by a de-ionized water rinse. These parameters will also be collected periodically during the injection procedure in order to assess overall effectiveness of the process. Observation wells MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, and MW1-18 as wells as proposed SVE wells SVE-1 and SVE-2 will also be periodically field screened for dissolved iron and hydrogen peroxide concentrations. Dissolved iron will be analyzed in the field using a Hach DR2800 spectrophotometer via EPA FerroVer Method #8008. This is a colorimetric method in which a small quantity of reagent (FerroVer powder) is added to a 10 milliliter volume of sample. If iron is present in the sample, an orange solution is formed after a brief reaction period. The detection range for this method is 0 to 3 mg/L of dissolved iron. A detailed methodology for the Hach 8008 method is provided in **Appendix B**. Hydrogen peroxide will be field analyzed using LaMotte Insta-test strips which have a measurement range of 0 to 100 ppm. It may be necessary to introduce additional Iron-EDTA solution if dissolved iron concentrations are observed to be insufficient within pilot test area #1 as described in **Section 3.4** below.

Observation wells MW1-6, MW1-7, MW1-18 and MW1-22, and proposed monitoring wells MP-1 and MP-2 will also be field tested for permanganate using the Hach Pocket Colorimeter II Manganese HR System and USEPA periodate oxidation method #8034 (provided in **Appendix C**).

These water quality parameters will also be collected from the listed monitoring wells after approximately 1 week and 6 weeks from the conclusion of the proposed injection activities. The water quality parameter



data will be evaluated to assess overall effectiveness of the process at each injection well location. The above field parameter plan is also presented on **Table 1**.

3.4 Catalyzed Hydrogen Peroxide (Pilot Test Area #1)

The CHP process is a non-selective process that may react with both target compounds and naturally occurring organic compounds at a near neutral level groundwater pH. The total volumetric quantities of activator and oxidant solutions that will be introduced into each injection well will be based on a target soil treatment volume around each injection well, an assumed 10 foot radius around each injection point, and an approximate 5 foot thickness. Based on these assumptions and a soil porosity of 0.25, the total pore space volume that is required to be addressed around each injection point is approximately 3,000 gallons.

The volume of liquid that can be injected into the formation should not exceed the pore volume (PV) of the anticipated radius of influence. A target of 70% of the PV is selected for the pilot study based on GES' experience at other similar sites having approximately similar lithology. Specifically, field measurements of both oxidant and dissolved oxygen concentrations have routinely indicated the presence of these parameters at observation wells located within 10 feet of a given injection point. These historical observations indicate that a sufficient volume of the existing groundwater was displaced by the liquid oxidants, allowing the oxidants to disperse radially around the injection points when up to 70% of the pore volume of oxidant fluid was injected. Therefore the total liquid oxidant volume required for this pilot test is approximately 2,000 gallons per injection well or approximately 4,000 gallons total.

Stainless steel wellhead injection assemblies (fitted with a flow control valves, check valves, and pressure gauges) will be used to connect to the top of each of the 2" diameter hydrogen peroxide injection points. The injection flow rate of the oxidant solution will initially be limited to 0.5 gpm. The flow rate will then gradually be increased up to the maximum pumping rate of the injection pump (approximately 5 gpm per well), depending on the observed well pressures and other field observations. Flow rates will be decreased if well pressures are observed to exceed 15 pounds per square inch (psi), or if excessive groundwater mounding is observed in the adjacent monitoring wells. Prior to initiating the injection activities described, each injection well will be purged for at least 15 minutes with pressurized air in order to clear silt and foreign materials that may be present on the well screens.

A target iron concentration of 250 mg/L will be used to activate the hydrogen peroxide injected in in the pilot test area. In order to achieve this iron concentration, a total volume of 200 gallons of Iron-EDTA solution (consisting of 13% iron by weight; trade name GrowMore™) at a concentration of approximately 20,000 mg/L will be introduced into each injection well prior to the injection of hydrogen peroxide. This will allow for an adequate concentration of dissolved iron to be stabilized in the groundwater and thereby be made available for the CHP process.

The total required volume of 2,000 gallons of hydrogen peroxide per injection point assumes that the formation has the capacity to accept the oxidants at a minimum flow rate of approximately 2 gallons per minute over the two day pilot testing period in pilot test area #1. If the average 2 gpm injection flow rate cannot be achieved, the total injection volumes that may be achieved may be lower than the targeted volumes proposed. Based on GES previous experience at other facilities, a hydrogen peroxide concentration range of 10-15% has proven to be the most optimal for field application. Concentrations below 10% are typically too low to provide adequate reactions with the chemicals of concern, while concentrations above 15% may result in rapid decomposition of hydrogen peroxide and increased groundwater temperatures. A hydrogen peroxide concentration of 10% will be utilized during the first



day of the pilot test. If favorable data is obtained, the hydrogen peroxide concentration will be increased to 15% on the second day.

During the injection of hydrogen peroxide solution into IP-1 and IP-2, groundwater quality parameters will be monitored as outlined above in the specified monitoring wells. Groundwater mounding will also be monitored in these wells as described in **Section 3.2** in order to ensure that the hydrogen peroxide solution does not break through to the surface.

A limited quantity of hydrogen peroxide solution may also be injected into several of the existing ¾-inch diameter injection points (previously installed in 2008) in order to compare injection flow rates, pressures and other data with the performance of the proposed HypeAir® injection wells. The decision to add the testing of the existing wells will be made based on the field observations during injection into the IP-1 and IP-2.

3.5 Sodium Permanganate Injection (Pilot Test Area #2)

On each day of the proposed injection activities in pilot test area #2, a total volume of up to 1,000 gallons of 10% sodium permanganate solution will be injected into injection wells IP-3 and IP-4. This volume represents half of the proposed volume of hydrogen peroxide to be injected in injection wells IP-1 and IP-2 in Pilot Test Area #1. Permanganate reacts with COCs occur at a slower rate than hydrogen peroxide, and therefore is expected to persist for a longer period in the subsurface, allowing for a greater area of influence with a lower injection volume. Groundwater quality parameters and residual permanganate will be monitored periodically as outlined above (**Section 3.3**).

A stainless steel wellhead injection assembly (fitted with a flow control valve, check valve, and pressure gauge) will be used to connect to injection wells IP-3 and IP-4. The injection flow rate of the oxidant solution will initially be limited to 0.5 gpm. The flow rate will then gradually be increased up to a maximum of 5 gpm, depending on the observed well pressures and other field observations. Flow rates will be decreased if well pressures are observed to exceed 15 psi, or if excessive groundwater mounding is observed in the monitoring wells. Prior to initiating the injection activities described, each injection well will be purged for at least 15 minutes with pressurized air in order to clear silt and foreign materials that may be present on the well screens.

A limited quantity of sodium permanganate solution may also be injected into several of the existing ¾-inch diameter injection points (previously installed in 2008) in order to compare injection flow rates, pressures and other data with the performance of the proposed injection wells.

3.6 Air Injection (Pilot Test Areas #1 & #2)

Compressed air will be sparged into the injection wells at flow rates of up to 10 scfm. This maximum flow rate is based on the capabilities of the equipment that will be brought to site. The process will be completed in order to enhance the radius of influence of the oxidant, and to enhance mixing of the various reagents within the subsurface. Air will be injected in each of the four injection pilot test wells for a minimum of one hour, following the injection of the hydrogen peroxide or sodium permanganate solution. The groundwater quality parameters outlined in **Section 3.3** will continue to be monitored during this process. Air flow rates may be adjusted as necessary based on observed conditions and monitoring data obtained.



Air injection may also be conducted at several of the existing $\frac{3}{4}$ -inch diameter injection points (previously installed in 2008) in order to compare injection flow rates, pressures and other data with the performance of the proposed injection wells.

3.7 Ozone Injection (Pilot Test Area #1)

Ozone gas will be generated using the available on-board HypeAir® equipment, and will be sparged into each of the proposed HypeAir® injection wells via the $\frac{3}{4}$ -inch diameter stainless steel injection point. Since ozone is highly reactive and incompatible with PVC, Teflon tubing with suitably rated compression fittings will be used to connect the discharge ports from the ozone generator to each injection well. Ozone and air sparging will be conducted concurrently with the injection of hydrogen peroxide. The total flow from the ozone generator is limited to approximately 0.3 scfm. Since this flow is too low for an effective application of sparging, pressurized air will be utilized to increase the total flow rate to a range of approximately 5 to 10 scfm. This will generate an ozone concentration output of approximately 1,000 to 2,000 ppm. Air flow rates will be adjusted as necessary based on site conditions and monitoring data obtained. Ozone injection will be conducted in the deeper injection screen of each nested well pair concurrent with the hydrogen peroxide injection in the shallower injection screens as described in **Section 3.4**, above.

3.8 Soil Vapor Extraction and Air Monitoring Program

During the pilot testing activities to be performed in Pilot Test Area #1 as described in **Section 3.4** above, GES will operate the on-board SVE system in order to collect subsurface vapors and off-gases that are generated. Soil vapors will be passed through an air/water separator or knockout tank in order to collect moisture and condensate in the influent stream. The knockout tank is equipped with a high level shut-off switch that turns off the SVE system and the injection pumps in the event of excessive moisture collection. The condensate/moisture that accumulates in the knockout tank will be containerized in a 55-gallon drum, and will be disposed of off-site at the conclusion of the pilot test. All extracted soil vapors will be directed through 200 pounds of granular activated carbon prior to atmospheric discharge via a minimum 15-foot high, 2-inch diameter stack.

In order to determine the most optimal vapor extraction operating conditions, vacuum will be applied at proposed extraction wells SVE-1 and SVE-2 in incremental steps up to the maximum attainable vacuum. The vacuum will be reduced if groundwater upwelling is observed. Vacuum transducers will be placed in selected monitoring wells in order to measure the extent of vacuum influence across the pilot test area. The extracted air flow stream will also be periodically screened for total VOC concentration, percent oxygen, percent LEL, and total air flow. The applied vacuum at each extraction well will also be recorded and adjusted throughout the injection activities as conditions warrant. While injecting air as described in **Section 3.6**, the total minimum vapor extraction air flow rate shall be a minimum of three times the air injection flow rate. If it is not possible to obtain this ratio between injected/extracted air, the air injection rate will be reduced accordingly. It may be necessary to further adjust operation of the SVE system based on the ambient air monitoring data collected as per **Section 3.2**, above.

4.0 POST-INJECTION SAMPLING AND PILOT TEST SUMMARY REPORT

Post-injection soil and groundwater samples will be collected from each of the monitoring wells in the study area approximately 6 weeks after completion of the pilot test. A total of four soil samples will also be collected within approximately 5 feet of each of the proposed injection wells. Sampling depths will be consistent with sampling depths for the pre-injection soil samples to be collected from the sampling



locations described in **Section 2.2**. The soil samples will be submitted for laboratory analysis of the COCs identified previously and total TAL metals. These data will be compared to the baseline soil analytical data from the installation of the proposed injection and monitoring wells described above. Groundwater samples will also be collected from each of the monitoring wells in the pilot test area and will be submitted for laboratory analysis of the COCs, total and dissolved TAL metals. The proposed post-injection soil and groundwater sampling and analysis plan is presented on **Table 1**.

Performance data from the pilot study that will be reviewed to evaluate the success of the application and will primarily consist of the parameter data collected at the observation wells (DO, ORP, pH, temperature and presence of oxidant and activator), and the oxidant and air injection flow rates. These data will allow GES to evaluate the radial influence around each injection well, and the rate at which the oxidants can be injected. GES will also evaluate the COCs in soil and groundwater to further determine the effectiveness of the application with respect to concentration reductions in both the dissolved and adsorbed phases.

Upon receipt of the post-injection soil and groundwater sampling results, GES will prepare a detailed summary report with recommendations for full-scale application, if warranted. The report is anticipated to include the following:

- Daily injection logs which will include duration of injection at each well, quantity of oxidants/activators injected, injection pressures and other relevant observations;
- Groundwater parameter data trends from before, during and after the pilot test;
- “As-built” well logs showing injection/SVE/monitoring well installation details;
- Pre and Post-injection sampling data (soil and groundwater);
- Approximate radius and depth of influence in areas tested;
- Estimates of injection and contaminant mass removal rates;
- Quantity and proposed locations of additional HypeAir/permanganate injection wells;
- Alternative Oxidant/Activator combinations that may be considered;
- Locations and quantity of additional soil vapor extraction and monitoring wells;
- Optimal oxidant and air injection flow rates;
- Optimal soil vapor extraction operational data; and,
- Modifications to air monitoring and health and safety procedures, if necessary



Tables

Table 1
Former AFP-51
Sampling Plan

Field Analysis/Sampling				Pre-Injection		During Injection	
Parameter	Containers per Sample (#, size, & type)	Preservation	Method	Matrix	Locations	# of Sample Locations	# of Sample Locations
VOCS, CO, O ₂ , LEL, H ₂ SO ₄	Well Headspace	NA	PID and 5 Gas Meter	Air/Vapor	HypeAir Inj Area (SVE-1, SVE-2, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-18)	9	9
Dissolved Oxygen, pH, ORP, Temperature, Conductivity.	Downwell	NA	YSI 556 or equivalent	Groundwater	Permanganate Inj Area (MP-1, MP-2, MW1-6, MW1-7, MW1-18, MW1-22)	6	6
Dissolved Iron	Grab Sample	NA	Hach DR2800 spectrophotometer, EPA FerroVer Method #8008	Groundwater	HypeAir Inj Area (SVE-1, SVE-2, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-18)	9	9
Hydrogen Peroxide	Grab Sample	NA	Lamotte Insta-test Strip	Groundwater	SVE-1, SVE-2, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-18	9	9
Permanganate	Grab Sample	NA	Hach Pocket Colorimeter II Manganese HR System/USEPA Periodate Oxidation Method #8034	Groundwater	MP-1, MP-2, MW1-6, MW1-7, MW1-18, MW1-22	6	6
Ozone	Breathing Zone	NA	Field Meter	Air/Vapor	NA	0	NA
VOCS	Breathing Zone	NA	Photo Ionization Detector	Air/Vapor	During Drilling activities in immediate work area where well is being installed	NA	NA
Dust/particulates	Breathing Zone	NA	Personal Particulate Meter	Air/Vapor	During Drilling activities in immediate work area where well is being installed	NA	0

Groundwater Sampling

Groundwater Sampling				Pre-Injection		During Injection	
Parameter	Containers per Sample (#, size, & type)	Preservation	Method	Matrix	Locations	# of Sample Locations	# of Sample Locations
TCL VOCs	3, 40ml VOA vials	HCL, 4°C	EPA 8260B	Groundwater	MP-1, MP-2, SVE-1, SVE-2	4	0
Alkalinity	2, 250 mL, plastic	None	EPA Method 310.1	Groundwater	MP-1, MP-2, SVE-1, SVE-2	4	0
TAL Metals (Total)	1, 250 mL, plastic	Nitric Acid	EPA Method 6010B	Groundwater	MP-1, MP-2, SVE-1, SVE-2	4	0
TAL Metals (Dissolved)	1, 250 mL, plastic	None, Filtered at Lab	EPA Method 6010B	Groundwater	MP-1, MP-2, SVE-1, SVE-2	4	0

Soil Sampling

Soil Sampling				Pre-Injection		During Injection	
Parameter	Containers per Sample (#, size, & type)	Preservation	Method	Matrix	Locations	# of Sample Locations	# of Sample Locations
TCL VOCs	50 grams, glass jar	4°C	EPA 8260B	Soil	MP-1, MP-2, SVE-1, SVE-2, IP-1, IP-2, IP-3, IP-4	8	0
TAL Metals	50 grams, glass jar	None	EPA Method 6010.B	Soil	MP-1, MP-2, SVE-1, SVE-2, IP-1, IP-2, IP-3, IP-4	8	0
Total Organic Carbon	50 grams, glass jar	None	SW846 Method 9060M	Soil	MP-1, MP-2, SVE-1, SVE-2, IP-1, IP-2, IP-3, IP-4	8	0

Table 1
Former AFP-51
Sampling Plan

Field Analysis/Sampling				1 Week Post Injection		6 Week Post Injection	
Parameter	Containers per Sample (#, size, & type)	Preservation	Method	Matrix	Locations	Locations	# of Sample Locations
VOCS, CO, O ₂ , LEL, H ₂ SO ₄	Well Headspace	NA	PID and 5 Gas Meter	Air/Vapor	HypeAir Inj Area (SVE-1, SVE-2, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-18)	HypeAir Inj Area (SVE-1, SVE-2, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-18)	9
Dissolved Oxygen, pH, ORP, Temperature, Conductivity.	Downwell	NA	YSI 556 or equivalent	Groundwater	Permanganate Inj Area (MP-1, MP-2, MW1-6, MW1-7, MW1-18, MW1-22)	Permanganate Inj Area (MP-1, MP-2, MW1-6, MW1-7, MW1-18, MW1-22)	6
Dissolved Iron	Grab Sample	NA	Hach DR2800 spectrophotometer, EPA FerroVer Method #8008	Groundwater	HypeAir Inj Area (SVE-1, SVE-2, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-18)	HypeAir Inj Area (SVE-1, SVE-2, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-18)	9
Hydrogen Peroxide	Grab Sample	NA	Lamotte Insta-test Strip	Groundwater	SVE-1, SVE-2, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-18	SVE-1, SVE-2, MW1-8, MW1-10, MW1-11, MW1-12, MW1-13, MW1-14, MW1-18	9
Permanganate	Grab Sample	NA	Hach Pocket Colorimeter II Manganese HR System/USEPA Periodate Oxidation Method #8034	Groundwater	MP-1, MP-2, MW1-6, MW1-7, MW1-18, MW1-22	MP-1, MP-2, MW1-6, MW1-7, MW1-18, MW1-22	6
Ozone	Breathing Zone	NA	Field Meter	Air/Vapor	NA	NA	0
VOCS	Breathing Zone	NA	Photo Ionization Detector	Air/Vapor	NA	NA	0
Dust/particulates	Breathing Zone	NA	Personal Particulate Meter	Air/Vapor	NA	NA	0

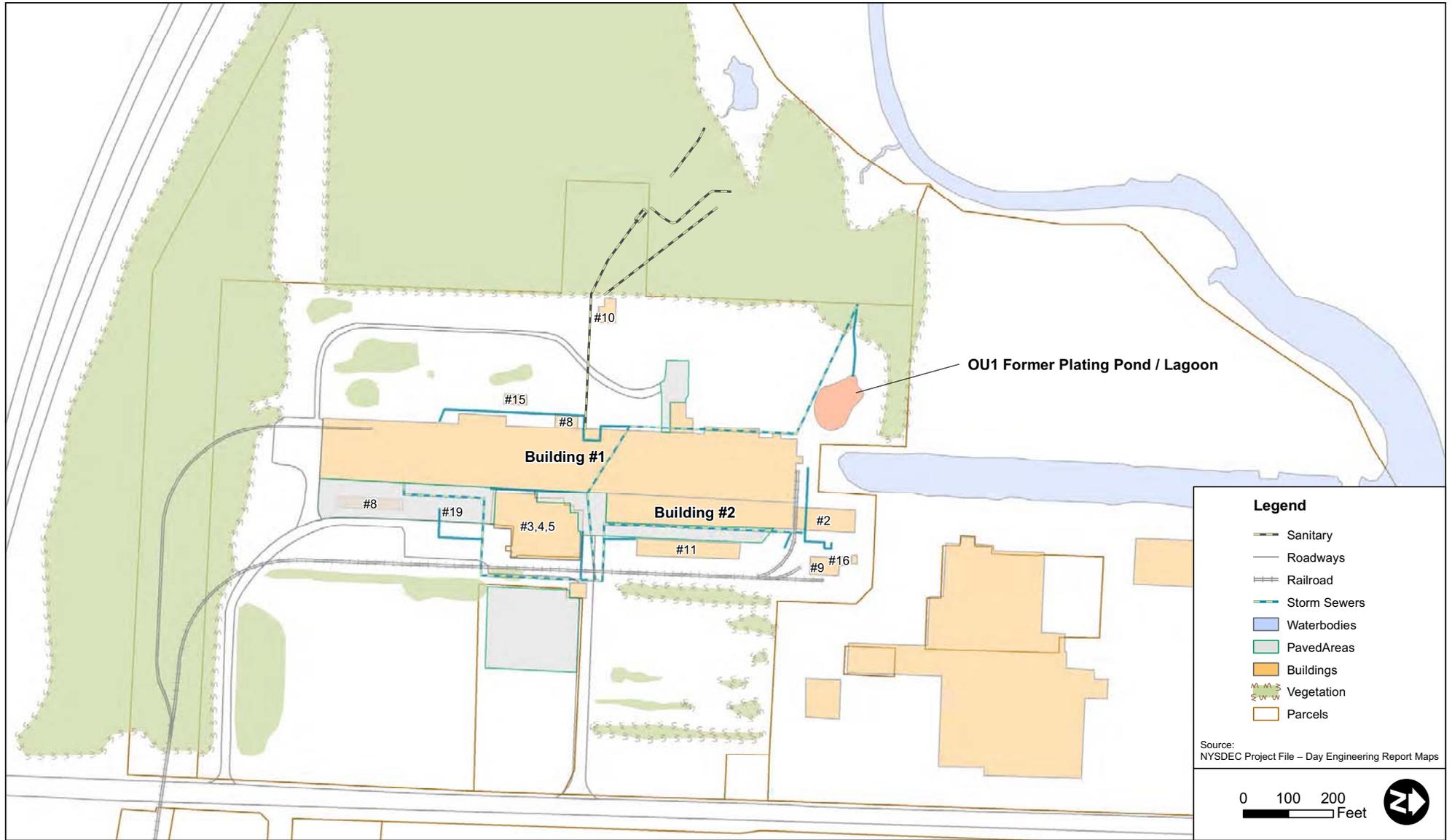
Groundwater Sampling

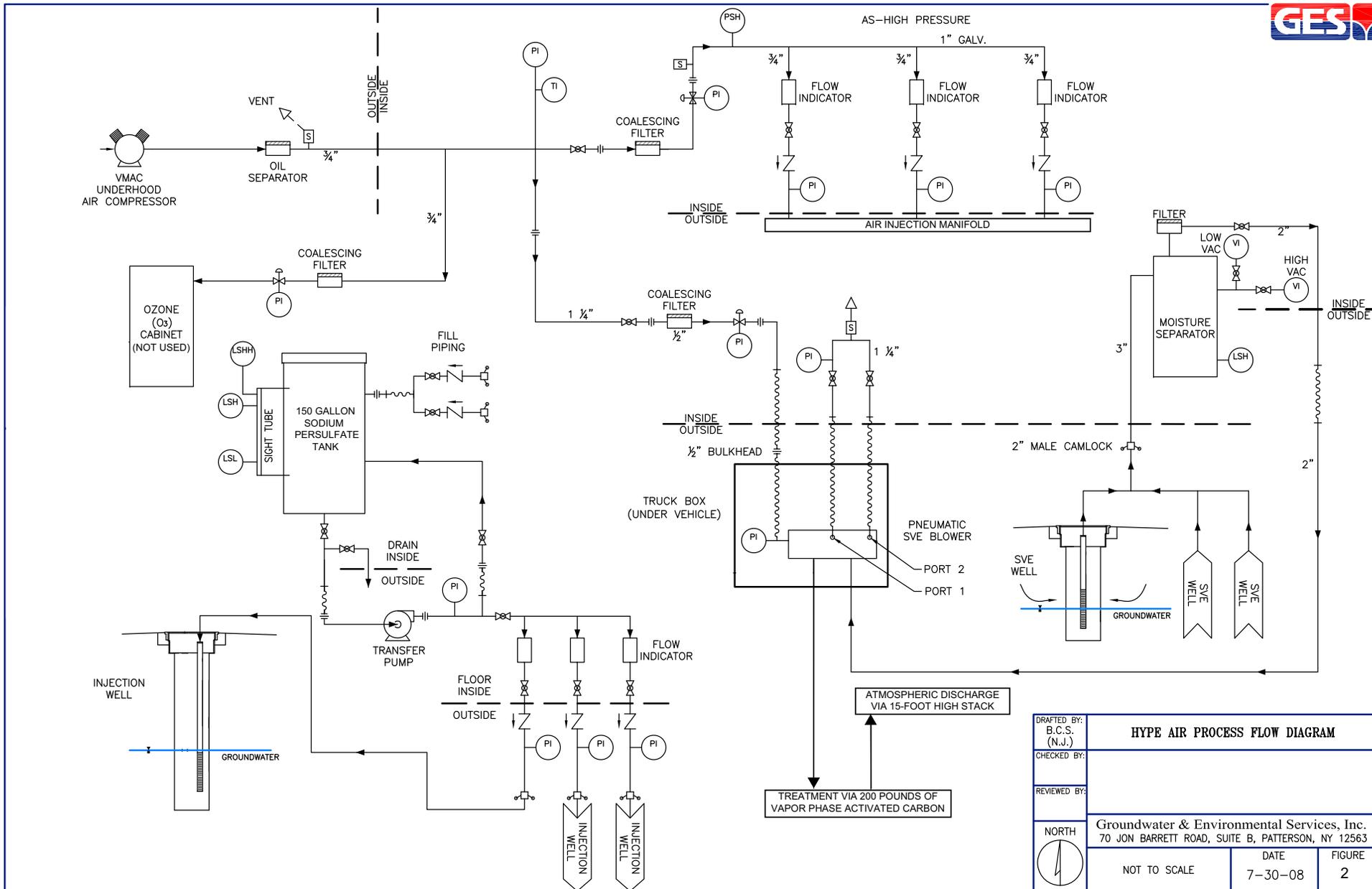
Parameter	Containers per Sample (#, size, & type)	Preservation	Method	Matrix	1 Week Post Injection		6 Week Post Injection	
					Locations	# of Sample Locations	Locations	# of Sample Locations
TCL VOCs	3, 40ml VOA vials	HCL, 4°C	EPA 8260B	Groundwater	None	0	MP-1, MP-2, SVE-1, SVE-2	4
Alkalinity	2, 250 mL, plastic	None	EPA Method 310.1	Groundwater	None	0	None	0
TAL Metals (Total)	1, 250 mL, plastic	Nitric Acid	EPA Method 6010B	Groundwater	None	0	MP-1, MP-2, SVE-1, SVE-2	4
TAL Metals (Dissolved)	1, 250 mL, plastic	None, Filtered at Lab	EPA Method 6010B	Groundwater	None	0	MP-1, MP-2, SVE-1, SVE-2	4

Soil Sampling

Parameter	Containers per Sample (#, size, & type)	Preservation	Method	Matrix	1 Week Post Injection		6 Week Post Injection	
					Locations	# of Sample Locations	Locations	# of Sample Locations
TCL VOCs	50 grams, glass jar	4°C	EPA 8260B	Soil	None	0	TBD	4
TAL Metals	50 grams, glass jar	None	EPA Method 6010.B	Soil	None	0	TBD	4
Total Organic Carbon	50 grams, glass jar	None	SW846 Method 9060M	Soil	None	0	None	0

Figures

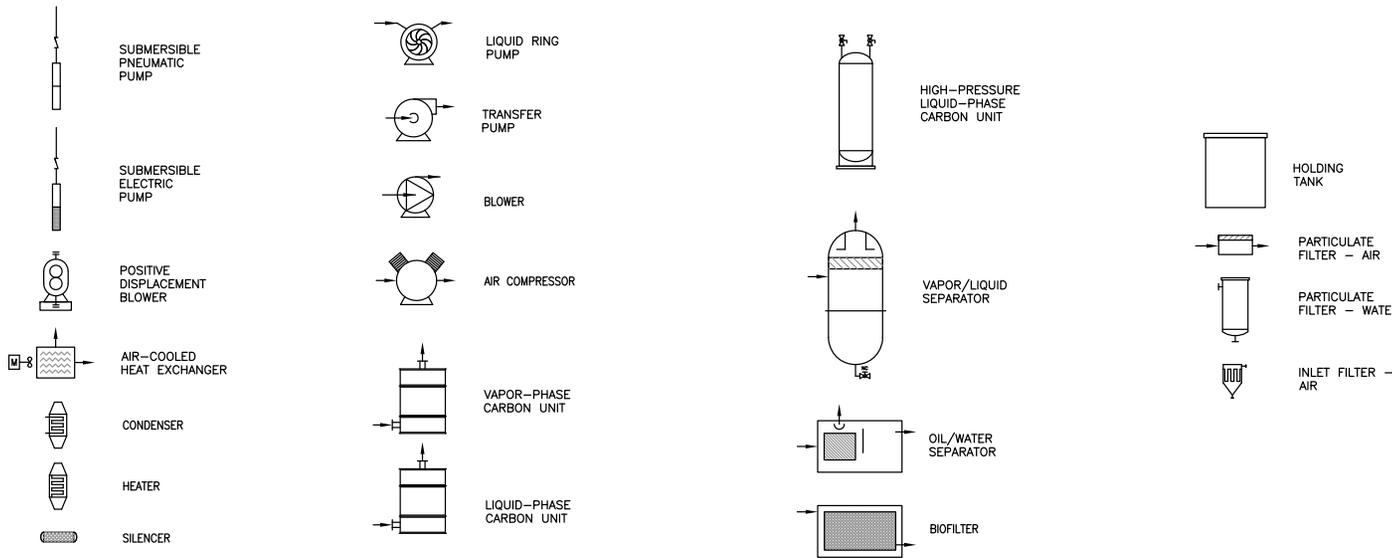




DRAFTED BY: B.C.S. (N.J.)	HYPE AIR PROCESS FLOW DIAGRAM	
CHECKED BY:		
REVIEWED BY:		
	Groundwater & Environmental Services, Inc. 70 JON BARRETT ROAD, SUITE B, PATTERSON, NY 12563	
	NOT TO SCALE	DATE 7-30-08



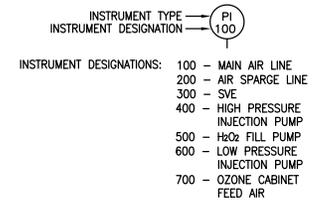
EQUIPMENT



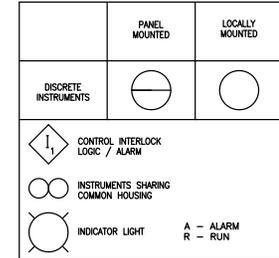
INSTRUMENTATION ABBREVIATIONS

FI	FLOW INDICATOR	PDS	PRESSURE DIFFERENTIAL SWITCH
FQ	FLOW TOTALIZER	PI	PRESSURE INDICATOR
FQI	FLOW INDICATING TOTALIZER	PSH	PRESSURE SWITCH HIGH
FSH	FLOW SWITCH HIGH	PSL	PRESSURE SWITCH LOW
FSL	FLOW SWITCH LOW	SP	SAMPLE POINT
LSLL	LEVEL SWITCH LOW LOW	TI	TEMPERATURE INDICATOR
LSL	LEVEL SWITCH LOW	TS	TEMPERATURE SENSOR
LSH	LEVEL SWITCH HIGH	TSH	TEMPERATURE SWITCH HIGH
LSHH	LEVEL SWITCH HIGH HIGH	TSL	TEMPERATURE SWITCH LOW
		VI	VACUUM INDICATOR

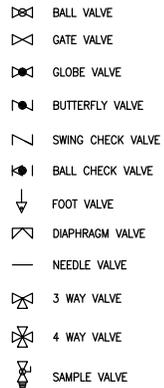
INSTRUMENTATION LABELING



GENERAL INSTRUMENTATION OR FUNCTION SYMBOLS



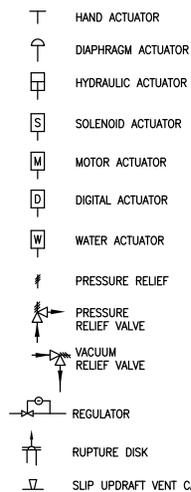
VALVES



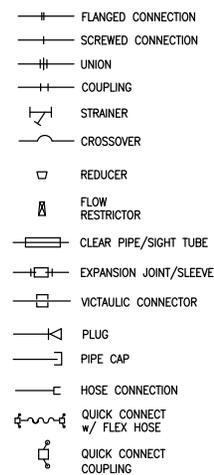
VALVE ABBREVIATIONS

N.C. - NORMALLY CLOSED
N.O. - NORMALLY OPENED
MAN - MANUAL

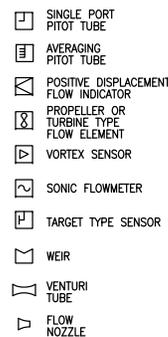
ACTUATORS / REGULATORS



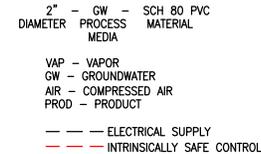
FITTINGS & PIPING



FLOW ELEMENTS



PIPE DESIGNATION



PIPING MATERIAL ABBREVIATIONS

CPVC	CHLORINATED POLYVINYL CHLORIDE
CS	CARBON STEEL PIPE
COP	COPPER
CM	CORRUGATED METAL PIPE
CI	CAST IRON PIPE
DI	DUCTILE IRON PIPE
GAL	GALVANIZED STEEL PIPE
POLY	POLYETHYLENE PIPE
PP	POLYPROPYLENE PIPE
PVC	POLYVINYL CHLORIDE PIPE
RC	REINFORCED CONCRETE PIPE
HOSE	RUBBER OR PLASTIC HOSE
SS	STAINLESS STEEL PIPE
VC	VITRIFIED CLAY PIPE

DRAFTED BY:
B.C.S.
(N.J.)

CHECKED BY:

REVIEWED BY:

PIPING & INSTRUMENTATION DIAGRAM LEGEND

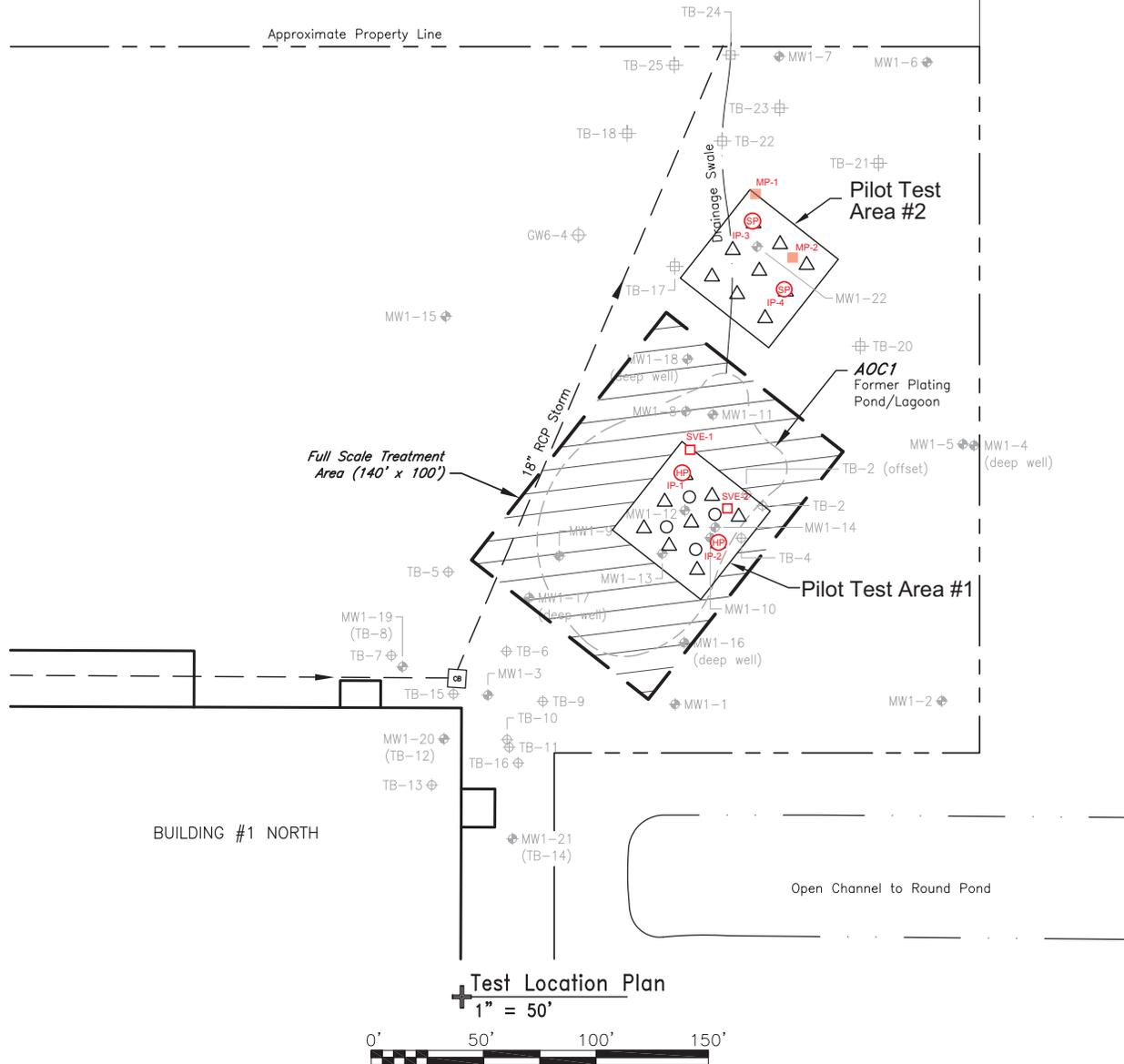
Groundwater & Environmental Services, Inc. 70 JON BARRETT ROAD, SUITE B, PATTERSON, NY 12563		
NOT TO SCALE	DATE 7-30-08	FIGURE 3

Ref1:
Ref2:
Ref3:

Pen Setting File: 800psFullcolor.ctb

Xerox432AnsIB-2; 11 x 17
Layout Name: OUI-PTP-E

Time Plotted: Tues Feb 8 12:50, 2008
File Name: Atkin_4800 Dewey_2806\Pilot_Layout.dwg



NOTES:

1. Monitoring Well MW1-1 thru MW1-10 are 2" PVC wells installed December 2002.
2. Monitoring Wells MW1-11 thru MW1-14 are 4" Stainless Steel wells installed July 2003.
3. Monitoring Well MW1-15 thru MW1-21 are 2" PVC wells installed March 2004.
4. Monitoring Well MW1-22 and Test Borings TB-17 thru TB-25 installed June 2007.
5. Drawing prepared from Figure GEN-2 in the general work plan dated June 2002 and site observations by representatives of Day Environmental, Inc. from December 2002 through April 2004.

LEGEND:

- ⊕ GW6-4 Well Location identified from a report titled "Former Air Force Plant No. 51, HTRW Investigation, Greece, NY" by Ogden Environmental And Energy Services Co., Inc, dated April 2000
- AOC1** Area Of Concern with Numbered Designation
- ⊕ MW1-1 Monitoring Well with Designation
- ⊕ TB-2 Test Boring with Designation
- △ Pilot Test Injection Well
- Pilot Test Vent Well
- ⊕ HP Proposed HypeAir Injection well
- ⊕ SP Proposed Sodium Permanganate Injection well
- Proposed Soil Vapor Extraction/Monitoring well
- Proposed Monitoring well

FIGURE 4
PROPOSED PILOT TEST WELL LOCATIONS

FIELD VERIFIED BY	JAD
DATE	01-2008
DRAWN BY	RJM
DATE DRAWN	02-21-2008
SCALE	1" = 50'
DATE ISSUED	03-04-2008

day
DAY ENVIRONMENTAL, INC.
ENVIRONMENTAL CONSULTANTS
ROCHESTER, NEW YORK 14614-1008
NEW YORK, NEW YORK 10165-1617

PROJECT TITLE
**4777 DEWEY AVENUE
GREECE, NEW YORK**

PROJECT NO.
2806S-01

DRAWING TITLE
OUI PILOT TEST PHASE REMEDIAL DESIGN PLAN

DRAWING TITLE
Tentative Pilot In-Situ Chemical Oxidation Treatment Areas

PROJECT NO.
2806S-01

PTP-E



Appendix A

Containment Berm Specifications



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DRUM FUNNELS

CONTAINER PRODUCTS

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GAS CYLINDER HANDLING

RADWASTE & DOE



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Ultra-Containment Berms®, Stake Wall Model

Portable Secondary Containment For Drums, Tanks, Equipment And Vehicles



- Stake Wall Model Containment Berms feature a unique design which allows the sidewalls to collapse in either direction and spring back automatically to their upright position! Roll drums over the sidewalls, drive trucks through them, and the sidewalls always return to vertical without assistance. [See how the sidewalls work](#)
- No set-up required - unfold and the sidewalls automatically spring into their vertical position.
- Rugged Construction - made of XR5, a polymer-coated fabric; offers excellent chemical resistance and durability. Polyurethane and other materials also available.
- Vertical sidewall supports located every 12". Each Support features a built-in "living hinge" at its base, which allows sidewalls to collapse in either direction. Collapsed sidewalls spring back automatically.



ULTRA-CONTAINMENT BERM, STAKE WALL MODEL			
Part Number	Description	Weight	Containment Capacity
8308	4' x 6' x 1'	44 lbs.	179 gallons
8309	6' x 6' x 1'	50 lbs.	269 gallons
8310	10' x 10' x 1'	100 lbs.	748 gallons
8337	15' x 50' x 1'	438 lbs.	5,610 gallons
8332	15' x 66' x 1'	563 lbs.	7,405 gallons
Options: Ground Tarps, Track Belts, Protective Storage Bags, Self-Bailer .			
Compliance: 40 CFR 264.175 and SPCC			
Chemical Compatibility Guide			
Material Specifications			
Frequently Asked Questions (FAQs)			
Download catalog page			
U.S. Patent No. 5,924,461			

To be used with the hydrogen peroxide totes

To be used with the sodium permanganate tanker truck

More photos:



[Camo Containment Berm](#) | [Collapsible Wall Containment Berm](#) | [Compact Containment Berm](#) | [Foam Wall Containment Berm](#) | [RapidRise Containment Berm](#) | [Stake Wall Containment Berm](#) | [Ultimate Containment Berm](#)





Appendix B

Description of EPA FerroVer Method #8008

Iron, Total

DOC316.53.01053

USEPA¹ FerroVer[®] Method²

Method 8008

0.02 to 3.00 mg/L

Powder Pillows or AccuVac[®] Ampuls**Scope and Application:** For water, wastewater and seawater; digestion is required for determining total iron¹ USEPA approved for reporting wastewater analysis, *Federal Register*, June 27, 1980; 45 (126:43459)² Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test preparation

How to use instrument-specific information

The [Instrument-specific information](#) table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

Before starting the test:

Digestion is required for determining total iron for EPA reporting purposes. Use the mild or vigorous digestion. Refer to the *Water Analysis Guide* for more information.

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. See the user manual for more information.

Adjust pH of stored samples before analysis.

For turbid samples, treat the blank with one 0.1-g scoop of RoVer Rust Remover. Swirl to mix.

Collect the following items:

Description	Quantity
Powder Pillow Test:	
FerroVer [®] Iron Reagent Powder Pillow	1
Sample Cells (see Instrument-specific information)	2

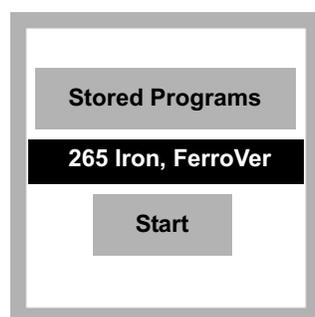
Iron, Total

Collect the following items: (continued)

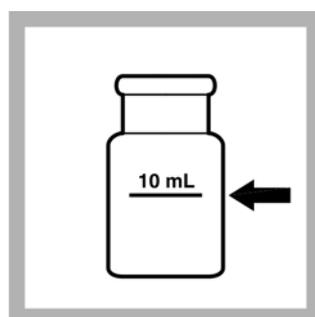
Description	Quantity
AccuVac® Ampul test:	
FerroVer® Iron Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample Cells (see Instrument-specific information)	1
Stopper of 18 mm tubes	1

See [Consumables and replacement items](#) for reorder information.

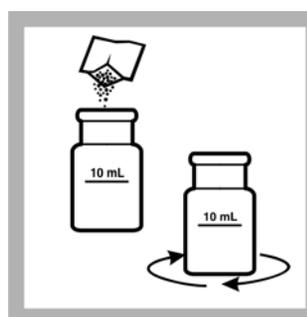
FerroVer method for powder pillows



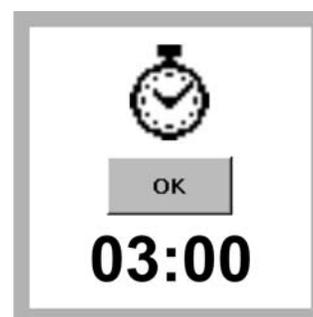
1. Select the test.
Insert an adapter if required (see [Instrument-specific information](#)).
Refer to the user manual for orientation.



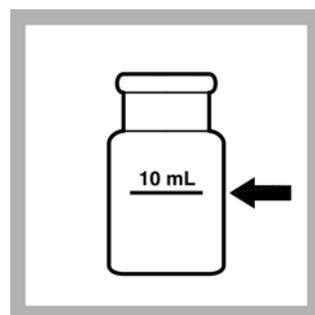
2. Prepared sample: Fill a clean sample cell with 10 mL of sample



3. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell. Swirl to mix.
Accuracy is not affected by undissolved powder.



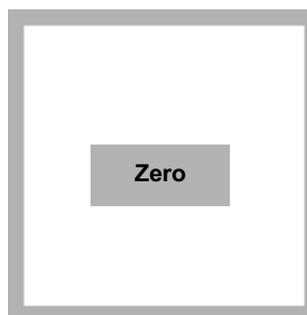
4. Start the instrument timer.
A three-minute reaction period will begin. An orange color will form, if iron is present.
(Allow samples that contain rust to react for at least 5 minutes.)



5. Blank preparation: Fill a second sample cell with 10 mL of sample.



6. When the timer expires, insert the blank into the cell holder.

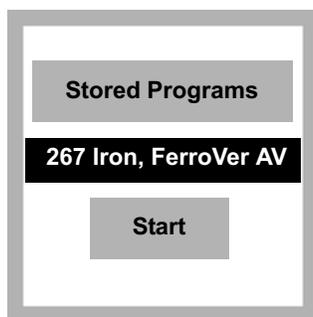


7. ZERO the instrument. The display will show:
0.00 mg/L Fe

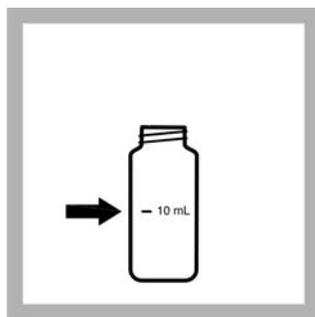


8. Insert the prepared sample into the cell holder.
READ the results in mg/L Fe.

FerroVer method for AccuVac® Ampuls



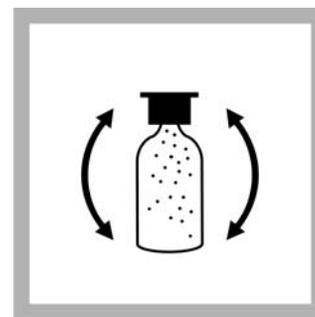
1. Select the test.
Insert an adapter if required (see [Instrument-specific information](#)).
Refer to the user manual for orientation.



2. **Blank Preparation:**
Fill a round sample cell with 10 mL of sample.



3. **Prepared Sample:**
Collect at least 40 mL of sample in a 50-mL beaker. Fill a FerroVer Iron AccuVac® Ampul with sample from the beaker. Keep the tip immersed while the Ampul fills completely.



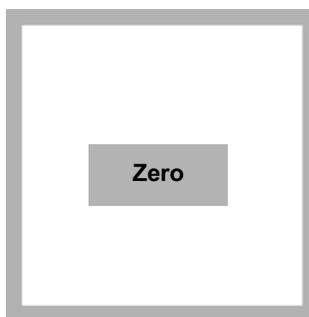
4. Quickly invert the Ampul several times to mix.
Accuracy is not affected by undissolved powder.



5. Start the instrument timer.
A three-minute reaction period will begin. An orange color will develop if iron is present.



6. Wipe the blank and insert it into the cell holder.



7. **ZERO** the instrument.
The display will show:
0.00 mg/L Fe



8. Wipe the Ampul and insert it into the cell holder.
READ the results in mg/L Fe.

Interferences

Table 2 Interfering substances

Interfering substance	Interference level
Calcium, Ca ²⁺	No effect at less than 10,000 mg/L as CaCO ₃ .
Chloride, Cl ⁻	No effect at less than 185,000 mg/L.
Copper, Cu ²⁺	No effect. Masking agent is contained in FerroVer Reagent.
High Iron Levels	Inhibit color development. Dilute sample and re-test to verify results.
Iron Oxide	Requires mild, vigorous or Digesdahl digestion. After digestion, adjust sample to pH 3–5 with sodium hydroxide, then analyze.
Magnesium	No effect at 100,000 mg/L as calcium carbonate.

Table 2 Interfering substances (continued)

Interfering substance	Interference level
Molybdate Molybdenum	No effect at 50 mg/L as Mo.
High Sulfide Levels, S ²⁻	<ol style="list-style-type: none"> 1. Treat in fume hood or well-ventilated area. Add 5 mL hydrochloric acid¹, ACS to 100 mL sample in a 250 mL Erlenmeyer flask. Boil 20 minutes. 2. Cool. Adjust pH to 3–5 with Sodium Hydroxide¹. Readjust volume to 100 mL with deionized water. 3. Analyze using <i>FerroVer method for powder pillows</i> or <i>FerroVer method for AccuVac® Ampuls</i>.
Turbidity	<ol style="list-style-type: none"> 1. Add 0.1 g scoop of RoVer® Rust Remover to the blank. Swirl to mix. 2. Zero the instrument with this blank. 3. If sample remains turbid, add three 0.2 g scoops of RoVer to a 75 mL sample. Let stand 5 minutes. 4. Filter through a Glass Membrane Filter and Filter Holder¹. 5. Use the filtered sample as the prepared sample and the blank.
Extreme Sample pH	Adjust pH to 3–5.
Highly Buffered Samples	Adjust pH to 3–5.

¹ See *Optional reagents and apparatus*.

Sample collection, preservation and storage

- Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately.
- To preserve samples, adjust the pH to 2 or less with concentrated nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature.
- Before analysis, adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution.
- Correct the test result for volume additions.
- If only dissolved iron is to be determined, filter the sample before acid addition.

Accuracy check

Standard additions method (sample spike)

Required for accuracy check:

- Iron Voluette® Ampule Standard, 25 mg/L
 - Ampule breaker
 - TenSette Pipet and Pipet Tips
1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
 2. Select Options>More>Standard Additions from the instrument menu.
 3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
 4. Open the standard solution ampule.
 5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to 10 mL of unspiked sample. Start the instrument timer. After the timer expires, read the result.

6. Prepare a 0.2 mL sample spike by adding 0.1 mL of standard to the 0.1 mL sample spike. Start the instrument timer. After the timer expires, read the result.
7. Prepare a 0.3 mL sample spike by adding 0.1 mL of standard to the 0.2 mL sample spike. Start the instrument timer. After the timer expires, read the result.
8. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard additions method for AccuVac Ampuls (sample spike)

1. Fill three mixing cylinders each with 50 mL of sample and spike with 0.2 mL, 0.4 mL and 0.6 mL of standard. Stopper and invert to mix.
2. Transfer 40 mL from each of the three mixing cylinders to three 50 mL beakers.
3. Analyze each standard addition sample as described in the [FerroVer method for AccuVac® Ampuls](#).
4. Accept each standard additions reading. Each addition should reflect approximately 100% recovery.

Standard solution method

Note: Refer to the instrument user manual for specific software navigation instructions.

Required for accuracy check:

- Iron Standard Solution, 100 mg/L
 - 100-mL volumetric flask
 - Class A volumetric pipet, 2 mL
 - Deionized water
 - Pipet filler
1. Prepare a 2.00-mg/L Fe standard solution as follows:
 - a. Pipet 2.00 mL of Iron Standard Solution, 100 mg/L, into a 100 mL volumetric flask.
 - b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
 2. Use the 2.00 mg/L Fe standard solution in place of the sample. Follow the [FerroVer method for powder pillows](#) test procedure.
 3. To adjust the calibration curve using the reading obtained with the Standard Solution, select Options>More>Standard Adjust from the instrument menu.
 4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value. Mixed-parameter standards are also available to simulate various matrices.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
265	2.00 mg/L Fe	1.99–2.01 mg/L Fe	0.021 mg/L Fe
267	2.00 mg/L Fe	1.98–2.02 mg/L Fe	0.023 mg/L Fe

Summary of method

FerroVer Iron Reagent converts all soluble iron and most insoluble forms of iron in the sample to soluble ferrous iron. The ferrous iron reacts with the 1-10 phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration. Test results are measured at 510 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
FerroVer® Iron Reagent Powder Pillows (for 10-mL sample)	1	100/pkg	2105769
OR			
FerroVer® Iron Reagent AccuVac® Ampuls	1	25/pkg	2507025

Required apparatus

Description	Quantity	Unit	Catalog number
Beaker, 50 mL	1	each	50041H
Sample cell, 10 mL round, 25 x 54 mm	1	each	2122800
Sample cell, 10 mL round, 25 x 60 mm	1	6/pkg	2427606
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402
Stopper for 18 mm tube	1	6/pkg	173106

Recommended standards

Description	Unit	Catalog number
Iron Standard Solution, 100 mg/L	100 mL	1417542
Iron Standard Solution, 10 mL Voluette® Ampule, 25 mg/L as Fe	16/pkg	1425310
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	2833749
Metals Drinking Water Standard, HR for Cu, Fe, Mn	500 mL	2833649
Water, deionized	4 L	27256
Pipet, TenSette, 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Flask, volumetric, Class A, 100 mL	each	1457442
Pipet, volumetric, Class A, 2.00 mL	each	1451536
Pipet Filler, safety bulb	each	1465100

Optional reagents and apparatus

Description	Unit	Catalog number
Beaker, 50 mL	each	50041H
Cylinder, mixing, 50 mL	each	189641
Hydrochloric Acid, concentrated	500 mL	13449
Nitric Acid, concentrated	500 mL	15249

Optional reagents and apparatus (continued)

Description	Unit	Catalog number
Sodium Hydroxide Standard Solution, 5.0 N	100 mL	245032
Glass Membrane Filter, 47 mm	100/pkg	253000
Glass Membrane Filter Holder	each	234000
RoVer Rust Remover	454 g	30001
Spoon, measuring, 0.1 g	each	51100



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Appendix C

Description of USEPA periodate oxidation method #8034

USEPA¹ Periodate Oxidation Method²

Method 8034

HR (0.1 to 20.0 mg/L)

Powder Pillows

Scope and Application: For soluble manganese in water and wastewater¹ USEPA Approved for reporting wastewater analyses (digestion required). *Federal Register*, 44(116)34 193 (June 14, 1979)² Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test preparation

How to use instrument-specific information

The [Instrument-specific information](#) table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Sample cell	Cell orientation
DR 5000	2495402	Fill line faces user
DR 3900	2495402	Fill line faces user
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right

Before starting the test:

Digestion is required for reporting wastewater analyses.

If only dissolved manganese is to be determined, filter the sample before acid addition.

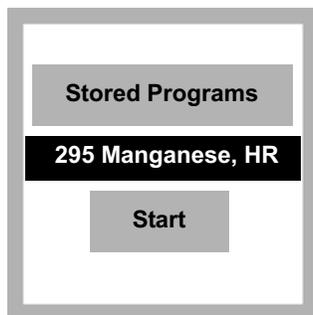
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

Collect the following items:

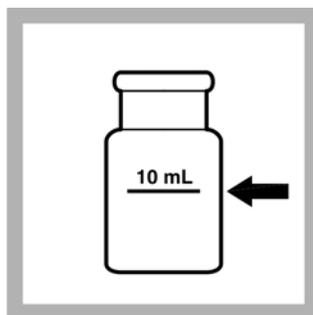
Description	Quantity
High Range Manganese Reagent Set	1
Sample Cells (see Instrument-specific information)	2

See [Consumables and replacement items](#) for reorder information.

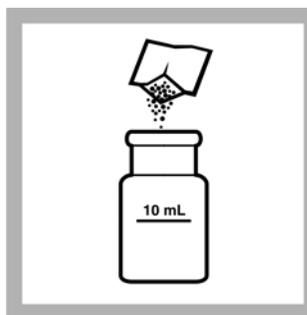
Periodate Oxidation method for powder pillows



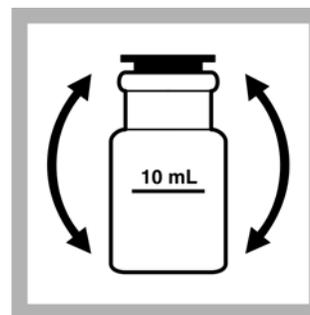
1. Select the test.
Insert an adapter if required (see [Instrument-specific information](#)).



2. **Prepared Sample:**
Fill a sample cell with 10 mL of sample.



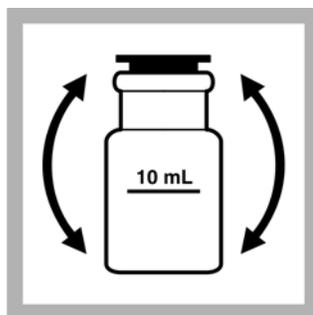
3. Add the contents of one Buffer Powder Pillow, Citrate Type for Manganese.



4. Stopper or cap and invert to mix.



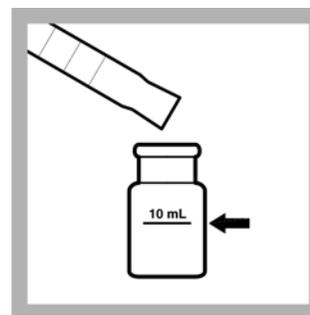
5. Add the contents of one Sodium Periodate Powder Pillow to the sample cell.



6. Stopper or cap and invert to mix.
A violet color will develop if manganese is present.



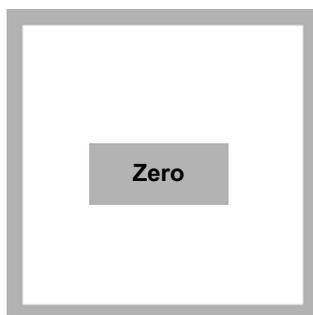
7. Start the instrument timer.
A two-minute reaction time will begin.



8. **Blank Preparation:**
Fill a second sample cell with 10 mL of sample.



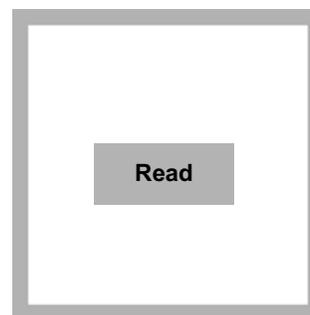
9. When the timer expires, insert the blank into the cell holder.



10. **ZERO** the instrument.
The display will show:
0.0 mg/L Mn



11. Within eight minutes after the timer expires, insert the sample into the cell holder



12. **READ** the results in mg/L Mn.

Interferences

Table 2 Interfering substances

Interfering substance	Interference level
Calcium	700 mg/L
Chloride	70,000 mg/L
Iron	5 mg/L
Magnesium	100,000 mg/L
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.

Sample collection, preservation and storage

- Collect samples in acid-washed plastic bottles. Do not use glass containers due to possible adsorption of Mn to glass.
- If samples are acidified, adjust the pH to 4–5 with 5.0 N Sodium Hydroxide before analysis.
- Do not exceed pH 5, as manganese may precipitate.
- Correct the test result for volume additions.

Accuracy check

Standard additions method (sample spike)

Required for accuracy check:

- Manganese Voluette® Ampule Standard, 250 mg/L Mn
 - Ampule breaker
 - TenSette Pipet
1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
 2. Select Options>More>Standard Additions from the instrument menu.
 3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
 4. Open the standard solution ampule.
 5. Use the TenSette Pipet to prepare spiked samples: add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 10-mL portions of fresh sample. Mix thoroughly.
 6. Follow the [Periodate Oxidation method for powder pillows](#) test procedure for each of the spiked samples using the powder pillows, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.
 7. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard solution method

Note: Refer to the instrument user manual for specific software navigation instructions.

Required for accuracy check:

- Manganese Standard Solution, 1000 mg/L
- Deionized water

Manganese

- 1 L Class A volumetric flask
 - Class A volumetric pipet, 10 mL
 - Pipet filler, safety bulb
1. Prepare a 10.0 mg/L manganese standard solution as follows:
 - a. Pipet 10.0 mL of Manganese Standard, 1000 mg/L, into a 1000 mL (1 liter) volumetric flask.
 - b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
 2. Use this solution in place of the sample. Follow the [Periodate Oxidation method for powder pillows](#) test procedure.
 3. To adjust the calibration curve using the reading obtained with the standard solution, select Options>More>Standard Adjust from the instrument menu.
 4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change	
			Portion of Curve	Concentration
295	10.0 mg/L Mn	9.6–10.4 mg/L Mn	Entire curve	0.1 mg/L Mn

Summary of method

Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration. Test results are measured at 525 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Manganese Reagent Set, High Range (100 tests), includes:	—	—	2430000
Buffer Powder Pillows, citrate type for Manganese	1	100/pkg	2107669
Sodium Periodate Powder Pillows for Manganese	1	100/pkg	2107769

Required apparatus

Description	Quantity	Unit	Catalog number
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402
Stopper, rubber	1	6/pkg	173106

Recommended standards

Description	Unit	Catalog number
Manganese Standard Solution, 1000 mg/L Mn	100 mL	1279142
Manganese Standard Solution, 250 mg/L Mn, 10-mL Voluette® ampule	16/pkg	1425810
Water, deionized	4 L	27256
Voluette Ampule breaker	each	2196800

Optional reagents and apparatus

Description	Unit	Catalog number
Manganese Standard Solution, 2 mL PourRite® Ampule, 25 mg/L	20/pkg	2112820
Manganese Standard Solution, 2 mL PourRite® Ampule, 10 mg/L	20/pkg	2605820
pH paper, 0–14	100/pkg	2601300
Pipe filler, safety bulb	each	1465100
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Pipet, TenSette, 1.0–10.0 mL	each	1970010
Pipet Tips, for TenSette Pipet 1970010	250/pkg	2199725
Pipet Tips, for TenSette Pipet 1970010	50/pkg	2199796
PourRite® Ampule breaker	each	2484600
Sodium Hydroxide, 5.0 N	100 mL	245032
Volumetric flask, Class A, 1000 mL	each	1457453
Volumetric pipet, Class A, 10 mL	each	1451538



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