

GUIDANCE DOCUMENT

Cost-Effective, Ultra-Sensitive Groundwater Monitoring for Site Remediation and Management: Standard Operating Procedures with QA/QC

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LIST OF ACRONYMS

bgs	below ground surface
C-18	octadecyl hydrocarbon
COC	Contaminant of Concern
ER	Environmental Restoration
EPA	Environmental Protection Agency (United States)
ESTCP	Environmental Security Technology Certification Program
ft	Feet
HLB	Hydrophilic-Lipophilic Balance
ICAL	Initial Calibration
IS2	<i>In Situ</i> Sampler
ml	Milliliter
MΩ	Mega-Ohm
NAPL	Non-Aqueous Phase Liquids
PAH	Polycyclic Aromatic Hydrocarbon
QA/QC	Quality Assurance/Quality Control
RL	Reporting Limit
SAX	Strong Anion Exchange
SDB	Styrene Divinylbenzene
SPE	Solid Phase Extraction
VOC	Volatile Organic Compound
WAX	Weak Anion Exchange

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1.0 INTRODUCTION

This document presents the standard operating procedures for the *In Situ* Sampling (IS2) Technology.

2.0 PRE-DEPLOYMENT WORK

As with any groundwater sampling method, the decision to apply the IS2 technology is based on the site characteristics and the type of data that is desired. Successful integration of the IS2 into a groundwater contamination monitoring plan requires the *a priori* development of a sampling plan to be executed in the field.

The primary product of pre-deployment work is a sampling matrix that describes the location, quantity of samples, and methods to be applied (Figure 1). A flow chart is provided in Figure 2 that provides an overview of the pre-deployment planning process. Answers to the questions asked through this process are used to populate the sampling matrix. Pre-deployment work ends with the mechanical preparation of the IS2 sampler.

Table 1. Example of a sampling matrix.

Wells	Analyte (Quantification Method)	Sorbent (Extraction Method)	Sample Type	Cartridge Quantity	Holding Time
W-31	PAHs (EPA 8310)	Oasis HLB, 6cc, 200 mg (oasis 127)	Quantification	3	14 days at 4 °C
			Breakthrough	3	
			Field Blank	1	
			Trip Blank	1	
			Method Blank	1	N/A
	Cr(VI) (EPA 218.7)	Phenomenex Strata SAX, 6cc, 200 mg (Hu & Deming, 2005)	Quantification	3	14 days at 4 °C
			Breakthrough	3	
			Field Blank	1	
			Trip Blank	1	
Method Blank			1	N/A	

2.1 PRE-DEPLOYMENT: CONFIRM APPLICABILITY

The IS2 uses solid phase extraction (SPE) and similar sorptive or partitioning-based chemistry to extract contaminants of concern (COCs) from water. This makes the IS2 capable of concentrating trace quantities of COCs from large volumes of water. It also makes the IS2 less suitable for use in wells where it may be in contact with non-aqueous phase liquids (NAPLs or "free product"), or where the chemical properties of the COC are not favorable (e.g., ethene).

Because a typical application of the IS2 uses sorptive chemistry for extraction, it is most readily applicable when the contaminants of interest are easily separated from an aqueous matrix by, and subsequently recovered from, a sorbent material.

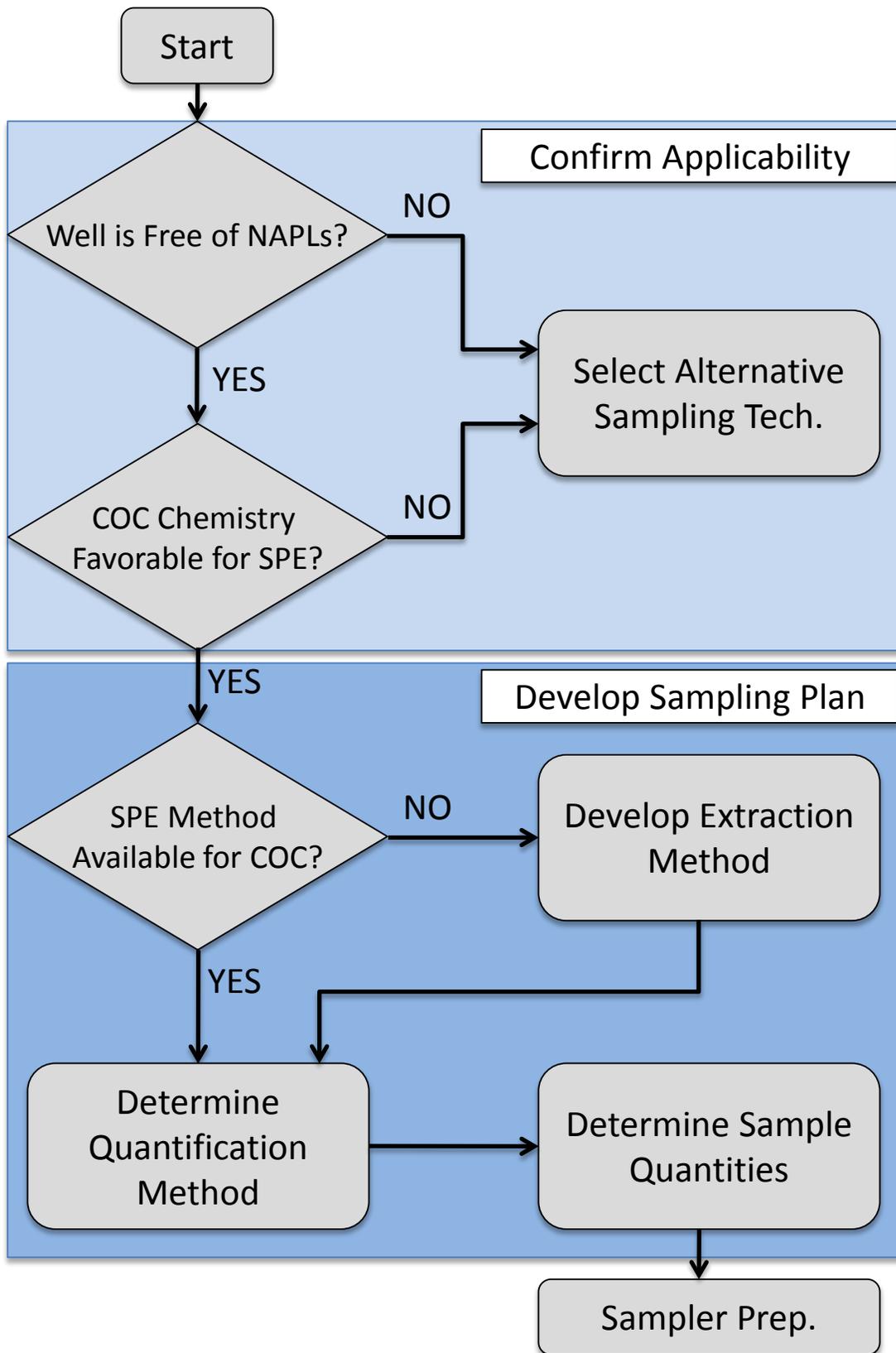


Figure 1. Pre-deployment flow chart for the IS2 technology.

The first step in pre-deployment planning is to confirm the applicability of the IS2 to the site. In confirming applicability, two questions are asked:

1. Is the well free of NAPLs?
2. Are the chemical properties of the contaminant favorable for SPE?

If the answer to both questions is affirmative, the IS2 is confirmed to be applicable to the site. The answer to the second question must be determined experimentally or according to the experience of the operator; it is often the case that contaminants which appear to have favorable properties for SPE will ultimately be difficult to capture due to the effects of commingled substances or secondary properties. This process is initiated by matching the salient features of the contaminant with a corresponding class of SPE material (Figure 3), but it must be noted that both exceptions and significant degrees of overlap may exist between these classes.

Table 2. Examples of contaminant properties with suggestions for SPE material.

Contaminant Property	Suggested SPE Material
Non-polar, organic	C-18
Non-polar, organic, aromatic	Styrene Divinylbenzene (SDB)
Organic Acids	Weak Anion Exchange (WAX)
Dissolved Metal Cations	Cation Exchange
Dissolved Metal Anions	Strong Anion Exchange (SAX)

2.2 PRE-DEPLOYMENT: DEVELOP SAMPLING PLAN

After confirming the applicability of the IS2 to the site, the next question is asked:

3. Is an SPE method available for the COC?

The speed with which IS2 Technology can be deployed to the field is greatly enhanced by the availability of well-defined extraction protocols for the contaminants of interest. Manufacturers of SPE cartridges maintain databases of application notes and methods for their products, which users of the IS2 are expected to leverage. If no methods are available from manufacturers or literature, it is up to the user to determine whether or not development of a method is worthwhile.

The Sampling Matrix (Figure 1) is now populated with the following data:

2.2.1 Wells

The wells to be sampled must meet the minimum dimensions for the IS2 sampler being deployed and should be in or outside of the dissolved plume, but free of NAPL.

2.2.2 Analyte and Quantification Method

The quantification method to be applied to each contaminant is recorded.

2.2.3 Sorbent and Extraction Method

The type of sorbent cartridge and the extraction method are recorded. SPE cartridges are described by the type of sorbent they contain, the mass of the sorbent, and the total volume of the cartridge. The pore size of the sorbent bed is frequently given.

2.2.4 Sample Type

The execution of the sampling plan returns quantification data for the COCs and four types of quality control data, which are discussed in Section 4.0: Data Types and Quality Control.

2.2.5 Sample Quantity

The IS2 sampler can be configured to generate a batch of replicate samples in parallel. For quality control, one Field Blank and one Trip Blank should be included with each batch of Quantification Samples loaded into the IS2 sampler.

2.2.6 Holding Time

Established holding time requirements for sample handling should be recorded for each COC. If no holding time is known, spiked quantification samples should be used to establish that a working period of at least 14 days.

2.3 PRE-DEPLOYMENT: GENERATE DOCUMENTATION

All sorbent cartridges prepared for the execution of the sampling plan must be labeled with a unique, consecutive number and a sample batch number.

A sample chain-of-custody form (Appendix A) must be generated in which each sample receives a line on which the persons responsible for the sample must record the following:

- The type of cartridge
- The date and time of preparation
- The date and time of loading into the IS2
- The date, time, and volume of sample processed
- The date and time of recovery
- The date and time of processing for quantification
- The name or initials of the person performing each of the above steps
- The date and time of any handoff of the samples between personnel, and the name or initials of the persons taking charge of the samples

2.4 PRE-DEPLOYMENT: SAMPLER PREPARATION

Preparation of the IS2 sampler includes decontamination, configuration and mode selection, calibration, and loading of the sorbent cartridges. Two pumps have been developed for use with

the IS2 sampler: a peristaltic pump and a syringe pump. Each pump has its own preparation requirements.

2.4.1 Decontaminating the Peristaltic Pump

Any parts of the sampler that make contact with the fluid stream and are intended for repeated use (e.g., Luer fittings) must be decontaminated by washing with water and a surfactant, rinsed with a clean solvent, and dried.

The process for decontaminating the peristaltic pump is as follows:

1. Remove pump cartridges from pump. Dispose of used pump tubing. Note that there are right-handed and left-handed cartridges.
2. Reset pump cartridge tension screw so that the head of the screw is flush with the surface of the cartridge.
3. Replace pump tubing, to prevent contamination between sampling events. Pump tubing is selected for chemical compatibility with contaminants and sized to enable desired flow-rate range. Attach clean Luer fittings on ends of pump tubing.
4. Program the tubing inner diameter setting on the pump controller to match the selected pump tubing.
5. Starting from the top of the pump and working around it, attach all of the right-handed cartridges, followed by all of the left-handed cartridges. Each of the three sides of the pump can accommodate a right- and left-handed cartridge.
6. Test pump operation at a low flow-rate (e.g., 100 ug/min). The cartridges should not flex when the pump rollers rotate. If flexure is observed, tighten the tension screw in half-turn increments until it stops. If flexure control requires significant tightening of the tension screw, the cartridges may be unacceptably worn and may be impossible to calibrate. Replace worn cartridges.
7. Starting at the top and working around and then down, attach the pump tubing inlets to the intake manifold. Cap unused manifold ports with Luer caps. Note the direction of the pump roller rotation, as it dictates which side of the cartridge (and which end of the tubing) is the inlet.
8. Connect a clean, chemically-compatible internal intake hose to the inlet of the intake manifold.

2.4.2 Decontaminating the Syringe Pump

Any parts of the sampler that make contact with the fluid stream and are intended for repeated use (e.g., Luer fittings) must be decontaminated by washing with water and a surfactant, rinsed with a clean solvent, and dried.

The process for decontaminating the syringe pump is as follows:

1. Remove the syringes from the pump.

2. Remove and replace the interconnect tubing that runs between the intake manifold, syringe, and sampling cartridges. Decontaminate or replace all luer fittings and check valves. If disposable syringes are used, replace with new stock.
3. If reusable glass syringes are used, wash the syringes in an industrial glassware washer using an appropriate decontamination sequence and detergents, or wash by hand with warm water and a surfactant, rinse with a clean solvent, and dry.
4. Replace syringes and fluid train (check valves, fluid lines, and fittings).
5. Set a 1 mm gap between the syringe plunger and the terminus of the syringe barrel by manually advancing the pump.
6. Test the operation of the pump over a short stroke length (e.g., 1 cm stroke over 20 seconds).
7. Connect a clean, chemically-compatible internal intake hose to the inlet of the intake manifold.

2.4.3 Priming the Peristaltic Pump

1. Connect the internal intake hose to a supply of 18-M Ω water.
2. Uncap or disconnect a pump inlet from one port on the intake manifold.
3. If water siphons through the system, recap the intake manifold. If not, attach a syringe to the free intake manifold and draw water into the manifold. Disconnect the syringe, recap inlet port, and check for leaks.
4. Test the pump for operation at a medium flow rate (e.g., 500 μ g/min). Observe the outlets of the pump tubes for liquid production.
5. If no channels produce liquid, check the pump rotation direction, using the controller to reverse direction if necessary.
6. If no channels produce liquid and the pump rotation direction is correct, observe the cartridges for movement. The cartridges should not flex when the pump rollers rotate. If flexure is observed, tighten the tension screw in half-turn increments until it stops. If flexure control requires significant tightening of the tension screw, the cartridges may be unacceptably worn and may be impossible to calibrate. Replace worn cartridges.
7. If a minority of pump channels fail to produce liquid, attach a syringe to the outlet of each no-flow pump tube and use the syringe to apply a vacuum until a small amount of liquid appears. Continue operating the pump, and verify that the channel is now operating.
8. With the pump off and the system primed, cap the internal intake hose.

2.4.4 Priming the Syringe Pump

1. Connect the internal intake hose to a supply of 18-M Ω water.
2. Working sequentially around the pump:
 - a. Remove a syringe.
 - b. Draw water into the syringe.
 - c. Tilt the syringe so that the air bubbles rise to the exit; evacuate the syringe.
 - d. Repeat until the intake system is purged of air.
 - e. Replace the syringe and repeat for the next syringe.
3. Cap the internal intake hose.

2.4.5 Calibrating the Peristaltic Pump

The control unit provides control via pulse-width modulation, with feedback from an optical rotary encoder. The control unit is calibrated with the sampling unit before deployment in the field, and is capable of maintaining very low, nearly continuous flows (microliter per minute) or extremely low flows using a pulsed operation mode. The flow rate is determined by the volume of water to be processed *in situ* and the expected aquifer flow rate through the well bore. In environments where the aquifer is essentially stagnant and communication with the surrounding media is poor, it is desirable to set the device to very low flow rates and/or operate it in effluent capture mode.

Calibration is required whenever new tubing is added and should be checked after any other components are replaced (e.g., when changing the length of the control cable or) or before any other operation. Note that the uncalibrated pump may deliver a significantly different volume that which was selected.

1. Connect the internal intake hose to a supply of 18-M Ω water.
2. Capture the effluent from the pump tubing in vessels with known tare weights (e.g., centrifuge tubes). Use of extension tubing may be convenient but is discouraged.
3. Select a low or medium flow-rate (e.g., 50 - 500 ug/min) on the controller, and specify a dispensation volume. Note that the target volume should be as close to the sampling volume as practical; error propagation will cause large target volumes to diverge when the pump is calibrated around very small volumes.
4. Operate the pump in volume-dispense mode and compare the volume dispensed by each channel with a balance. Most channels should dispense similar amounts; of those dispensing similar amounts, the most productive channel should be selected as the target for calibrating the other channels. Note that the actual amount of water dispensed is irrelevant, as the pump is not calibrated, so long as the channels dispense similar amounts.
5. If one channel dispenses significantly more than the others, or continues to dispense when the pump is turned off, it may be siphoning. Tighten the cartridge tension screw 1 - 2 turns.
6. For other channels that dispense less than the target channel, tighten the setscrew 0.5 - 1 turn.
7. Repeat Step 5 until all of the channels dispense the same volume with no more than 5% deviation from the mean.
8. Using the controller's calibration function, input the mean volume acquired in Step 6.
9. Repeat Steps 5 - 7 until the mean of the channels is within 5% of the target volume.
10. Verify that the pump dispenses the expected amount using the control cable to be used in the field. Cables exceeding 100 ft in length may cause dispensation problems at very low flow rates; this can be mitigated by using higher flow rates in pulses instead of continuous, low flow modes.
11. With the pump off and the system primed, cap the internal intake hose.

2.4.6 Calibrating the Syringe Pump

An onboard motor controller provides control for the syringe pump. The surface package provides a 24-V DC power supply and an interface for sending commands to the pump. Calibration is handled in the interface software, and calibration settings are saved. As long as the syringe pump is paired with its calibration file, the calibration of the pump only needs to be tested before deployment.

1. Connect the internal intake hose to a supply of 18-M Ω water.
2. Capture the effluent from the syringes in vessels with known tare weights.
3. In the calibration pane of the interface software:
 - a. Enter a single-stroke test volume and pump command.
 - b. Alternatively, a large volume pump sequence can be programmed and used to generate a large volume water sample.
4. Determine the volume of water pumped by the syringes and enter the mean value into the calibration input on the interface software.
5. Repeat until the difference between pumped and programmed values meets quality requirements.

2.4.7 Loading the Sorbent Cartridges

1. Prepare sorbent cartridges according to the method and quantity specified in the sampling plan.
2. Attach Luer connectors to the sorbent cartridges and array them in a cartridge holder.
3. Attach the cartridge holder to the IS2 sampler internal framework. Connect the outlet side of the pump tubing to each quantification cartridge. Leave field blank cartridges uncapped.

2.4.8 Effluent Capture or Effluent Discharge

1. To operate the IS2 sampler in effluent capture mode, prepare a clean effluent capture cartridge. The effluent capture cartridge is a frame that supports up to six Tedlar bags which are connected to the effluent lines leaving the SPE cartridges.
2. To operate the IS2 sampler in effluent discharge mode, prepare an outlet manifold and outlet and internal outlet hose.
3. Connect the outlet of the SPE cartridges to either the effluent capture cartridge or the outlet manifold.

2.4.9 Preparing the Sampler for Deployment

1. The IS2 deployment shell has two end caps, each with four ports for passing fluid lines or control cables. One port in the upper cap is dedicated to the controller. Attach fluid pass-throughs or plug to the remaining ports.
2. Taking note of the location of the fluid pass-throughs in the caps, arrange the internal intake and outlet (if using effluent release mode) hoses to reach the correct end of the internal framework.
3. Insert the IS2 sampler internal framework into an empty shell.

4. Screw the threaded caps onto the shell.
5. Connect fluid pass-throughs and control cables from the end caps to the IS2 internal framework.
6. Bolt the end caps to the threaded caps.
7. Cap the fluid pass-throughs.
8. Decontaminate the exterior of the shell by washing it with an approved cleaning solution. Rinse thoroughly with deionized water.

3.0 DEPLOYMENT

3.1 DEPLOYMENT: INSERTION

The IS2 sampler may be operated with or without purging the well; it is up to the sampling manager to determine the necessity of purging, based on the particular hydrogeology of the site.

After insertion, the well may be under pressure from the displaced volume of water. It is similarly up to the sampling manager to determine whether or not an equivalent volume of water should be removed from the well pre-insertion, or if the well should be given time to equilibrate after insertion, based on the particular hydrogeology of the site.

3.1.1 Shallow Site Insertion

When the sampling depth is less than 60 ft bgs, the sampler can be inserted manually. Three personnel are required for insertion of the IS2 sampler into the well.

1. Verify the depth to groundwater in the well.
2. Verify that the IS2 sampler is responding to commands from the controller without error messages.
3. Verify that the fluid pass-throughs are uncapped.
4. Assemble pre-cut deployment cable to match the sampling depth in the well, typically the middle of the screened interval. Stretch the deployment cable out straight across the ground.
5. Attach the deployment hanger to the deployment cable. The deployment hanger is critical, as it prevents the device from being lost in the well.
6. Attach the deployment cable to the sampler.
7. One member of the team is stationed at the far end of the deployment cable, holding deployment hanger. This member inserts the device into the well by walking the deployment hanger to the well.
8. The second member of the team places the sampler in the well to begin the insertion process. As the device is inserted into the well, the control cable is unreeled. This member fastens the control cable to the deployment cable at intervals (e.g., at the connections between pre-cut deployment cable sections) to ensure that the deployment cable carries the weight of the control cable.
9. The third member of the team uses the above-well pulley to keep the device centered over the well during insertion.
10. The well may be allowed to equilibrate for a period of time determined by the sampling manager.

3.1.2 Deep Site Insertion

When the sampling depth is greater than 60 ft bgs, the sampler should be inserted using a crane to ensure that the installing technicians are not overburdened with the weight of the sampler and its accompanying cables.

1. Verify the depth to groundwater in the well.
2. Verify that the IS2 sampler is responding to commands from the controller without error messages.
3. Verify that the fluid pass-throughs are uncapped.
4. Prepare the appropriate number of pre-cut deployment cables to match the sampling depth in the well.
5. Attach a deployment cable to the sampler.
6. Use the crane to lift the free end of the cable, position the sampler over the well, and lower the sampler into the well.
7. Secure the cable at the wellhead by passing a length of steel rod through the eyelet on the cable and resting the rod across the wellhead.
8. Disconnect the crane from the cable. Connect the next length of deployment cable to both the crane and the previous cable.
9. Use the crane to lift the free end of the cable, remove the temporary securement rod, and lower the sampler into the well.
10. Repeat 7 through 9 until the sampler is at the desired depth.
11. Attach the deployment hanger to the deployment cable. The deployment hanger is critical, as it prevents the device from being lost in the well.
12. The well may be allowed to equilibrate for a period of time determined by the sampling manager.

3.2 DEPLOYMENT: OPERATION

The peristaltic pump control unit can be powered by either a standard 120-V AC connection or by a 120-V AC inverter connected to a 12-V batteries pack. The syringe pump control unit can be powered by either a standard 120-V AC connection or by a 24-V battery pack. The choice of power source is dependent on the accessibility of the location.

After the equilibration period (if applicable), the controller dispenses a volume of water at a pre-selected rate. Start the dispensation process and verify that the sampler is operating without error messages.

3.3 DEPLOYMENT: RECOVERY

Three personnel are required for recovery of the IS2 sampler from the well.

1. One member of the team uses the above-well pulley to keep the device centered over the well during recovery.
2. The second member of the team lifts the sampler by walking the deployment handle away from the well.
3. The third member of the team removes the sampler from the well when it emerges.
4. The exterior of the shell should be decontaminated by washing with an approved cleaning solution and rinsing with deionized water.
5. If the assembly facility is close, the fluid pass-throughs can be capped and the sampler returned for disassembly. Otherwise, the end caps are unbolted, connections to the pass-throughs are disconnected, the threaded caps removed, and the IS2 internal framework is

removed. The sample cartridges and effluent capture bags (if equipped) are removed, capped, and placed on ice for return to the laboratory.

3.4 INVESTIGATION-DERIVED WASTE

Hazardous waste generated in the course of performing a deployment of the IS2 sampler includes the used peristaltic pump tubing in the sampler, disposable gloves and other soiled PPE, and the water used for decontamination of the instrument. All of this waste will be disposed of as appropriate for the type of contaminant encountered at the site, and in consultation with the site management.

4.0 DATA TYPES AND QUALITY CONTROL

A sampling plan must account for the collection, handling, and analysis of the primary quantification samples as well as quality control (QC) data. Four types of QC data are used to provide information on the limits of quantification associated with the methods used herein. An IS2 sampling plan thus accounts for five types of data, all of which are processed with replicates:

- Quantification Samples
- Breakthrough Warning Samples
- Blanks
- Standards
- Spike Samples

4.1 QUANTIFICATION SAMPLES

Quantification Samples (or quantification cartridges) are SPE cartridges that are prepared in the laboratory, placed in the IS2 sampler, loaded with groundwater *in situ*, returned to the laboratory and processed for quantification.

Sample Replicates enable the analyst to estimate precision. Quantification Sample Replicates are generated by the IS2 sampler. Quantification Sample Replicates duplicate the entire chain of preparation and sampling and incorporate variability in method execution, including variability in the accuracy of IS2 sampler water delivery channels. At least three Quantification Sample Replicates are generated in each deployment.

4.2 BREAKTHROUGH DETECTION SAMPLES

To determine the contaminant concentration in the subsurface, the operator of the IS2 must be able to reliably determine the volume of water processed and the mass of contaminants captured by the SPE cartridge. Saturation of the SPE cartridge, and the resulting loss of contaminant mass due to breakthrough, poses a unique failure mode for the IS2. This limitation is largely eliminated by including Breakthrough Detection Samples.

4.2.1 Breakthrough Detection Cartridges

Breakthrough Detection Cartridges are SPE cartridges prepared as quantification cartridges. For each Quantification Sample, a Breakthrough Detection Cartridges is included in series, downstream. Breakthrough Detection Cartridges are recovered and processed in the same manner as Quantification Samples. Each Breakthrough Detection Cartridges should return results that are non-detect for the target analytes; in doing so, the Breakthrough Detection Cartridges guarantees that the Quantification Sample accounts for all of the analyte mass in the volume of processed water.

If a Breakthrough Detection Cartridges is determined to have a detectable quantity of the target analyte, any concentration data derived from the associated Quantification Sample must be appropriately flagged and noted as under-representative of the true value.

4.2.2 Breakthrough Detection in Effluent

When the IS2 is operated in effluent capture mode, the post-cartridge effluent can also be analyzed for breakthrough detection. This method is only applicable for contaminants that are not subject to degradation or volatilization during storage in the effluent capture bag. Examples of such substances include metal salts.

4.3 BLANKS

Field Blanks are SPE cartridges prepared as quantification cartridges, uncapped and placed inside the IS2 sampler for the duration of the deployment, but not loaded with groundwater. Upon return to the laboratory, they are loaded with a clean matrix and processed for quantification. The Field Blank provides information regarding contamination associated with ambient conditions during handling and operation of the IS2 sampler. One Field Blank is prepared for every group of quantification cartridges loaded into the IS2 sampler.

Trip Blanks are SPE cartridges prepared as quantification cartridges, capped and placed inside the IS2 sampler for the duration of the deployment without being exposed to the environment. Upon return to the laboratory, they are loaded with a clean matrix and processed for quantification. The Trip Blank provides information regarding contamination associated with the sample packaging, transportation, and storage. One Trip Blank is prepared for every group of quantification cartridges loaded into the IS2 sampler.

Method Blanks are SPE cartridges prepared as quantification cartridges, loaded with a clean matrix in the laboratory, and processed for quantification. Method Blanks provide information regarding systemic contamination during handling and processing.

Upon analysis, concentrations data for blanks is multiplied by ten (raised an order of magnitude). Quantification samples of this magnitude are considered similar to blank concentrations and flagged as such.

4.4 STANDARDS

Initial Calibrations (ICAL) are performed using a range of calibration standards. The lowest-concentration standard is used to define the reporting limit (RL) for each analyte. Continuing calibration standards consisting of selected parts of the calibration standard are evaluated for consistency with the original ICAL as part of each batch analysis.

4.5 SPIKE SAMPLES

Method development for some analytes may require the generation of spike samples for each batch of field samples.

Surrogate Spikes are field samples to which are added defined amounts of compounds chemically similar to the analytes of interest. These might be isotope-labeled standards or other analogues, and enable the analyst to estimate the recovery efficiency of the extraction and analysis methods.

5.0 REFERENCES

URS Corporation. (2011). *Final Site ST012 Former Liquid Fuels Storage Area Groundwater Monitoring Report, February 2011 Event, Former Williams AFB, Mesa, Arizona.* (Williams AR #1458). Austin, TX.

