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# Wastewater Response Protocol Toolbox:

Planning For and Responding To Wastewater Contamination Threats and Incidents

December 2011

Module 3: Site Characterization and Sampling Guide This page intentionally left blank.

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# **1** Introduction

Site characterization and sampling are activities that should be initiated in response to a 'Possible' contamination threat in order to determine whether or not the threat is 'Credible.' Site characterization is the process of collecting information concerning a 'Possible' contamination event. If a suspected contamination site has been identified, it will likely be designated as the primary investigation site. Additional or secondary sites may be identified to investigate the potential spread, or source, of a suspected contaminant. For example, this could include monitoring of the influent pump station wet well at the treatment plant headworks if contamination is suspected in the wastewater collection system. The results of site characterization are critically important to the threat evaluation process. Note that in some cases, the evidence or observations gathered during the site characterization could be sufficient to elevate the threat evaluation from 'Possible' to 'Credible' and even 'Confirmed.'

Module 3 describes recommended procedures for carrying out the site characterization activities. These procedures may be adapted to a utility's specific needs consistent with any applicable laws or regulations.



There are two broad phases of site characterization: planning and implementation. The Incident Commander is typically responsible for planning while the Site Characterization Team is typically responsible for actually implementing the Site Characterization Plan. This module provides information for those involved in either the planning or implementation phases of site characterization. While the target audience is primarily wastewater utility managers and staff, other organizations may be involved in site characterization. Therefore, this module may also be useful for a variety of first responders including police, fire, HazMat responders, FBI and EPA criminal investigators, National Guard Civil Support Teams, and environmental response teams from EPA and other government agencies.



# **2 Overview of Recommended Site Characterization Process**

# **Process Overview**

The recommended site characterization process includes five stages. These are shown in the flowchart in Figure 3-1, and are described in the narrative that follows.



Figure 3-1. Site Characterization Process

### **Customize the Site Characterization Plan**

A Site Characterization Plan should be customized for a specific threat, from the utility's generic Site Characterization Plan. The generic Site Characterization Plan should be developed as part of a utility's prior preparation for responding to both intentional and accidental contamination threats, and should be designed to be adaptable to a variety of situations. The generic plan may contain information on pre-entry criteria (i.e., under what circumstances a particular team, such as a utility team, may execute the site characterization), communications, team organization and responsibilities, safety, field testing details, sampling details, and a protocol for exiting the site. The customized plan should guide the team during site characterization activities and be based on the specific circumstances of the current threat warning. The Site Characterization Team typically will use the customized plan as the basis for their activities at the investigation site. A template for the development of a Site Characterization Plan is provided in Appendix 8.

plan, it is important for the Incident Commander to conduct an initial assessment of site hazards, which is critical to the safety of the Site Characterization Team.

The initial assessment of site hazards will impact the makeup of the team. Under low hazard conditions, a utility team may perform site characterization. If there are obvious signs of more hazardous conditions (radiological, chemical, or biological contamination), then teams trained in hazardous materials safety and handling techniques (HazMat) may need to conduct an initial hazard assessment and clear the site for entry by utility personnel. Alternatively, the HazMat team may decide to perform all site characterization activities themselves. The composition of the Site Characterization Team should be consistent with the role that the utility has assumed beforehand in threat/incident response. Obvious signs of hazard would provide a basis for determining that a threat is 'Credible.' Furthermore, the site might be considered a crime scene if there are obvious signs of hazards and human intervention. In this case, law enforcement may take over the site investigation.

During the development of the customized

Four hazard categories are considered in the context of site characterization:

**Low Hazard** - no obvious signs of radiological, chemical, or biological contaminants present at the site (i.e., in the air or on surfaces). Contaminants that may be present are assumed to be dilute and confined to the wastewater.

**Radiological Hazard** - presence of radiochemical isotopes or emitters tentatively identified, at the site, in the air or in the wastewater (i.e., through the use of field radiation detectors).

**Chemical Hazard** - presence of highly toxic chemicals (e.g., chemical weapons or biotoxins) or volatile toxic industrial chemicals, tentatively identified at the site in the air or in the wastewater, with a potential risk of exposure through dermal or inhalation routes.

**Biological Hazard** - presence of pathogens, tentatively identified at the site, with a potential risk of exposure through dermal or inhalation routes.

Figure 3-2 illustrates how information from recommended site characterization activities may be used to refine the hazard assessment, which in turn may influence the course of the site characterization.





# Approach the Site

Before entering the site, an initial assessment of conditions and potential hazards should be conducted at the site perimeter. As part of this assessment, the Site Characterization Team, upon arrival at the perimeter, should conduct a field safety screening and observe site conditions.

The purpose of the field safety screening activities is to identify potential environmental hazards that might pose a risk to the Site Characterization Team. The screening may include tests for radioactivity and atmospheric screening for ambient combustible gases, toxic gases, and volatile organic compounds (VOCs). Flammable or explosive gases can be detected using a Lower Explosive Limit (LEL) meter and/or a combustible gas detector. Non-specific VOCs can be detected with a Photoionization Detector (PID) Total Organic Vapor Detector. Specific VOCs can be detected and identified with field portable gas chromatography (GC) or gas chromatography-mass spectrometry (GC-MS). The team should also conduct a visual inspection of the site to detect signs of hazard (e.g., dead animals, dead vegetation, discarded equipment, gloves, and containers).

If the team detects signs of hazard, they should stop their investigation and contact the Incident Commander to report their findings. If no immediate hazards are identified during the approach to the site, the Incident Commander will likely direct the team to enter the site and perform the site characterization (Figure 3-3).

Observations made during the approach to the site should be documented using a form such as the Site Characterization Report Form in Appendix 9. The results of the field safety screening should be documented using a form such as the Field Testing Results Form included in Appendix 10.



Figure 3-3. Operator Using a Field Meter for Site Characterization.

# Characterize (Investigate) the Site

During this stage, the team should repeat the field safety screening (at the site itself), conduct a detailed visual investigation of the site, and perform rapid field testing of the wastewater that is suspected of being contaminated. Rapid field testing may include the collection of samples based on the process outlined in the section below. Details observed during the visual inspection of the site can also be documented using the form in Appendix 9.

Rapid field testing of the wastewater has three objectives:

- 1. Provide additional information to support the threat evaluation process.
- 2. Provide tentative identification of contaminants that would need to be confirmed by laboratory testing.
- 3. Determine if hazards tentatively identified in the wastewater require special handling precautions for sample collectors.

The field testing performed on wastewater should be based on the circumstances of the specific threat and should be consistent with the training and resources of the Site Characterization Team. A core set of rapid field tests includes measurement of pH, conductivity, and radioactivity (including alpha, beta, and gamma radiation). Abnormal pH, conductivity, or radioactivity values may indicate a problem.

In addition to the core tests, the Site Characterization Team may conduct expanded field testing of wastewater commensurate with their training and resources (Figure 3-4). Expanded field testing may include screening for combustible gases in the headspace of a wastewater sample using an LEL gas detector. It may also include non-specific screening for VOCs using a sample headspace total organic vapor PID detector, or specific detection and identification of VOCs using a portable GC/ MS. Screening for gases in a manhole can include measurements at two inches below the lip and repeated measurements after lowering the probe to a point just above the wastewater surface. Toxicity screening may be conducted using acute toxicity screening tests, and biotoxins and pathogens may be detected using Polymerase Chain Reaction (PCR) technology.

It is important to note that negative rapid field test results are not a reason to forgo sample collection since field testing is limited in scope and can result in false negatives. This is especially true given the complicated analytical matrix presented by wastewater. It is also important to emphasize that any field detectors or kits used during an emergency should be evaluated and characterized with respect to performance, and a baseline established before an emergency for the monitored parameter. Use of detectors or equipment that have not been characterized may lead to greater uncertainty with respect to how to respond, especially if the tests produce false positive results.

Results of rapid field testing of the wastewater can be documented using the Field Testing Results Form in Appendix 10.



Figure 3-4. Operator Conducts Field Testing at a Treatment Plant.

# **Collect Samples**

Following rapid field testing of the wastewater, samples of the suspect wastewater should be collected for potential laboratory analysis. The purpose of sampling from a suspected contamination site is to obtain and preserve a sample of the wastewater at a particular time and location so that it can be analyzed later if necessary. The decision to send samples to a laboratory for analysis should be based on the outcome of the threat evaluation. If the threat is determined to be 'Credible,' then samples should be immediately delivered to a laboratory for analysis. On the other hand, if the threat is determined to be 'Not Credible,' then samples should be secured and stored for a predetermined period in the event that it becomes necessary to analyze the samples at a later time.

In order to sample effectively, sampling requirements should be considered during the development of the customized Site Characterization Plan. Factors to consider during the development of the sampling approach include:

- Which contaminants or contaminant classes will be analyzed for?
- What type of samples will be collected (i.e., grab or composite)?
- When and where will samples be collected?
- Are any special precautions necessary during sample collection?

Under low hazard conditions, no special sampling techniques may be necessary beyond good safety practices as outlined later in this module. If the site is characterized as a radiological hazard during field safety screening or the rapid field testing of wastewater, then samples should be collected



for radiological analysis by personnel trained and equipped to work at radioactive contamination sites. If the site is characterized as a chemical hazard, dilution of samples collected for chemical analysis may be an appropriate sampling strategy to reduce risk during sample transport and analysis. Finally, if the site is characterized as a biological hazard, pathogen sampling may require the collection of a large volume of wastewater for subsequent concentration in the lab.

Critical information for each sample should be documented. The same information captured on the sample labels should be transferred to a sample documentation form to serve as a sample inventory. Appendix 11 contains an example documentation form. Additionally, sample custody should be closely tracked and documented using a chain of custody form. See Appendix 12 for an example of this form.

EPA has recently published additional guidance on sample collection entitled *Sampling Guidance for Unknown Contaminants in Drinking Water* (EPA-817-R-08-003, November 2008) (see www.epa.gov/ watersecurity; search under Water Laboratory Alliance). While this document is intended for drinking water applications, it may also be useful for wastewater sampling. Table 3-1 presents an example of a sample collection kit, while Table 3-2 provides a detailed listing of the sample containers included in the kit. The sample collection kit described in this section is intended to illustrate the types of materials and supplies that might be useful during sampling activities. However, the design of a specific kit should be tailored to the needs and sampling objectives of the user.



Table 3-1: Example Design of an Emergency Wastewater Sample Collection Kit

ltem	Quantity	Notes
Field Resources and Documentation		
Field Guide	2	Resource for field personnel
Health and safety plan	2	If required for the site
Sample labels	48	Waterproof (filled out in advance, if possible)
Sample documentation forms	24	For recording sample information
Custody tape (or seals)	2 rolls	Used on sample or shipping containers
Chain of custody forms	24	For documenting sample custody
Lab marker	2	Waterproof, 1 red, 1 black
General Sampling Supplies		
Sample containers	Table 3-2	For collecting samples
Device for grab sampling	1	For sampling large water bodies
10 liter HDPE container	4	For collection of large volume water samples
Lab grade tape	3 rolls	For temporary labeling in the field
Miscellaneous glassware	N/A	Beakers, graduated cylinders, spatula, etc.
Collapsible cooler	1	For sample storage
Rigid shipping container	1	For shipping by overnight service if needed

# Table 3-1 (cont.): Example Design of an Emergency Wastewater Sample Collection Kit

Item	Quantity	Notes
1 qt. zippered freezer bags	1 pack 100	For double bagging ice and sample containers
Thermometer	2	For checking water temperature
Paper towels	2 rolls	Wiping wet containers and containing spills
Reagents (may need to be kept separ	ate from the re	st of the kit)
Laboratory grade water	5 liters	For sample dilution in the field
6 Molar ACS grade hydrochloric acid (HCI)	25 mL	In dropper bottle for preservation of samples for organic analyses
6 Molar trace metal-grade nitric acid (HNO <sub>3</sub> )	25 mL	In dropper bottle for preservation of samples for trace metals analysis
10 Normal sodium hydroxide (NaOH)	25 mL	In dropper bottle for preservation of samples for cyanide analyses
pH paper in ranges from 0 – 4 and 10 – 14	50 strips	For checking pH of samples preserved with acid or base (sensitive to 0.5 pH units)
Safety Supplies		
Splash resistant goggles	2	One per individual (minimum)
Disposable gloves	1 box	Nitrile or polyethylene, powder-free
Disposable shoe covers	2 pairs	One pair per individual (minimum)
Disposable laboratory coats	2	One per individual (minimum)
Clear, heavy duty plastic trash bags	4	For disposal of lab coat, gloves, etc.
Rinse water	20 liters	For general use and first aid
Antiseptic wipes	1 container	For cleaning hands, sample containers, etc.
Bleach solution (at least 5%)	1 gallon	For decontamination if necessary
Squirt bottle	2	For use with rinse water or lab grade water
First aid kit	1	For general first aid
Flashlight/headlamp	3	For working at night or in dark locations

# If the threat is determined to be 'Credible,' then samples should be immediately delivered to a laboratory for analysis.

Table 3-2: Sample Containers for Emergency Wastewater Sample Collection \*

Sample Type	Container Size	Container Type	No.	Preservative	Analytical Technique	Reference Methods
CHEMISTRY – BAS	SIC SCREEN (I	<b>Established Techniques</b> )				
Organic Analytes						
Volatiles	40 mL	Glass w/Teflon faced septa	5	1:1 HCl to pH < 2	P&T - GC/MS	8260B
				See method	P&T – GC/PID/ELCD	8021B
Semi-volatiles	1 L	Amber w/Teflon-lined screw caps	4	6M HCI. See method	SPE GC/MS	8270D 3535A
Quarternary nitrogen compounds	IL	Amber PVC or silanized glass	4	Sulfuric acid to pH2	SPE HPLC – UV	8321B
Carbamate Pesticides	40 mL	Glass w/Teflon faced septa	4	Potassium dihydrogen citrate sample pH to $\sim 3.8$	HPLC-fluorescence	8318
Metals/Elements	125 mL	Plastic (i.e., HDPE)	0	Trace metal grade nitric acid. See method.	ICP-MS	6020A
					ICP-AES	6010C
					AA	7010
Organometallic compounds	125 mL	Plastic (i.e., HDPE)	7	Nitric acid to pH 2. See method	AA – cold vapor	7471B
Cyanide	1 L	Plastic	7	Sodium hydroxide to pH 12. See method.	Wet Chemistry	9012A
Radiological	2 L	Plastic	0	None – mark samples not preserved	Gross alpha, gross beta, gamma isotopes, specific radionuclides	7110B

\* Analytical techniques and reference methods are covered in more detail in Module 4.

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Sample Type	Container Size	Container Type	No.	Preservative	Analytical Technique	Reference Methods
CHEMISTRY – EXI	PANDED SCRI	<b>EN (Exploratory Techniqu</b>	es)			
Unknown organics (volatile)	40 mL	Glass w/Teflon faced septa	5	None – mark samples not preserved	P&T-GC/MS	See Module 4
Unknown organics (general)	١L	Amber Glass	4	None – mark samples not preserved	Prep: SPE, SPME, micro LLE, direct aqueous injection, headspace Analysis: GC/MS. GC,	See Module 4
					HPLC, LC-MS	
Unknown inorganics	١L	Plastic	5	None – mark samples not preserved	ICP-MS	See Module 4
Immunoassays	1 L	Amber Glass	2	Consult manufacturers instructions	Consult manufacturers instructions	None
PATHOGENS – EP	ANDED SCREI	EN (Established and Explor	atory Te	chniques)		
Pathogens -culture	100 mL	HDPE (plastic)	2	TBD	Per target pathogens	See Module 4
Pathogens – PCR	100 mL	HDPE (plastic)	2	TBD	Per target pathogens	See Module 4
<b>BASELINE WATEH</b>	A QUALITY PA	ARAMETERS (See Section 3	3-4)			
Water quality: Chemistry	1 L	Plastic	-	None – mark samples not preserved	Conductivity, pH, alkalinity, hardness, turbidity	Standard methods
Surrogates	١L	Amber Glass	5	None – mark samples not preserved	TOC, ultraviolet absorbance, color, chlorine demand	Standard methods
Toxicity	125 mL	Glass	0	Consult manufacturers instructions	Rapid toxicity assay (several vendors)	None

# **Exit the Site**

Upon completion of site characterization activities, the team should prepare to exit the site. At this stage, the team should make sure that they have documented their findings, collected all equipment and samples, and resecured the site (e.g., locked doors, hatches, and gates).

If the site is considered to be a hazardous site, special procedures for exiting the site may be required by HazMat officials. For example, personnel and equipment may be required to undergo decontamination prior to exiting the site, and access to the site is likely to be tightly controlled.

If the site is considered a crime scene, the site may be secured by law enforcement, and qualified investigators may be responsible for collecting and preserving any physical evidence (such as empty containers, or discarded equipment).

The site characterization activities presented in this module range from relatively simple activities, such as visual inspection of the site, to complex activities, such as field testing of the air, environmental surfaces, and wastewater for unusual contaminants. The wastewater utility should decide in advance the extent of site characterization activities that they will perform within their own organization and those that would be provided by external organizations. For example, a utility may choose to develop a capability for performing the visual inspection and core field testing at low hazard sites. The utility may make arrangements with HazMat responders to provide support during the characterization of potentially more hazardous sites. The utility may also arrange with a contract lab to provide sample kits and sample containers. It is critical that the utility plan ahead of time for those



site characterization activities that they will take responsibility for, and make arrangements with agencies that will support the utility in the event that a situation exceeds the utility's resources and capabilities. Tabletop and operational-based drills and exercises provide training opportunities to improve coordination between the utility and response agencies.

# **3 Safety and Personnel Protection**

Proper safety practices are essential for minimizing risks to the Site Characterization Team and must be established prior to an incident in order to be effective. Field personnel involved in site characterization activities should have appropriate safety training to conform with applicable laws and regulations including work safety regulations under the Occupational Safety and Health Administration. These include OSHA 1910.120 (http://www.osha.gov), which deals with hazardous substances.

Basic good safety practices should be incorporated into a set of concise safety guidelines for personnel responsible for performing site characterization activities. These guidelines may be formalized into a health and safety plan (HASP).

3-12

The appropriate level of personal In other can be observed or during the assessment of site hazards that might pose a risk to the Site Characterization Team. Site hazard assessment is conducted during the development of the customized Site Characterization Plan and continues throughout the period of time that the team In other can be observed or during the development of the customized Site Characterization Plan and continues throughout the period of time that the team In other can be observed or during the development of the customized Site Characterization Plan and continues throughout the period of time that the team In other can be observed or during the development of the customized Site Characterization Plan and continues throughout the period of time that the team In other can be observed or during the development of the customized Site Characterization Plan and continues throughout the period of time that the team In other can be observed or during the development of the customized Site Characterization Plan and continues throughout the period of time that the team In other can be observed or during the development of the customized Site Characterization Plan and continues throughout the period of time that the team In other can be observed or during the development of the customized Site Characterization Plan and continues throughout the period of time that the team In other can be observed or during the development of the customized Site Characterization Plan and continues the team In other can be observed or during the development of the customized Site Characterization Plan and continues the team In other can be observed or during the development of the customized Site Characterization Plan and continues the team In other can be observed or during the development of the customized Site Characterization Plan and continues the team In other can be observed or during the development of the customized Site Characterization Plan and continues the team In other customized Site Characterization Plan and co

throughout the period of time that the team occupies the investigation site. Two general scenarios are considered, one in which there are no obvious signs of immediate hazards, and one in which there are indicators of site hazards.

In most cases the investigation site will not present a significant hazard and basic equipment and training will be sufficient to conduct site characterization activities safely. This would typically be the case for a routine security breach such as an open manhole cover. Under these conditions it may be reasonable to presume that any contaminants that might be present are confined to the wastewater and are present at dilute concentrations. Risk to personnel may be minimized through the use of good safety practices, including:

- Do not eat, drink, or smoke at the site
- Do not smell wastewater samples
- Use basic personal protective equipment
  - Splash proof goggles
  - Disposable gloves
  - Disposable foot covers
  - Disposable lab coat
- Avoid skin contact with wastewater
- Fill sample containers slowly to avoid volatilization or aerosolization of contaminants
- Minimize time that personnel are on site

In other cases obvious signs of hazards may be observed at the time the threat is discovered or during the approach to the site. Under these conditions, only personnel with proper equipment and training (e.g., HazMat teams) should enter the site.

# 4 Roles and Responsibilities for Site Characterization

The Incident Commander, Operations Section Chief and the site characterization Team Leader are key personnel in site characterization. The Incident Commander should have overall responsibility for managing response to the threat, and is responsible for planning and directing site characterization activities. The Incident Commander may also approve the Site Characterization Team to proceed with their activities at key decision points in the process. The Operations Section Chief is responsible for all field activities and serves as the liaison between the Incident Commander and the Site Characterization Team Leader. The Site Characterization Team Leader should be responsible for implementing the Site Characterization Plan in the field and supervising site characterization personnel.

Depending on the nature of the contamination threat, other agencies and organizations may be involved or assume responsibility during planning and implementation of site characterization activities. Some of these organizations and roles are described below.

# Wastewater Utility

The utility may provide the Incident Commander unless another organization is so designated to provide that role.

## HazMat Response Teams

In coordination with utility staff, these HazMat Teams may assume responsibility for oversight of site characterization activities in situations where hazardous materials are suspected.

### **Technical Assistance Providers**

The wastewater primacy agency, EPA hazardous material responders, or other specially trained response teams may be consulted for technical assistance and in some cases be requested to take responsibility for planning, oversight, and implementation of site characterization activities.

#### Laboratories

Laboratories are responsible for timely analysis of samples collected by the Site Characterization Team in response to a contamination threat.

#### Local Law Enforcement Agencies

These agencies may assume responsibility for incident command in situations where criminal activity, excluding a federal crime, is suspected.

#### FBI

The FBI is expected to assume incident command for the investigation aspects of the situation when terrorism is suspected. If the FBI becomes involved they would likely make the credibility determination.

### EPA

The EPA may provide technical advice for site characterization or other components of the Threat Management process, and may provide personnel for site characterization if requested by a state regulatory agency. In cases where a contamination threat or incident is not an act of terrorism, EPA's CID will typically be the lead federal agency for law enforcement in the response.

# 5 Summary

Once the determination has been made that a contamination event is 'Possible,' it is appropriate to conduct a site characterization to help determine whether the threat is 'Credible.' Site characterization is the investigation of the suspected site of contamination as well as other locations where contaminants may have spread or originated. Site characterization should be carried out systematically and involves customization of a general Site Characterization Plan followed by the actual investigation. The investigation includes physical inspection of the site, field safety screening of the environment, rapid testing of the suspect wastewater, and sample collection. While it is important to conduct a thorough investigation of the site and collect representative samples, it is also important to minimize the risk faced by the Site Characterization Team. Module 3: Site Characterization and Sampling Guide suggests a protocol to accomplish all of these goals.

# 6 Appendices

The following are examples of forms that may be used to facilitate the public health response:

- Site Characterization Plan Template
- Site Characterization Report Form
- Field Testing Results Form
- Sample Documentation Form
- Chain of Custody Form

These forms can be found in the Appendices located at the end of the Toolbox.