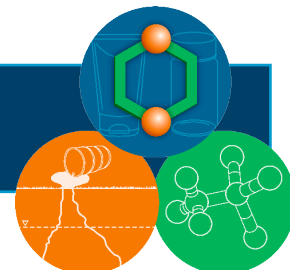




# Sampling and Analysis

## 1,4-Dioxane



### 1 Sampling

In general, conventional equipment has not demonstrated problems when sampling for 1,4-dioxane (Chemical Abstracts Service [CAS] Registry Number #123-91-1) in different matrices. However, this fact sheet provides precautionary information for groundwater sampling and equipment decontamination procedures when 1,4-dioxane is a contaminant of concern. Proper quality control should always be in place to monitor potential issues.

#### Precautions When Sampling for 1,4-Dioxane in Groundwater

Practitioners have evaluated and used a variety of methods and devices for sampling 1,4-dioxane in groundwater. These methods include low-flow purging, grab samplers, and passive diffusion samplers.

Some of these sampling methods have shown comparable concentrations for 1,4-dioxane in laboratory and side-by-side field tests (ITRC 2006); however, the commonly used passive diffusion bag (PDB) sampler using a single low-density polyethylene membrane for volatile organic compounds (VOCs) does not diffuse the 1,4-dioxane molecule and should not be used for 1,4-dioxane sampling. Two commercially available passive diffusion samplers have been effective for sampling 1,4-dioxane:

- Rigid Porous Polyethylene (RPP) passive diffusion samplers
- Dual-membrane passive diffusion bag (DMPDB) samplers

There are two commercially available grab samplers for sampling 1,4-dioxane:

- HydraSleeve™ Sampler
- Snap™ Sampler

Prior to initiating sampling activities, field personnel should discuss the proposed methodology with regulators due to the different approaches available for sampling.

#### Equipment Decontamination

1,4-Dioxane is a common impurity in detergents (see the *History of Use and Potential Sources* fact sheet). In early 2014, 1,4-dioxane was detected at elevated concentrations in leachate from a widely used decontamination detergent. Because manufacturing processes can introduce 1,4-dioxane into cleaning products through ethoxylation, individuals should take care to prevent residual detergents or surfactants from remaining on sampling equipment. In studying the potential presence of 1,4-dioxane in detergents used for decontamination of sampling equipment, researchers demonstrated that some common products were free of 1,4-dioxane when used according to the manufacturer's instructions (DiGuseppi et al. 2015). The collection of equipment blanks is useful for the detection of any residual 1,4-dioxane on sampling equipment.

The Interstate Technology and Regulatory Council (ITRC) has developed a series of six fact sheets to summarize the latest science and emerging technologies regarding 1,4-dioxane. The purpose of this fact sheet is to:

- describe potential concerns when sampling for 1,4-dioxane
- identify the common analytical methods available for 1,4-dioxane in different matrices, including water, solids, and air
- highlight the benefits and limitations of the available analytical methods

*Note: ITRC is developing a 1,4-dioxane guidance document for publication in late 2020. The guidance document will provide additional details on 1,4-dioxane sampling and analysis.*

## Sampling and Analysis: 1,4-Dioxane *continued*

Disposable equipment reduces the likelihood of cross-contamination and eliminates the need for equipment decontamination. In addition, the extended residence time of passive samplers allows sampler materials to reach chemical equilibrium with the surrounding groundwater, minimizing the potential effects of leaching or adsorption related to equipment materials.

### Sampling Containers, Preservation Requirements, and Holding Times

Table 1 provides a summary of the typical sampling containers, preservation methods, and holding times when sampling for 1,4-dioxane in different matrices.

**Table 1.** Containers, preservation, and holding times for 1,4-dioxane\*

Matrix	Analytical method	Typical collection volumes and containers	Preservative	Holding time (Schep et al. 2009)
Aqueous	SW-846 USEPA Method 8260 (USEPA 2018a)	Three 40-mL VOA vials with PTFE-lined screw caps (1–40 mL for analysis; remaining vials for screening and backup)	No headspace, HCl to pH <2, cool to 0°C–6°C. If residual chlorine is present, pre-preserve vials with sodium thiosulfate (3 g/40 mL).	<b>Analysis:</b> 7 days from collection if pH ≥ 2; 14 days from collection if pH < 2.
Aqueous	SW-846 USEPA Method 8270 (USEPA 2018b)	Two 1-L amber glass containers with PTFE-lined screw caps (1 L for extraction and 1 L for backup) <sup>†</sup>	Cool to 0°C–6°C.	<b>Extraction:</b> 7 days from collection. <b>Analysis:</b> 40 days from extraction. Extract may be frozen for up to 1 year to arrest HT.
Drinking water	USEPA 522 (USEPA 2008)	Two 500-mL glass containers with PTFE-lined screw caps (1 container for extraction and the other for backup)	Sodium sulfite (50 mg/L) and sodium bisulfate (~1 g/L), pH < 4, cool to ≤10°C.	<b>Extraction:</b> 28 days from collection. <b>Analysis:</b> 28 days from extraction if extracts are stored in the dark at -5°C.
Solid	SW-846 USEPA Method 8260 (USEPA 2018a)	Three 40-mL VOA vials with PTFE-lined screw caps (2–40 mL for low-level analysis and 1–40 mL for medium-level analysis) or three EnCore samplers	Vials are weighed and preserved prior to addition of the sample. <b>Low-level preservative</b> = 1 g sample: 1 mL water (with or without sodium bisulfate). <b>Medium-level preservative</b> = 1 g sample: 1–2 mL methanol (typically, 5 g of soil in 5–10 mL methanol). Sample extruded into pre-preserved vials on site; preservative must cover sample; low-level vials are stored at <-7°C and medium-level vials are stored at 0°C–6°C. <b>EnCore samplers:</b> Must be preserved as described above within 48 hours.	<b>Analysis (low-level):</b> 48 hours from collection if not frozen. <b>Analysis (low-level):</b> 14 days from collection if frozen within 48 hours of collection. <b>Analysis (medium-level):</b> 14 days from collection.

## Sampling and Analysis: 1,4-Dioxane *continued*

Solid	SW-846 USEPA Method 8270 (USEPA 2018b)	One 4- to 8-oz glass jar with a PTFE-lined screw cap	Cool to 0°C–6°C. May be frozen at lab to <-10°C.	<p><b>Extraction:</b> 14 days from collection. If sample is frozen to arrest HT, HT extends to up to 1 year from collection.</p> <p><b>Analysis:</b> 40 days from extraction. Extract may be frozen for up to 1 year to arrest HT.</p>
Air	USEPA TO-15 (USEPA 1999b)	One 1- to 6-L evacuated, passivated stainless	None	<p><b>Analysis:</b> 30 days from collection</p>
Air	USEPA TO-17 (USEPA 1999a)	Two sorbent tubes	Sealed tubes wrapped in aluminum foil (uncoated side facing the tube) and placed in a clean amber glass container. Store at <4°C.	<p><b>Analysis:</b> 30 days from collection</p>
<p>* Preservation and holding time requirements are standard VOC/SVOC holding times for the listed method references.</p> <p>† Check with the laboratory on whether a reduced volume option is available for Method 8270.</p> <p><b>Definitions:</b> HCl: hydrochloric acid; PTFE: polytetrafluoroethylene; SVOC: semivolatle organic compound; USEPA: U.S. Environmental Protection Agency; VOA: volatile organic analyte; VOC: volatile organic compound.</p>				

## 2 Analysis

Detection of 1,4-dioxane by routine VOC analytical procedures may be complicated because of its high solubility in water and chlorinated solvent interferences, both of which can lead to low bias and problems achieving the required sensitivity. (Refer to the *Environmental Fate, Transport, and Investigation Strategies* fact sheet for more information on the unique properties of 1,4-dioxane.) Table 2 summarizes the methods commonly used to measure 1,4-dioxane in samples of various matrices, including water, solids, and air (indoor air, outdoor air, and soil vapor). The table also includes comments and issues for each method to assist users in choosing the best analytical approach for their project.

## Sampling and Analysis: 1,4-Dioxane *continued*

**Table 2.** Summary of commonly used analytical methodologies for 1,4-dioxane

Analytical Method Reference	Typical RLs	Relative cost of analysis	Comments/additional considerations
<b>WATER MATRICES</b>			
SW-846 USEPA Method 8260: VOC (USEPA 2018a)	200–500 µg/L	\$	Ambient purge and trap with full-scan GC/MS (1) Due to poor purging efficiency, RLs may be too high to achieve regulatory standards. (2) 1,4-Dioxane-d8 should be used as internal standard.* (3) The isotope dilution method compensates for poor purge efficiency.
	2–5 µg/L	\$	Heated purge and trap (40°C–80°C) with SIM GC/MS (1) RLs may be too high to achieve regulatory standards. (2) Interferences can result in dilutions due to potential contamination of the instrument, resulting in elevated RLs for 1,4-dioxane. (3) 1,4-Dioxane-d8 should be used as internal standard.* (4) Interferences may also cause elevated recoveries of the internal standard, 1,4-dioxane-d8. (5) The isotope dilution method compensates for poor purge efficiency. (6) Improved precision and accuracy through recovery correction.
SW-846 USEPA Method 8270: SVOC (USEPA 2018b)	5–10 µg/L	\$\$	Full-scan GC/MS (1) RLs may be too high to achieve regulatory standards. (2) May result in low-biased data and poor recoveries of 1,4-dioxane due to poor extraction efficiency.
	0.15–0.4 µg/L	\$\$	SIM with isotope dilution GC/MS (1) Suggested isotope: 1,4-dioxane-d8. (2) The isotope dilution method compensates for poor extraction efficiency. (3) Improved precision and accuracy through recovery correction.
USEPA 522 (USEPA 2008)	0.05–0.1 µg/L	\$\$	Used for drinking water Solid-phase extraction (SPE) and SIM GC/MS (1) Generally required method for analysis of drinking water samples.
<b>SOLID MATRICES</b>			
SW-846 USEPA Method 8260: VOC (USEPA 2018a)	0.2–0.5 mg/kg <sup>†,‡</sup>	\$	Ambient purge and trap with full-scan GC/MS (1) 1,4-Dioxane-d8 should be used as internal standard.* (2) Elevated RLs are due to poor purging efficiency.
	0.002–0.005 mg/kg <sup>†,‡</sup>	\$	Heated purge and trap (40°C–80°C) with SIM GC/MS (1) Not routinely needed for solid samples. (2) 1,4-Dioxane-d8 should be used as internal standard.*
SW-846 USEPA Method 8270: SVOC	0.05–0.2 mg/kg <sup>†</sup>	\$\$	Full-scan GC/MS (1) May result in low-biased data and poor recoveries of 1,4-dioxane due to poor extraction efficiency.

## Sampling and Analysis: 1,4-Dioxane *continued*

(USEPA 2018b)	0.05–0.2 mg/kg <sup>‡</sup>	\$\$	Full scan with isotope dilution GC/MS (1) Suggested isotope: 1,4-dioxane-d8. (2) The isotope dilution method compensates for poor extraction efficiency. (3) Improved precision and accuracy through recovery correction.
<b>AIR MATRICES</b>			
USEPA TO-15: VOC	0.7–1.0 µg/m <sup>3</sup>	\$\$\$	Full-scan GC/MS
(USEPA 1999b)	0.4–1.0 µg/m <sup>3</sup>	\$\$\$	SIM GC/MS
USEPA TO-17: VOC (USEPA 1999a)	1.1–11 ng/tube	\$\$\$	Thermal desorption/full-scan GC/MS
<p>* When 1,4-dioxane-d8 is used as an internal standard in 8,260 analyses, this is comparable to isotope dilution.          † Assumes samples are preserved using the low-level preservation method in Table 1.          ‡ RLs assume 100% solids content; RLs and results should be corrected for percent solids.</p> <p><b>Definitions:</b> \$–\$\$\$: relative costs of analysis; µg/L: micrograms per liter; GC/MS: gas chromatography/mass spectrometry; RL: reporting limit; SIM: selective ion monitoring.</p> <p><b>Notes:</b> Other analytical methods may be available but are not commonly used on environmental samples. Analytical laboratories should be consulted for proper containers and preservatives for all matrices. Check with the appropriate regulatory agency for any state-specific requirements.</p>			

Analytical methodologies that use a combination of an extraction preparation, selective ion monitoring (Simonich et al. 2013), and isotope dilution generate accurate and low-level measurements of 1,4-dioxane. SIM offers greater sensitivity (lower reporting limits [RLs]). Using 1,4-dioxane-d8 for isotope dilution yields a “recovery corrected” final result that can compensate for the analyte’s poor recovery characteristics at low levels as well as potential matrix interferences. For this reason, the isotope dilution analytical approach typically improves accuracy and precision. However, the ultimate analytical method selected will depend on the project’s objectives.

It’s important to review the required action levels set by the applicable regulatory agency and to contact the laboratory to ensure success in the following areas:

- achievement of the required sensitivity
- performance of the desired method
- possession of the appropriate certifications, as required by the regulatory agency

### 3 References

The references cited in this fact sheet, and the other ITRC 1,4-Dioxane fact sheets, are included in one combined list that is available on the ITRC web site.

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