



Recommendations for the Low Level Analysis of 1,4-DIOXANE in Aqueous Samples

Background

A widely used industrial chemical, 1,4-dioxane may be present in a variety of commercial and consumer products either as an ingredient or as a by-product of manufacture. 1,4-dioxane has been widely used as a stabilizer for 1,1,1-trichloroethane in particular, and may have been used with other chlorinated solvents as well (USEPA 505-F-13-001). The U.S. Environmental Protection Agency (USEPA) has classified 1,4-dioxane as “likely to be carcinogenic to humans” by all routes of exposure (USEPA IRIS 2010) and has established 0.35 ug/L as a health advisory level (HAL).

While the USEPA has not established a Maximum Contaminant Level (MCL) for 1,4-dioxane, many states have established screening criteria and action levels for 1,4-dioxane in potable and non-potable water at concentrations in the 0.2 to 0.7 ug/L range. 1,4-Dioxane is completely miscible with water, making it very mobile in the environment and difficult to analyze for in the laboratory, especially at less than 1.0 ug/L concentrations.

Analytical Options for Aqueous Low Level 1,4-Dioxane Analysis

Alpha Analytical recommends a SVOC (i.e., extractable) approach for low level 1,4-dioxane analysis in water. There are two analytical methods that Alpha Analytical offers for 1,4-dioxane analysis with reporting limits of 0.15 ug/L: Modified Method 8270 GC/MS in selected ion monitoring (SIM) mode utilizing isotope dilution, and Method 522 GC/MS SIM (without isotope dilution). Both of these methods have the added advantage of having no interference from additional chlorinated solvent compounds that may be present in the sample.

Modified Method 8270 SIM, Isotope Dilution

Superior chromatographic performance and greater sensitivity can be achieved if Method 8270 is modified for 1,4-dioxane specifically. This approach references a modified EPA Method 8270/1625 GC/MS SIM with the mass spectrometer operated in the selected ion mode with use of the isotope dilution technique for greater analytical certainty. Isotope dilution techniques incorporate a deuterated form of the target analyte (d-1,4-dioxane), which is spiked into every sample to act as a target-specific internal standard to normalize recoveries and assist with 1,4-dioxane quantitation. Alpha Analytical can achieve an aqueous reporting limit of 0.15 ug/L utilizing modified EPA Method 8270.

EPA Method 522

EPA Method 522 has been promulgated by EPA for the analysis of 1,4-dioxane in drinking water. This procedure also utilizes GC/MS SIM (without isotope dilution) but it utilizes a different preparative procedure. Aqueous samples are extracted by a solid phase extraction (SPE) technique. This methodology was developed specifically for the drinking water matrix but can be used for other relatively clean aqueous matrices as well. For public water supply samples, Method 522 must be used as written for analysis of 1,4-dioxane in potable water samples. Alpha Analytical can achieve an aqueous reporting limit of 0.15 ug/L utilizing EPA Method 522.

Method 8260 SIM

Alpha Analytical offers, but does not recommend, analysis of 1,4-dioxane utilizing Method 8260 GC/MS SIM. Analysis of 1,4-dioxane using this analytical approach is known to exhibit poor analytical performance and sensitivity in aqueous samples. As such in 2013 the USEPA moved 1,4-dioxane from the TCL VOC list to the TCL SVOC list because of its inherent chemical properties. Also, any dilutions required for TCL VOCs by 8260 GC/MS SIM analysis due to high concentrations of chlorinated solvents present in the sample will adversely affect the 1,4-dioxane reporting limit. For these reasons, sample analyses that require a reporting limit in the 0.1 to 0.7 ug/L range for 1,4-dioxane should not utilize this analytical approach.

Additional Considerations

Sample collection considerations should be evaluated if low level 1,4-dioxane analysis is required. Practitioners are encouraged to address any questions with the laboratory in advance of field sampling as sampling containers and preservation will vary between these analytical approaches. Practitioners are also advised to incorporate field blanks made using 1,4-dioxane free water into their sampling programs at a conservative frequency whenever low level analysis is required. Additionally, practitioners should recognize that detergents used for decontamination of sampling equipment may be a source of 1,4-dioxane.

Bottle Requirements

Analytical Method	Alpha RL	Container	Preservative	Hold Time
EPA Method 8260-SIM	3.0 ug/L	3 x 40 mL Vial	HCl preserved, 4 degrees C	14 Days (to Analysis)
EPA Method 8270-SIM M Isotope Dilution	0.15 ug/L	2 x 500mL Amber glass, teflon lined	Unpreserved, 4 degrees C	7 Days (to Extraction)
EPA Method 522	0.15 ug/L	2 x 500mL Amber glass, teflon lined	Na2O3S/NaHSO4 preserved, 4 degrees C	28 Days (to Extraction)

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