

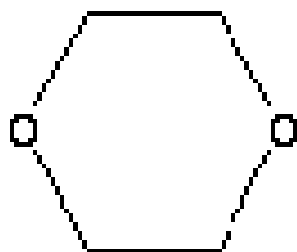


Challenges and Options for the Analysis of 1,4-Dioxane

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1,4-Dioxane



Formula: $C_4H_8O_2$

CAS Number: 123-91-1

Molecular Weight: 88.1

Boiling Point: 101.1 °C

Water Solubility - Miscible

Synonyms

- p-dioxane
- diethylene oxide
- 1,4-diethylene oxide
- dioxyethylene ether
- diethylene ether
- ethylene glycol ethylene ether

1,4-Dioxane



- Primarily used as a solvent stabilizer for 1,1,1-Trichloroethane (TCA) and other chlorinated solvents.
- Prevents breakdown of solvent due to
 - Light
 - Heat
 - Oxygen
 - Acids
- Also used in lacquers, paints, resins and automotive coolants

1,4-Dioxane



- Sources of 1,4-dioxane are anthropogenic as it does not occur naturally in the environment
- Classified as Class B2 probable carcinogen
- Inhalation and ingestion are the primary routes for human exposure

1,4-Dioxane



- 1,4-Dioxane is listed as a volatile compound under SW-846, Method 8260B. However, until recently (SOM01.2) it was not included in the Target Compound List (TCL) or the Priority Pollutant List (PPL).
- Site investigations may have failed to account for it's presence.

1,4-Dioxane



An enforceable Maximum Contaminant Level (MCL) has not been established for 1,4-dioxane but several states/agencies have established drinking water guidelines;

EPA (health advisory/RSL)	0.35 ug/l/0.46 ug/l
New Hampshire	0.25 ug/l (limit for all public water)
Massachusetts	0.3 ug/l (GW clean-up standard)
New Jersey	0.4 ug/l (GW Quality standard)
California	1 ug/l Notification level
Florida	3.2 ug/l Minimum criteria
Colorado	3.2 ug/l DW criteria (proposed 0.35 ug/l)

Analytical Methodologies



Would like to talk about four that we perform in our laboratory;

- VOC – 8260
- VOC – 8260 SIM
- SVOC – 8270
- SVOC – 8270 SIM

1,4-Dioxane as a Volatile



SW-846 8260B

- Water solubility results in poor purge efficiency
- Poor purge efficiency yields elevated reporting limits

	<u>Water</u>	<u>Soil</u>
MDL	70 ug/l	70 ug/kg
LOQ	250 ug/l	250 ug/kg

1,4-Dioxane as a Volatile



SW-846 8260B

- Poor purge efficiency also results in calibrations with low relative response factors (RRF), typically in the range of 0.08 to 0.11
- Data validators will often reject data with RRFs below 0.1

1,4-Dioxane as a Volatile



SW-846 8260B

- Method Performance/Recoveries

	<u>Water</u>	<u>Soil</u>
LCS	51%-129%	57%-126%
MS/MSD	43%-131%	39%-180%

1,4-Dioxane as a Volatile



SW-846 8260B

Advantages

- Can be used with limited sample volume
- Can be cost effective to acquire along with full 8260 list

Disadvantages

- High Limits
- Data rejected due to poor RRF

1,4-Dioxane as a Volatile



SW-846 8260B SIM

- Use of Selected Ion Monitoring (SIM) allows for better sensitivity for 1,4-dioxane
- Uses isotope dilution (1,4-dioxane d8) so compensates for poor purge efficiency
- Typically see RRFs of 0.9 to 1.1



1,4-Dioxane as a Volatile

SW-846 8260B SIM

- Better Sensitivity than 8260B

Water

MDL 0.2 ug/l

LOQ 0.4 ug/l

- Method Performance/Recoveries

Water

LCS 70% - 130%

MS/MSD 70% - 130%

1,4-Dioxane as a Volatile



SW-846 8260B SIM

Advantages

- Better sensitivity than regular 8260
- Data will more readily meet typical data quality objectives

Disadvantages

- Separate analytical run, so increases cost
- A modified 8260 – not all regulators accept
- Highly contaminated samples can cause interferences

1,4-Dioxane as a Semi-Volatile



SW-846 8270C

- Better sensitivity than typical 8260 analysis

	<u>Water</u>	<u>Soil</u>
MDL	1 ug/l	100 ug/kg
LOQ	5 ug/l	330 ug/kg

- Typically see RRFs of 0.5 to 0.8

- Method Performance/Recoveries

	<u>Water</u>	<u>Soil</u>
LCS	45% - 78%	31% - 55%
MS/MSD	39% - 73%	11% - 59%

1,4-Dioxane as a Semi-Volatile



SW-846 8270C

- Waters – solvent extraction with CH_2Cl_2

SW-846 3510C

- Soils – solvent extraction with 1:1 CH_2Cl_2 /Acetone

SW-846 3550B

SW-846 3546

1,4-Dioxane as a Semi-Volatile



SW-846 8270C

Advantages

- Better sensitivity than regular 8260
- Technique is more capable of dealing with difficult matrices
- Data will more readily meet typical data quality objectives
- Can be cost effective to acquire with full 8270 analysis

Disadvantages

- 1,4-dioxane elutes early in chromatogram – analyst experience
- Best recoveries are in 50% to 70% range

1,4-Dioxane as a Semi-Volatile



SW-846 8270C SIM

- Best sensitivity of the techniques discussed

	<u>Water</u>	<u>Soil</u>
MDL	0.05 ug/l	0.7 ug/kg
LOQ	0.2 ug/l	1.7 ug/kg

- Typically see RRFs of 0.5 to 0.8
- Method performance/recoveries similar to 8270 full scan

1,4-Dioxane as a Semi-Volatile



SW-846 8270C SIM

Advantages

- Best sensitivity of the techniques
- LOQ exceeds (lower than) current drinking water guidelines
- Data will readily meet typical data quality objectives

Disadvantages

- Separate analytical run, so increases the cost
- Some regulators will not accept 1,4-dioxane as a semi-volatile

Summary



- There are several approaches that can be used to meet new water quality standards without going to unique instrumentation
- The particular method and application that is best will depend upon
 - regulator acceptance
 - sample matrix considerations
 - overall project scope considerations (\$)

Questions

