

Method 0040 for Volatile Total Organics as Unspeciated Mass in Stack Gas Emissions

Sampling and Field Procedure for Volatile Unspeciated Mass from Method 0040 Samples in Stack Gas

Sample Name:	Stack Gas Volatile Unspeciated Mass
Sampler:	Stack sampling specialists
Process Sample Location:	Stack Port
Sampling & Health & Safety Equipment:	Sampling and safety equipment is as follows: <ul style="list-style-type: none">▪ Tedlar[®] gas bags▪ Stack gas metering pump▪ Heated stack probe and condensate traps▪ "Lung Sampler" for bag sampling operation▪ Glass 40 ml volatile organic analysis (VOA) vials with screw-top caps and Teflon[®]-lined septa▪ Safety glasses or face shield▪ Gloves and other safety equipment as required
Sample Collection Frequency:	Continually integrated sample collected for approximately three (3) hours from the stack for each integrated sampling run.
Sampling Procedures:	<p>Collect an integrated sample of approximately 30 liters in a clean, new Tedlar[®] bag using an appropriate sampling rate.</p> <p>A standard Method 0040 stack gas sampling train is assembled and leak checked at the stack sampling location. A new 40 liter Tedlar[®] Bag is placed in the sampling chamber and an initial leak check of the sampling system is conducted. Ice water is circulated through the gas sample condenser, and the condensate trap is submerged into the system ice bath. The ice bath will be maintained at approximately 4°C. The sampling rate will be maintained for a total sampling volume of approximately 30 liters. The collected bag sample is to represent an integrated stack gas collection being taken for a minimum of a two (2) hour period, and up to three (3) hours while the companion organics trains (i.e. semivolatiles, PAHs, volatiles, dioxin and furans) are being operated. When the final sample volume is achieved, a final leak check of the train will be performed and the train will be prepared for sample retrieval from the train.</p>

Volatile Condensate Handling - The condensate trap will be removed from the sampling train and its aqueous contents will be transferred to either a 20 mL or a 40 mL VOA vial, whichever is appropriate in terms of volume and requiring the least amount of organic-free water to top off the vial, and provide a zero headspace volatiles sample.

Sample Preservation:

Tedlar[®] Bag Sample Preservation and Handling - The Tedlar[®] Bag is stored in a Coleman type "cooler" equipped with an adjustable heat source. The bag should be stored in the heated storage chest at approximately 60°C. Bags will be stored in the dark and will not be allowed to chill at any time. The holding time for volatile unspiciated mass bag samples is six (6) hours.

Volatile Unspiciated Mass Condensates will be stored on ice at approximately 4°C until analyzed at the performing laboratory. The holding time for condensates is 14 days from collection. Sodium Thiosulfate (Na₂S₂O₃) should be added to the condensate samples as a preservative against residual chlorine. A 0.008% solution of Na₂S₂O₃ should be used.

Quality Assurance:

One (1) Tedlar[®] bag will be collected from each run. All sample analyses will be analyzed in duplicate and the average result used for the final result. One (1) train blank and one trip blank will be collected for each test condition. A field spike and a field spike duplicate will be prepared and analyzed for each test condition.

A D.I. water condensate rinse will be collected with the train blank, and a D.I. water trip blank will be collected one time during each test condition. Tedlar[®] bags may not be reused after the introduction of stack gas. Unused bags are required, and all bags are required to be tested for leaks and background contamination.

Method References:

Method 0040 - Sampling of Principal Organic Hazardous Constituents from Combustion Sources Using Tedlar[®] Bags. Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

EPA Guidance for Total Organics, Second Edition, Draft (EPA Report Number and Publication Date were not identified on the draft report): Appendix E: "EPA Revised Method 0040 - Sampling of Principal Organic Hazardous Constituents from Combustion Sources Using Tedlar[®] Bags".

Analysis Procedure for Volatile Unspeciated Mass from Method 0040 Samples in Stack Gas

Sample Name (Matrix):	Volatile Unspeciated Mass (Carbon 1 - Carbon 7) Method 0040 Tedlar [®] Bags Method 0040 Condensates
Sample Holding Times:	Tedlar [®] Bags - 6 hours Condensates - 14 days
Analysis Procedures:	<p>A GC equipped with an FID detector is set up at the CPT test site for the analysis of the Tedlar[®] bag samples. Calibration is completed using methane through heptane standards. Bag samples are introduced into the gas sample bags followed by introduction into the column. The total unspeciated mass is calculated relative to propane (C-3) and reported as propane.</p> <p>Condensate samples are analyzed at the fixed base laboratory by direct injection on a GC equipped with an FID.</p>
Method References:	<p>Method 0040 - "Sampling of Principle Organic Hazardous Constituents from Combustion Sources Using Tedlar[®] Bags". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.</p> <p>Method 5030B - "Purge and Trap for Aqueous Samples". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.</p> <p>Method 8015B - "Nonhalogenated Organics Using GC/FID". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.</p>

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SAMPLE PRESERVATION AND HOLDING TIMES

All samples requiring refrigeration should be placed on ice (when required for preservation) in coolers during and after sampling and will be stored at a temperature of approximately 4°C until analyzed. In addition to cooling all samples that require low temperature preservation, chemical preservatives should be used, as required, in samples for specific analyses according to EPA protocols. The holding times and preservation techniques are either those recommended in Title 40 CFR Section 136.3, Table 11, "Required Containers, Preservation Techniques, and Holding Times," or those presented by EPA in Table 3-1 of the *Handbook - Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration* (EPA-625/6-89-023).

Measurement	Matrix	Preservation ^a	Holding Time ^b
Volatiles (including unspciated mass)	Tedlar [®] bags	Do not chill, Warm to ~60°C	6 hours
	Condensate and aqueous liquid samples	Chill with ice 4 °C ± 2°C, 0.008% Na ₂ S ₂ O ₃	14 days to analysis

Notes:

- ^a CPT samples requiring refrigeration will be preserved on ice from the time of collection through delivery to the analytical laboratory.
- ^b Holding times are calculated from the date of collection.

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Recommended Quality Measurements for a 3 Run CPT

Analytical Parameter (Analysis)	Sample Name or Type	Total No. of Field Samples	Analytical Procedure Description (Method)	Laboratory QC Measurement Type	Frequency of Applied QC Measurement Type	Total No. of Laboratory QC Measurements	Field QC Measurement Type	Total No. of Field QC Samples	Total No. of Laboratory Analyses ^a
Volatile Unspeciated Mass (Carbon-1 through Carbon-7)	Method 0040 Tedlar [®] Bags	3	GC/FID (Modified SW-0040), SW-8015B	Field Spikes	Two field spikes per test condition	2 bags per test condition	Train blank	1 bag	7
				Duplicate Analyses	Every Tedlar [®] bag	7	Trip blank	1 bag	
	Method 0040 Condensate	3	Purge and trap GC/FID (SW-5030B, SW-0040, SW-8015B)	Matrix Spike	One blank spike and blank spike duplicate per test condition	2	Trip blank	1	7
						Train blank	1		

^a Total laboratory analyses includes all field samples collected and all laboratory and field QC samples that are analyzed. This number may not be calculated easily by adding the totals from the columns above; however, the total number presented represents the required total analyses for the sample and quality assurance analytical program.

^b Surrogate spikes will be applied to all samples including matrix spikes, duplicates, and blank analytical aliquots.

Recommended Analytical Quality Control Checks, Frequencies, Target Acceptance Criteria, and Corrective Action

Parameter/Method	QC Check	Frequency	Target Criteria	Corrective Action
Tedlar® Bag Samples for Total Organics (Method SW-0040 and Guidance for Total Organics)	Initial Calibration (minimum five (5) standards in duplicate)	Prior to analysis	Precision of duplicate standards as %Difference <ul style="list-style-type: none"> • %D ≤ 5% Fit of standard curve <ul style="list-style-type: none"> • Correlation coefficient ≥ 0.995 	(1) Correct problem (2) Repeat initial calibration
	Continuing Calibration Verification (CCV) standards in duplicate	Daily, and after every ten (10) samples	Accuracy <ul style="list-style-type: none"> • %Recovery: 90 – 110% Precision of duplicate standards as %Difference <ul style="list-style-type: none"> • %D ≤ 5% 	(1) Recalibrate instrument (2) Reanalyze affected samples if possible, or flag data and discuss in final report
	Laboratory Method Blank	Once per sample batch (maximum 20 samples)	Target analyte concentrations <ul style="list-style-type: none"> • Concentrations < Reporting Limit (RL) 	(1) Flag data associated with method blanks (2) Discuss in final report
	Field Spike/ Field Spike Duplicate (FS/FSD)	Once per test series (maximum 20 samples)	Accuracy <ul style="list-style-type: none"> • %Recovery: 70 – 130% Precision (if applicable) <ul style="list-style-type: none"> • RPD ≤ 35% 	(1) Flag FS/FSD data (2) Discuss in final report

Parameter/Method	QC Check	Frequency	Target Criteria	Corrective Action
Volatile Condensates for Total Organics (Methods SW-0040 and Modified SW-8015B, Guidance for Total Organics)	Initial Calibration (minimum five (5) standards)	Prior to analysis	Fit of standard curve <ul style="list-style-type: none"> Correlation coefficient ≥ 0.995 	(1) Correct problem (2) Repeat initial calibration
	Continuing Calibration Verification (CCV)	Daily, and after every ten (10) samples	Accuracy <ul style="list-style-type: none"> %Recovery: 85 – 115% 	(1) Repeat CCV (2) If still unacceptable, recalibrate instrument (3) Reanalyze affected samples
	Laboratory Method Blank	Once per sample batch (maximum 20 samples)	Target analyte concentrations <ul style="list-style-type: none"> Concentrations < Reporting Limit (RL) 	(3) Flag data associated with method blanks (4) Discuss in final report
	Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	Once per digestion batch (maximum 20 samples)	Accuracy <ul style="list-style-type: none"> %Recovery: 70 – 130% Precision (if applicable) <ul style="list-style-type: none"> RPD $\leq 35\%$ 	(1) Flag data (2) Discuss in final report

***SUMMARY OF FIELD QUALITY CONTROL SAMPLE REQUIREMENTS
 COMPREHENSIVE PERFORMANCE TEST - 3 RUNS***

Sample	QC Sample Type	Frequency	QC Sample Total
Volatile Unspeciated Mass (Method 0040)	Train Blanks	One per test condition	1
	Trip Blanks	One per test condition	1
	Condensate Trip Blank	One per sample shipment	1
	Field Spike/Field Spike Duplicate	One set per test condition	2

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Method 0040 Recommended Sample Collection Methods, Frequency, and Containers for a 3 Run CPT

Sample Name (Matrix)	Analysis	Type of Container(s)	Sampling Method	Sampling Frequency	Test Samples	Field QC Samples	Total Field Samples Collected
Volatile Unspeciated Mass (Tedlar® Bags) C₁-C₇	Volatile unspeciated mass	Tedlar® bags	Modified Method 0040 ^a	1 Tedlar® bag per run taking 2 to 3 hours per bag.	3	1 trip blank, 2 field spikes, 1 train blank	7
Volatile Unspeciated Mass (Condensate) C₅-C₇	Volatile unspeciated mass	40-mL volatile organic analysis vial	Modified Method 0040 ^a	Collect the Method 0040 condensate at the end of each run.	3	1 train blank, 1 trip blank	5

^a Method 0040 is appropriate for sampling stack gases for principal organic hazardous constituents. Taken from “Sampling of Principal Organic Hazardous Constituents from Combustion Sources Using Tedlar® Bags”. Taken from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, (SW-846), Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996). U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. 20460. Also, see “Guidance for Total Organics - Final Report”, prepared by Radian Corporation, Research Triangle Park, North Carolina for National Exposure Research Laboratory, Air Measurements Research Division Methods Branch, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, March 1996.