Effective Date: 10/04/2016
Review/Revision Date: 10/03/2016
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VO-008-1.5

ROUTINE INSTRUMENT SETTINGS FOR VOLATILES ANALYSIS

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ROUTINE INSTRUMENT SETTINGS FOR VOLATILES ANALYSIS

1. SCOPE AND APPLICATION

1.1. The settings for analytical instrumentation used within the FDEP volatiles (VOC) laboratory are provided in this Standard Operating Procedure (SOP). The values are guidance and may vary somewhat from instrument to instrument in order to optimize performance. Once established, the settings must not be altered for a particular instrument calibration. If the settings are altered or the equipment is serviced, then a new calibration must be prepared.

1.2. FDEP Test IDs and equivalent EPA methods covered by this SOP:

Test	Matrix	EPA Method Reference and Description	Instrument Set-up
W-VOC-MS	Water	Method 624 analysis, non-preserved	Refer to Section 2.1.1
W-VOC-MS-A	Water	Method 624 analysis, acid-preserved	Refer to Section 2.1.1
W-VOC-MS-R	Water	Method 8260C analysis, non-preserved	Refer to Section 2.1.1
W-VOC-MS-R-A	Water	Method 8260C analysis, acid-preserved	Refer to Section 2.1.1
S-VOC-MS	Soil	Method 8260C analysis, Sediment	Refer to Section 2.1.2
WAS-VOC-MS	Waste	Method 8260C analysis, Waste	Refer to Section 2.1.1
TCLP-VOC	Waste	Method 8260C analysis of TCLP Extract	Refer to Section 2.1.1
SPLP-VOC	Waste	Method 8260C analysis of SPLP Extract	Refer to Section 2.1.1
T-VOC-MS	Tissue	Method 8260C analysis, Tissue	Refer to Section 2.1.3
W-8015	Water	Method 8015C analysis, acid preserved	Refer to Section 2.1.4
W-8260-AA	Water	Method 8260C analysis, non-preserved	Refer to Section 2.1.4
W-ETHYL-AC	Water	Method 8260C analysis, acid-preserved	Refer to Section 2.1.4

1.3. Refer to the specific analytical method SOPs for details regarding sample preparation and analysis, apparatus/equipment, and reagents/chemicals.

SOP **VO-002** Analysis of VOCs in Water by EPA Method 624/8260C
SOP **VO-003** Analysis of VOCs in Sediment, Tissue, and Waste by EPA 8260C
SOP **VO-004** Analysis of Polar Organic Compounds in Sediment and Waste
SOP **VO-005** TCLP and SPLP Sample Preparation
SOP **VO-006** Analysis of Polar Organic Compounds in Water

2. VOC INSTRUMENT SETTINGS

The following settings are for an Agilent 7890 Gas Chromatograph (GC) and 5975C Inert Mass Spectrometer (MSD) equipped with a Tekmar Purge & Trap System (or similar equipment).

The operation, cleaning, and scheduled maintenance procedures prescribed by the equipment manufacture are followed as provided in the Operator's Manuals. Maintenance contracts, provided by the manufacture, are purchased for the Gas Chromatograph/Mass Spectrometer Systems. Documentation of cleaning, maintenance, or system modifications is recorded in a maintenance logbook which accompanies each instrument system.

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2.1. Purge & Trap Setting - Tekmar Atomx

2.1.1. EPA 624/8260C Water Method

• Helium Purge Flow Rate: 37 mL/min	• Drypurge Time = 2 min
• Trap Type: VOCARB 3000 or Trap #9	• Drypurge Temp = 20ºC
• Sample Volume: 15 mL	• Drypurge Flow = 100 mL/min.
• Sample Heater = On	• GC Start = Start of Desorb
• Sample Sparger Temp = 37ºC	• Desorb Preheat = 245ºC
• Sample Preheat Time = 1.0 min.	• Desorb Time = 1.0 min.
• Line Temp = 125ºC	• Desorb Temp = 250ºC
• Valve Temp = 125ºC	• Desorb Flow = 100 mL/min.
• Purge Ready Temp = 35ºC	• Sample Drain = On
• Purge Temp = 30ºC	• Bake Time = 15 min.
• Sample Mount Temp = 90ºC	• Bake Temp = 260ºC
• Condenser Line Temp = 40ºC	• Condenser Bake Temp = 210ºC
• Condenser Des Temp = 20ºC	• Standby Flow = 10 mL/min.
• Purge Time = 11 min.	

2.1.2. EPA 8260C Sediment Heated Purge Method

• Helium Purge Flow Rate: 37 mL/min	• Soil Vial Mix Speed: Fast
• Trap Type: VOCARB 3000 or Trap #9	• Presweep Time = 0.25 min
• Water Volume: 5 mL	• Sweep Water Time = 0.25 min
• Sparger Sample Heater = On	• Sweep Water Flow = 100 mL/min
• Sparger Temp = 40ºC	• Drypurge Time = 3.0 min
• Sample Vial Temp = 55ºC	• Drypurge Temp = 20ºC
• Sample Preheat Time = 1.0 min.	• Drypurge Flow = 100 mL/min.
• Line Temp = 130ºC	• GC Start = Start of Desorb
• Valve Temp = 130ºC	• Desorb Preheat = 245ºC
• Purge Ready Temp = 40ºC	• Desorb Time = 2.0 min.
• Purge Temp =20ºC	• Desorb Temp = 250ºC
• Sample Mount Temp = 90ºC	• Desorb Flow = 100 mL/min.
• Condenser Ready Temp = 45ºC	• Sample Drain = On
• Condenser Purge Temp = 20ºC	• Bake Time = 10 min.
• Condenser	• Bake Temp = 260ºC
• Purge time = 12 min.	• Condenser Bake Temp = 240ºC
• Purge Flow = 40 mL/min	• Bake Flow = 300 mL/min
• Water Heater Temp = 90ºC	• Drain Flow = 300 mL/min
• Soil Valve Temp = 105ºC	• Standby Flow = 10 mL/min.

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2.1.3. EPA 8260C Tissue Heated-Purge Method

The tissue heated purge settings were established using a Tekmar Atomx instrument that incorporates both autosampler and purge & trap functions into one sample introduction system.

• Helium Purge Flow Rate: 37 mL/min	• Soil Vial Mix Speed: Fast
• Trap Type: VOCARB 3000 or Trap #9	• Presweep Time = 0.25 min
• Water Volume: 5 mL	• Sweep Water Time = 0.25 min
• Sparger Sample Heater = On	• Sweep Water Flow = 100 mL/min
• Sparger Temp = 40ºC	• Drypurge Time = 3.0 min
• Sample Vial Temp = 55ºC	• Drypurge Temp = 20ºC
• Sample Preheat Time = 1.0 min.	• Drypurge Flow = 100 mL/min.
• Line Temp = 130ºC	• GC Start = Start of Desorb
• Valve Temp = 130ºC	• Desorb Preheat = 245ºC
• Purge Ready Temp = 40ºC	• Desorb Time = 2.0 min.
• Purge Temp =20ºC	• Desorb Temp = 250ºC
• Sample Mount Temp = 90ºC	• Desorb Flow = 100 mL/min.
• Condenser Ready Temp = 45ºC	• Sample Drain = On
• Condenser Purge Temp = 20ºC	• Bake Time = 10 min.
• Condenser	• Bake Temp = 260ºC
• Purge time = 12 min.	• Condenser Bake Temp = 240ºC
• Purge Flow = 40 mL/min	• Bake Flow = 300 mL/min
• Water Heater Temp = 90ºC	• Drain Flow = 300 mL/min
• Soil Valve Temp = 105ºC	• Standby Flow = 10 mL/min.

2.1.4. EPA 8260C/8015C Water Heated-Purge Method (*- trap options and temperatures)

• Helium Purge Flow Rate: 37 mL/min	• Drypurge Time = 2 min
• Trap: VOCARB 3000 or Trap 1A *	• Drypurge Temp = 20ºC
• Sample Volume: 15 mL	• Drypurge Flow = 100 mL/min.
• Sample Heater = On	• GC Start = Start of Desorb
• Sample Sparger Temp = 60ºC	• Desorb Preheat = 245ºC or 210ºC *
• Sample Preheat Time = 1.0 min.	• Desorb Time = 1.0 min.
• Line Temp = 125ºC	• Desorb Temp = 250ºC or 220ºC *
• Valve Temp = 125ºC	• Desorb Flow = 100 mL/min.
• Purge Ready Temp = 35ºC	• Sample Drain = On
• Purge Temp = 30ºC	• Bake Time = 5 min.
• Sample Mount Temp = 90ºC	• Bake Temp = 260ºC or 230ºC *
• Condenser Line Temp = 40ºC	• Condenser Bake Temp = 230ºC
• Condenser Des Temp = 20ºC	• Standby Flow = 10 mL/min.
• Purge Time = 11 min.	

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2.2. GC/MS Settings - Agilent 7890 Gas Chromatograph and 5975C Inert Mass Spectrometer

2.2.1. Analytical Column: J & W DB624, 30 m x 0.25 mm ID x 1.4 µm film thickness, or equivalent.

2.2.2. Gas Chromatograph Program Settings

The same GC program is used for both routine water, tissue, and sediment analysis.

• Initial Temperature = 35°C	• Injector Temperature = 150°C
• Initial Time = 3 min.	• Ramp 2 Rate =19°C/min
• Ramp 1 Rate = 6°C/min.	• Ramp 2 Final Temp = 220°C
• Ramp 1 Final Temp = 100°C	• Ramp 2 Final Time = 0 min.
• Ramp 1 Final Time = 0 min.	• Post Run =70°C
• Carrier Gas Flow Rate = 1.2 ml/min	• Post Run Flow = 1.0 mL/min
• Split Mode Ratio = 30:1	• Gas Saver On at 20 mL/min

2.2.3. Mass Spectrometer Conditions

2.2.3.1. Full Scan Water and Sediment Methods

MSD Quad Temp = 150°C	Filament Delay = 1 min
MSD Source Temp = 270°C	Acquisition Time = 20 min
MSD Transfer Line Temp = 150°C	Scan Range: 40-260 amu
MSD Scan Mode = EI	Scan Rate: 6.0 scans/sec
MSD Acquisition Mode = Full Scan	

2.2.3.2. Selected Ion Monitoring (SIM) Tissue Method

MSD Quad Temp = 150°C	Filament Delay = 1 min
MSD Source Temp = 270°C	Acquisition Time = 20 min
MSD Transfer Line Temp = 150°C	Scan Range: specific selected mass ion
MSD Scan Mode = EI	Scan Rate: 6.0 scans/sec
MSD Acquisition Mode = SIM	

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