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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:

W-VOC-MS -	EPA Method 624 analysis, non-preserved water sample
W-VOC-MS-A -	EPA Method 624 analysis, acid-preserved water sample
W-VOC-MS-R -	EPA Method 8260C analysis, non-preserved water sample
W-VOC-MS-R-A -	EPA Method 8260C analysis, acid-preserved water sample
S-VOC-MS -	EPA Method 8260C analysis, preserved sediment sample
WAS-VOC-MS -	EPA Method 8260C analysis, preserved waste sample
TCLP-VOC -	EPA Method 8260C analysis of TCLP Extract
SPLP-VOC -	EPA Method 8260C analysis of SPLP Extract
T-VOC-MS -	EPA Method 8260C analysis, preserved tissue sample

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.

2.1. <u>Purge & Trap Setting - Tekmar Stratum or Atomx</u>

2.1.1. EPA 624/8260C Water Method

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• Helium Purge Flow Rate: 37 mL/min	• Drypurge Time = 2 min
• Trap Type: VOCARB 3000 or Trap #9	• Drypurge Temp = 20ºC
• Sample Volume: 5 or 20 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.	

2.1.1. EPA 8260C Sediment Heated Purge Method

All the conditions are the same as the water method except the following setting. The flow conditions within the P&T are much less because the primary flow is coming from the autosampler in the soil method.

• Purge Flow = 3 mL/min
• PrePurge Time = 0.50 min
• Drypurge Time = 1.0 min.
• Drypurge Flow = 3 mL/min.

2.1.2. 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 = 40 ºC	• 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

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• 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 = 105 ºC	• Standby Flow = 10 mL/min.

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 = 35ºC	• Injector Temperature = 150ºC
• Initial Time = 3 min.	• Ramp 2 Rate =19ºC/min
• Ramp 1 Rate = 6ºC/min.	• Ramp 2 Final Temp = 220ºC
• Ramp 1 Final Temp = 100ºC	• Ramp 2 Final Time = 0 min.
• Ramp 1 Final Time = 0 min.	• Post Run =70ºC
• 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 = 150ºC	Filament Delay = 1 min
MSD Source Temp = 270ºC	Acquisition Time = 20 min
MSD Transfer Line Temp = 150ºC	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 = 150ºC	Filament Delay = 1 min
MSD Source Temp = 270ºC	Acquisition Time = 20 min
MSD Transfer Line Temp = 150ºC	Scan Range: specific selected mass ion

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MSD Scan Mode = EI	Scan Rate: 6.0 scans/sec
MSD Acquisition Mode = SIM	

2.3. P&T Autosampler – 4552 Analytical/Arcon

These settings are applicable for the autosampler only and do not apply to the purge & trap.

2.3.1. 624/8260C Water Method:

Standard: Yes
Sample Volume: 5 or 20 mL
Dilution = 1
Rinses: 3 times
Rinse Volume: 7 or 25 mL
Stir Time: 0 min.
Settle Time: 0 min.
Syringe Flush: 2 times
Start Delay = 0.0

2.3.2. 8260C Heated Purge Sediment Method:

Standard: Yes	Desorb Time: 1 min.
Flow Rate = 40 mL/min.	Syringe Flush: 2 times
Line Heat = 125^{o}C	Sample Volume: 5 ml
Sample Heat = 40^{o}C	Dilution = 1
PreHeat = 0.50 min.	Rinses: 1 time
Water volume = 5 ml	Rinse Volume: 7 mL
Pre-Purge = 0.0 min	Settle time: 0 min.
Purge Time = 11 min.	Start Delay = 0.0
Stir Speed: High	

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