Agilent CrossLab
From Insight to Outcome

Consumable Workflow Ordering Guide

Semi-Volatile Organic Compounds (SVOC) in Water Using Helium or Hydrogen Carrier Gas

Semi-volatiles compounds encompass a wide range of bases, neutrals and acids, phenols, and polycyclic hydrocarbons (PAHs) that demonstrates higher boiling points, usually greater than that of water with correspondingly low vapor pressure. Sources of these compounds include pesticides and herbicides, ingredients in cleaning agents, and industrial solvents, to name a few. These contaminants are important to monitor, especially in water supplies as they are bio-accumulative and hazardous to health.

Challenges of SVOC analysis

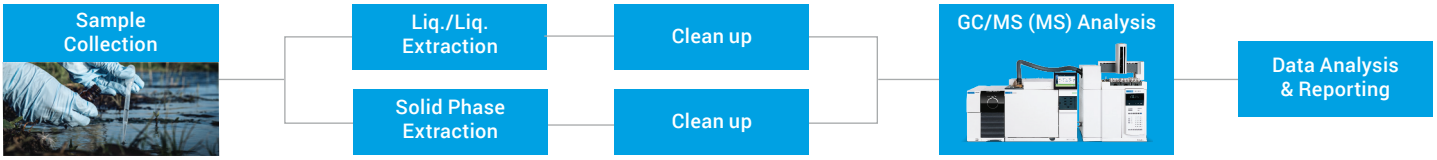
Reactive Analytes

Environmental analysis for SVOCs focuses on the extraction, identification, and quantitation of contaminants. One of the challenges of SVOC analysis stem from the highly active nature of these analytes. Many reactive SVOCs interact with hot active surfaces of the GC flow path and breakdown, exhibiting poor peak response and asymmetrical peak shape while resulting in poor column lifetime and frequent system maintenance. Laboratories risk large amounts of system downtime for maintenance and recalibration for these tests, by not using products specifically designed for this type of analysis.

Agilent J&W DB-UI 8270D GC column is inert and has its chemistry optimized to deliver excellent peak shape for trace-level analysis of active SVOC. Agilent Ultra Inert supplies minimize analyte degradation and enable accurate, sensitive, and reliable trace-level multi-residue analysis is required according to EPA (525, 625, and 8270) guidelines. While Agilent J&W DB-5ms UI and HP-5ms UI columns have been historically used in some labs, they do not undergo the quality assurance tests with SVOC analytes, which the DB-UI 8270D GC columns do during the manufacturing process.

Carrier Gas Supply Issues

Recent pressure on the helium (He) supply has required organizations to actively investigate hydrogen (H_2) carrier gas, but most GC/MS and GC/MS/MS analyses have reduced sensitivity and hydrogenation or dechlorination in the existing mass spectrometry products. New advances in mass spectrometer design have reduced hydrogenation and dechlorination reactions in the source. The Agilent HydroInert source retains the ability to analyze a wide calibration range, for some compounds from 0.02 to 100 µg/mL, and meet the U.S. Environmental Protection Agency (EPA) method 8270 calibration criteria when using H_2 carrier gas.



Molecular Weight Discrimination

Another challenge of analyzing semi-volatile analytes is molecular weight discrimination. This can occur if:

a. The injection port temperature is set too low (<300 °C) and there is incomplete sample vaporization in the inlet

b. The splitless injection hold time is not optimized to effectively transfer all the samples onto the head of the analytical column or

c. The wrong inlet liner is chosen. Chromatographically, this will be observed as a lower response of the higher molecular weight SVOCs

Recommended parameters to overcome molecular weight discrimination

a. Injection volume: 1–2 µL

b. Inlet temperature: 300–320 °C

c. Purge time activation: 45–90 seconds splitless

d. 4 mm splitless inlet liners - fritted or glass wool

e. Pulsed Splitless at 20 to 50 psi for 0.9 min to transfer high boiling SVOCs onto the column. “Cold trapping” on the liquid phase is often applied for higher molecular, higher boiling analytes such as PAHs for splitless/PTV/MMI type of injections. An initial oven temperature of 75 °C usually provides good quality peak shapes for a variety of sample solvents

f. Minimize inlet (and system) dwell time by operating at higher column flows
0.18 mm: 1.5 mL/min He
0.25 mm: 2.0 mL/min He
Note: Although 0.18 and 0.25 mm id GC columns can handle higher flow rates, this will lead to decreased MS sensitivity. Exceeding 1.5 mL/min is not recommended for the HES source

Best practices for optimizing a GC/MS for SVOC analysis

- Use retention gaps and/or inlet backflushing to eliminate sample carryover, reduce maintenance, and cut the analysis cycle times
- Use of Ultra Inert sintered fritted liners provide a significant barrier to challenging matrix while minimizing the risk of formation of new active sites from glass wool breakage or movement of glass wool due to pressure changes in the inlet (Application note: 5994-0953)
- Perform the analysis in constant flow mode
- Keep heated zones well insulated and hot to reduce the potential for system cold spots and resultant signal loss
- Keep MS transfer line and ion source at temperatures above 300 °C. Too low temperatures will result in PAH tailing
- While 0.25 mm id is more commonly used, a 0.18 mm id allows for faster analysis time with no loss in resolution (Application note: 5991-0250EN)
- Use Agilent JetClean to substantially reduce the need for manual source cleaning especially with high-matrix samples. Continuous cleaning of the source with hydrogen (0.33 mL/min) has been demonstrated to significantly improve calibration linearity and precision of response over time for SVOC analysis (JetClean e-book: 5991-9517EN)
- Allow standards to come to room temperature before diluting or prepping calibration mixtures since heavier molecular weight SVOCs can fall out of solution during refrigerated storage
- Use a 9 mm extractor lens to minimize the surfaces available for deposition of the SVOCs – this works best for phenols, PAHs, and other active compounds, providing better signal-to-noise ratios and linear range, and resulting in less tailing (Application note: 5994-0350). The 6 mm extractor lens works better for less active analytes and may be used to balance MDL with linear range/tailing

Best practices for transitioning from helium to hydrogen

- Use the Hydroinert source for Inert Plus mass spectrometer
- Stainless steel tubing is recommended. If only copper tubing is available use fresh copper lines.
- Always use gas filters, especially with H_2 generators.
- Decrease inlet temperature when working with sensitive compounds or use cold splitless injection with multimode inlet.
- Hydrogen viscosity is lower than helium, causing lower inlet pressures. It is advisable to:
  - Change column dimensions to 0.18 mm IDs, or
  - Increase the carrier gas flow rate
- If changing to 0.18 mm ID columns, the injection mode may need to be changed to a split injection to avoid column overload.
- Optimal carrier gas flows depend on the pump, as pumping capacity for hydrogen is lower than helium.
  - Optimal range: 0.5 to 1.0 mL/min
  - Maximum recommended flow rate for turbo pump: 2.0 mL/min
- Use constant flow methods.
- For GC/MS/MS systems running hydrogen, no quench gas (helium) is used. Please remove the helium plumbing from the back of the electronics pressure control module (EPC) and set the quench gas to 0.00 mL/min in any GC/MS/MS method.

Parameter	Value
Injection Volume	1 µL
Multimode Inlet	Split 20:1 250 °C (hold 0.3 min) ramp 200 °C/min to 350 °C (hold for run length) Postrun: 350 °C/min with 100 mL/min split flow
Column Temperature Program	40 °C (hold 0 min), 30 °C/min to 320 °C (hold 2 to 2.7 min*) Post run: 320 °C hold for 2 min
Carrier Gas and Flow Rate	H_2 at 1.2 mL/min**, constant flow
Transfer Line Temperature	320 °C
Ion Source Temperature	300 °C
Quadrupole Temperature	150 °C
Collision Gas and Flow Rate	Nitrogen, 1.5 mL/min
Quench Gas	No quench gas is used with H_2 carrier gas
EMV Mode	Gain factor
Gain Factor	1 (optimized for each system)
Scan Type	dMRM

Table 1. Instrument conditions for 8890 GC/7000E inert plus triple quadrupole GC/MS with Agilent HydroInert source.

Parameter	Value
Injection Volume	1 µL
Inlet	230 °C Split 10:1
Column Temperature Program	40 °C (0 min hold) 30 °C/min to 320 °C (hold 2 min)
Carrier Gas and Flow Rate	H_2, 1.2 mL/min constant flow
Transfer Line Temperature	320 °C
Ion Source Temperature	300 °C
Quadrupole Temperature	150 °C
Scan	35 to 500 m/z
Tune	etune.u
Gain Factor	0.5
Threshold	0
A/D Samples	4

Table 2. Instrument conditions for Agilent 8890 GC/5977 Inert Plus GC/MSD.

Easy selection and ordering information

This guide provides recommendations for Agilent products by regulatory method, so you can find what you're looking for quickly. To add items to your *Favorite Products** list at the Agilent online store, simply click the *MyList* links in each header below. Then, enter the quantities for the products you need. Your list will remain under *Favorite Products* for your use with future orders.

If this is your first time using *Favorites Products* you will be asked to enter your email address for account verification. If you have an existing Agilent account, you will be able to log in. If you don't have a registered Agilent account, you will need to register for one. This feature is valid only in regions that are e-commerce enabled. All items can also be ordered through your regular sales and distributor channels.

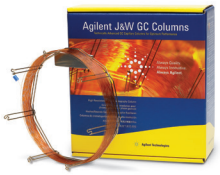
Standards*

Description	Part Number	EPA Method
SVOC-EPA 8270D/E standards	various (view MyList)	EPA ground-water standards
SVOC-EPA 525 standards	various (view MyList)	EPA drinking water standards
SVOC-EPA 625 standards	various (view MyList)	EPA wastewater standards

Not available in all countries. Please contact your local sales representative for availability. For more information on the full Agilent SVOC standards portfolio visit www.agilent.com/chem/standards

MyList of GC Columns

Description	Part Number	EPA Method
DB-UI 8270D, 30 m x 0.25 mm x 0.25 µm (recommended)	122-9732	8270D/E, 525.2, 525.3, 625.2
DB-UI 8270D, 20 m x 0.18 mm x 0.18 µm	121-9723	8270 D/E, 525.2, 525.3, 625.2 and 8270E H_2 GC/TQ
DB-5ms UI, 30 m x 0.25 mm x 0.25 µm	122-5532UI	525.2, 525.3, 625.2
DB-5ms UI 20m x 0.18 mm x 0.36 µm	121-5523UI	8270 D/E H_2 GC/MS

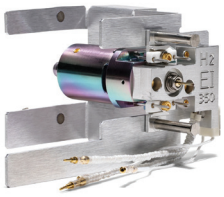


MyList of GC Supplies

Description	Part Number
BTO inlet septa, 11 mm, 50/pk	5183-4757
BTO inlet septa, 11 mm, 100/pk	5183-4757-100
Liner UI 4 mm glass-bottom frit, taper, 1/pk	5190-5112
Liner UI 4 mm glass-bottom frit, 5/pk	5190-5112-005
Liner Ultra Inert, low pressure drop, glass wool (recommended for H_2 carrier gas)	5190-2295
Ultra Inert gold seal, with washer, 1/pk	5190-6144
Ultra Inert gold seal with washer, 10/pk	5190-6145
Self-tightening column nut, collared, inlet	G3440-81011
Self-tightening column nut, collared, MSD	G3440-81013
Replacement collar for self-tightening nut	G3440-81012
Graphite vespel ferrules	5181-3323
ALS syringe, Blue Line, 10 µL, fixed needle, 23-26/42/cone, PTFE-tip plunger	G4513-80203

MyList of HydroInert Source for Transition to H_2 Carrier Gas

Description	Part Number
Hydroinert complete source assembly for 5977	G7078-67930
Hydroinert complete source assembly for 7000 TQ	G7006-67930
Hydroinert GC/MSD upgrade	5505-0083
Hydroinert GC/TQ upgrade	5505-0084
Stainless steel installation kit	19199S



MyList of MS Supplies

Description	Part Number
Filament	G7005-60061
Drawout plate, 9 mm, inert source (recommended)	G3440-20022
Extraction lens, 9 mm, extractor source (recommended)	G3870-20449
Drawout plate, 6 mm, inert source	G2589-20045
Extraction lens, 6 mm, extractor source	G3870-20448
9 mm Hydroinert extraction lens (recommended for H_2 carrier gas)	G7078-20909
Extraction lens insulator (recommended for H_2 carrier gas)	G3870-20445

MyList of Vials and Caps

Description	Part Number
Agilent A-Line certified amber (screw top) vials, 2 mL, 100/pk	5190-9590
2 mL screw top amber, write-on spot, deactivated, certified, 100/pk	5183-2072
Screwcaps, blue, certified, PTFE/silicone/PTFE septa, 100/pk	5182-0723
250 µL glass/polymer feet	5181-8872

MyList of Gas Filters

Description	Part Number
Gas Clean carrier gas kit for 7890	CP17988
Agilent big universal trap (recommended for H_2 carrier gas)	RMSH-2-SS
Gas Clean carrier gas kit for 8890 and 8860	CP179880
Gas Clean carrier gas purifier replacement cartridge	CP17973
Agilent Gas Clean purifier kit for carrier gas	CP17976

Sample preparation considerations for SVOC analysis

The sample matrix dictates the technique needed to successfully extract target contaminants or contaminant groups. Generally, an aqueous sample of a known volume is extracted with solvent or diluted with solvent. Techniques for extracting water-based samples include separatory funnel liquid-liquid extraction (LLE), continuous liquid-liquid extraction (CLE), solid-phase extraction (SPE), automated SPE (ASPE), and solid-phase microextraction (SPME).

For larger numbers of samples, Solid-phase microextraction (SPME) with automation results in less sample manipulation, decreases solvent consumption and reduces analysis time per sample. Agilent SPME Arrows, available on the PAL3 series RDI and RTC systems, have higher mechanical robustness and larger surface area capacity than their fiber counterparts, increasing trace level sensitivity, shortening extraction time, and increasing throughput. Both SPME fibers and arrows can be used for manual sampling.

MyList of Automated Sample Preparation Supplies

Description	Part Number
Agilent SPME Arrow PDMS 100 µm, 1.1 mm	5191-5862
Agilent SPME Arrow PDMS 100 µm, 1.5 mm	5191-5866
SPME Fiber PDMS 7 µm	5191-5870
SPME Fiber PDMS 30 µm	5191-5871
SPME Fiber PDMS 100 µm	5191-5872
Manual injection kit for SPME fiber and SPME arrow	5191-5877
PAL3 Alignment Ring (for manual injection)	G7371-67001

MyList of CTC/CombiPAL & SPME Headspace Supplies

Description	Part Number
Inlet liner Ultra Inert, splitless, straight, 2 mm id, for SPME Arrows	5190-6168
Inlet liner Ultra Inert, straight, 0.75 mm id, for SPME fiber	5190-4048
Sample loop, headspace, 1.00 mL, inert	G4556-80106
Sample probe, deactivated, for Agilent 7697A headspace sampler	G4556-63825
Headspace syringe CTC/CombiPAL, 1.0 mL	G6500-80107
Headspace syringe CTC/CombiPAL, 2.5 mL	G6500-80109
Headspace syringe CTC/CombiPAL, 5.0 mL	G6500-80111
Fused silica tubing, deactivated, 5 m, 0.32 mm, 0.43 mm od	160-2325-5
Ferrule, polyamide, graphite 1/32 inch, 5/pk	0100-2595
Fitting, internal reducer, 1/16 to 1/32 inch	0100-2594
Vial, crimp top, headspace, clear, certified, flat bottom, 10 mL, 100/pk	5182-0838
Vial, headspace, certified, crimp, clear, flat bottom, 20 mL, 100/pk	5182-0837
Vial, crimp top, headspace, amber, graduation marks and write-on spot, flat bottom, certified, 10 mL, 100/pk	5190-2287
Vial, crimp top, headspace, amber, flat bottom, certified, 20 mL, 23 mm x 75 mm, 100/pk	5067-0226
Cap, crimp, headspace, aluminum, PTFE/silicone septa, 20 mm, 100/pk	5183-4477
Screw cap, headspace, steel, magnetic cap, PTFE/silicone septa (top white, bottom blue), 18 mm, 100/pk	5188-2759

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