Sulfurs in SilcoCan™ Canisters

Long-Term Stability of Very Low-Level (1-20ppbv) Reactive Sulfurs

tech tip

Alternative Analytical Techniques

Trace level sulfur compounds also can be analyzed using Restek's Rt-XLSulfur™ packed or micropacked columns. The specially designed packing material is optimized for low ppbv sulfur compound analysis.

For an example chromatogram, visit our website and enter GC_PC00436 in the search function.

Introduction

Collection and measurement of sulfur-containing volatile organic compounds (VOCs), such as hydrogen sulfide, methyl mercaptan, ethyl mercaptan, and dimethyl disulfide in the atmosphere is very difficult because of their low concentrations and high reactivity. Sulfur VOCs can react not only with each other, but also with the vessels in which they are collected. This causes low recoveries. In Tedlar® bags, the stability of low-level (100ppbv) sulfur VOCs is poor, even within 24 hours of sampling.[1] Sulfur compounds react with the metal surface in electropolished canisters, so these canisters are unsuitable for collecting and storing low-level sulfur VOCs.[2]

When you need to perform sensitive air monitoring analyses, use SilcoCan™ canisters to collect and store samples. SilcoCan™ canisters, which feature a Siltek®-treated surface, offer superior storage stability. We evaluated the stability of sulfur VOCs in SilcoCan™ canisters at very low levels (1–20ppbv) for six days, under dry or humid conditions, to demonstrate the excellent ability of SilcoCan™ canisters to store low-level sulfur VOCs (Figure 1). Hydrogen sulfide, methyl mercaptan, and ethyl mercaptan rapidly degraded in electropolished canisters.

Analytical System

High resolution capillary gas chromatography (GC) in conjunction with sensitive, selective detectors such as sulfur chemiluminescence detectors (SCD) or flame photometric detectors (FPD) offers many advantages for trace analysis of sulfur VOCs. For this study, we used an Rtx®-1 capillary column, a Siltek® treated six-port Valco® valve, and a Siltek® treated 1mL sample loop and 1/16" sample pathway. A representative chromatogram for the sulfur compounds is shown in Figure 2.

three simple words...

Plus 1™
Exceeding your expectations in everything we do.

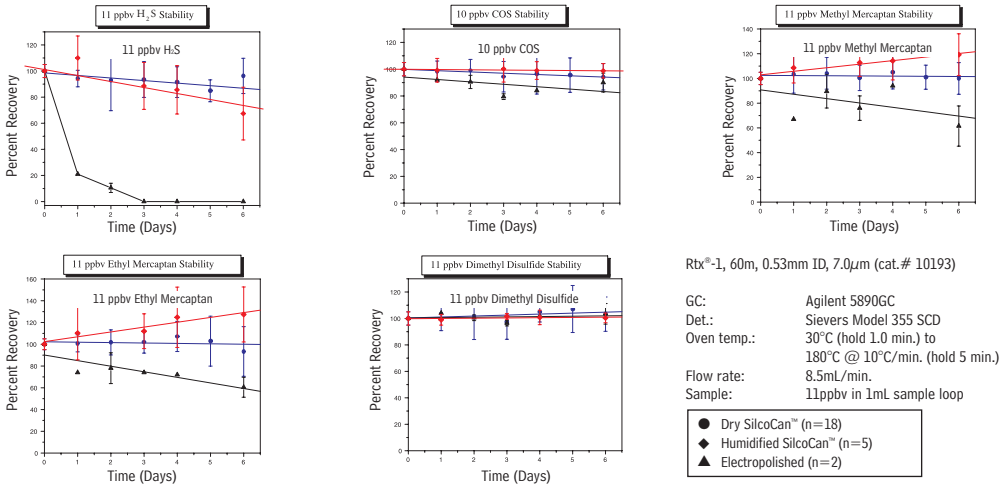
Innovation
Turning visions into reality™.

Execution
On-time delivery of products and services.

Restek's vision is to be the company that chromatographers trust by providing the highest quality, most innovative products and services throughout the world.

We will soon reach our goal of 100% employee ownership. As owners, our success depends on your success.

Figure 1 Stability data demonstrate ability of SilcoCan™ canisters to store low-level organic sulfur compounds in real-world conditions.



Standards: Dry standards were made by adding 2mL of a 100ppm stock sulfur standard to each precleaned and evacuated canister, then pressurizing to 30psig with ultra-pure nitrogen. The resultant concentrations are listed in Table 1. Humidified standards were made by injecting 100µL deionized water into the evacuated canisters prior to adding 2mL of stock standard. This produced 50% RH.

Figure 2 Rtx®-1 column is ideal for trace-level sulfur analysis.

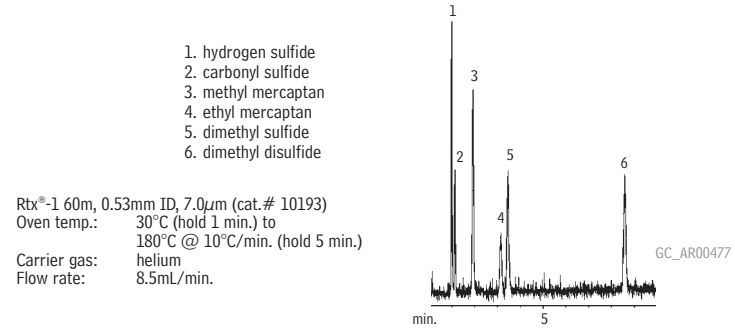


Table 1 Concentrations of sulfur compounds used in stability study.

Compound	Stock Conc. (ppmv)	Standard Conc. (ppbv)	Standard Conc. as Sulfur (ppbv)
hydrogen sulfide (H_2S)	105	11.51	10.83
carbonyl sulfide (COS)	98	10.74	5.73
methyl mercaptan (CH_3SH)	101	11.07	7.38
ethyl mercaptan (CH_3CH_2SH)	101	11.07	5.71
dimethylsulfide (CH_3SCH_3)•	99	10.85	6.81
dimethyldisulfide (CH_3SSCH_3)	100	10.96	7.46

• Internal standard

Stability

A 55ppbv reference standard (11ppbv each sulfur compound, dry standard) was analyzed three times each day for six days. Eighteen SilcoCan™ canisters and two electropolished canisters were used. The sulfur VOCs showed excellent stability in the SilcoCan™ canisters. The electropolished canisters allowed rapid degradation of hydrogen sulfide, methyl mercaptan, and ethyl mercaptan (Figure 1).

Humidity Effects

Five SilcoCan™ canisters that were used in the stability test were cleaned according to US Environmental Protection Agency (EPA) Compendium of Toxic Organic Method TO-14 and reused for the humidity study.[3] After adding 100µL of deionized H_2O to each canister, the relative humidity was 50%. Two mL of the stock sulfur standard was added to each canister and aliquots were analyzed over six days (Figure 1). The results show no difference in the performance by the SilcoCan™ canisters for storing humidified sulfur VOCs as compared to dry standards.

Conclusion

This study confirmed the stability of very low-level sulfur VOCs (1-20ppbv) in SilcoCan™ canisters. Using dry or humidified conditions, sulfur compounds exhibited virtually no loss in SilcoCan™ canisters after six days. Sampling with electropolished canisters leads to degradation of hydrogen disulfide, methyl mercaptan, and ethyl mercaptan.

Rtx®-1 Column (fused silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	4.00	-60 to 280/300°C	30-Meter	10198
0.53mm	7.00	-60 to 240/260°C	60-Meter	10193

For other Rtx®-1 column dimensions, please request our general catalog or visit our website.

Rt-XLSulfur™ Packed/Micropacked Columns

- Optimized for low pbbv sulfur analyses.
- Eliminate the need for Teflon® tubing.
- Column and end-fittings are Sulfinert®-treated for maximum inertness.

Rt-XLSulfur™ Packed Columns

Purchase installation kit separately. Please see our catalog.

OD	ID (mm)	1-Meter*	2-Meter*
1/8"	2.0mm	80484-	80485-
3/16"*	3.2mm	80482-	80483-

*Please add configuration suffix number to cat.# when ordering. Please see our catalog.

Rt-XLSulfur™ Micropacked Columns

Purchase installation kit separately. Please see our catalog.

OD	ID (mm)	1-Meter	2-Meter
1/16"	1.0mm	19804	19805
0.95mm	0.75mm	19806	19807

References

1. Quang Tran, You-Zhi Tang; *Stability of Reduced Sulfur Compounds in Whole Air Samplers*, 1994 AWMA/EPA International Symposium of Measurement of Toxic and Related Air Pollutants.

2. Hoyt, Steven; Longacre, Vivian; and Stroupe, Michale; *Measurement of Oxygenated Hydrocarbons and Reduced Sulfur Gases by Full Scan GC/MS: EPA TO-14* in: *Sampling and Analysis of Airborne Pollutants*, Eric Winegar, and Lawrence Keith, editors. CRC Press, 1993 384pp (Restek cat. #20468).

3. Method TO-14A, *Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters with Subsequent Analysis by Gas Chromatography* in: *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*. US EPA, Jan 1997.

References 1 and 3 not available from Restek.

SilcoCan™ Air Monitoring Canisters

Siltek® treated - ideal for low-level reactive sulfur compounds (1-20ppb)

- Unsurpassed inertness, even for sulfur-containing or brominated compounds.
- Sizes from 1 to 15 liters support a wide range of sampling needs.
- Optional vacuum/pressure gauge for monitoring canister pressure.
- For critical applications, order a Siltek® treated valve - add suffix "-650" to the catalog number of the canister.

For ultimate inertness, we treat SilcoCan™ air monitoring canisters with our unique Siltek® passivation technology. Even highly active components, at low parts-per-billion concentrations, can be stored without loss. The valve is a high quality, metal-to-metal seal, 2/3-turn valve with metal diaphragms. Both stainless steel and Siltek®-treated valves are available, in both the 2-port and 3-port configurations.

Description	qty.	cat.#
1L Volume		
SilcoCan™ Canister, 1/4" Valve	ea.	24180
SilcoCan™ Canister, Siltek®-Treated 1/4" Valve	ea.	24180-650
SilcoCan™ Canister with Gauge, 1/4" Valve	ea.	24140
SilcoCan™ Canister with Gauge, Siltek®-Treated 1/4" Valve	ea.	24140-650
3L Volume		
SilcoCan™ Canister, 1/4" Valve	ea.	24181
SilcoCan™ Canister, Siltek®-Treated 1/4" Valve	ea.	24181-650
SilcoCan™ Canister with Gauge, 1/4" Valve	ea.	24141
SilcoCan™ Canister with Gauge, Siltek®-Treated 1/4" Valve	ea.	24141-650
6L Volume		
SilcoCan™ Canister, 1/4" Valve	ea.	24182
SilcoCan™ Canister, Siltek®-Treated 1/4" Valve	ea.	24182-650
SilcoCan™ Canister with Gauge, 1/4" Valve	ea.	24142
SilcoCan™ Canister with Gauge, Siltek®-Treated 1/4" Valve	ea.	24142-650
15L Volume		
SilcoCan™ Canister, 1/4" Valve	ea.	24183
SilcoCan™ Canister, Siltek®-Treated 1/4" Valve	ea.	24183-650
SilcoCan™ Canister with Gauge, 1/4" Valve	ea.	24143
SilcoCan™ Canister with Gauge, Siltek®-Treated 1/4" Valve	ea.	24143-650

All Restek canisters are originally equipped with high-quality Parker Hannifin diaphragm valves. Each valve is helium leak-tested to 4 x 10^{-9}cc/sec. The all-stainless steel construction eliminates contamination and the valve operates at temperatures from -100°C to 250°C. Compression outlet fitting, indicator plate to display open or closed position, 1/4" inlet and outlet.

Miniature Air Sampling Canisters

These 1000cc canisters are suitable for sampling volatile organic compounds in air according to US EPA Methods TO-14 and TO-15.

Description	Volume	qty.	cat.#
Sulfinert®-Treated Miniature Canister with Quick-Connect Stem Fitting	1000cc	ea.	24195
Sulfinert®-Treated Miniature Canister with Sulfinert®-Treated Quick-Connect Stem Fitting	1000cc	ea.	24196
Sulfinert®-Treated Miniature Canister with Metal-Seated Diaphragm Valve	1000cc	ea.	24198
Sulfinert®-Treated Miniature Canister with Sulfinert®-Treated Diaphragm Valve	1000cc	ea.	24199
Sulfinert®-Treated Miniature Canister with Nut & Ferrule	1000cc	ea.	24208

Also available: 400cc canisters. See our catalog or website.

The ultimate in controlled heating, for reliably cleaning your air canisters!

Air Canister Heating Jacket

- Heats entire canister, and valve.
- Two temperature settings, 75°C and 150°C.*
- Prevents sample condensation, for accurate sub-sampling.
- Easily fits canister up to 6 liters.
- Lightweight; comfortable to the touch when heated.
- Connect up to five Canister Heating Jackets to one 15 amp circuit.

Description	qty.	cat.#
Air Canister Heating Jacket (110 volt)	ea.	24123

*Not CE certified.

Dimensions: 2.75" diameter, 11.92" long (7 x 30.3cm)

Passive Air Sampling Kits

- Improved design eliminates leaks at the filter.
- Excellent for sampling times from 1 hour to 125 hours, or grab sampling.

Available in six sampling flow ranges, Restek's passive air sampling kit incorporates all hardware necessary to collect air samples, and is easy to assemble for field sampling.* The stainless steel kit is ideal to partner with the Restek TO-Can™ air sampling canister for TO-14A and TO-15 methods. Use the Siltek®-treated version with the Restek SilcoCan™ air sampling canister when collecting low-level volatile sulfur compounds, or other active compounds.

Canister Volume					Flow	Orifice	Siltek®-Treated	Stainless Steel
400cc	1 Liter	3 Liter	6 Liter	15 Liter	(sccm)	size	Complete Sampling Kits	Complete Sampling Kits
8 hour	24 hour	48 hour	125 hour	—	0.5–2	0.0008"	24217	24216
2 hour	4 hour	12 hour	24 hour	60 hour	2–4	0.0012"	24160	24165
1 hour	2 hour	6 hour	12 hour	30 hour	4–8	0.0016"	24161	24166
—	1 hour	4 hour	8 hour	20 hour	8–20	0.0020"	24162	24167
—	—	2 hour	3 hour	8 hour	20–40	0.0030"	24163	24168
—	—	—	1 hour	3 hour	40–80	0.0060"	24164	24169

*Air sampling canisters sold separately.

free literature

For detailed information about using, cleaning, and certifying passive sampling trains in air sampling applications, request our technical guide *A Guide to Passive Air Sampling*.

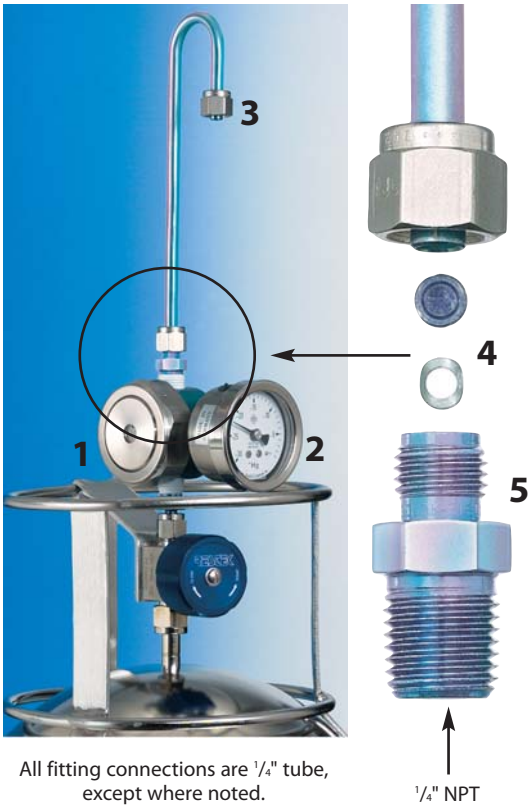
Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

lit. cat.# 59977B

1. Veriflo™ SC423XL flow controller
Designed to maintain a constant mass flow as the pressure changes from 30" Hg to 5" Hg. All wetted parts of the flow controller can be Siltek®-treated.

2. Stainless steel vacuum gauge
Monitors canister pressure change during sampling.

3. 1/4-inch Siltek® sample inlet
Stainless steel nut prevents water droplets from accumulating at the edge of the tubing, where they could be pulled into the sampling train.



All fitting connections are 1/4" tube, except where noted.

4. 2-micron frit filter and washer
Replaceable. Available in stainless steel, or Siltek®-treated for optimum inertness.

5. Interchangeable critical orifice
Sapphire critical orifice controls the flow with very high precision. Available in stainless steel, or Siltek®-treated for optimum inertness.

For individual components, see our catalog or website.