

Sulfinert® Treated Systems Preserve ppb Levels of Active Sulfur Compounds

By Gary Barone, Martin Higgins, David Smith (Restek Performance Coatings Division);
Shawn Rowan and Warren J. Gross (O'Brien Corporation); and Phil Harris (Harritec LLC.)

- Sulfinert® treatment prevents adsorption of sulfur compounds, ensuring representative sampling.
- Improved accuracy allows precise control of downstream processes, for better efficiency and profitability.
- Shorter cycles translate directly into increased sample throughput.

Many volatile sulfur compounds adsorb strongly to metal surfaces in sampling, storage, and transfer apparatuses. In addition to causing inaccurate values, adsorption can prolong analysis cycle times. To compare quantitative losses of active sulfur species, we sampled, stored, and transferred low ppmv to low ppbv concentrations of active sulfur gases, using control (untreated) and Sulfinert® treated system components.

Preventing Sulfur Compound Losses During Storage

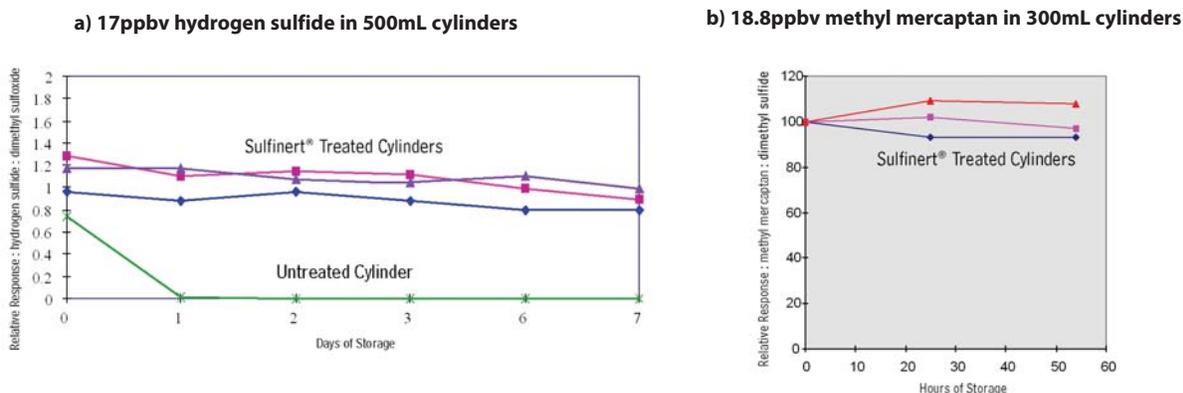
Figure 1a depicts results from a comparison in which a gas containing 17ppbv of hydrogen sulfide was stored for 7 days in untreated or in Sulfinert® treated stainless steel sample cylinders. The response ratio for hydrogen sulfide, relative to a stable reference material, dimethyl sulfide, is steady at approximately 1:1 for at least 7 days in Sulfinert® treated cylinders. The data show a Sulfinert® treated system will reliably store ppb levels of the active sulfur-containing compound during transport from the sampling site to the analytical laboratory. In contrast, hydrogen sulfide degraded rapidly in the untreated cylinder, and was lost totally within 24 hours. In a similar study in which gas containing 18.8ppbv methyl mercaptan was stored for 60 hours in Sulfinert® treated sample cylinders, recovery of the active sulfur compound was equally high relative to the stable reference material, dimethyl sulfide, as shown in Figure 1b.

Sample Transfer: Adsorption of Sulfur Compounds to Tubing

Comparison of Sulfinert® treated electropolished stainless steel tubing (TrueTube® EPS tubing, O'Brien Corporation, St. Louis, MO), untreated electropolished stainless steel tubing (TrueTube® EP tubing, O'Brien Corporation), and raw commercial grade 316L stainless steel tubing showed Sulfinert® treated electropolished tubing has the inertness necessary for quantitatively transferring low ppmv to low ppbv concentrations of sulfur compounds.

To determine whether an active sulfur-containing compound in a gas stream would adsorb to active sites on the transfer tubing surface, we monitored the length of time that elapsed before recovery values for a sulfur compound exiting a 100-foot (30.5-meter) length of tubing were stable and accurate, using helium containing 0.500ppmv methyl mercaptan as the test material, at a flow rate of 40cc/minute. Figure 2 shows Sulfinert® treated electropolished tubing did not adsorb methyl mercaptan to any measurable extent, delivering a representative sample with no delay. When adsorption of sulfur-containing compounds is prolonged, desorption from the surface also is slow. This "memory" of adsorbed compounds can cause long delays in re-equilibrating a sample pathway. In Figure 3, Sulfinert® treated tubing shows the lowest retention of sulfur compounds, by several orders of magnitude. Samples can be evaluated, with accurate results and with no delay between them.

Figure 1 Sulfur compounds are stable in Sulfinert® treated cylinders, assuring a representative sample is delivered.



The authors thank Shell Research and Technology Centre, Amsterdam, for the data used in evaluating sulfur gas uptake and the memory effects of tubing substrates.

Figure 2 Sulfinert® treated tubing (red) does not adsorb methyl mercaptan (500ppbv), giving accurate results with no delay.

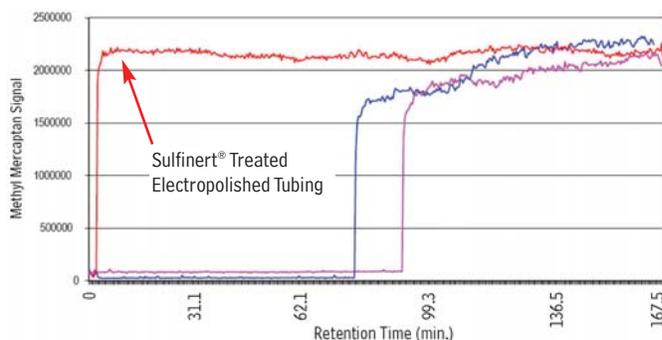
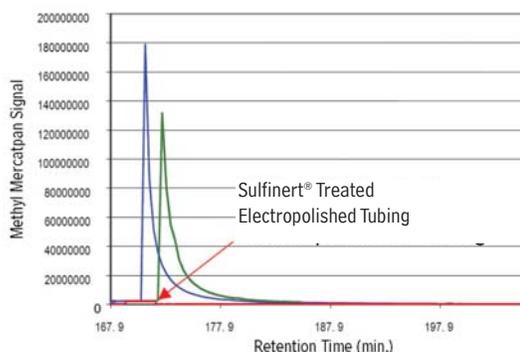


Figure 3 Sulfur memory is prolonged in raw commercial grade stainless steel tubing, increasing cycle time and reducing accuracy.



Sulfinert® Treated Alta-Robbins Sample Cylinder Valves

- All wetted parts are Sulfinert® treated for inertness.
- Compatible with Sulfinert® treated Swagelok® sample cylinders.
- Large, durable, Kel-F® seat ensures leak-free operation; temperature range: -40°C to 120°C.

Description	qty.	cat.#
1/4" NPT Exit	ea.	21400
1/4" Compression Exit	ea.	21401
1/4" NPT with Dip Tube*	ea.	21402
1/4" NPT with 2850psi Rupture Disc	ea.	21403
1/4" NPT Male Inlet x 1/4" Female Outlet with 2850psi Rupture Disc	ea.	21404

*To order catalog #21402 (Sulfinert Alta-Robbins Sample Cylinder Valve, 1/4" NPT with Dip Tube), please call Customer Service at 800-356-1688, ext. 3, or contact your Restek representative. Specify dip tube length or % outage when ordering (maximum length = 5.25" / 13.3cm). Note: End of part will not be treated after cutting tube to length.

Sulfinert® Treated Swagelok® Sample Cylinders

Ideal for collecting and storing samples, such as natural gas or beverage-grade carbon dioxide, because active compounds remain stable during transport.

Size	qty.	cat.#
75cc	ea.	24130
150cc	ea.	24131
300cc	ea.	24132
500cc	ea.	24133
1000cc	ea.	24134
2250cc	ea.	21394

Conclusion

We obtained more accurate data, with no delay between samples, by using Sulfinert® treated electropolished tubing in the sampling-storage-transport system. In contrast, we obtained significantly less accurate data, with delays of more than two hours between samples, by using untreated tubing. Improved accuracy and reliability of data for sulfur mean downstream processes can be more precisely controlled, with associated cost savings. Shorter cycles translate directly into more samples collected and analyzed in a given period of time. Analysts charged with monitoring sulfur levels can significantly improve efficiency and profitability by using Sulfinert® treated tubing and components.

Acknowledgement

The authors thank O'Brien Corporation for arranging the research studies and supplying the electropolished tubing. To learn more about O'Brien Corporation, visit www.obrien-analytical.com.

**This article is an analytical summary.
For the complete study, visit us at
www.restek.com or call 800-356-1688,
ext. 5 and request lit. cat.# 59082.**

