Applications Note

Improving Gas Chromatography with a SilcoNert®-treated Sample Flow Path

An inert sample flow path is imperative for accurately quantifying sample profiles that are now more complex than ever before. Regulatory requirements coupled with increasingly difficult samples and sampling environments require solutions that cannot be achieved with any selection of raw material alone; metal components provide adequate robustness but quickly adsorb important molecules that must be analyzed at trace levels. In order to accurately quantify these critical compounds, the entire sample flow path throughout the analyzer must be inert to ensure that the whole sample reaches the detection point.

SilcoNert® 2000 Coating

SilcoNert® 2000, previously named Sulfinert®, is an amorphous silicon (a-Si) based coating that is applied via chemical vapor deposition (CVD). The deposition is performed under vacuum as a proprietary blend of precursor gases react to molecularly bond the coating to the substrate. This process allows for a completely non-line-of-sight coating which penetrates small orifices and easily coats complex geometries. The result is a robust, fully adhered coating that can move with components that must be configured to meet the physical demands of the analytical system.

Although naturally inert, silicon alone is not adequately resistant to some of the more reactive compounds such as hydrogen sulfide (H_2S), mercury (Hg), or ammonia (NH_3), especially when these analytes must be quantified at very low - even parts-per-trillion - levels. To address this, SilcoTek functionalizes the silicon surface with hydrocarbon molecules in a secondary deposition process to increase inertness and reduce the effects of porosity in the layer. This, the final SilcoNert® 2000 surface, is the most inert coating available to the chromatography market.

When to use SilcoNert®

A SilcoNert-treated flow path is especially important for highly reactive species, e.g. sulfurs, NOx, SOx, stack gas, alcohols, pharmaceuticals, explosives, chemical weapons, volatile organic compounds (VOCs), OP pesticides, chlorinated pesticides, herbicides, and more. SilcoTek® is dedicated only to providing coating services and can make any stainless steel, glass, ceramic, titanium or exotic alloy inert, even outside of a standard GC configuration. Any surface that contacts your sample should be coated to ensure reliable results.

Ideal Applications

- Trace sulfurs, mercury, and ammonia
- Moisture
- Natural gas testing
- NOx and SOx
- Stack gas monitoring
- Acidic streams
- Environmental sampling/compliance
- Refinery and petrochemical monitoring
- Down-hole oil and gas sampling
- Any time complete inertness is required

Experimental - Organosulfur Compounds

Certain species e.g. H_2S tend to readily adsorb to steel surfaces much more drastically than others, causing false and unstable analytical readings [1]. Prior to inert coating technology, passivation was a frequently chosen technique for stablizing the analytical system. Studies have shown that, for gas phase transport at low temperature, flowing sulfur compounds through the sample pathway will cause them to adsorb onto active sites of the steel, creating a passive surface for the actual sample to flow through afterward (a.k.a. "priming") [2]. However, this is an extremely limited, time-consuming solution that is not feasible for meeting the now rigorous analytical demands in applications dealing with odorants, beverage grade CO_2, and the production of ethylene and propylene, amongst others. In conjunction with Restek® Corporation, we have examined the adsorption effects of sulfur species on stainless steel sample storage and transfer components in simulated applications via GC.

Got used liners? SilcoTek offers redeactivation services!
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Figure 1: Block diagram of analytical system

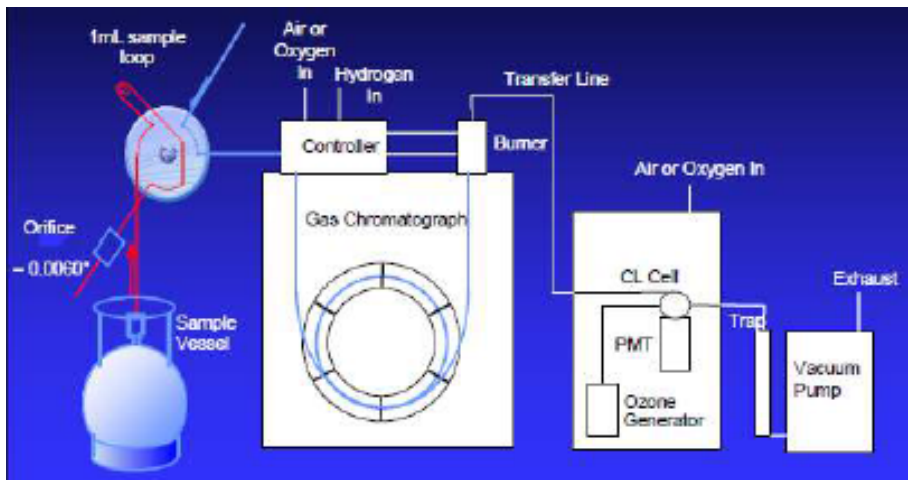


Figure 2: List of sulfur compounds and concentrations

		Conc	Conc	Conc as S
Compound Name	Formula	(ppmv)	(ppbv)	(ppbv)
hydrogen sulfide	H2S	105	11.51	10.83
carbonyl sulfide	COS	98	10.74	5.73
methyl mercaptan	CH3SH	101	11.07	7.38
ethyl mercaptan	CH3CH2SH	101	11.07	5.71
dimethylsulfide	CH3SCH3	99	10.85	6.81
dimethyl disulfide	CH3SSCH3	100	10.96	7.46

Results

As shown in the data below, SilcoNert® 2000 treated sample holding vessels show virtually no loss of H_2S compounds after 6 days. Additionally, 11 ppbv methyl mercaptan did not adsorb or degrade after 14 days.

Figure 3: H2S at 11.51 ppbv 6-day stor-

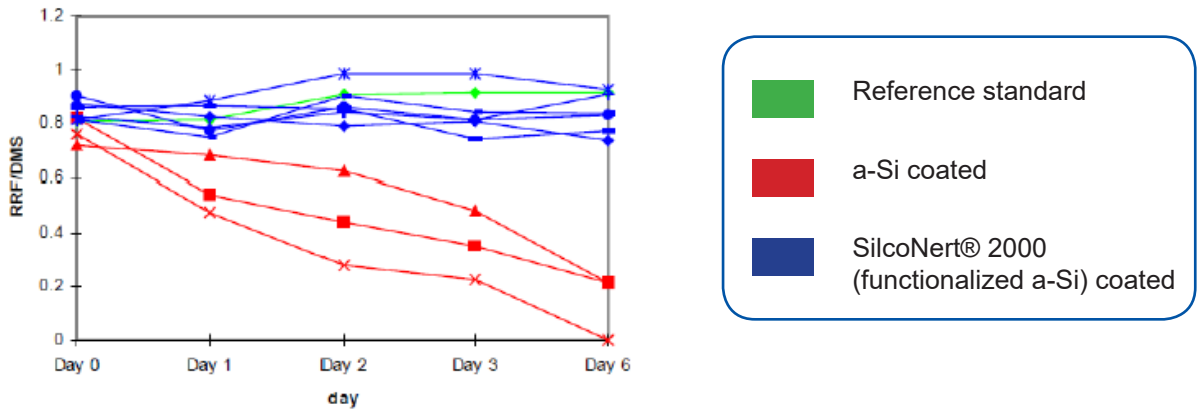
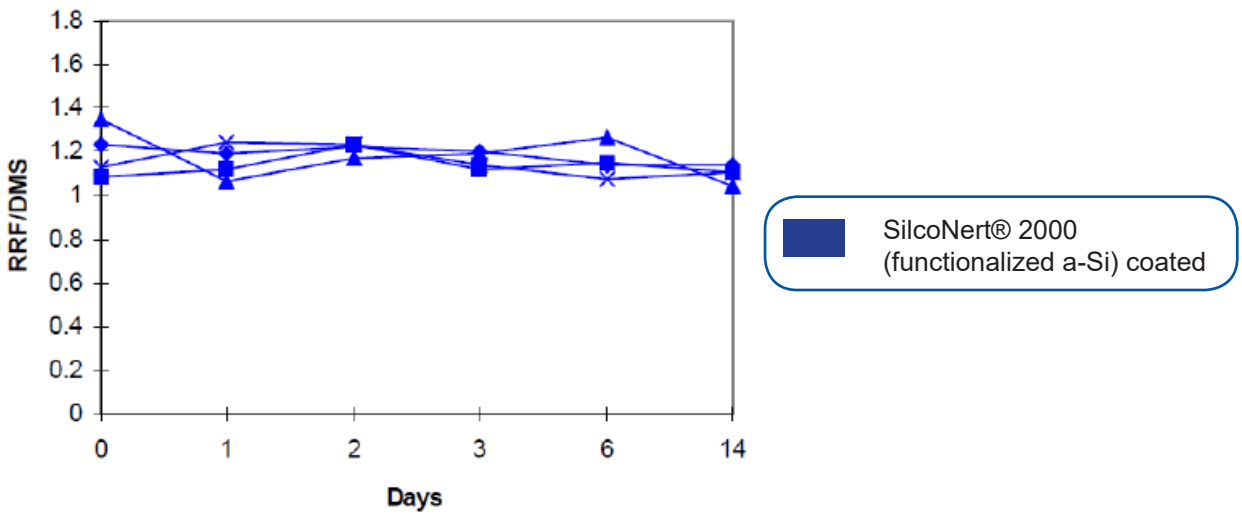


Figure 4: Methyl mercaptan at 11.51 ppbv
14-day storage



SilcoNert® 2000-treated components are especially optimal when single digit ppb or even ppt levels of impurities must be tested; other surface treatments simply are not suited for analysis at these trace concentrations. A GC outfitted with a SilcoNert-coated flow path produced chromatographic results of sulfur testing in beverage-grade CO_2, beer, and refinery gas which are illustrated in figures 5, 6, and 7 below, respectively.

Figure 5: Beverage-grade CO_2 at 20 ppbv, sulfur standard added

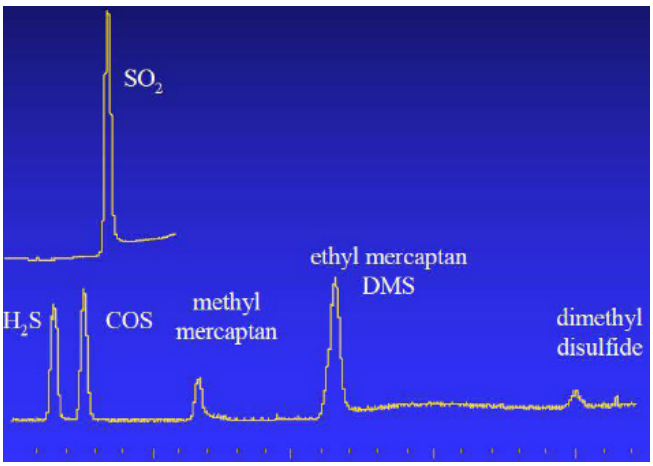


Figure 6: Typical run to determine beer's sulfur content

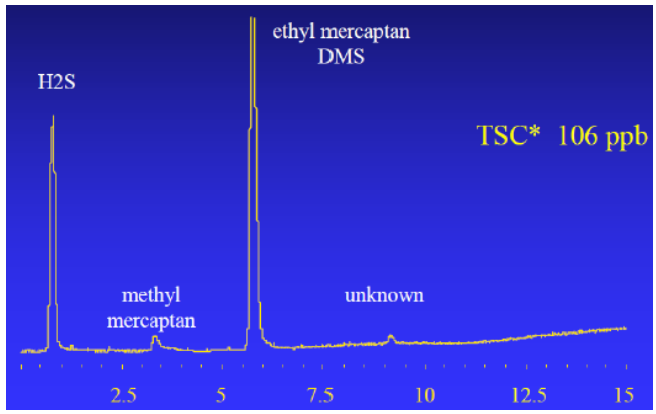
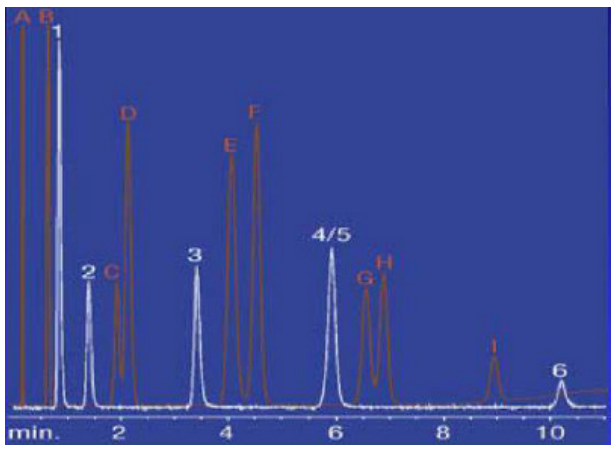


Figure 7: Sulfur and hydrocarbon analysis of refinery gas



Rt-XL Sulfur Micropacked 50 ppb

1. Hydrogen sulfide
2. Carbonyl sulfide
3. Methyl mercaptan
4. Ethyl mercaptan
5. Dimethyl sulfide
6. Dimethyl disulfide

A. Methane
B. Ethane
C. Propylene
D. Propane
E. Isobutane
F. Butane
G. Isopentane
H. Pentane
I. Hexane

SilcoNert® 2000 provides any compatible surface with industry-leading inertness with drastically improved durability compared to glass and PTFE linings. Coat any surface that touches the sample to ensure the best results.

The SilcoTek® Difference

SilcoTek is a company solely dedicated to improving the performance of customer-supplied products. Our unwavering focus to coatings has expanded our reach to a variety of industries outside of gas and liquid chromatography, including semiconductor manufacturing, petrochemical, refining, oil and gas exploration, automative, aerospace, and more. This drives an endless commitment to our customers to provide them with a service that is fast, effective, and mistake-free. Our ZIP code (**Z**ero disappointments, **I**ntegrity in all we do and **P**lus 1 customer service) embodies these everyday beliefs and is the reason why customers love to work with us.

SilcoTek's coating process is unlike any other available on the market. Aside from the actual deposition of the coating, our workflow is designed to treat your parts quickly and with quality, documenting every step of the way. Once received, we perform a thorough inspection to confirm that your parts arrived at our facility just the way they left your's. Then, the parts' surfaces are prepared using our proprietary aqueous cleaning method that thoroughly removes contaminants even as simple as fingerprints. Parts are then re-inspected, coated, cleaned, and rigorously inspected once more, all to ensure you say "wow" when you open the box and experience the new upgraded performance of your SilcoTek-treated components.

Coating your parts is easy!

1. Request a quote on SilcoTek.com or by emailing SilcoD@SilcoTek.com. Simply list the parts requiring treatment and include drawings or photographs.

2. After helping you choose the best coating, a SilcoTek representative will send you a quote with a customized shipping label to include on your box.

3. Send us your parts. In 10 business days or fewer, you will get them back; this time, they will be ready to perform better than ever.

That's it!

Visit www.SilcoTek.com to learn more

SilcoTek's line of coating solutions:

SilcoNert®
The world's most inert coating

Dursan®
Durable, corrosion resistant, and inert, all in one

Silcolloy®
High purity, acidic corrosion resistance

SilcoKlean®
Resists carbon build up

SilcoGuard®
Low outgassing, ideal for ultra high vacuum

REFERENCES

[1] Barone, G., Smith, D., and Higgins, M. "Improving the reliability of analytical and sampling systems in challenging and corrosive environments. Presented at the 57th Analysis Division Symposium in Anaheim (2011).

[2] Biela, B., Moore, R., Benesch, R., Talbert, B., Jacksier, T. "The Do's and Don'ts in the Analysis of sulfur for Polyolefin Producers", Presented as paper 081 at the Gulf Coast Conference, Galveston Island, TX, October 22, 2003.

Information subject to change without notice

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QUALITY ISO 9001:2008 CERTIFICATION

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