Keys to a High-Performance Analytical System

How to Improve Sampling System Reliability and Results with Inert Coatings

SilcoTek®

E-book #1 of 4 in

The Ultimate Guide to Reliable Sampling with SilcoTek® Inert Coatings

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1. Introduction

SilcoTek® became a coatings company because we have a passion for helping people and businesses conquer their most difficult material challenges. We help our customers improve analytical results, improve yield and efficiency, and to develop innovations that push the scientific and industrial world forward.

Why Should I Read This E-book?

Because you'll learn how to prevent common sampling and analytical problems and learn ways to save time and money. You will learn how to:

- Improve sample system reliability
- Improve system response and process control
- Reduce troubleshooting
- Get the results you need the first time, every time

Our scientists and engineers are dedicated to teaching the customer how to improve the material performance of their products and processes. Read on and benefit from our over 250 years of analytical experience.

Paul Silvis, Head Coach

2. 5 Signs your Analytical System is Not Performing Optimally

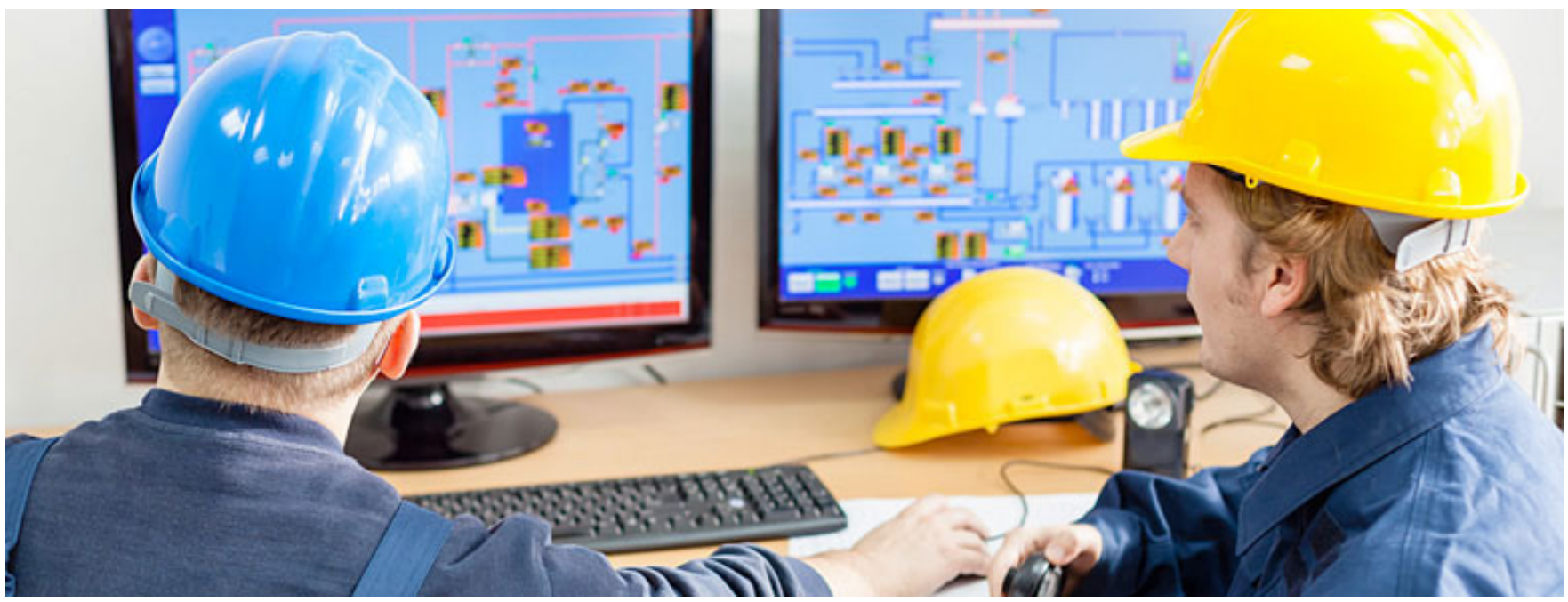


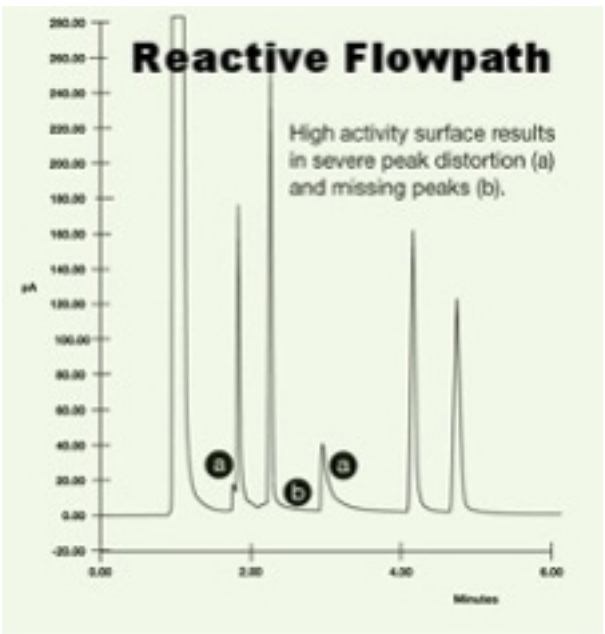
Image courtesy of https://cocareeractiontools.com/job/process-engineer

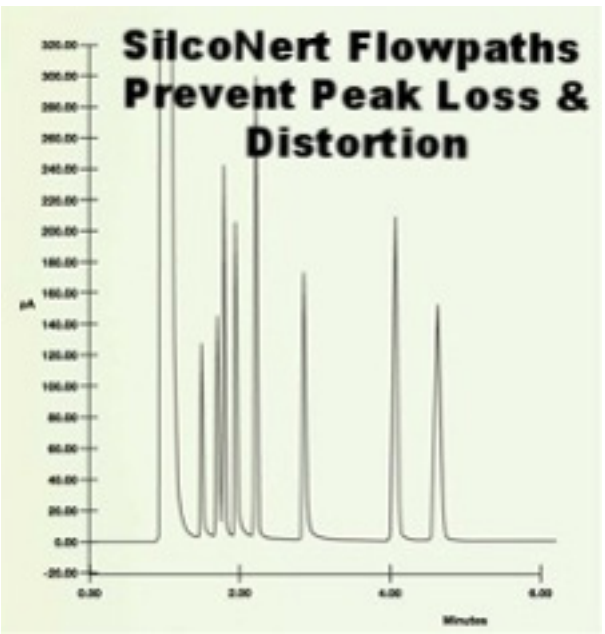
An under-performing sampling system can cost an operator more than just time and money. A lousy system can be the cause of poor yield, slow response, bad decisions, and regulatory or contract violations. We all know when a sample transfer system is running like a top, but how do you know when a system is not doing its job?

Here are 5 signs you've got an under-performing analytical system.

1. Calibration errors

Excessive stabilization time, loss of low or high side calibration standard, slow recovery, poor peak shape and outright failure to get a calibration signal response can all be signs that your calibration system is not performing. A reactive calibration flowpath will result in lost peaks minimal peak area or peak tailing.

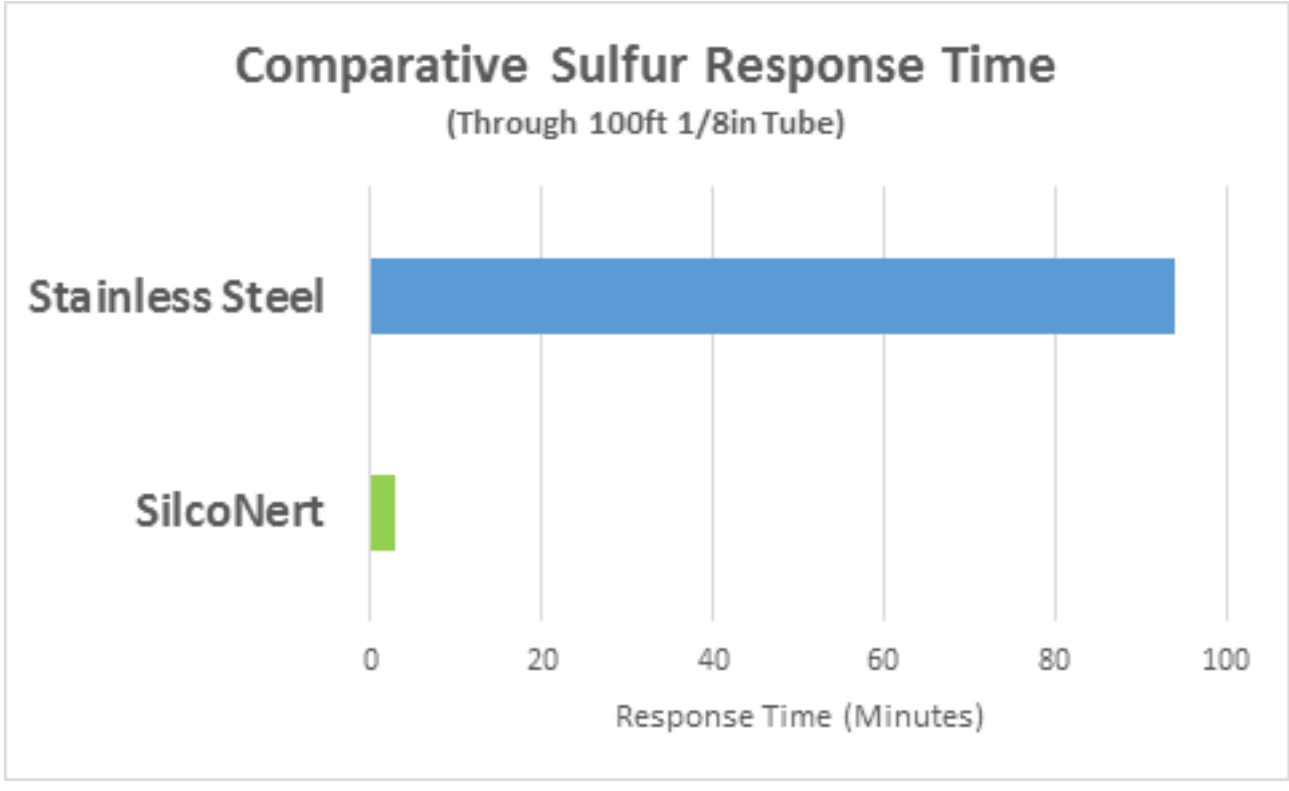




An inert flowpath will prevent signal loss, improve response and improve peak quality; making calibration faster and easier.

2. Slow response

Tubing can be a huge source of sample loss and adsorption. How disruptive can reactive tubing be? Here's a comparative signal response graph plotting the response signal of a sulfur sample through 100ft of 1/8in tubing[1].



Active sites in the uncoated tube grab onto the sulfur sample, preventing it from reaching the analyzer for 90 minutes! Excessive delay in signal response will make process control almost impossible.

3. All is well...*too* well

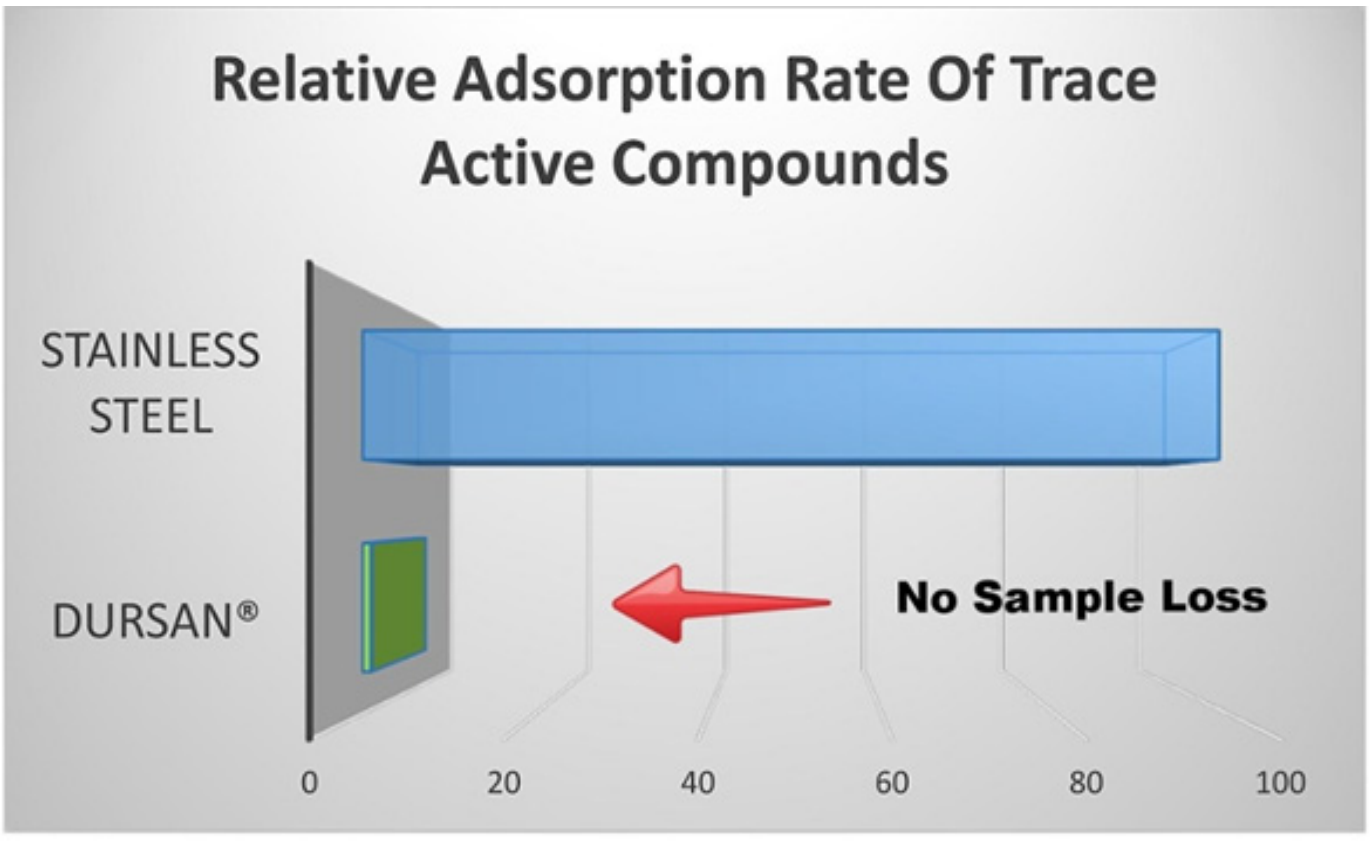
If you've never seen a high sulfur reading, or results are "rock steady" and never changing, it's a sign that the sample system is adsorbing analyte or is saturated and desorbing at a consistent rate. If you hear technicians state "we never have sulfur in our product," it could mean the sample system is adsorptive and not allowing your analyzer to detect sulfur. Poor response can make life easier sometimes, right up until regulatory or customer auditors show up and reveal the ugly truth.

4. Excessive maintenance

A reactive flowpath can be caused by corrosion. Poor system design or installation can result in moisture accumulation and corrosive particulate generation. Interaction with reactive iron oxide particles can grab onto a sample and prevent accurate signal response. If maintenance staff is constantly fixing system failures or replacing components due to corrosion, it could be a sign that your system could be causing adsorptive particle generation.

5. Variable response

If the system is showing no response and then an unexplained huge signal, the system may be adsorbing and then desorbing/releasing analyte. Seemingly random spikes can be a sign your system is holding active compounds then as the system becomes saturated, the surface may release or desorb analyte, causing sudden unexplained increases in response.

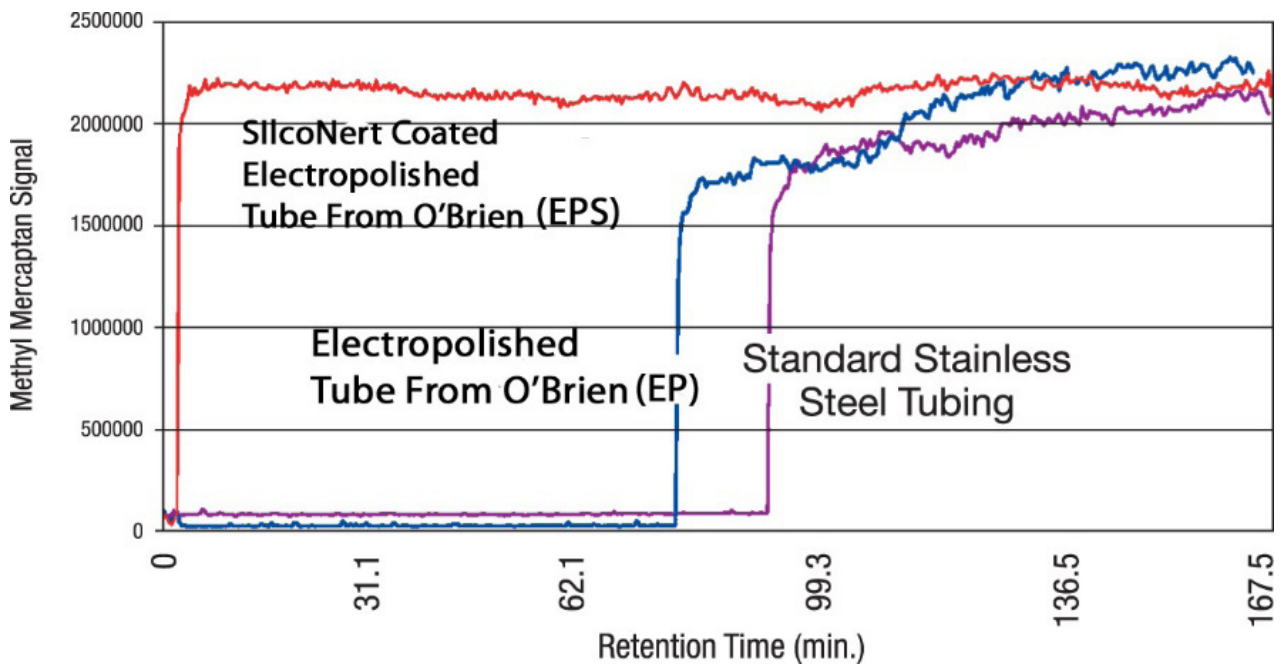


Want to learn more about adsorption and desorption of active compounds?

Read the complete study by Shell and O'Brien Analytical.

[1] Data courtesy of Shell Research & Technology Centre, Amsterdam, and O'Brien Analytical, USA

3. 7 Tips for Improving Analytical Sampling and Reliability



Analyzing highly active sulfur compounds like methyl mercaptan (CH_3SH) once required "priming" or "seasoning" of the system to get an accurate signal, adding hours to overall analysis times. Today, inert-coated sample flow paths allow for instant and accurate readings of these reactive analytes (red line above).

An inert flowpath is key to reliable process sampling and lab analysis. SilcoTek® is dedicated to providing coating services and can make 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.

If you're not getting the sample results you expect, here are tips for how to improve analytical sampling system performance.

1. Make the flow path inert

SilcoNert® 2000 and Dursan® inert coatings improve sample reliability of active and unstable compounds. Click on the links in the table below to read studies about how inert coatings help real-world applications.

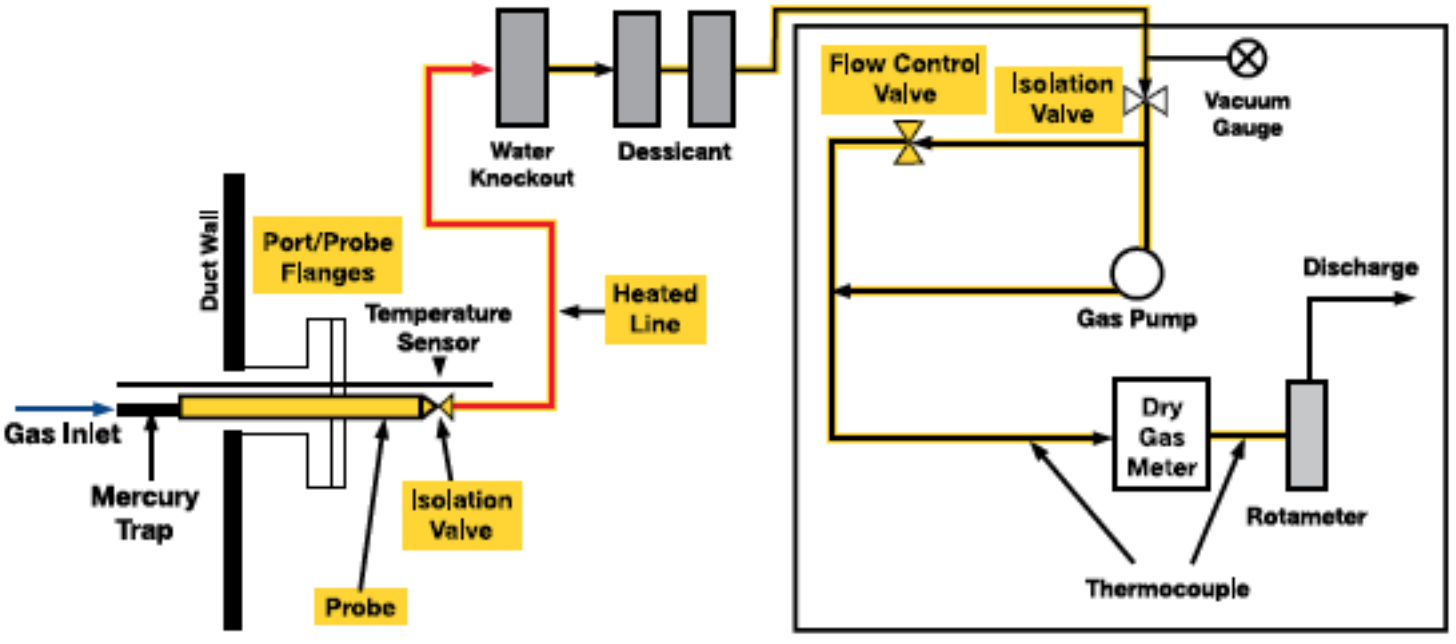
Pesticides	NO_x / SO_x / Stack	Bioanalytical & Pharma
Semi Volatiles	VOCs	Explosives / Chemical Agents
Amines	H_2S & Sulfur	Mercury

Coating of wetted flow pathways of analytical and process sampling instrumentation minimizes surface interaction and break-down of active compounds, allowing for fast, accurate, and reliable detection.

2. Coat the entire sample pathway

A single uncoated filter or fitting can adsorb a significant amount of a reactive chemical, especially when sampling trace sulfur or other highly active compounds. The entire sample flowpath should be made inert to assure complete transfer of the sample to the instrument. Coat all components like:

- Injectors
- Fittings and valves
- Tubing
- Separators/Filters
- Weldments
- Probes
- Liners



You can purchase components like these off the shelf that have already been inert-coated by SilcoTek®. Click here to see the list of SilcoTek's Approved OEM Partners and the products they offer.

3. Check the calibration system

A slow calibration cycle, unstable readings, and outright calibration failures can be a sign of a reactive flowpath.

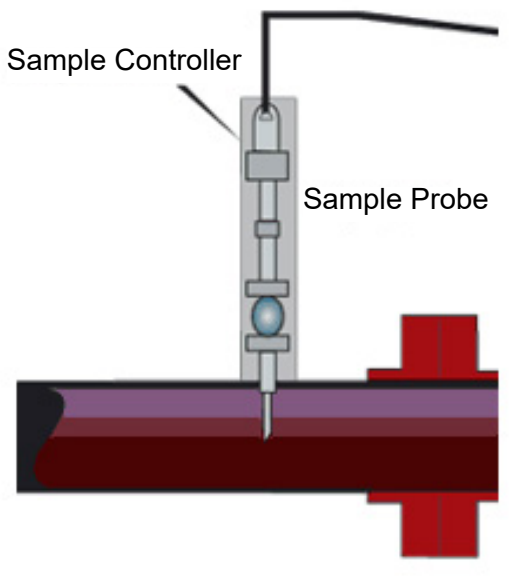
- Look for exposed stainless steel surfaces. They may be adsorbing the sample.
- Check calibration gas. It may be expired or contaminated.
- Check the regulator. It may not be functioning properly or may not be sized for the target pressure range.
- Check fritted filters. Are there filters in the regulator inlet? Uncoated metal frits can retain significant amounts of reactive calibration gas.

4. Check the sample probe location

Improper sample location can distort results. Studies and presentations by Welker Engineering show that sample location influences the quality of results. Position the stack probe to assure a representative sample.

Position the probe:

- Away from the wall
- Away from turbulent flow
- From center of stream if possible
- Away from any flow disruptions



5. Is the heat trace tubing inert and installed per manufacturer specifications?

• Reactive tubing surfaces can adsorb sample.

• Dips, kinks, and off spec heating can distort results or cause reactive corrosion or moisture accumulation.

• Cold spots in heat trace tubing can cause condensation of sample and moisture.

• PTFE tubing can distort, cold flow, or fail if exposed to elevated temperatures.

• Check for contamination and particulates. Steam cleaning can inject active particles into sample collection systems, which can cause loss of sample and poor test results.

6. Are regulators designed for inertness?

• Check with your regulator manufacturer to be sure it's designed for analytical use.

• Check for flow or pressure problems, especially at extreme low side and high side testing. A worn spring or diaphragm can restrict flow and distort results.

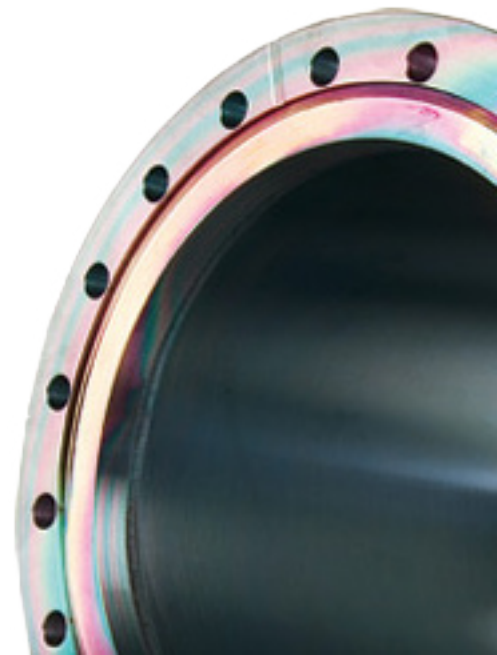
• Check for a fritted filter at the regulator inlet. It may be clogged or if not coated will be adsorbing active compounds.

SilcoTek® teams up with its approved partners to offer inert-coated regulators from stock.

7. Are GC setup conditions, column, and injection volume within spec?

Most often, poor sampling results can be traced to the sample system, but what if it's the instrument? Troubleshooting an underperforming gas chromatograph can be a daunting challenge. Is the column the issue? Is injection volume within spec? Is the carrier gas contaminated?

There are any number of issues and solutions for fixing a problem GC. Following accepted GC troubleshooting guidelines can help improve sample performance and get you back on track.



4. What Is An Inert Surface? 3 Ways to Tell If a Flowpath Is Inert

A web search will tell you that an inert substance is not chemically reactive, has no ability to react, or does not produce a chemical reaction. That sounds suspiciously absolute.

So what is an "inert surface" and how do I know if I have one?

Digging further into the definition the term gets less clear. You'll find that given enough energy and under the right conditions almost anything will react. It's all about the conditions and specific chemicals under which the surface should be non-reactive. So be weary of claims of absolute inertness.

One other key factor to consider is surface adsorption. A flowpath may not react chemically with a target compound, but will it allow other substances to stick or adhere to the surface?

Adsorptive surfaces will retain a substance on the surface, creating a film of adsorbate that will eventually desorb or be released back into the sample stream. Adsorption is caused by physisorption (van der Waals forces), chemisorption (covalent bonding), or electrostatic attraction. Adsorption can be just as important a factor as surface inertness.

Here are 3 tips to determine if a surface is reacting with your sample.

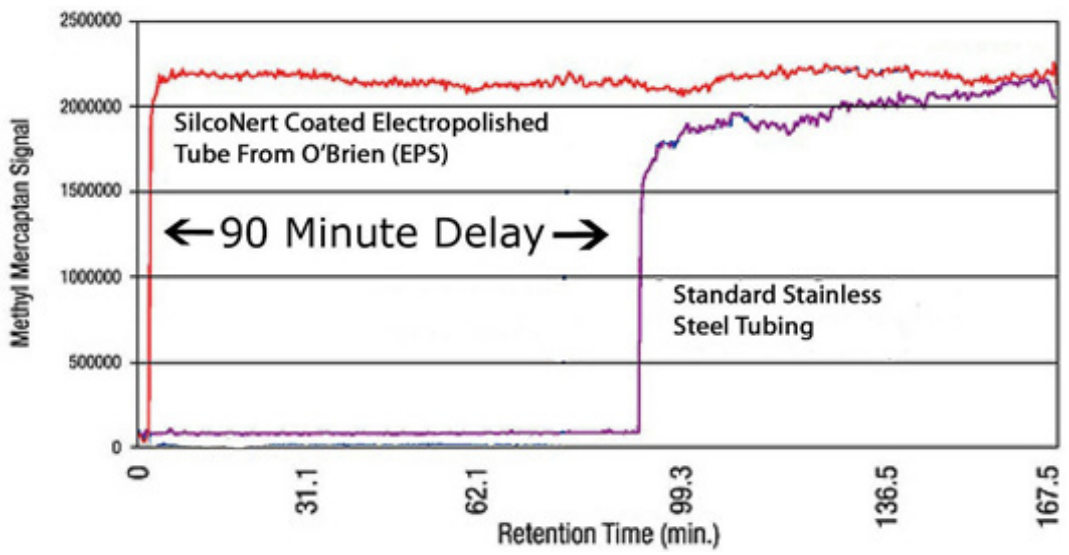
Test for the relevant target compounds.

If a manufacturer claims a surface is non-reactive but does not produce data relevant to your application, test the surface under controlled conditions to be sure the surface is truly not reacting with your compounds. Test both adsorption (loss of compounds) and desorption (gain of compounds) in a stream.

Here are 2 examples of how to test for surface reactivity and desorption

Example 1

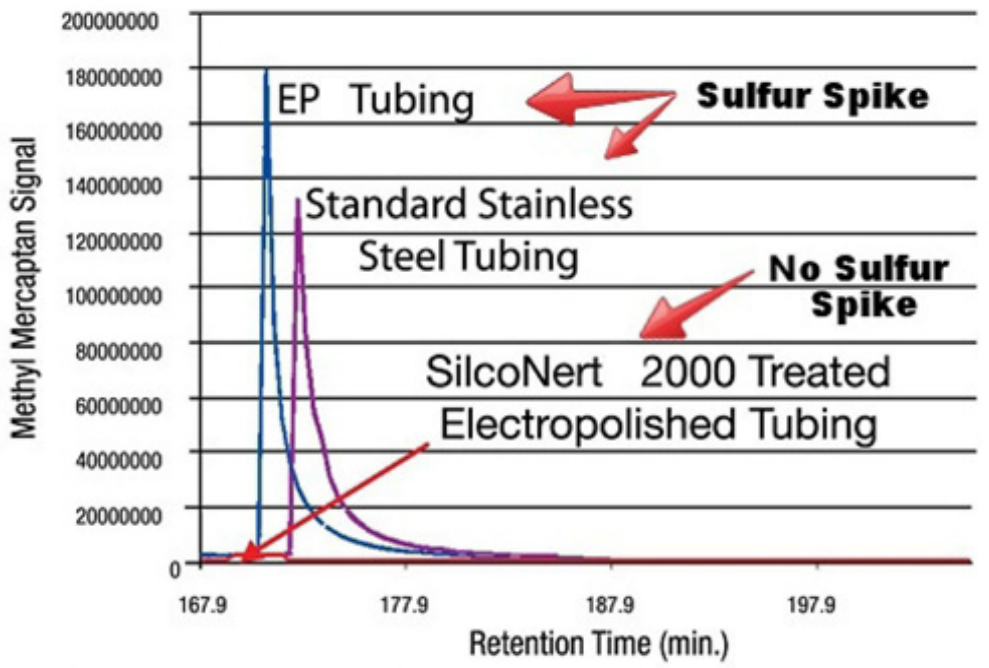
Example 1 is a comparison of a coated and uncoated 1/8 in. tube. Take a 100ft length and coil it. Flow a sample of your analyte (in this case sulfur in gas form) through the tube. Analyze the output to determine if the output matches the known input concentration.[1]



Adsorption will delay or even prevent accurate testing. Reactive surfaces like stainless steel can significantly impact results, so an inert flow path is a critical feature in the sampling system for maximum reliability.

Example 2

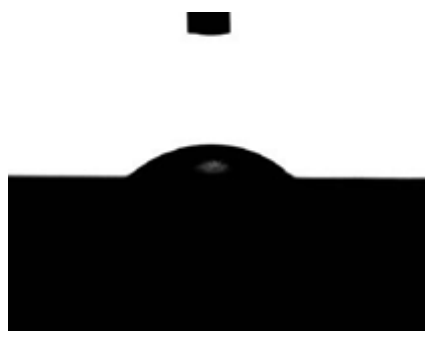
To determine desorption or retention of your target compound, flow nitrogen or another “non-reacting” gas through the tube and analyze the output for your target analyte.[1]



Desorption or delayed release of compound from a reactive surface can lead to false or misleading results.

Surface Energy Effects

Surface energy can play an important role in retention of compounds. Easily wetting surfaces with low contact angle and high surface energy may react more readily with target compounds and cause adsorption or corrupt the sample.



Low energy surfaces that don't wet and produce a high contact angle may be less reactive to your analyte.

Want to learn more about the impact that moisture and surface energy have on analytical results?

[Click here to view our white paper.]

Heat Effects

Analysts often test surfaces under similar conditions to "real world" field exposure. Unfortunately a thorough evaluation can take a lot of time. If time is short and you need to know if a surface flowpath is non-reactive, test it under elevated temperature. A heated surface will increase the rate of chemical reaction and surface reactivity. Heat will bring out the worst in a surface.

Want more details about how to prevent surface interactions and adsorption?

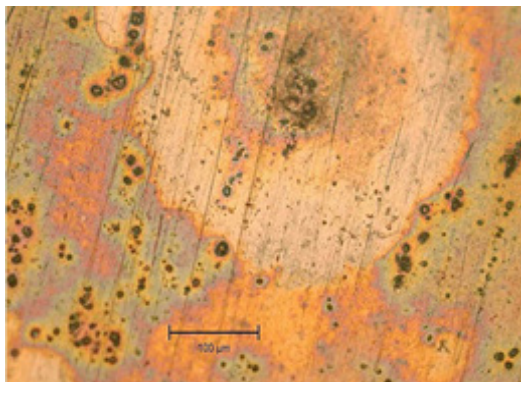
[Read our presentation] **and learn about the benefits of an inert flow path.**

[1] Data courtesy of Shell Research & Technology Centre, Amsterdam, and O'Brien Analytical, USA

5. How to Reduce Contamination and Improve Reliability

Customers experiencing material contamination and sampling reliability issues frequently ask: How can I improve flow path inertness and sample system reliability? Replacing chromatograph columns isn't a big job, but replacing 100 meters of sample tubing routed high up among the pipe-work would be a major headache."

1. Sample integrity



The biggest consideration regarding contamination and sampling reliability would be sample condition. How dirty or clean is the sample stream? If there is a lot of particulate coming into the line, then cleaning would be needed to remove contaminants and sample filtration is needed. If sample particulates build up, they could become adsorptive and degrade test results. Once particulates are removed, an inert coating should be applied to the entire flow path to minimize particulate stiction and sample adsorption. Never clean an analytical sample flow path with steam. Steam, especially process steam systems, contain particulates that will adsorb test samples and disrupt system calibration. Particulates can also damage inert coatings if used in the flow path.

2. Manage Moisture

Another area of potential poor reliability is the accumulation of moisture in sampling systems. If there is any point of condensation in the system and there are acids or bases that form in water from the sample stream/water contact, the subsequent corrosive attack and reactivity of the acid/base will decrease the coating life and adversely impact sample reliability.

Poorly designed or installed systems can form a cold spot or dip in the heat trace tubing which resulted in moisture condensation and accumulation. HCl or Sulfuric acid formation can result. The acid can attack the tube surface, eventually eating a hole (in the inert coating layer and the tubing) and creating active iron particulates and ultimately tube failure. Manage moisture by coating the surface with an inert, hydrophobic coating to reduce the surface energy and increase moisture repelling properties.

3. Keys to Reliable Sampling

For most applications a well-designed sample system featuring an inert coating will last many years without issue. A study by BP, presented at the 2011 ISA-AD Conference in Houston, TX, highlighted a SilcoNert® 2000 coated flare gas sampling line that had been delivering accurate results without issue for 2 years. This is a very dirty application with the potential of many contaminants and failures; however, with first-rate design and maintenance, the system continues to provide accurate, reliable sampling results. Keys to reliable sampling are:

• Eliminate potential cold spots in heat trace tube.

• Proper installation, avoid dips or areas for moisture accumulation in tubing.

• Install filtration, avoid build-up of active particulates.

• Proper material selection for the environment
  - Inert coatings
  - Materials specified for the sampling environment

• Proper coating selection for the sampling application:
  - SilcoNert® 2000 for low level sulfur/H2S sampling
  - Dursan® for extreme environments (acid/base exposure, particulate abrasion, etc)

3. Heat Trace References

For design and installation pointers, ask the heat trace companies about their experiences with installing and servicing lines used in your application. SilcoTek works extensively with O'Brien Corporation and Thermon Manufacturing Company. They are the market leaders and have a great deal of technical expertise and experience with installation and maintenance of reliable sampling systems.

6. Conclusion

So, what are the keys to a truly high-performance analytical system? It takes an understanding of each system component and an integrated approach to system design and operation. Keys to success are:

- Listening to your system. Watch for signs of performance issues.
- Maintaining and building reliability into the system.
- Understanding the signs of reactivity and system contamination.
- Coating the flow path with an inert coating that will prevent adsorption and contamination while improving system reliability.

Want to learn more about sample systems and how to improve results? Well you're in luck! This is book 1 in a 4 book series on improving analytical sampling. Stay tuned for the next e-book in your email inbox. Upcoming topics will discuss:

- An introduction to inert coatings
- Managing the sampling environment
- Troubleshooting & protecting the sampling system
- Maintaining an inert surface

Received this e-book from a colleague but want to make sure you get the rest of the series? Click here to subscribe.

Start Improving Process Sampling Now

You've read the e-book, now it's time to take action! Contact our technical service team so we can help you select the best coating and evaluation methods for your application. We'll even send you free coated samples for you to put to the test. Click the link below to get started!

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