

#### Performance Comparison of Surface Treatments for Monel®, Stainless Steel, and Aluminum Flow Path Materials

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# Background



- The use of surface treatments is commonly used in sampling and analytical systems to improve sensitivity and performance.
  - Electropolishing to smoothen tubing surfaces
  - PTFE (Teflon®) for lining sample cylinders, valves, fittings, and tubing
- 1990s: Ultra-low-sulfur fuel (ULSD & ULSG) standards lowered detection limits. This increased demand for more inert sample transfer components.
- Industry needed better alternatives to PTFE capable of higher temperatures with greater adhesion, density, and durability.
  - Today, we see the same push to regulate PFAS materials



# Sulfurs and Steel

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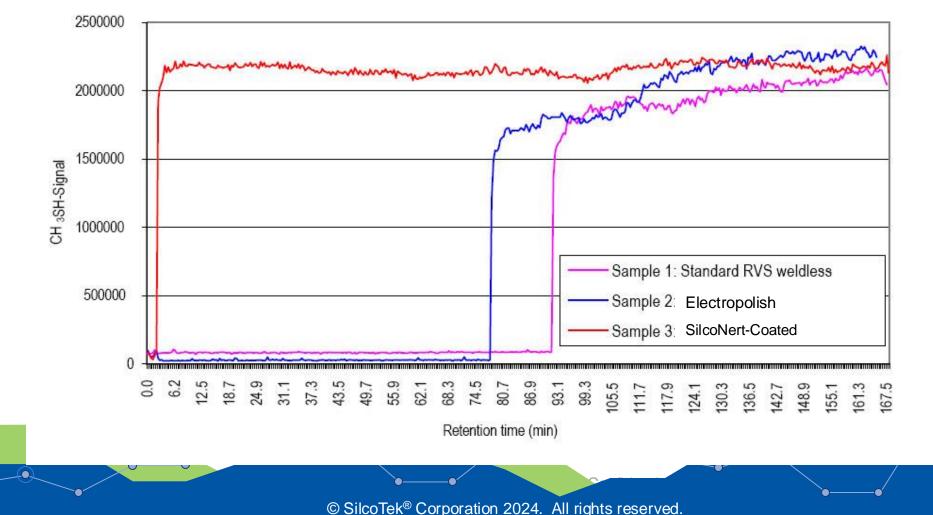


- Most chemical pathways consist of steel and stainless steel, which is problematic for analyzing sulfurs.
- Priming is a commonly used technique to self passivate surfaces.
- This process can take hours given concentration changes, and low-level analysis is often not possible.
- Coatings are proven solutions to improve reliability, speed, and accuracy of analytical results.
- Essentially... the smoother the better. Reducing surface area = lower chance of contact points.

### Instant Signal Response with SilcoNert-Coated Tubing – No Priming Required



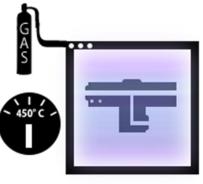
Methyl Mercaptan at 0.5ppm



# What are SilcoTek coatings?

- Amorphous silicon coatings applied via thermal CVD (chemical vapor deposition).
- Non-line of sight coating process ensures all surfaces are evenly and completely coated.
- Ability to coat complex geometries and a large variety of materials.
- Coatings are chemically bound to the substrate material.
  - Will not delaminate of flake off
- Nanometers thick and does not require retooling or recalibration of parts.
- Scalable, versatile, and highly reproducible.







# Today

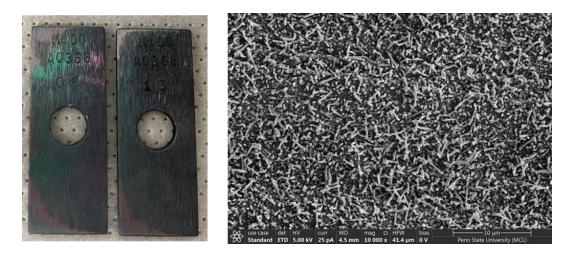


- SilcoNert 2000 (previously Silcosteel<sup>®</sup> and Sulfinert<sup>®</sup>) can make any stainless-steel analytical system component chemically inert.
- Other materials are now desired in chemical processes that have historically been incompatible with these inert silicon-based CVD coatings:
  - Monel<sup>®</sup> and Inconel are used for corrosion benefits to withstand harsh chemistries
    - However, there is still a need to measure sulfur compounds like  $H_2S$
  - Aluminum is used to reduce cost and weight
- Current supply chain restraints have led refineries and system integrators to ask "do we *need* electropolished tubing?"



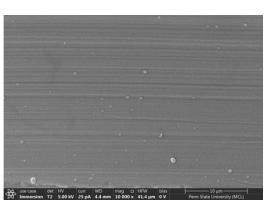
# SilcoNert 2000 Coated Monel®

Original SilcoNert 2000 process applied to Monel<sup>®</sup> 400 (top) showed poor appearance, adhesion, and surface quality (SEM 10k magnification). Reaction with substrate caused micro-crystallization of silicon.



New Monel<sup>®</sup> compatible SilcoNert 2000 process (bottom) shows clean, complete coating film growth that passes standard quality checks.







# Monel<sup>®</sup> Sample Cylinder Storage Test

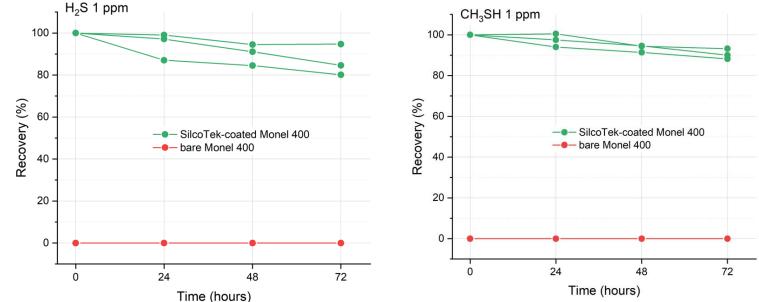
All traces of H2S and methyl mercaptan were completely gone at day zero.

Even the carbonyl sulfide (reference compound) was completely adsorbed.

Carbonyl sulfide is used because it does not adsorb to SS.

Monel is much more active than SS and presents a higher challenge in sulfur analysis.

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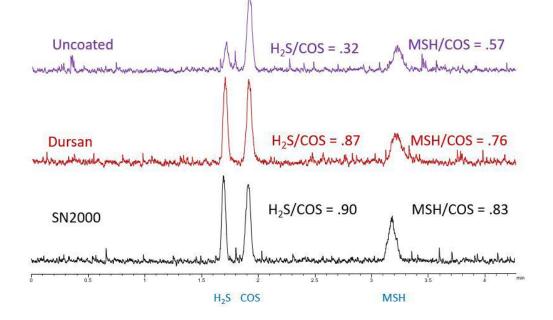
## SilcoNert 2000 Coated Inconel 600

We tested coated Inconel (SilcoNert 2000 and Dursan) and uncoated Inconel using active sulfur gases known to adsorb to metallic surfaces.

Uncoated showed a high degree of adsorption of reactive sulfur gases. Adsorption is highly reduced or eliminated when the flow path is coated with either Dursan or SilcoNert 2000.

Less adsorption leads to improved peak shapes.



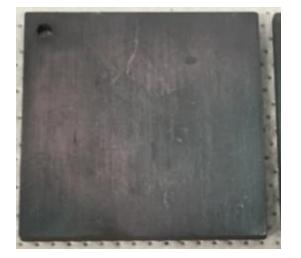


Silco



### Aluminum

- Aluminum is commonly used for gas cylinders and is a desirable alternative to steels, due to its cost, weight, and material quality.
- Like Monel<sup>®</sup>, aluminum has historically been a challenge for silicon CVD coatings to be applied.
  - Original coating process catalyzes silicon crystallization and promotes "nanowire" growth on the substrate.
  - This leads to an inconsistent and unpredictable coating output and creates a negative impact on appearance and performance (especially corrosion resistance).

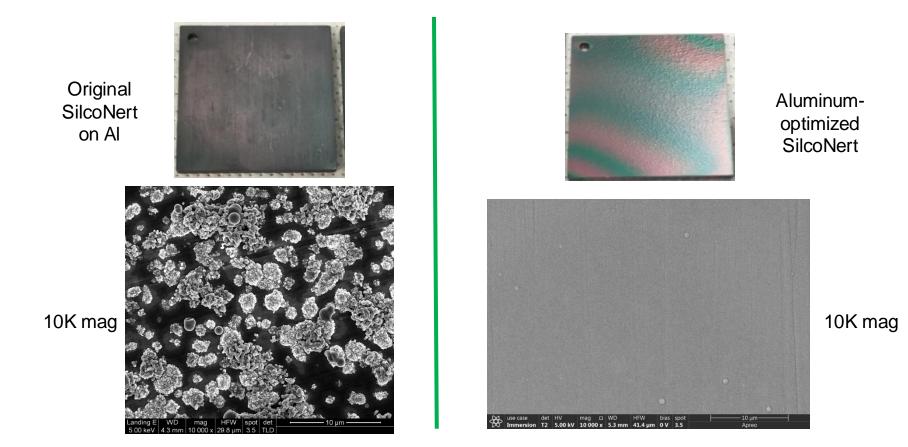






### Original SilcoNert vs. Hybrid SilcoNert on Aluminum

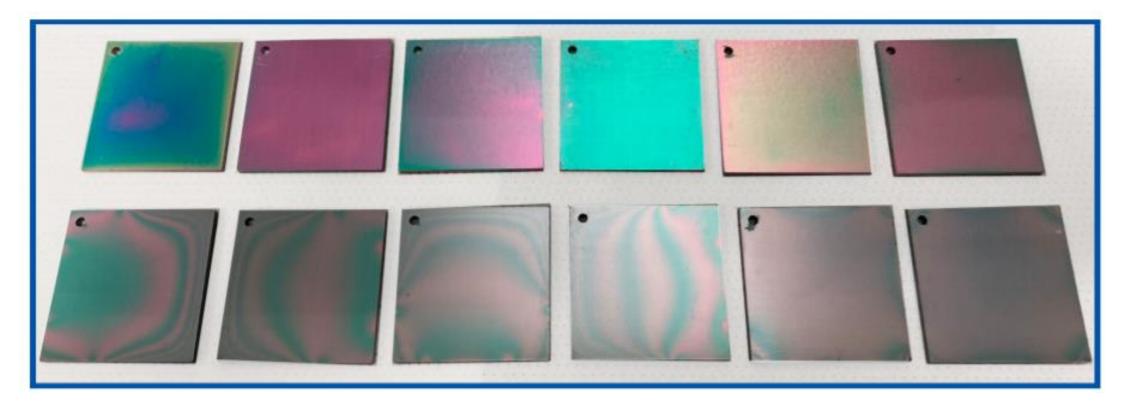




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# Coated aluminum using new optimized SilcoNert 2000 process



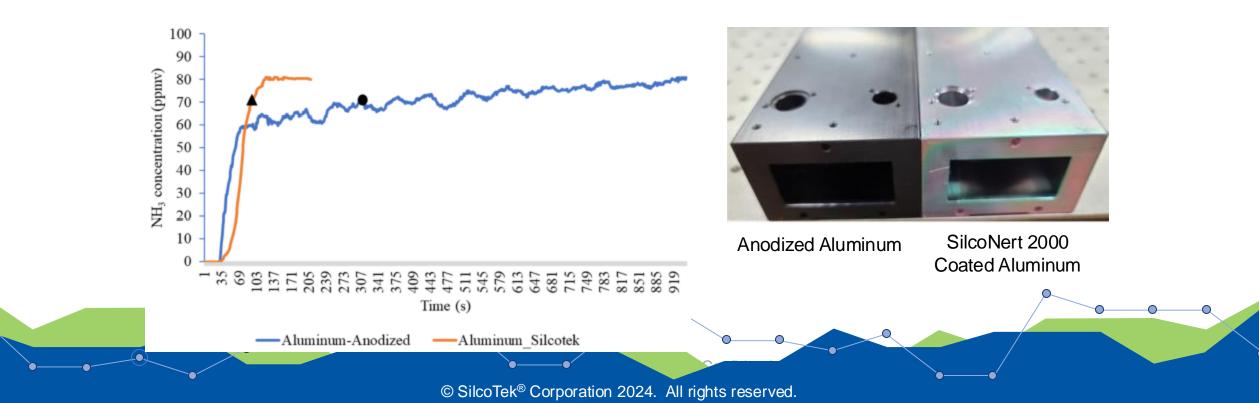




# Ammonia Analysis using Aluminum



- Ammonia injection comparison between an anodized chamber and SilcoNert 2000 chamber.
- SilcoNert 2000 coated chamber quickly reached the 90% mark while the anodized aluminum experienced high variability before and after the 90% mark (while taking dramatically more time).
- While an anodized aluminum chamber had inconsistent results, a chamber coated with SilcoNert 2000 yielded fast and consistent results.



# Is Electropolishing (EP) Required?



- EP is NOT required to deposit SilcoNert 2000 or other silicon coatings.
- However, data shows that a smoother surface reduces active sites and can make notable improvements (especially at ppb-levels of analysis<sup>2</sup>).
- How does SilcoNert 2000 coated stainless steel tubing without EP compare to non-coated tubing with EP?

<sup>2</sup>Harris, P. Pelligrini, M. Mass transport in sample transport lines adsorption desorption effects and their influence on process analytical measurements. https://www.silcotek.com/hs-fs/hub/22765/file-13441094.pdf?



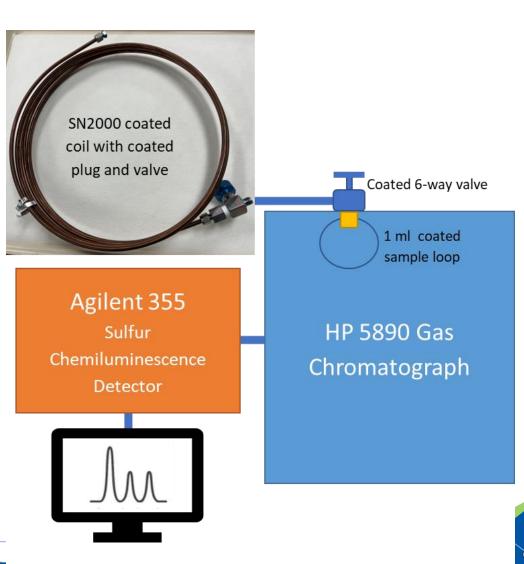


# EP + SilcoNert vs. No EP + SilcoNert

- 20' lengths of 1/4" OD stainless steel tubing.
  - No EP + no SiloNert
  - No EP + SilcoNert
  - EP + no SilcoNert

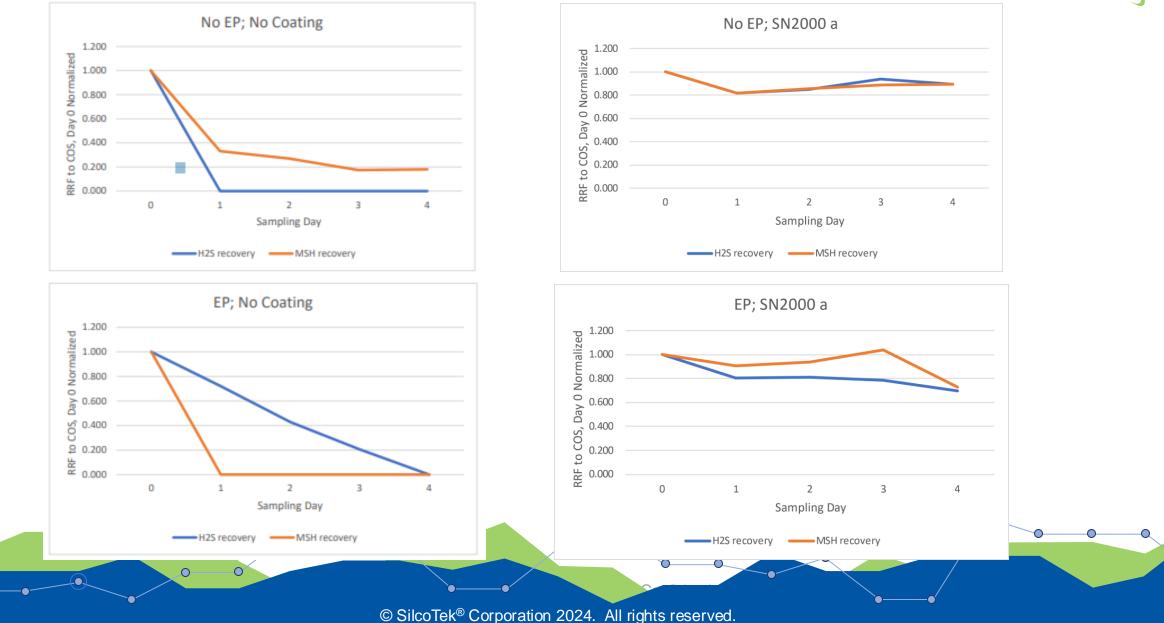
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- EP + SilcoNert
- Connected to HP 5890 GC with Agilent 355 SCD
- Test mix containing hydrogen sulfide (H<sub>2</sub>S), carbonyl sulfide (COS), and methyl mercaptan (MeSH) at 20ppbv



# **Results**: EP showed no significant impact on the inertness of the analytical system for measuring 20ppb sulfurs mix. **No EP + SilcoNert performed best**.





## Takeaways on Electropolishing



- A quality electropolish (EP) will always result in smoother surfaces and thus fewer potential sites for chemical activity.
- An ultra smooth surface can be important for extremely sensitive analysis (ex. ppb or ppt level moisture).
- In most analytical applications, EP + coating does not show a statistically significant advantage in testing.
  - In fact, EP + coating was less inert in the previous test. This may be caused by high variation in EP quality.
- This information may help the industry overcome rising costs and material lead times.



## Conclusion



- Samples are becoming more complex while detection limits are continuously lowered to meet evolving technical and regulatory needs.
- Inert coatings within sampling and analytical flow paths are required to enjoy the many benefits of metallic sample system construction.
- As a solutions provider to the chemical analysis industry, SilcoTek continues to invest in its capabilities to meet the industry's requirements.
- SilcoNert 2000 coating is now available on materials that have historically been impossible to coat like Monel<sup>®</sup>, aluminum, and more.





# Thank you!

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