



SilcoTek's Inert CVD Coatings for Analytical Applications

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Coating

SilcoNert®

Ultimate chemical inertness and compatibility

Dursan®

Inert, durable, corrosion resistant

Common Applications

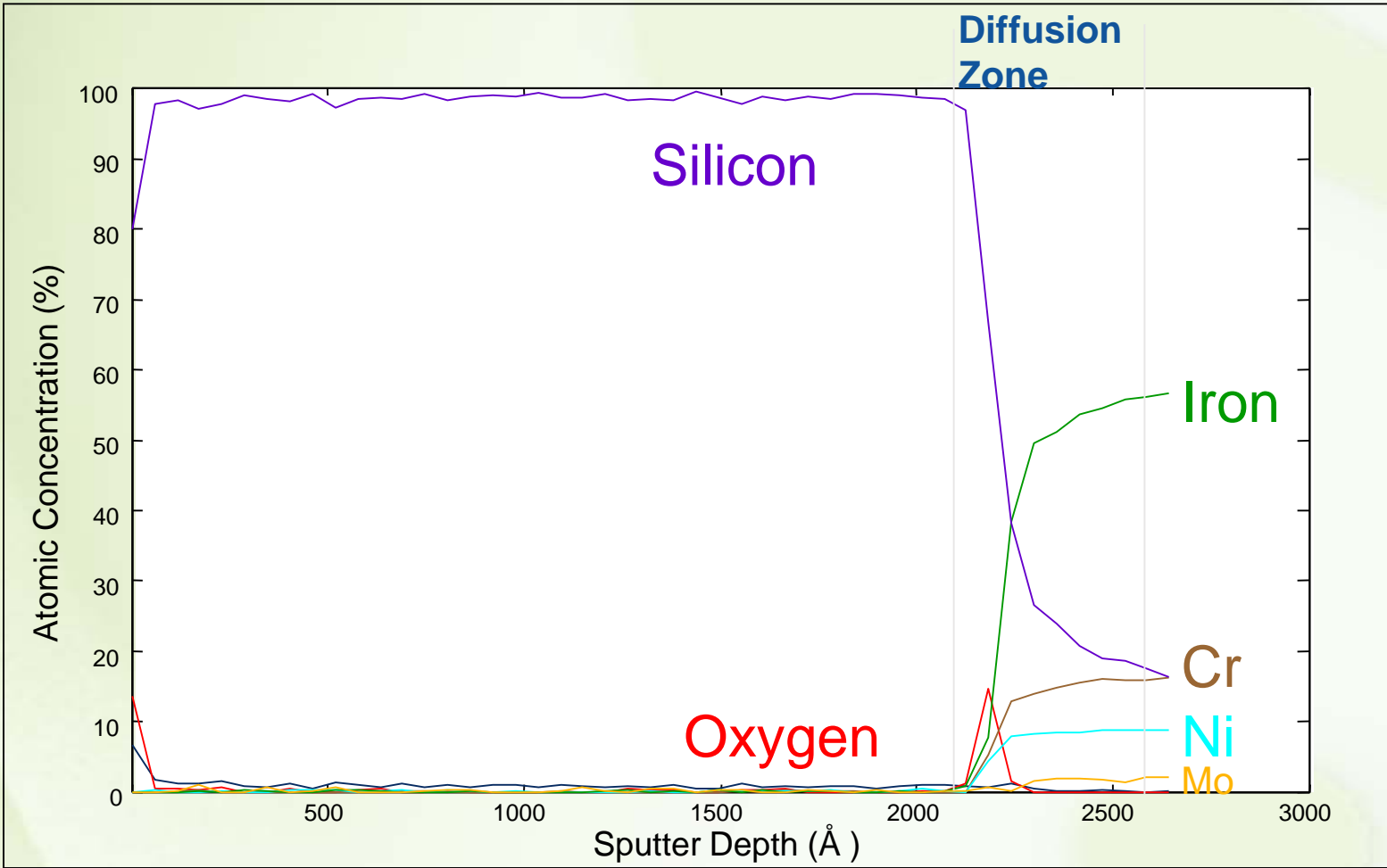
- Analytical chemistry
- Environmental
- Process analytical

- HPLC & Bioanalytical
- pH 0-14
- Refinery/Petrochemical
- Abrasive applications

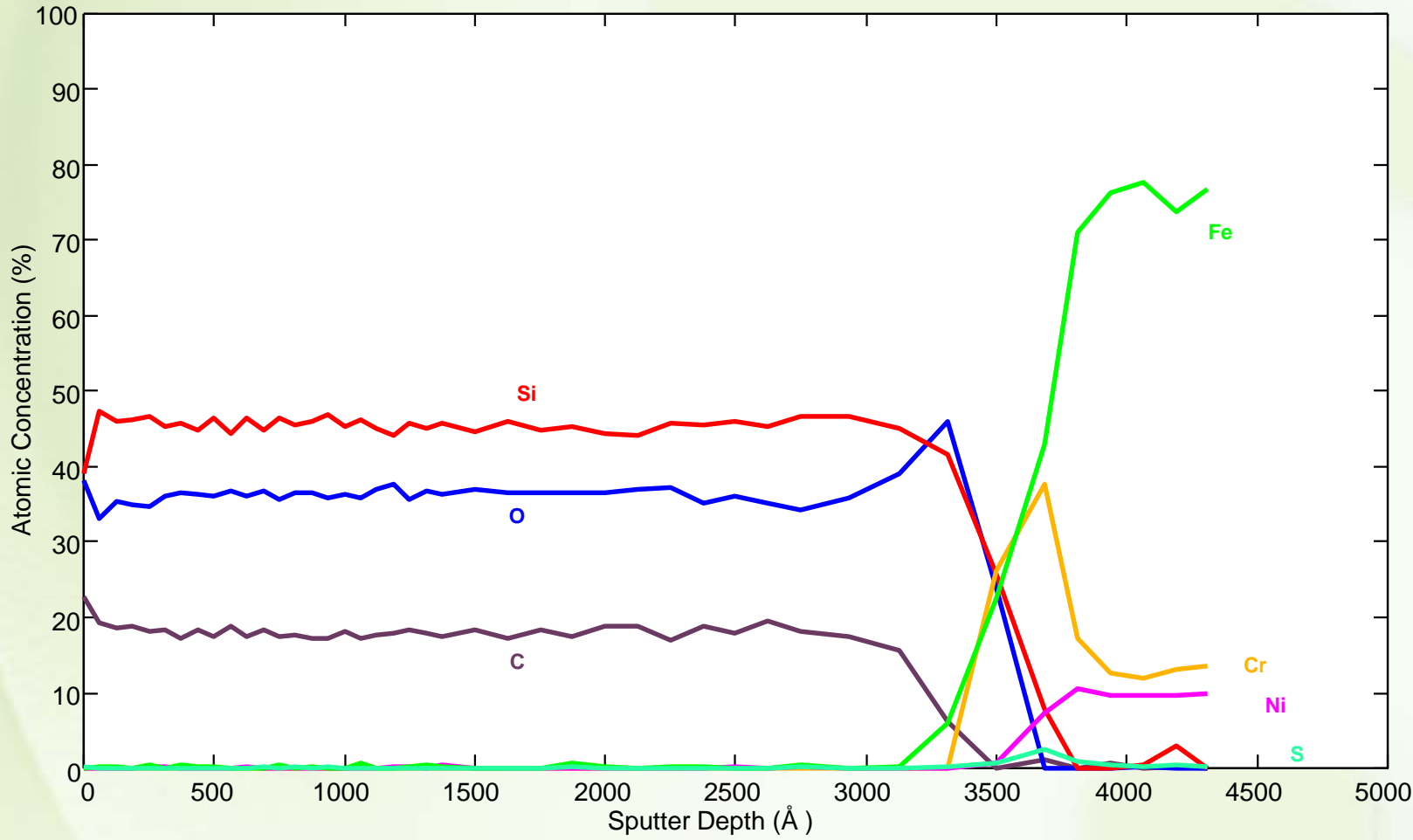
Coating Properties

Composition and Physical Properties

Auger Depth Profile: Silco-



Auger Depth Profile: Dursan



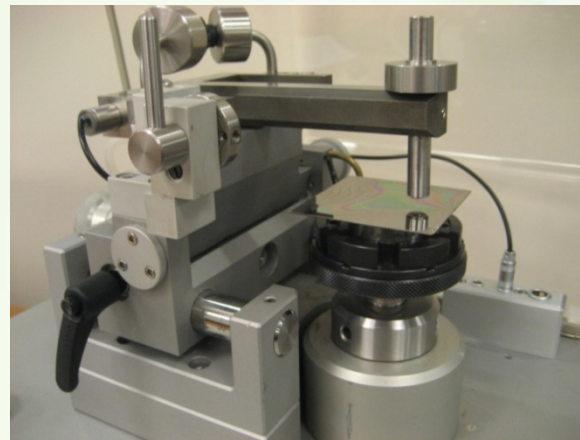
SilcoTek Coatings vs. PTFE

| Property | Silco- Coatings | Dursan | PTFE |
|------------------|-----------------------------|------------------|------------------|
| Max Temperature | >900°C, substrate dependent | 450°C | 260°C |
| Min Temperature | -196°C | -100°C | -240°C |
| Low pH limit | 0 | 0 | 0 |
| High pH limit | 7 | 14 | 14 |
| Thickness | 0.12um to 0.8um | 0.3um to 1.5um | 25um |
| Adhesion | Very Good | Very Good | Poor |
| Wear Resistance | 90% of Stainless | 2x 316 Stainless | 10% of SS (est.) |
| Moisture Contact | 72-90° | 104-140° | 125° |
| Inertness vs. SS | Superior | Good | Excellent |

Wear Resistance

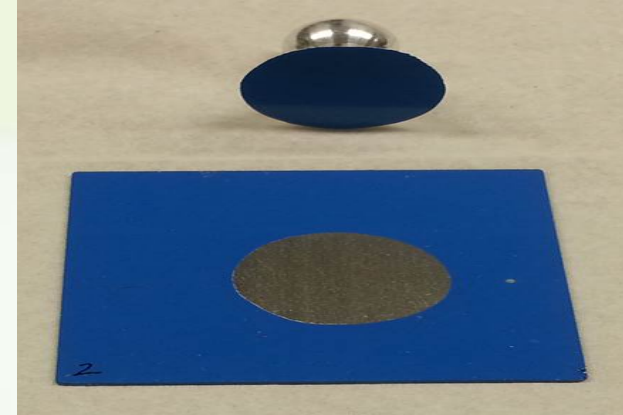
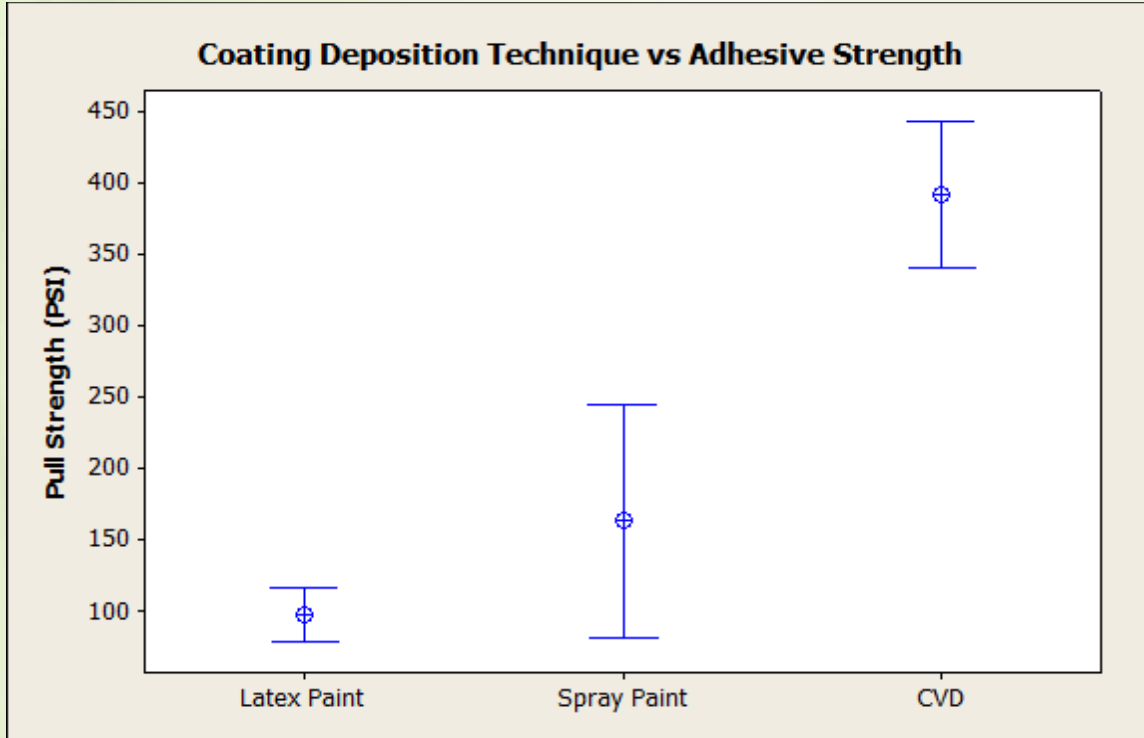
| | <u>Avg. Coeff. Friction</u> | <u>Wear Rate (x10⁻⁵mm³/Nm)</u> |
|-------------|-----------------------------|--|
| Uncoated SS | 0.589 | 13.810 |
| Dursan | 0.378 | 6.129 |
| Silcolloy | 0.7 | 14.00 |

- ASTM G133
on 316 SS
mirror finish



| | |
|---------------|--------|
| Load | 2.0 N |
| Duration | 20 min |
| Speed | 80 rpm |
| Radius | 3mm |
| Revolutions | 1,554 |
| Ball Diameter | 6mm |
| Ball Material | SS 440 |

Adhesion



Reliable Sampling, Testing and Analysis with SilcoNert®

Applications, data, and benefits



Why is an inert flow path critical?

- Reduce costs and downtime
 - No re-testing needed
 - Accurate analytical profile of all trace compounds
- Trust your sample
 - Eliminate false negatives
 - Get a reliable sample from field to lab
- Immediate, reliable response

Stainless Steel not Good Enough

- Stainless steel common for analytical pathways
 - Good structure, resists corrosion
 - Poor chemical properties for chemists
- Coatings address many failures of bare Stainless Steel:
 - Chemical reactivity
 - Aggressive corrosion
 - Abrasion
 - Moisture



Stainless Steel

- Stainless steel adsorbs trace compounds:
 - Adsorbs/reacts with reduced sulfurs, methylmercaptan, H₂S
 - Causes loss of ammonia and mercury
 - Leads to “tailing” of polar organics e.g. alcohols

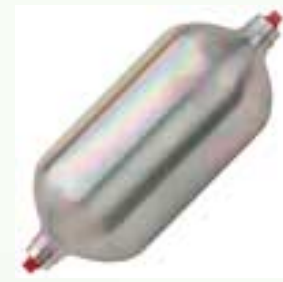
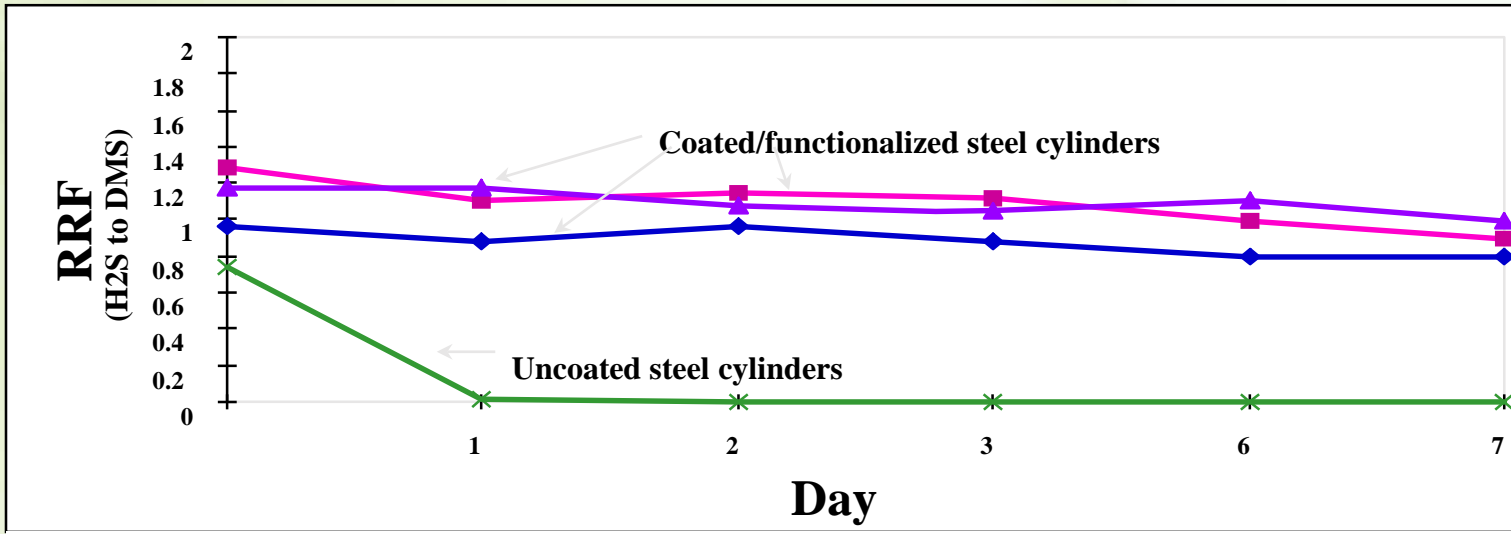
Copper and Brass

- Brass substituted for stainless in cost cutting efforts
- No sulfur data from a system containing these materials is to be trusted.
- Copper alloys cannot benefit from the Silicon/Silco- based coatings.
- Test data on Monel® and sulfurs well-presented by Hashem, et. al.,¹ from Schlumberger-Oilphase DBR
 - Samples at 50ppm (relatively high nowadays) are scavenged in Monel® tubing

¹Hashem, et. al, "Low-Level Hydrogen Sulphide Detection using Wireline Formation Tester", International Petroleum Technology Conference, IPTC 11582, (2007)

Solution: **SilcoNert**[®]

- **Performance:** part-per-billion (ppb) analysis
- **Protection:** prevents moisture contamination
- **Confidence:** a reliable sample you can trust

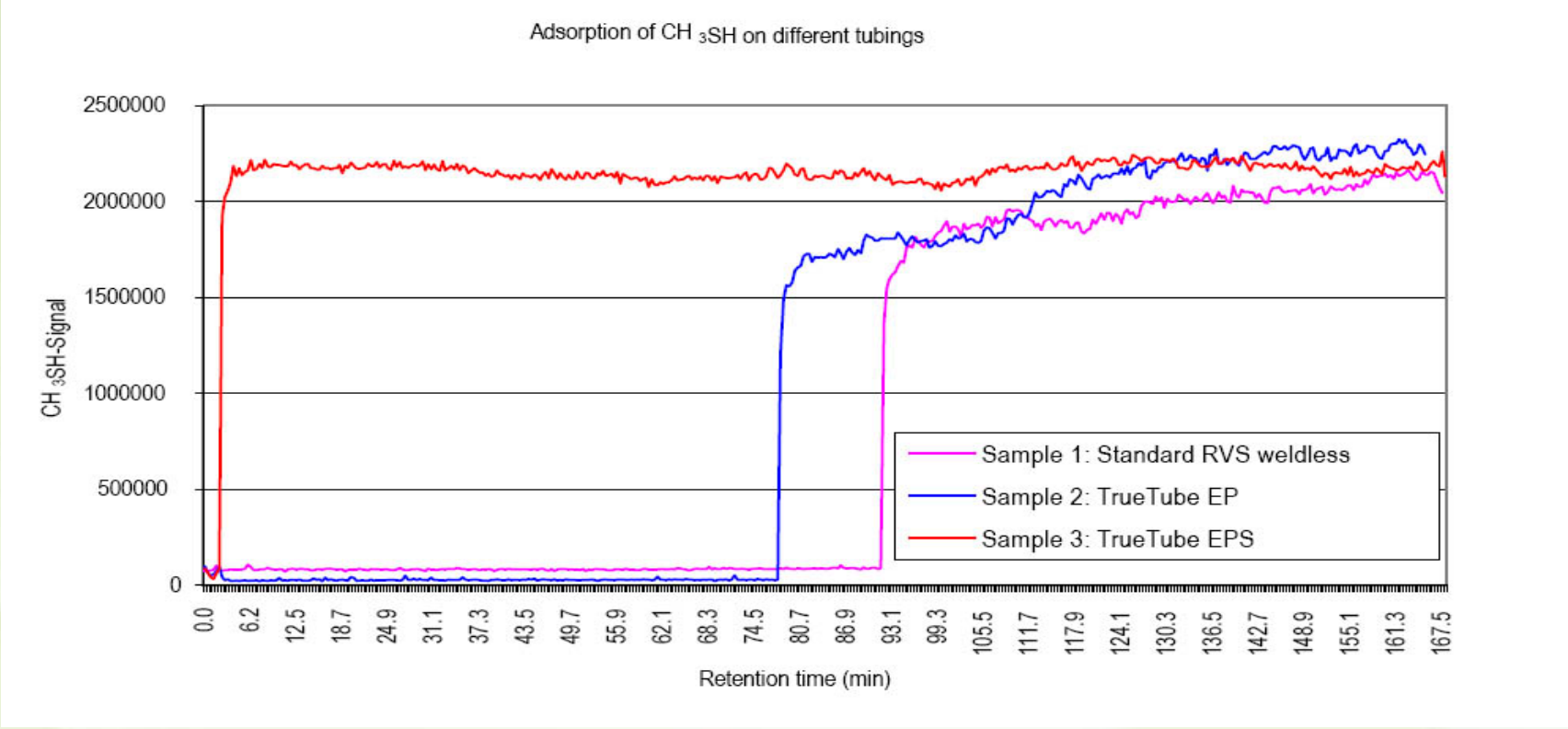


17ppb H₂S in Helium

Example: CH₃SH Flow-Through

- 100' 1/8" x .020" tubing
 - Standard seamless 316L
 - Electropolished 316L
 - SilcoNert[®] coated EP 316L
- 0.5ppmv methylmercaptan in helium
- SCD detection
 - Data courtesy of Shell Research Technology Centre, Amsterdam

No transfer loss



Current Applications

- [Refinery flare gas testing](#)
- [Oil and Gas well downhole sampling](#)
- [Ethylene/Propylene catalyst poisons](#)
- [Coal Fired Boiler Flue Gas testing](#)
- [Ammonia slip](#)
- [Tier 3 gasoline and diesel regulation](#)
- [HPLC](#)
- [Bioanalytical](#)



Refinery flare gas

- Davidson, et. al.² published data on refinery flare gas monitoring systems for stability over a year.
- This was then California Rule 1118, which is direct predecessor of Subpart –Ja regulations
- System showed great stability over 1 year reporting period.
- No impact from upsets on system performance

² Davidson, T.; et. al. "Performance of Environmental Monitor For Total Sulfur and High Heating Value of Refinery Flare Gas System." ISA 56th Analysis Division Symposium 2011, League City, TX (2011)

Refinery Flare gas Study finding continued

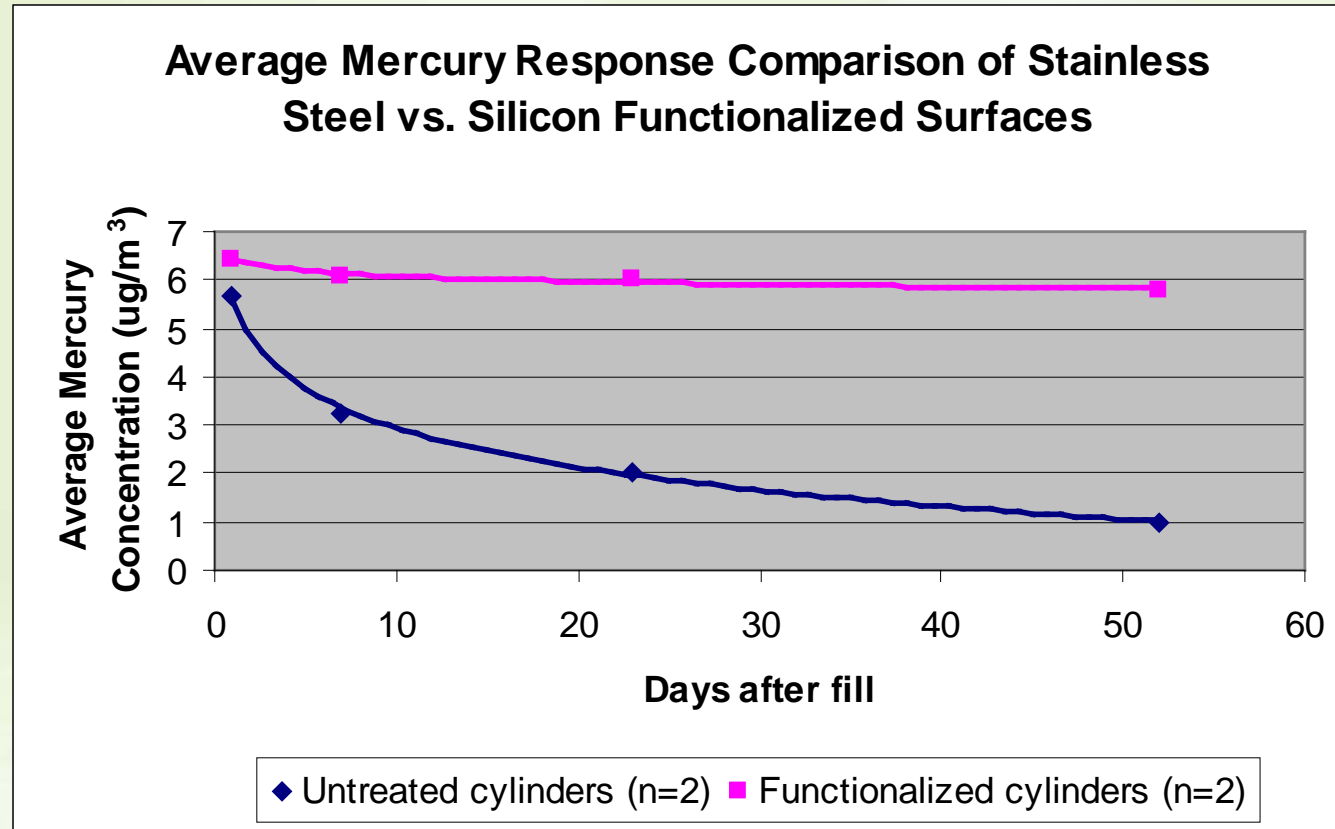
- Monitoring range from 1 to 150,000 ppm total sulfur
- Vent gas measure at middle 50% of flare with angled coated probe to get representative sample
- Concluded need for stable instrumentation, heated sample system and inert coating

Oil and Gas Well Sampling

- Need to quantify sulfur content of new wells
- Growing concerns now requiring monitoring of mercury content as well
- Presentations by Schlumberger in 2007¹ and 2013³ highlight the application of coatings to provide stable sample bottles.
- Any level of mercury (measured in $\mu\text{g}/\text{m}^3$) is of interest because of mass volumes being pumped
- Need to quantify sulfur content of all wells for quality and safety standards

³ Harfoushian, J. "Quantification of Low Levels of Mercury in Gas Reservoirs Using Advanced Sampling and Analysis Techniques" Society of Petroleum Engineers Annual Conference, SPE166220 (2013)

5 $\mu\text{g}/\text{m}^3$ Hg Standard Stable for Weeks



Ethylene/Propylene

- Study presented by Biela, et. al. from Air Liquide⁴
- Sulfur contamination causes catalysis poisoning relating directly to reduced yields
- H₂S (hydrogen sulfide) and COS (carbonyl sulfide) coming over in polymer-grade Ethylene and Propylene
- Conversion of COS in furnace to H₂S and then contact with catalysts.

⁴ Biela, B.; et. al. "The Do's and Don'ts in the Analysis of Sulfur for Polyolefin Producers"; Gulf Coast Conference, Galveston, TX, Paper 081 (2003)

Ethylene/Propylene

- Poison levels very low (Propylene)
 - 10ppb COS
 - 50ppb CS₂
 - 1ppm Dimethyl Sulfide (DMS)
- Manufacturing Specifications for monomers: 50ppb H₂S (ethylene); 20ppb COS (propylene)
- Sampling systems and standards are necessary to keep yield high

Coal Flue Gas

- Emissions of mercury in Coal Flue Gas from Boilers is now a monitored pollutant
- Also effluent from refining, petro activities that are monitored
- Problem is the oxidation of mercury and inability to analyze due to loss
- Coatings applied to sample probes, transfer lines, inertial filters eliminated adsorption
- Studies done on oxidized mercury Hg^{+2} demonstrate 100% transfer of these adsorptive compounds in coated transfer lines⁵.

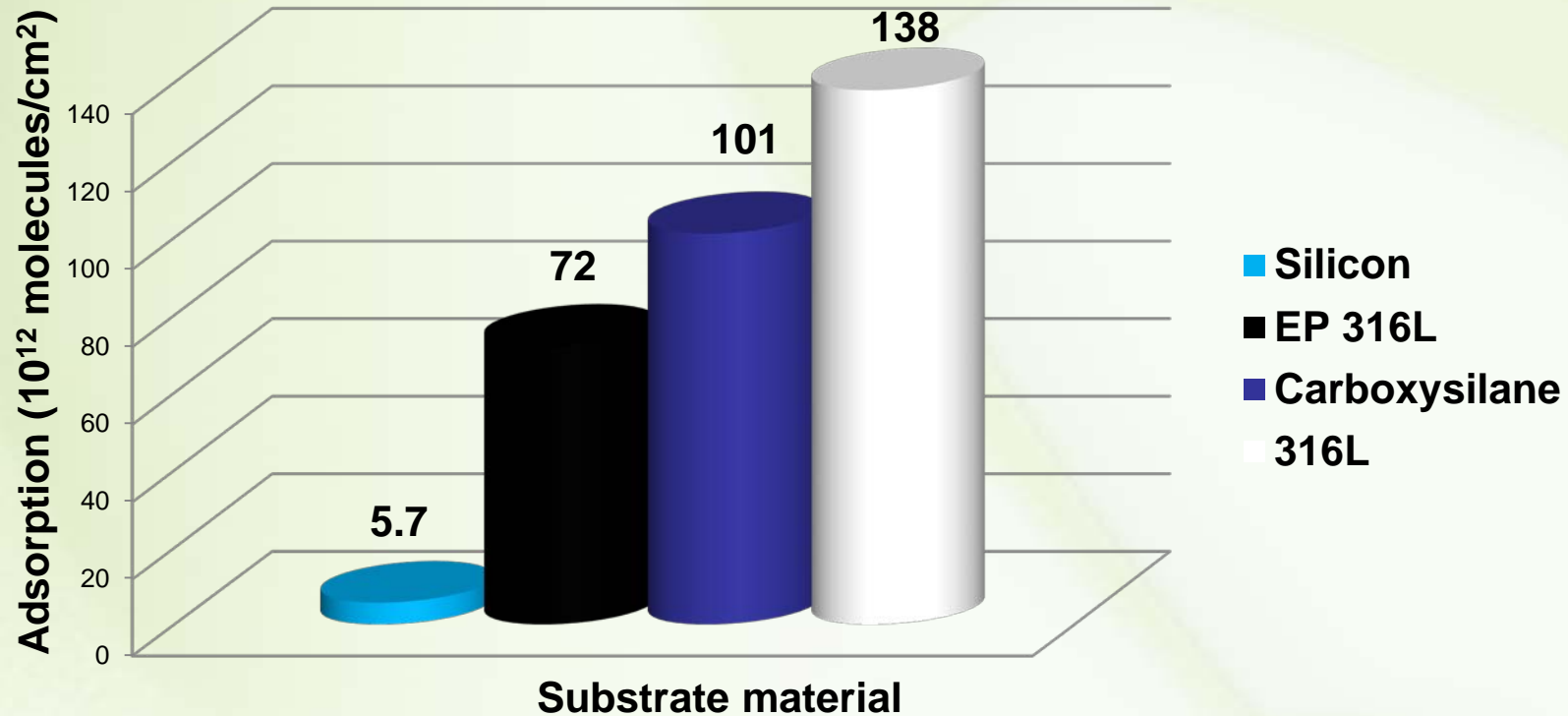
⁵ Lan, X.; et. al. "Seasonal and Diurnal Variations of Total Gaseous Mercury in Urban Houston, TX, USA"; *Atmosphere*, 5, pgs. 399-419 (2014)

Ammonia

- Ammonia injection common means of reducing sulfur emission
 - Coal fired boilers
 - Sulfur scrubbers
- Becoming a pollutant of concern
- Can also be harmful to equipment

Reducing adsorption of ammonia⁶

Adsorption totals of ammonia on different substrates

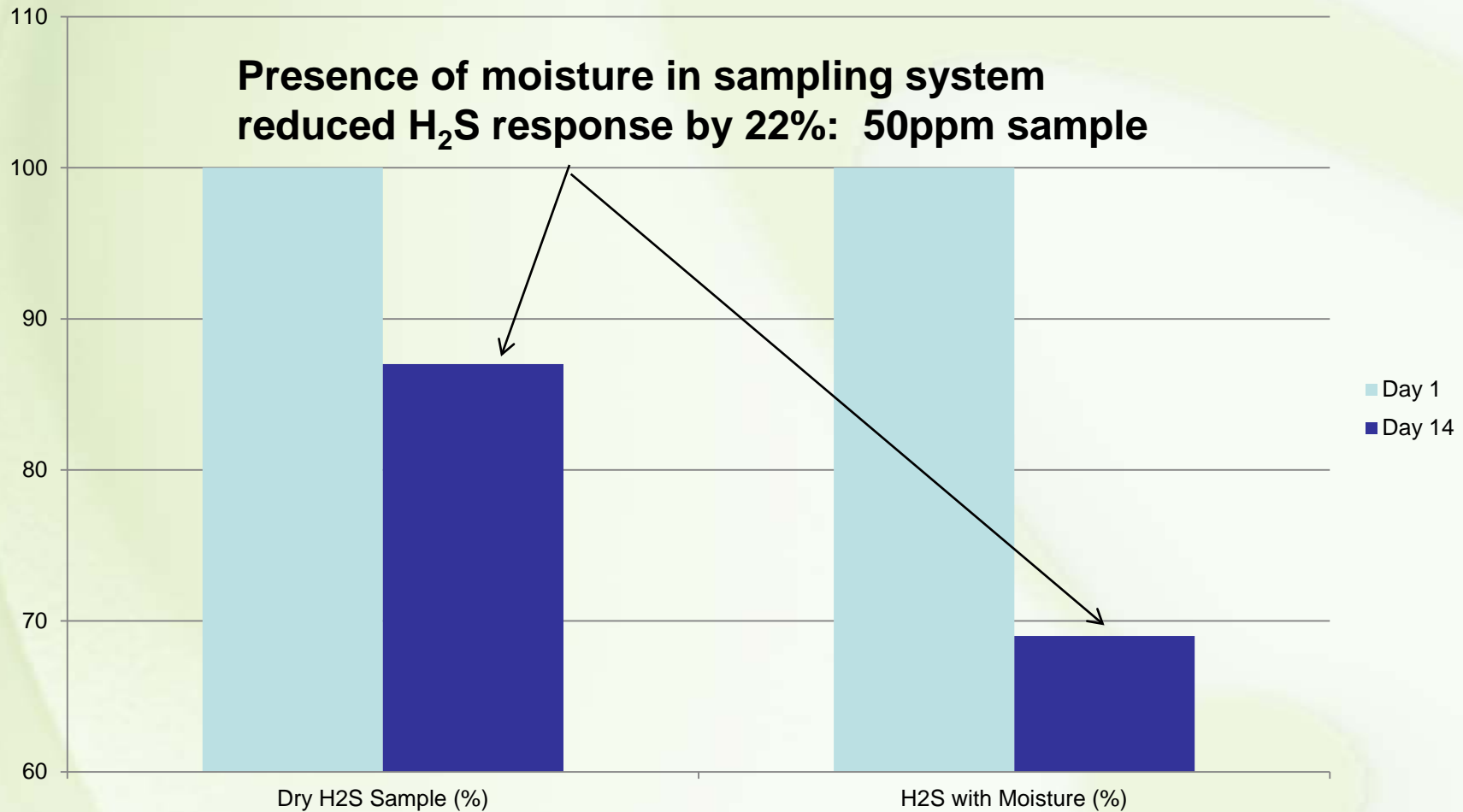


⁶Vaittinen, et. Al., "Adsorption of ammonia on metal and polymer Surfaces", University of Helsinki, 2013

Tier 3 Fuel Standards

- Sulfur in Gasoline from 30ppm to 10ppm starting 2017
- Old tricks of priming sample system will not work
- Coatings will play important role
- Common sources of sulfur adsorption:
 - Probes
 - Tubing
 - Metal filters
 - Sample Cylinders
 - Regulators
 - Fittings
 - Valves
- Get testing systems ready now.

Impact of Moisture



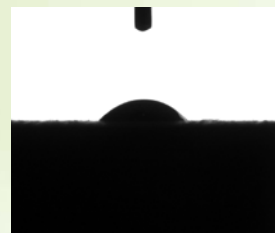
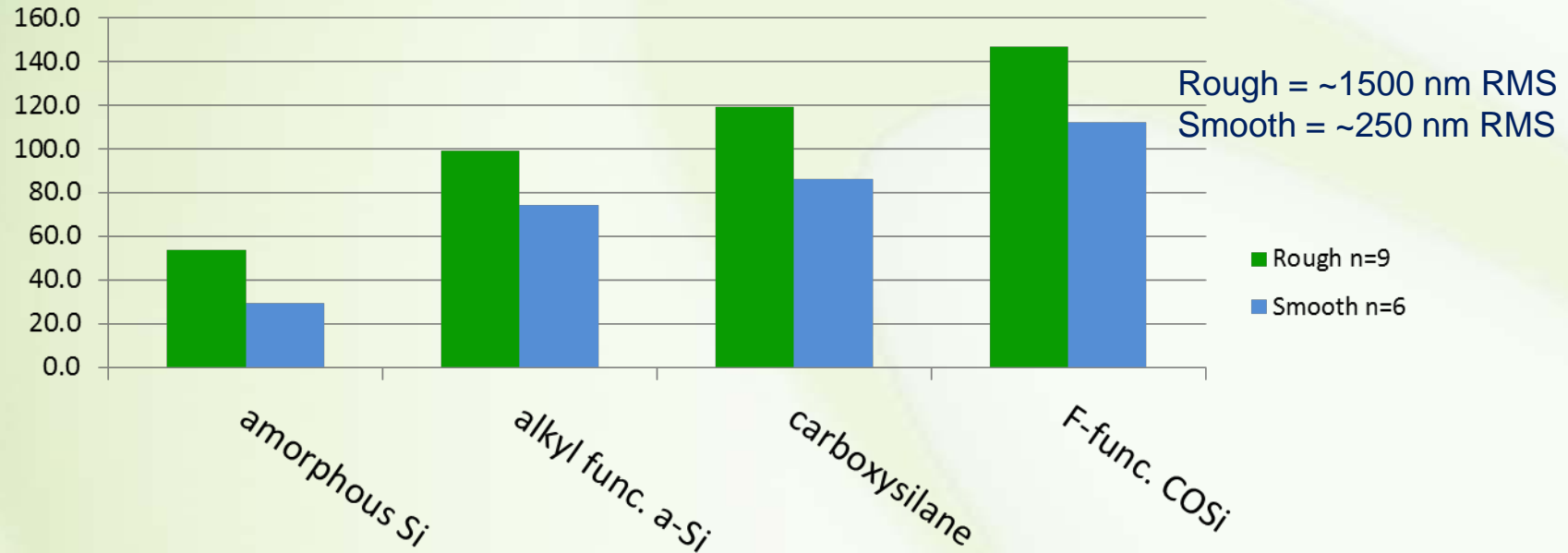
Example: Moisture Wet-Up

- 1ppm moisture, 0.35slpm
- Amount of time to equilibrate a 1ppm moisture sample through 100 feet of dry tubing:
 - Commercial Seamless 316L tubing
 - 180 minutes (96% equilibrated)
 - Electropolished (EP) Seamless 316L tubing:
 - 60 minutes (98% equilibrated)
 - SilcoNert coated EP seamless 316L tubing
 - 30 minutes (98% equilibrated)

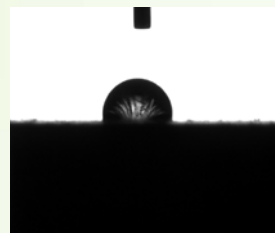
Example: Moisture Dry-Down

- Time to dry 100' tubing wetted with 1ppm of moisture when connected to a dry purge
 - Commercial Seamless 316L tubing:
 - 175 minutes
 - Electropolished Seamless 316L tubing:
 - 65 minutes
 - SilcoNert coated e-polished seamless 316L tubing
 - 35 minutes

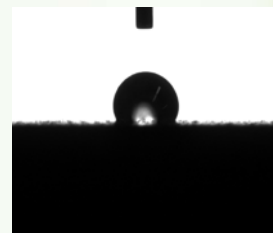
Hydrophobicity Measurement



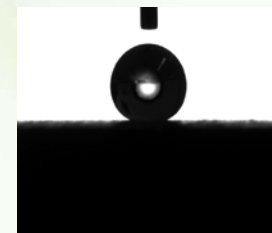
amorphous Si
Silcolloy
49°



alkyl func. a-Si
SilcoNert
101°



Carboxysilane
Dursan
121°



F- func.
FluoroDursan
163°

SilcoNert[®] Summary

- Most inert coating available on the market
 - Allows for as low as parts-per-trillion analysis
- Functionalized a-Si surface is stable at high temperatures (450°C)
 - Can be bent and flexed, allows for testing in challenging environments
- Upgrade system without redesign or remanufacture

**When a robust surface
is as critical as an inert
surface:**

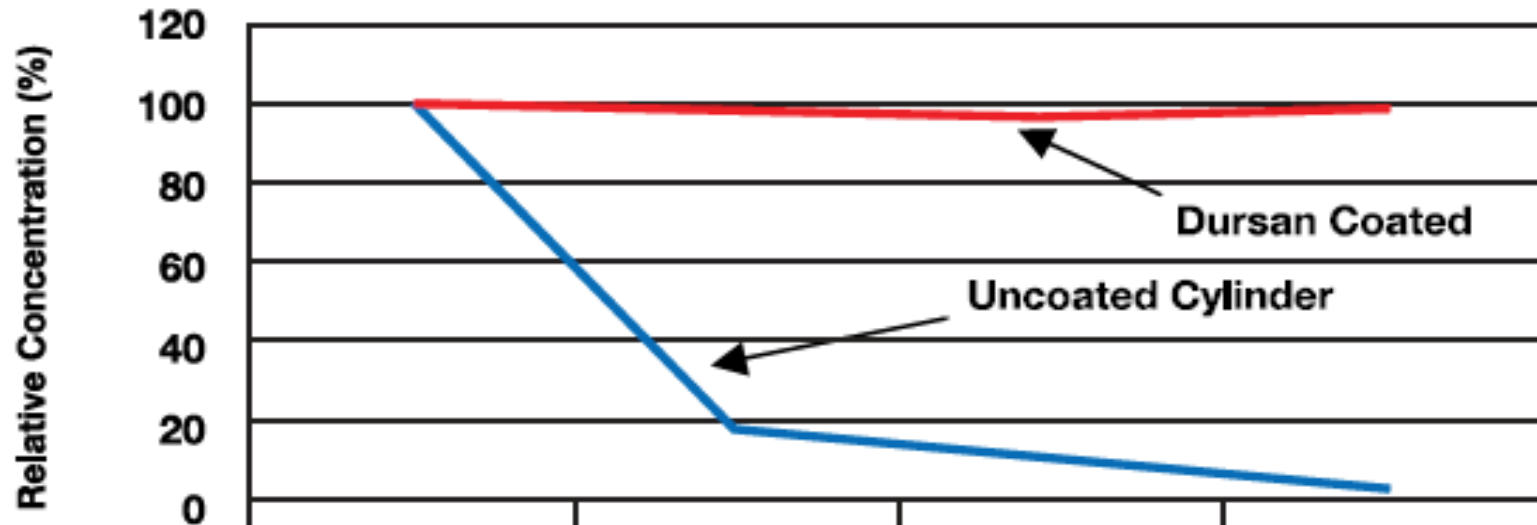
The Dursan logo is centered on the slide. It features the word "Dursan" in a blue sans-serif font, with a green swoosh element above the "r" and "s". A registered trademark symbol (®) is located to the right of the word.

Dursan®

- Developed to withstand rigors of down-hole oil and gas sampling
- Protection from the full pH range 0-14
- Tough + inert surface suitable for more robust analytical applications:
 - HPLC, refining, clinical diagnostics, etc.

H₂S Stability: Dursan vs. Stainless Steel

50ppmv, 300cc cylinder



| | 0:00 | 25 hours | 50 hours | 75 hours |
|-------------------|------|----------|----------|----------|
| — Stainless Steel | 100 | 19 | 10 | 4 |
| — Dursan | 100 | 98 | 96.6 | 98.3 |

Future Work

- Data Proving Dursan's benefits for HPLC:
 - Bio-inert
 - pH stable 0-14
 - Corrosion resistant
 - More robust than PEEK and PTFE
 - Better physical durability
 - Higher temperature capability (450 °C)

SilcoTek Coatings as a Solution

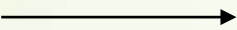
- Drastically increase analytical reliability, accuracy, and throughput
- A required treatment for ultimate surface inertness
- Trusted in environmental labs, refineries, oil and gas fields, and process environments since the 1990s

White Paper: Improving Analytical and Sampling Reliability

Analytical and/or process system flow paths are typically constructed of stainless steel or glass, but these materials contribute to poor sampling reproducibility and accuracy. Inert surfaces are critical to repeatable and precise results.



Download the white paper



Download Now

Contact SilcoTek

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