# Welcome to the webinar!

## How to Improve LC and GC with SilcoTek's Inert Coatings

We will begin 2 minutes past the hour

Your Presenter:

**Dr. David Smith**

SilcoTek R&D Manager

David's team is responsible for developing and improving coating solutions for SilcoTek customers. A founding member of the original Restek Performance Coatings division and pioneer of SilcoTek's SilcoNert 2000 (formerly known as Sulfinert) coating technology, he has been helping chromatographers solve problems with sample recovery, reliability, and overall performance since 1992.

# Welcome!

Webinar will be recorded and emailed to you

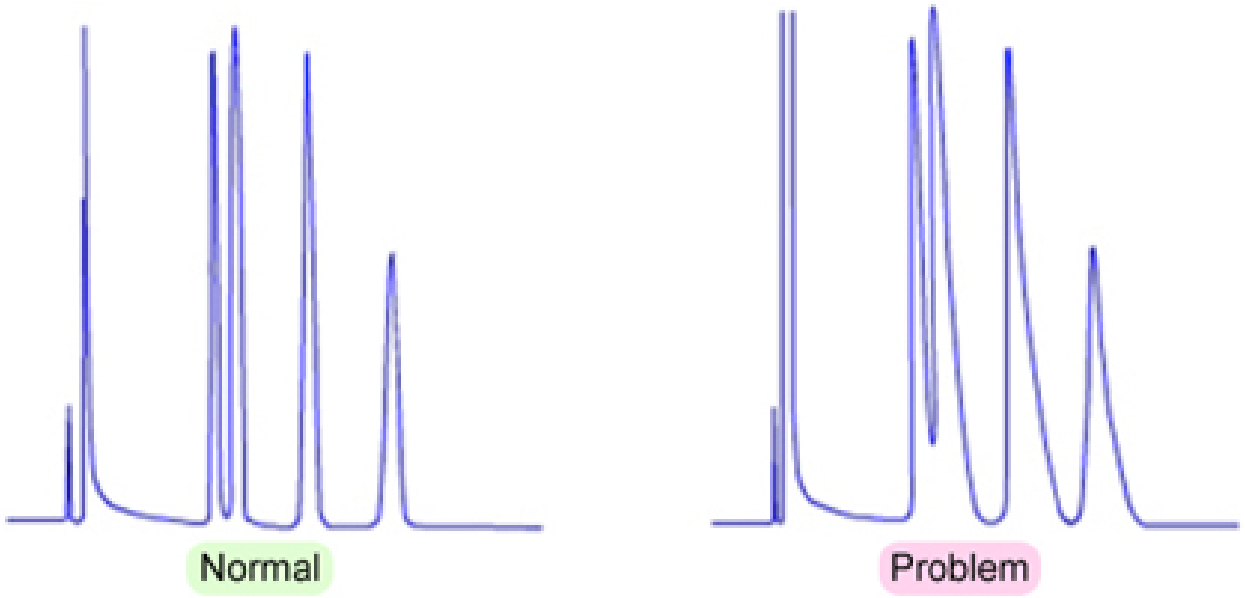
- You can find it any time on SilcoTek.com

#AskSilcoTek on Twitter (@SilcoTek)

**?** Send questions via message bar

# Outline

- SilcoTek's History with Chromatography
- GC and SilcoNert 2000
  - Inert pathways and Coatings
  - GC System, Components, Data
- LC and Dursan
  - Inert pathways and Coatings
  - LC System, Components, Data
- Conclusions, Questions



# Brief History – Our Start in Chromatography

RESTEK® invents SilcoSteel® in 1987 to make stainless steel capillary columns act like glass

Paul Silvis – Founder of Restek® and SilcoTek®.
Current President of SilcoTek.

# SilcoTek History

- Throughout the 1990s, a team dedicated to the SilcoSteel® technology began to experiment with custom coating for various uses.
- Demand grew for coatings outside of chromatography applications.
- **2009:** Restek coatings group splits off and forms SilcoTek®, an independent company

# **2013:** 3,400 $m^2$ state-of-the-art coating facility opens, tripling previous coating capacity

# Why do you need an inert pathway?

- Reduce loss of active compounds
- Avoid false negatives because active compounds can be quickly lost on active surfaces.
- Improved sample transfer
- Prevent carryover and cross-contamination
- Sample integrity maintained from field to lab
- Immediate response during process changes
- Creates savings when used in feedback / feed forward monitors

# How does a CVD Coating Help?

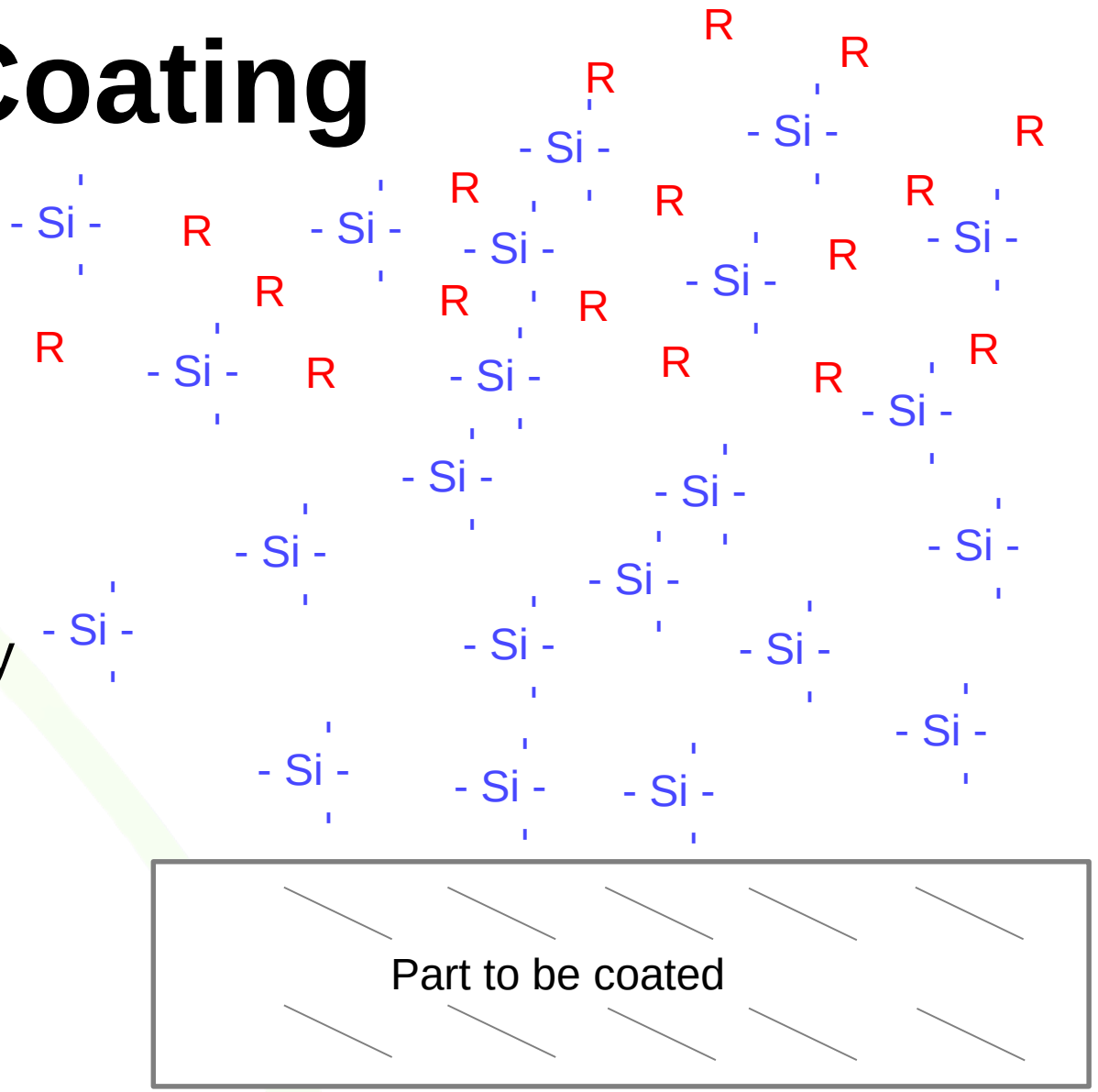
- Control of the surface
  - Deposit a pure, known material
  - Allows for further chemistry to tailor surface properties
- Covers all exposed surfaces
  - Active metallic substrates eliminated
  - Eliminate Si-OH activity on glass substrates

# What are SilcoTek Coatings?

- Barrier Coatings - Prevent interaction with the surface of a part
- Why?
  - Inertness to active compounds
  - Hydrophobicity
  - Prevent protein sticking
- SilcoNert 2000 (Primarily GC)
- Dursan (Primarily LC)

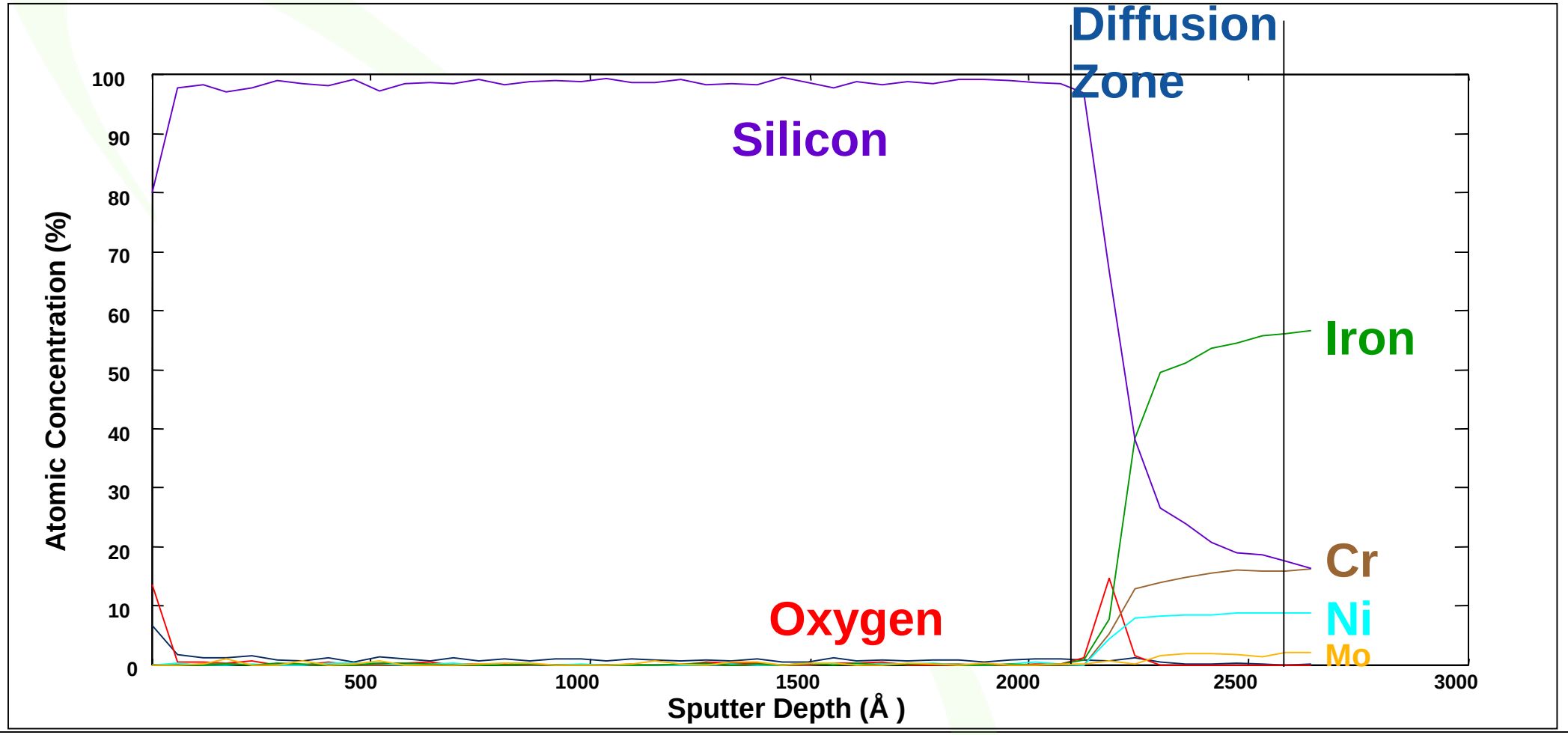
# The Coating

- Coatings consist of a Base Layer and a Surface Layer
  - Base is 150-1600nm of:
    - Si (SilcoNert) or
    - Si – C – O (Dursan)
  - Functionalized surface – Chemistry is key to performance




- SilcoNert 2000 (SN2000)…
  - aka Siltek
  - aka Sulfinert

# AES Depth Profile: SN2000 – Surface Functionalized Amorphous Si



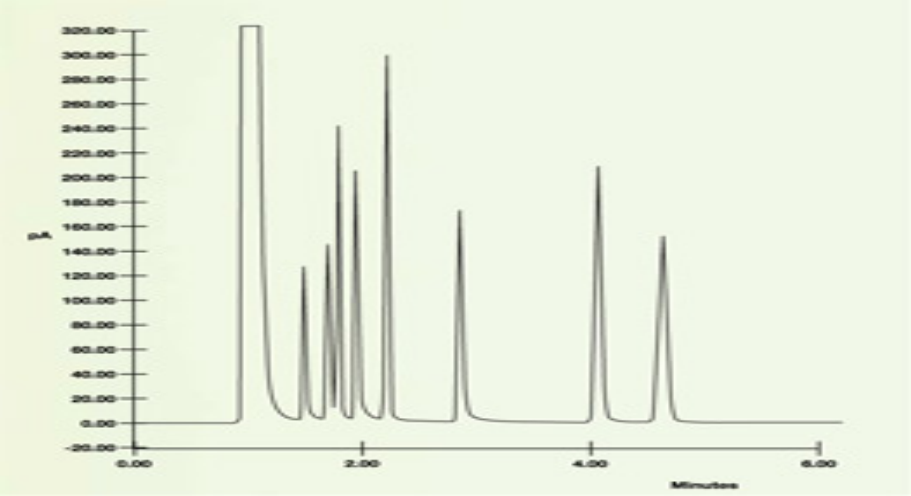
# How Can Chromatography Benefit?

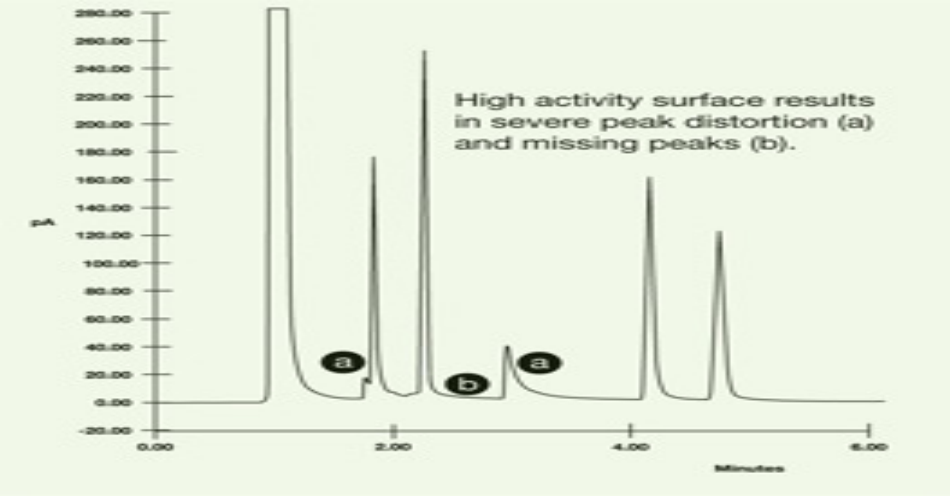
- Gas Chromatography
  - Inertness
    - Reproducibility
    - Adsorption
    - Peak Integration
  - Anti-Corrosion (minor)
  - Prevent Carry-Over

- Liquid Chromatography
  - Inertness
    - Reproducibility
    - Adsorption
    - Peak Integration
  - Anti-Corrosion
  - Prevent Carry-Over

# Create a chemically inert flow path

- Accurate analytical profile of all trace compounds
- Eliminate false negatives
- Get a reliable sample from field to lab
- Used in manufacturing process systems and analytical laboratories

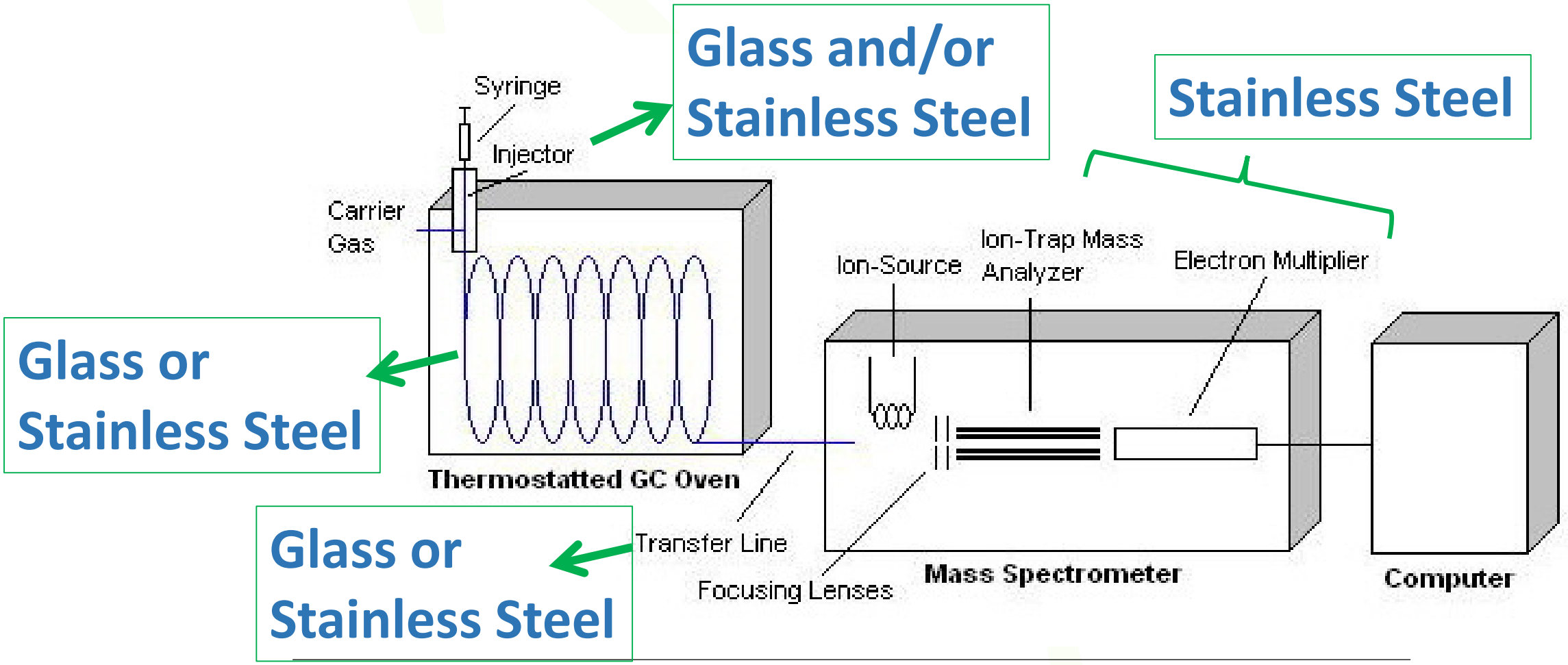




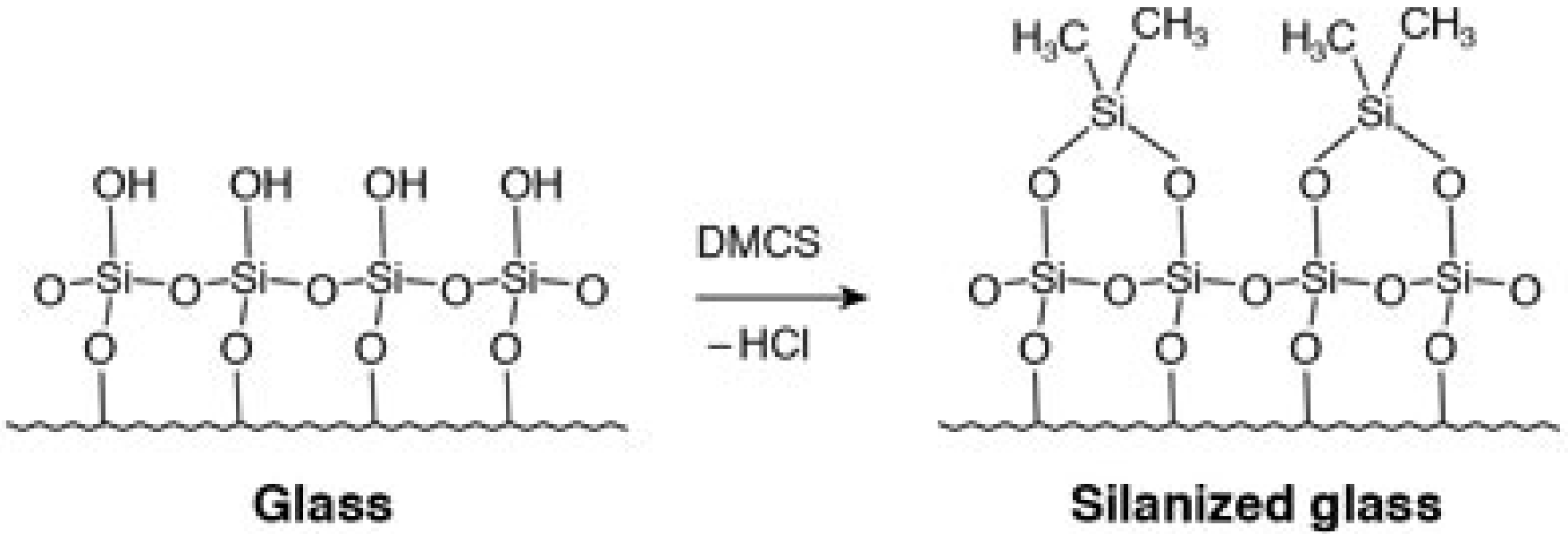
# Examples of Bad GC Actors

- Sulfur Compounds
- Phenols
  - Dinitrophenol
  - Pentachlorophenol
- Semi-Volatiles
- Pesticides
- Amines

# Simple GC System Schematic



# Simplified Glass Surface and Classic Deactivation



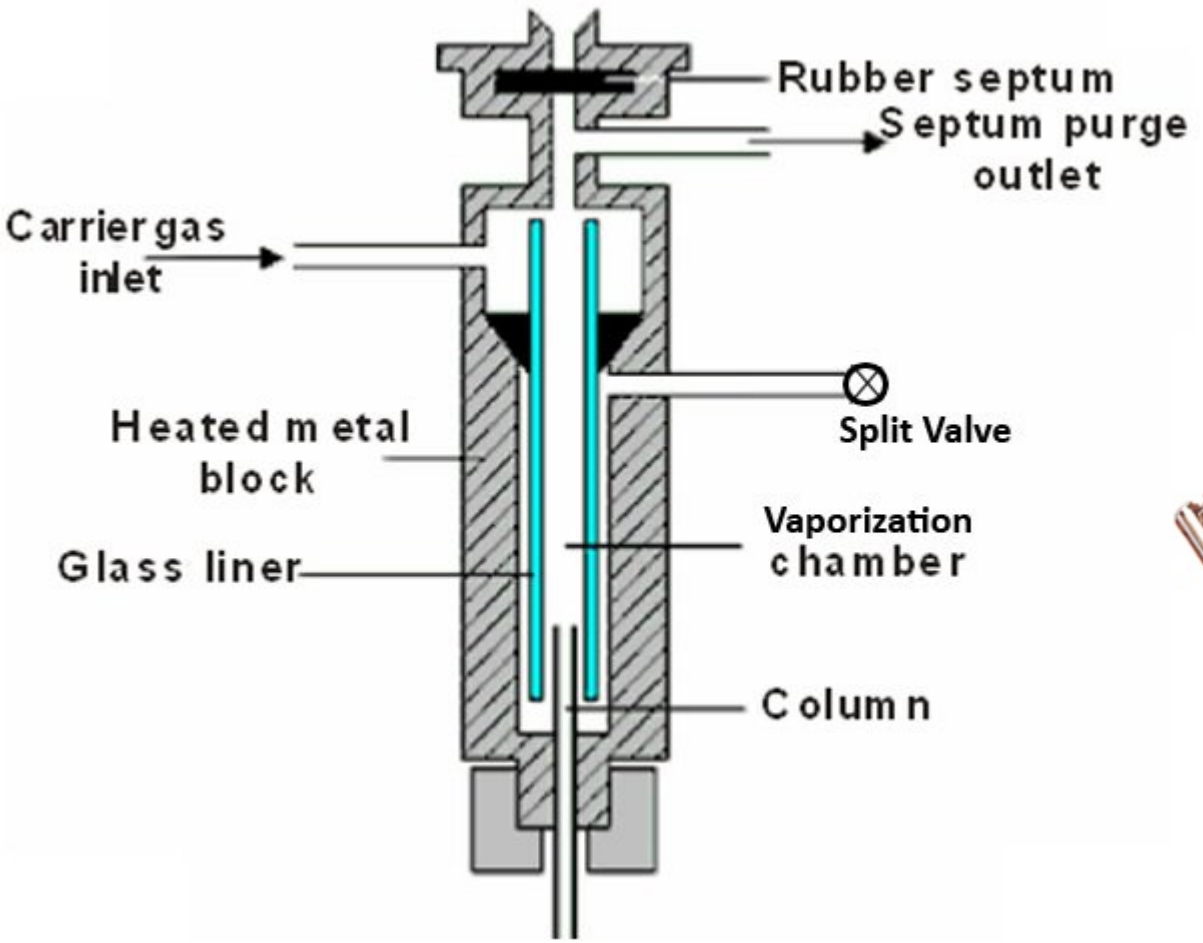

- Silanols are acidic and must be deactivated for inertness
- 100% silanols cannot be deactivated through this method, but a coating can be a game-changer…

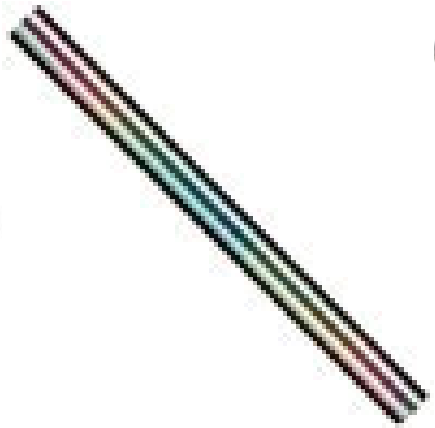
# Stainless Steel Surfaces

- Stainless steel common for analytical pathways
  - Good structure, resists corrosion
  - Poor chemical properties for chemists
    - 316 SS:  68%Fe; 17%Cr; 12%Ni; 2%Mn; Si; C
    - Chemically reactive, adsorptive, catalytic

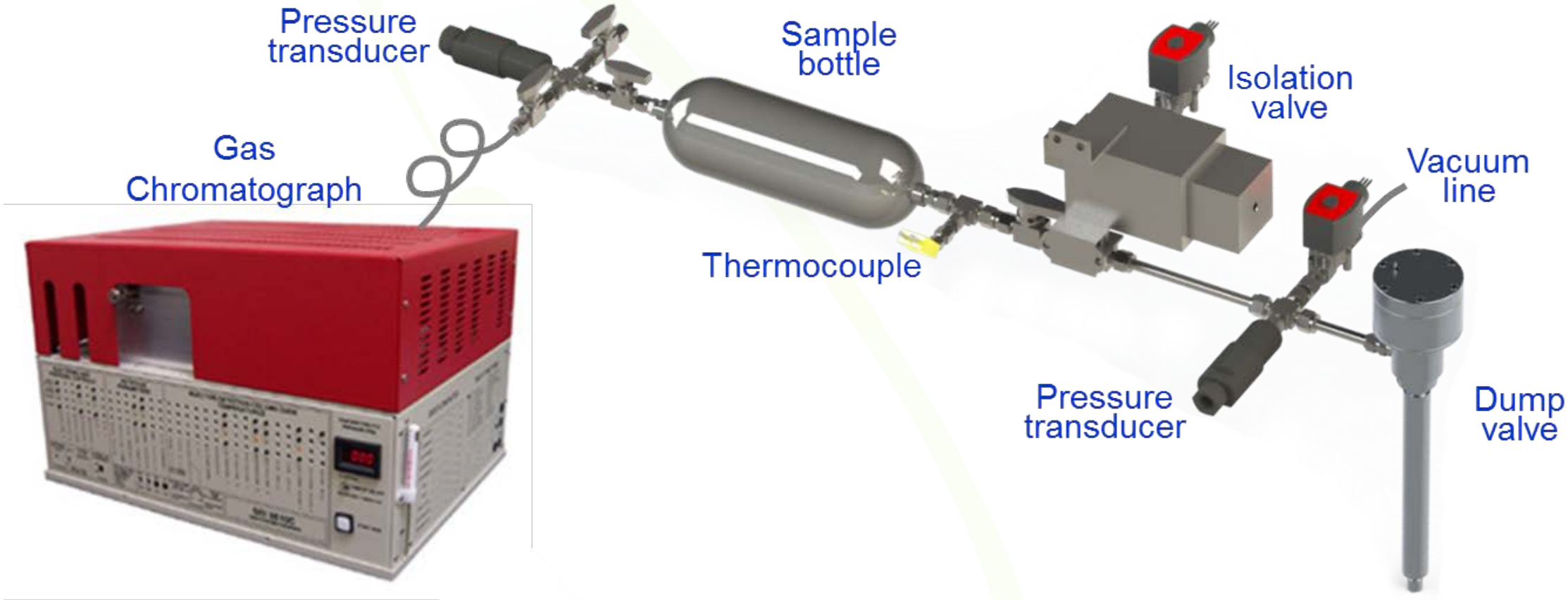
- Coatings address many failures of bare SS:
  - Chemical reactivity
  - Aggressive corrosion
  - Moisture

# Injection Port Inertness

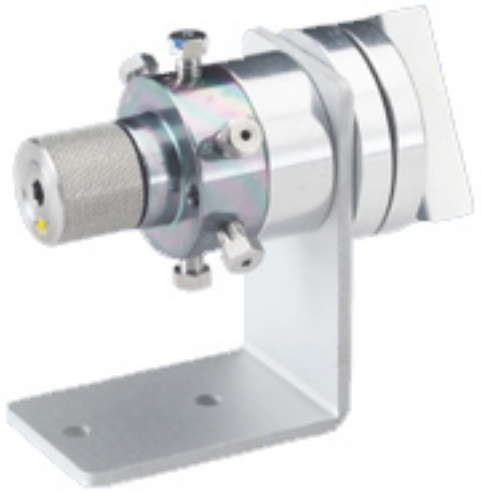


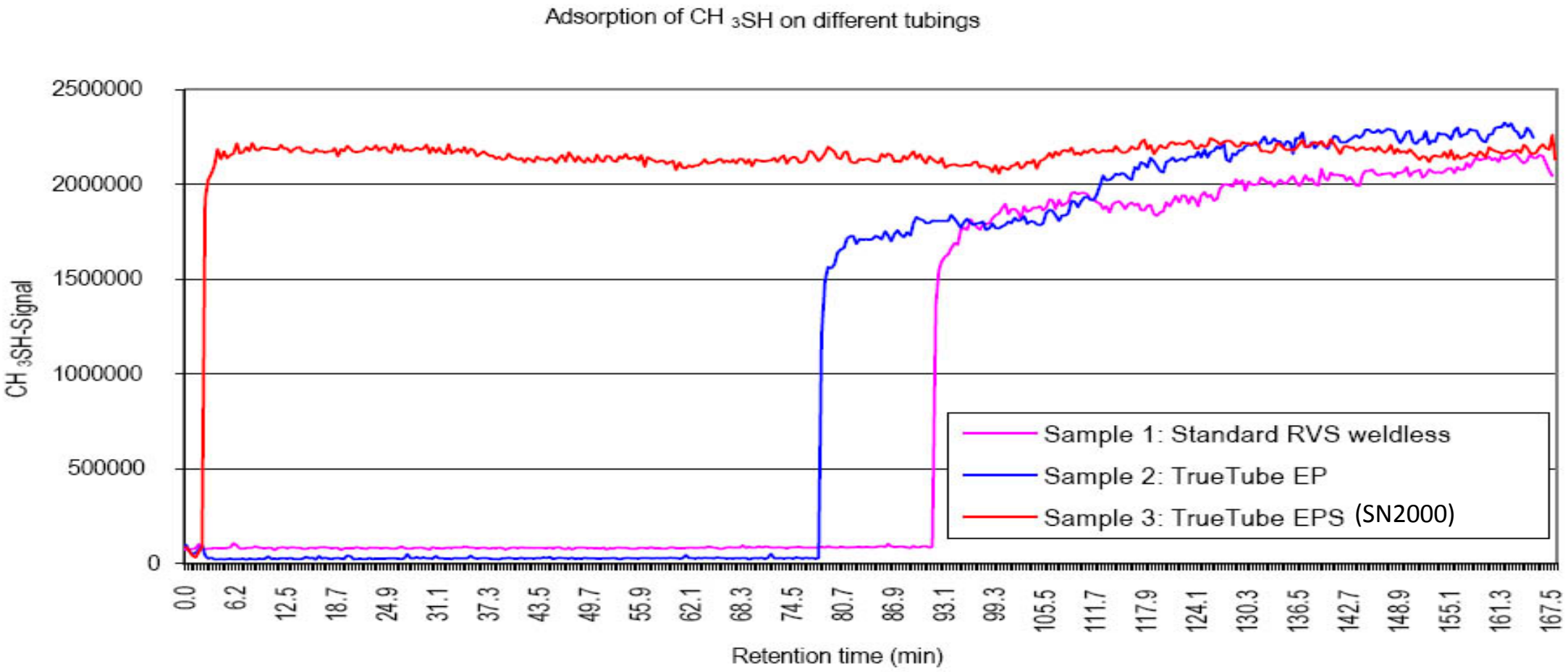
# Sampling Systems – Are they inert?



# Is it in the flow path?

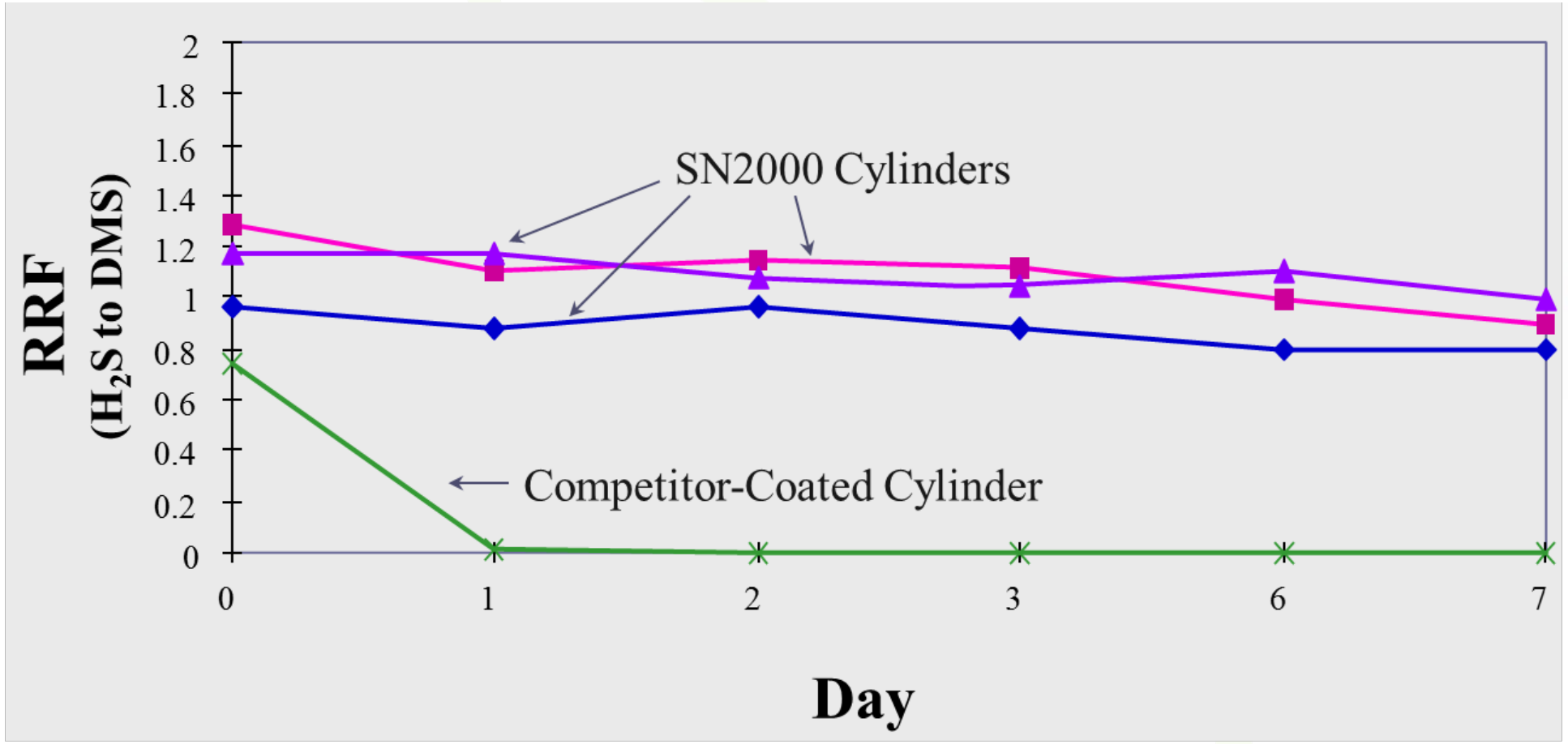


# SilcoNert 2000 Improves Transfer

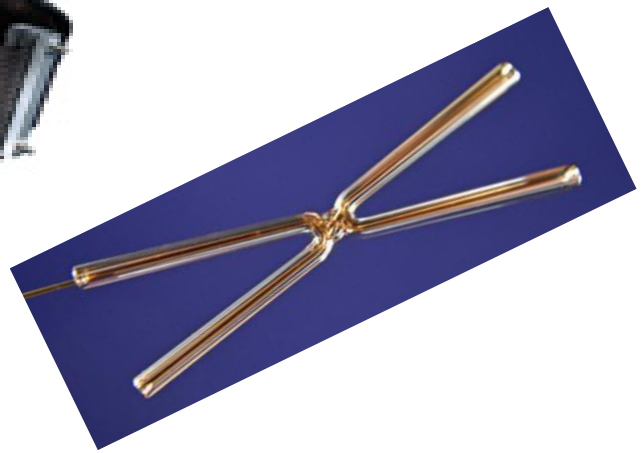


# Sulfur Inertness Comparison

## -$H_2S$ Static Storage at 17ppbv




# GC Columns, Guard Columns, Transfer Lines, Connectors



# Coatings for Detector Components

- Mass Spec
  - Ion Trap
  - Quad/TOF ion sources
- FID
- TCD

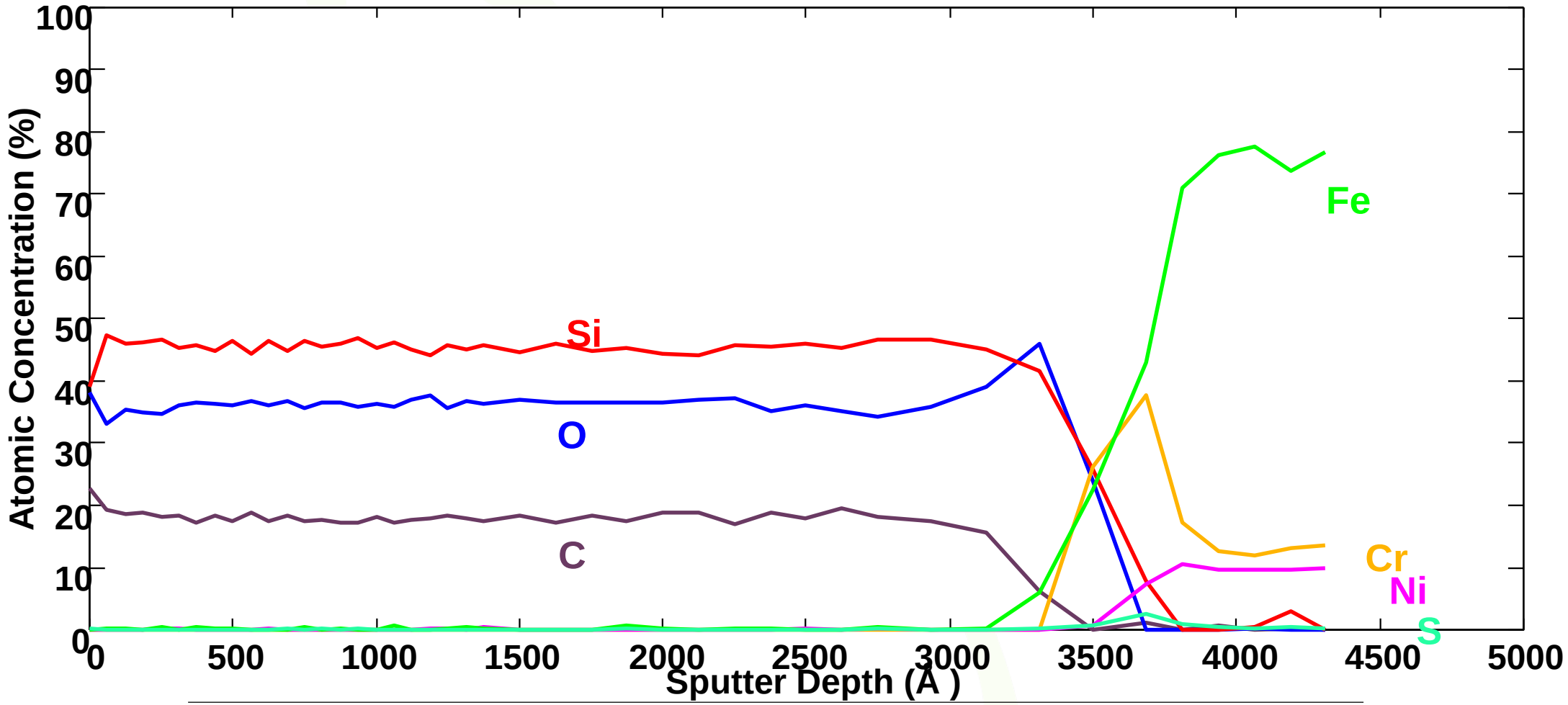
# GC Summary

- Many opportunities for prevention of active compound adsorption
  - Sampling system, transfer
  - Injection
  - Analytical column
  - Connectors
  - Transfer / guard columns
  - Detector components
- SilcoTek coatings can help create the ultimate inertness for GC analytical systems
  - Patented – no other CVD coatings on stainless steel have shown inertness surpassing SN2000 / Siltek / Sulfinert

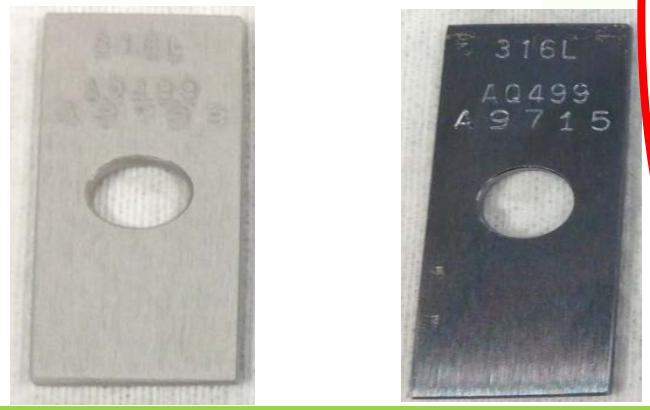
# Coatings for HPLC

- Growing Area of Application – New Opportunities
- Stainless Steel vs. PEEK Issues
  - Stainless Steel
    - Acid Corrosion (Halogenated Solvents - HCl, HBr)
    - Ion Chromatography
    - Anionic Compounds (phosphates may chelate)
  - Peek
    - Temperature (Tg 148°C)
    - Halogenated solvents
    - Tetrahydrofuran
- Dursan Solutions
  - Inertness
  - Anti-Corrosion,
    - wide pH range
    - Resistant to saltwater corrosion
  - Anti-Biofouling
  - Robust

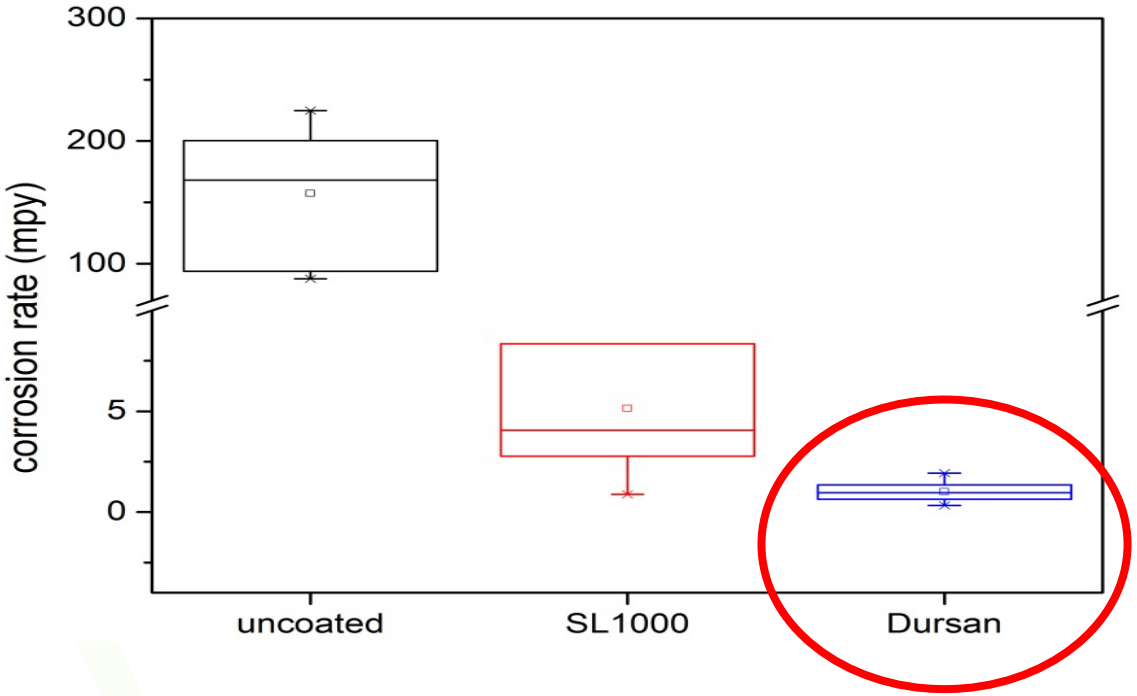
# Auger Depth Profile: Dursan – Surface Functionalized Amorphous SiOC



# 6M HCl Corrosion Resistance, 24hr @ RT


uncoated | SL1000-coated | Dursan-coated



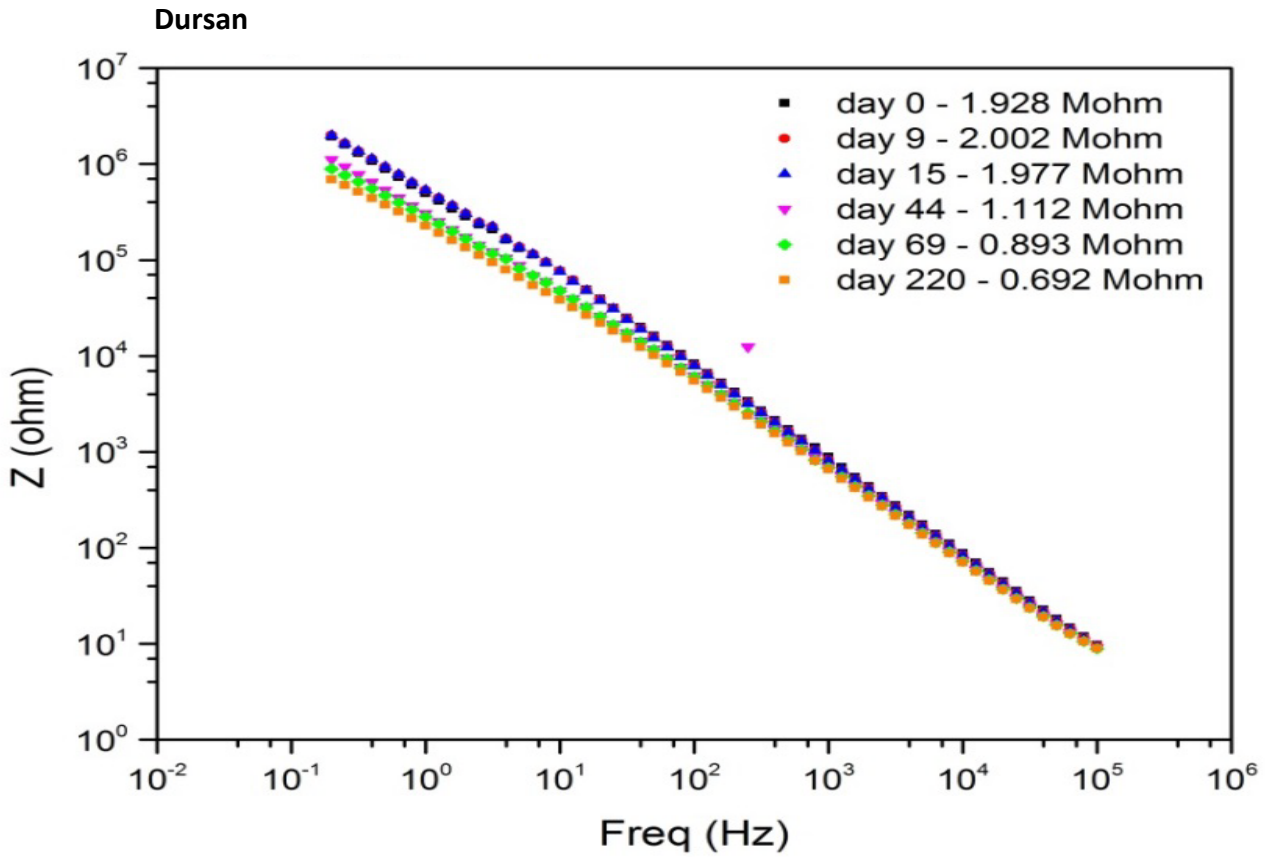
# Exposure to Caustic Base

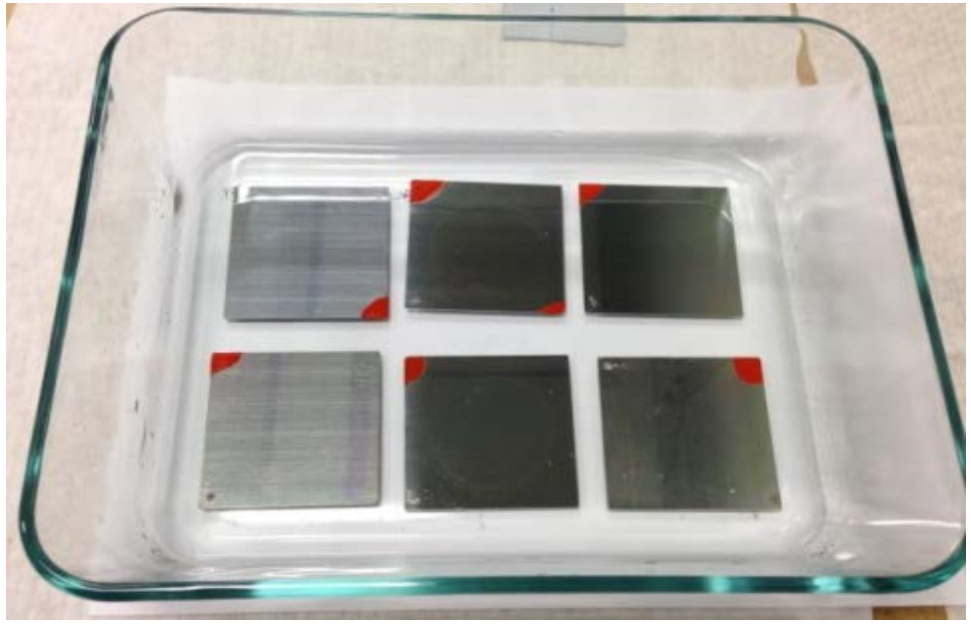
- *1M KOH; 24hr; 22ºC*

| *ASTM G31* | 316L SS | Silcolloy (also SN2000) | Dursan |
|---|---|---|---|
| MPY | 0 | 3.40 | 0.01 |
| Improvement Factor Over Silicon | unaffected | Dissolution | 261 |

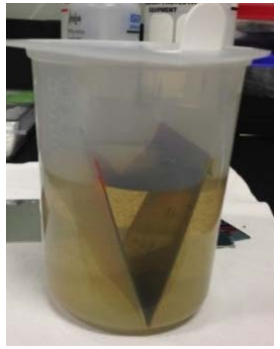
# Corrosion performance comparison: EIS

- Dursan in 5% NaCl monitored with EIS



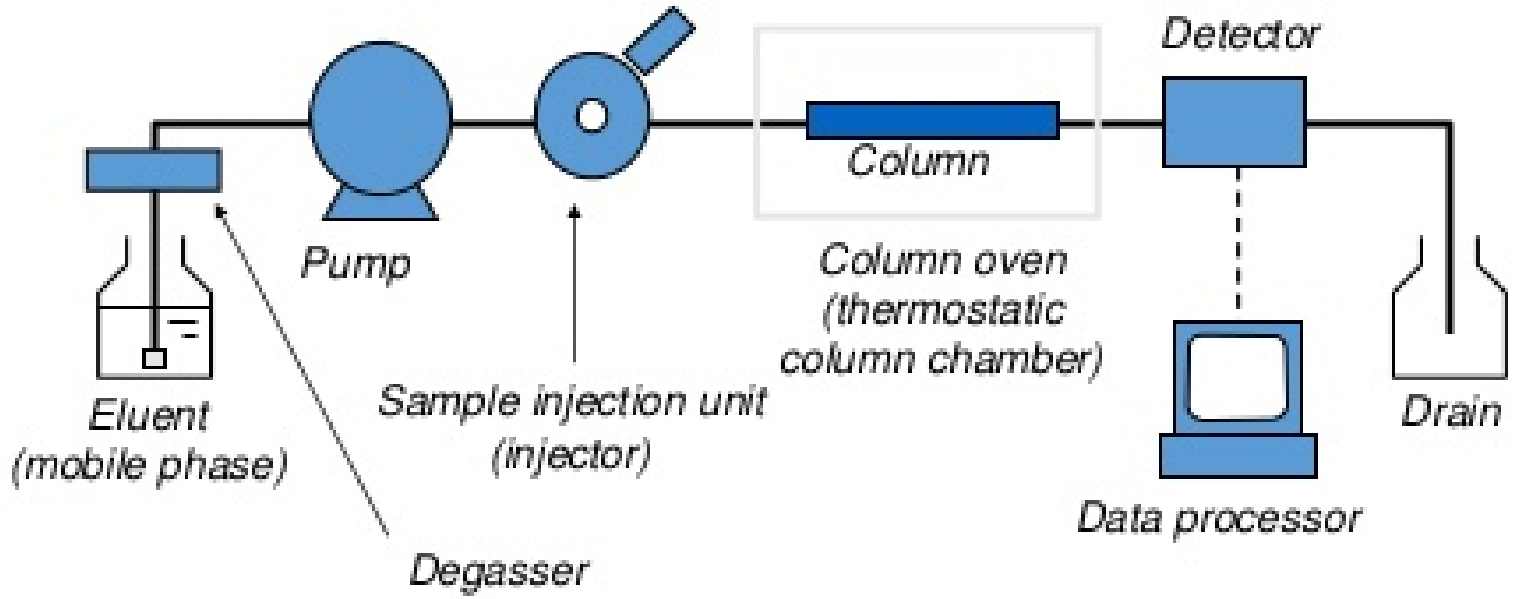



Dursan 420 days in salt water



Inferior Coating 20 days in salt water

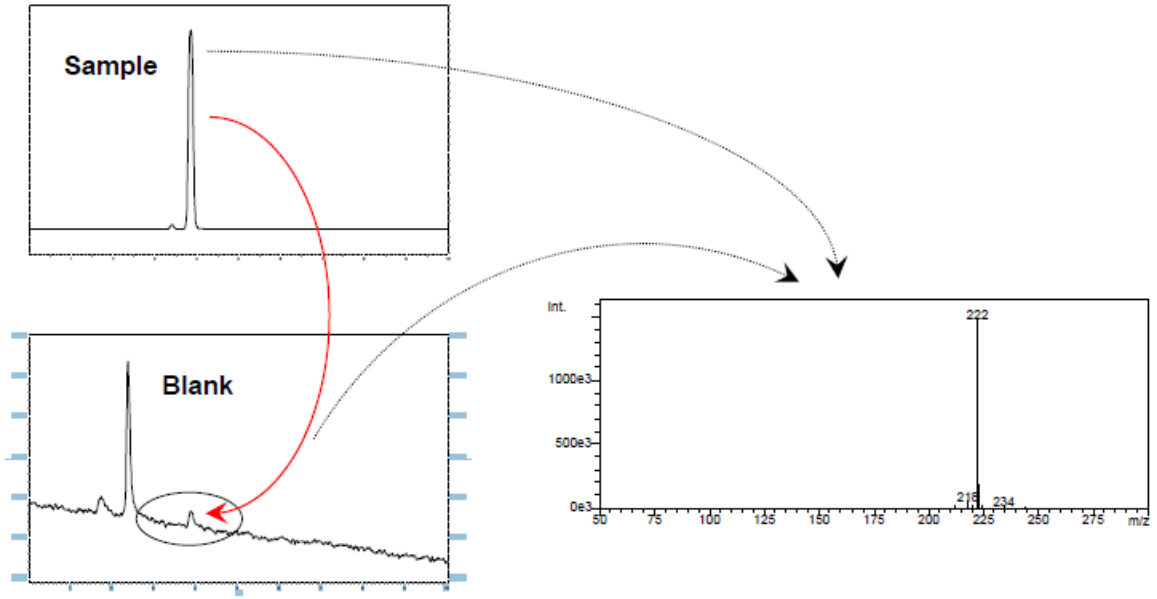
# HPLC - System Schematic



# Carryover – Injection system

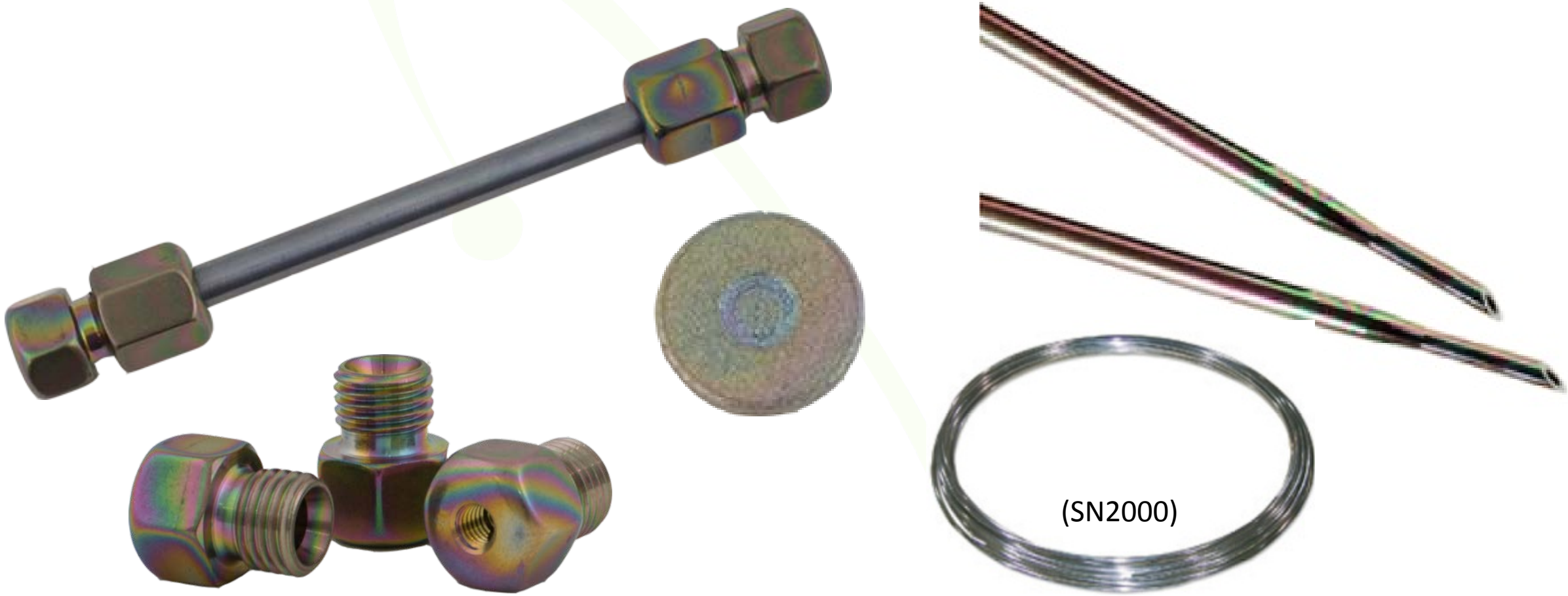
## What is Sample Carryover?

- Previously injected sample that elutes upon subsequent analyses due to chemical/physical characteristics of the sample, analysis system or both. As a result, peaks attributed to the previously analyzed sample may be observed in the subsequent chromatogram(s) which may co-elute or interfere with desired analytes. Also the MS spectrum at the background area shows the profile of a sample compound.

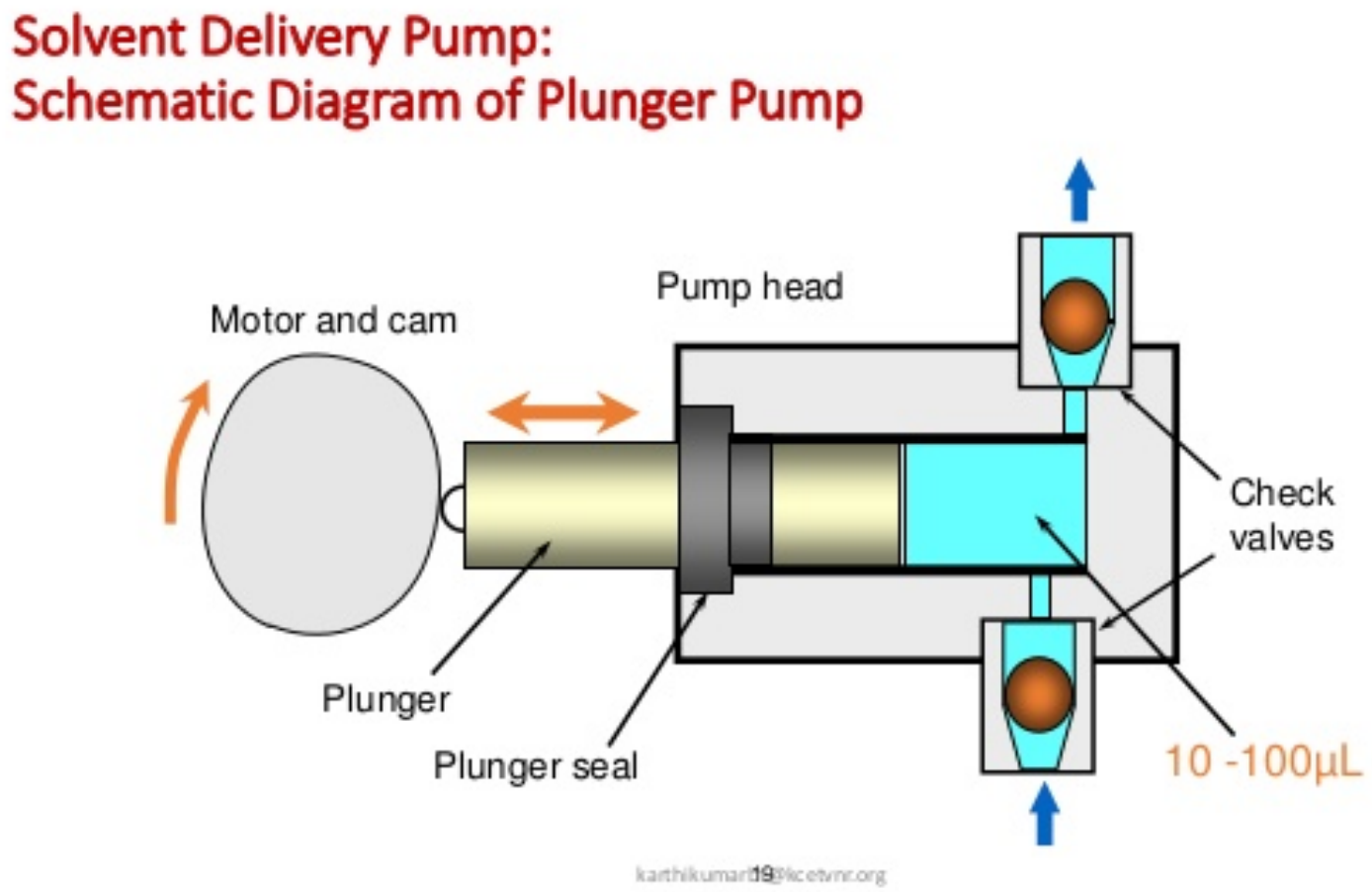



Hedgepeth, W.; Aiello, M.; Ellis, R.; Schreiber, A.; Caraiman, D.; Sakuma, T. "High Sensitivity MS Determination of Carryover in a New Autosampler Design" Shimadzu Scientific Instruments.

# Dursan for HPLC Columns, Fittings, Frits, Sampling Components

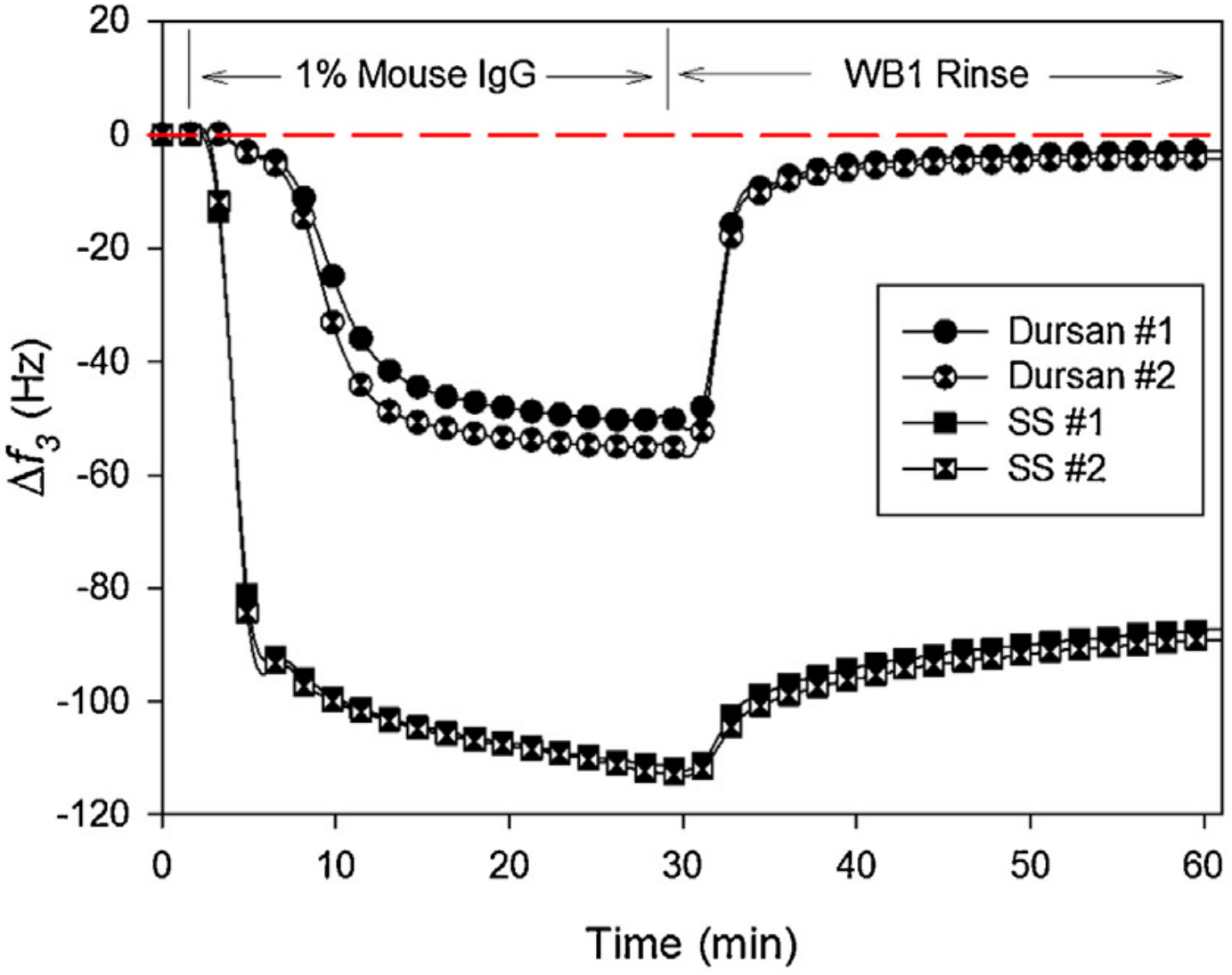


# HPLC Pump Schematic



# Anti-Biofouling

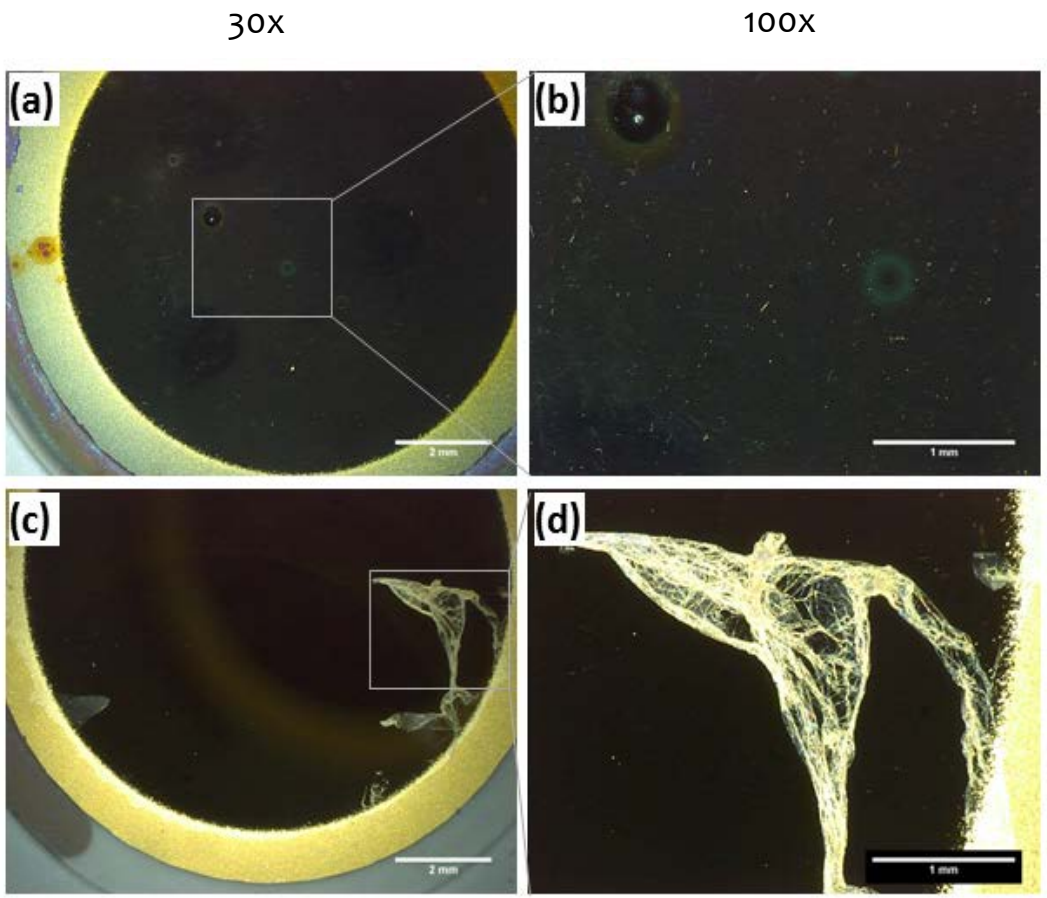
- Quartz crystal microbalance w/ dissipation (QCMD)
- Mouse Immunoglobulin G
- WB1:  Wash buffer with non-ionic surfactant (PBS with Brij 35)
- Dursan vs. Stainless steel sensor




Vaidya, S.V.; Yuan, M.; Narvaez, A.R.; Daghfal, D.; Mattzela, J.; Smith, D. *Appl. Surf. Sci.* **2016**, *364*, 896-908.

# Dursan Robustness

- Dursan-coated vs. Fluoropolymer- coated sensors
- Before vs. After sonication 10 min in EtOH




Dursan

AF1600

Vaidya, S.V.; Yuan, M.; Narvaez, A.R.; Daghfal, D.; Mattzela, J.; Smith, D. *Appl. Surf. Sci.* **2016**, *364*, 896-908.

# Conclusions, Questions

- LC – Improve difficult analyses
  - Sampling system inertness, transfer, carryover
  - Challenging mobile phases
    - Acidic, salts, incompatible solvents
  - Bio-inertness
  - Robust
- Dursan – Patent applied:  no other CVD deposition equal

- How can we help?  What are your challenges?

# Thank you!

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www.SilcoTek.com