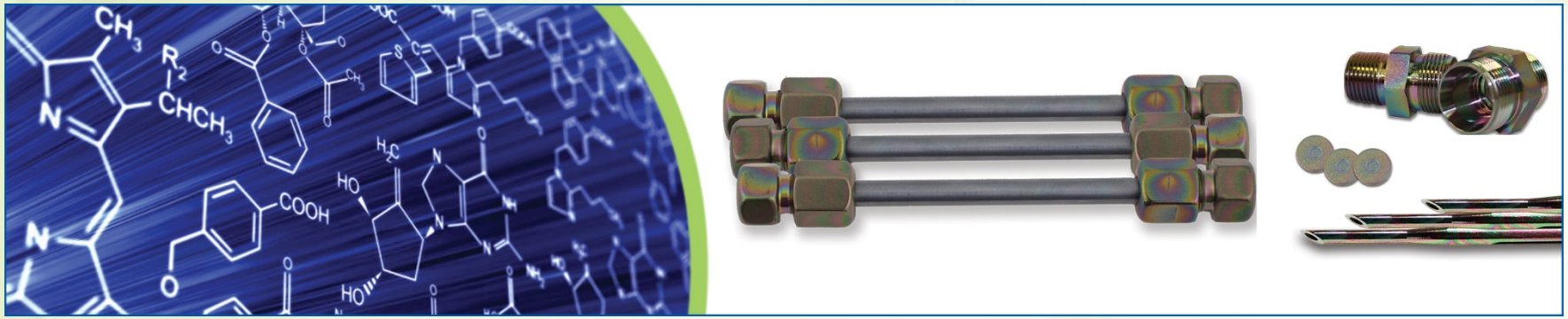
Metal-Free HPLC with Dursan® Bio-Inert Coating



Technology, Applications, and Data

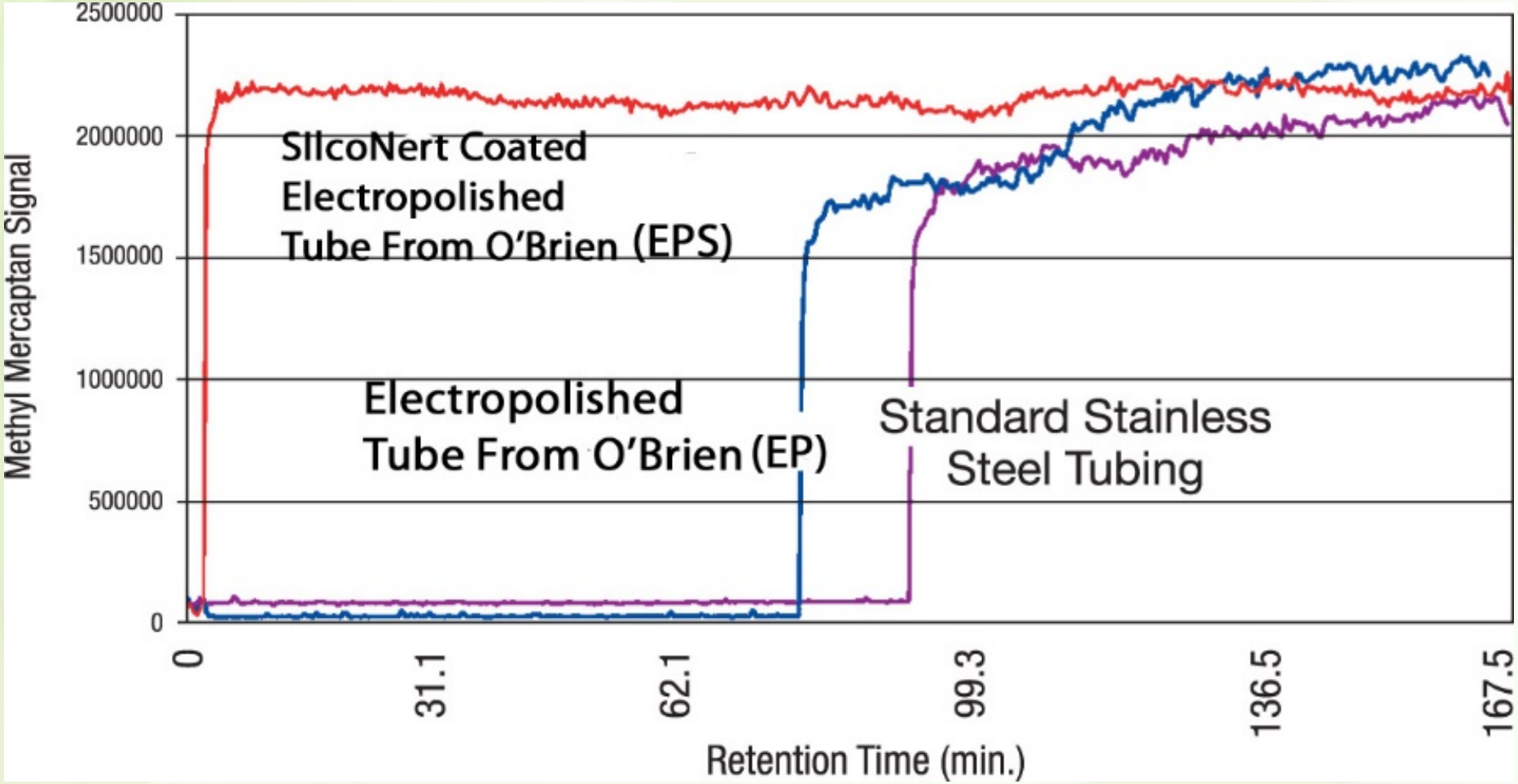
Presentation Overview

- Background: The need for an inert flow path
- Overview of SilcoTek Technology
- Issues with Stainless Steel and PEEK
- Materials properties of Dursan®
- Protein adsorption studies on Dursan®
- Dursan® used in HPLC columns

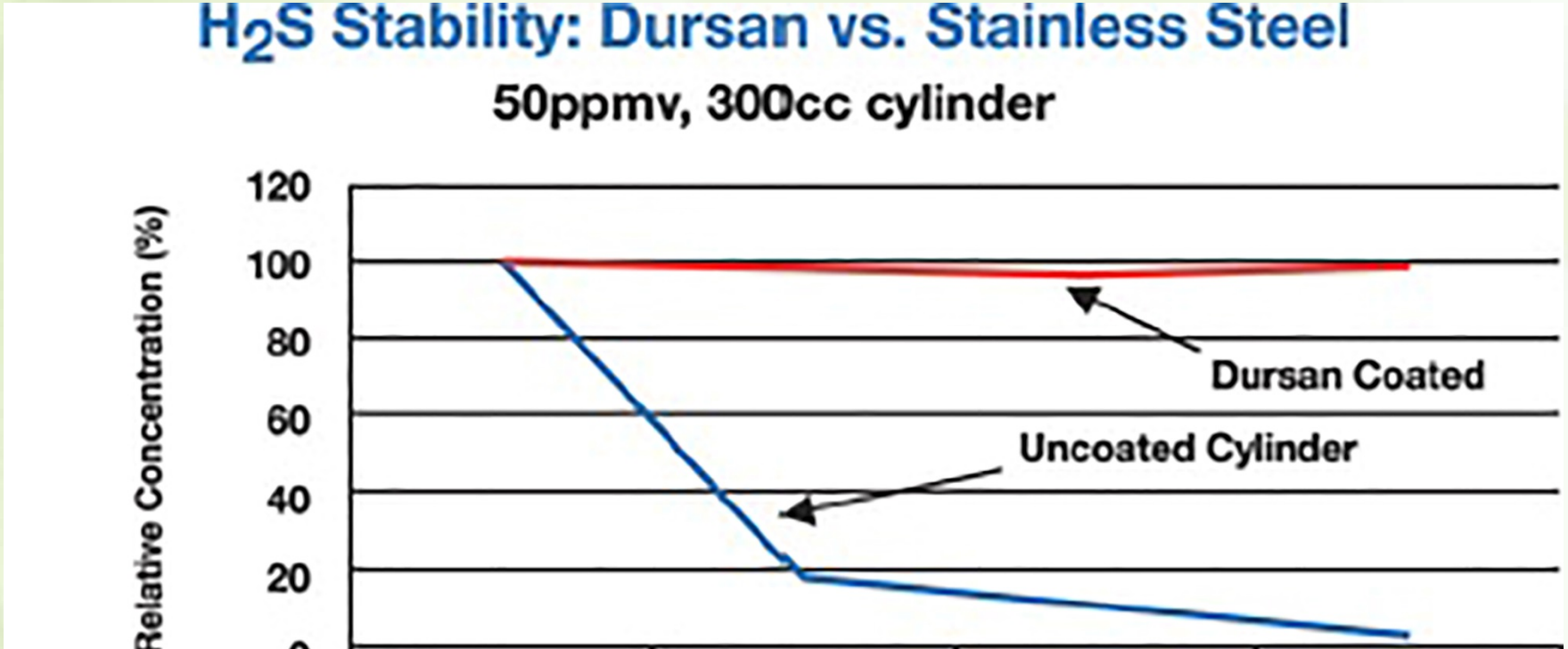
Why is an inert flow path critical?

- Reduce costs and downtime
  - No need to re-test
  - Accurate profile of all components, both reactive and non-reactive
- Trust your results
  - Eliminates false negatives
- No additional molecules/ions introduced
  - Stainless steel: chemical reactivity, corrosion, and abrasion can all lead to the introduction of molecules and ions that are not in your sample.

Inertness leads to faster response time

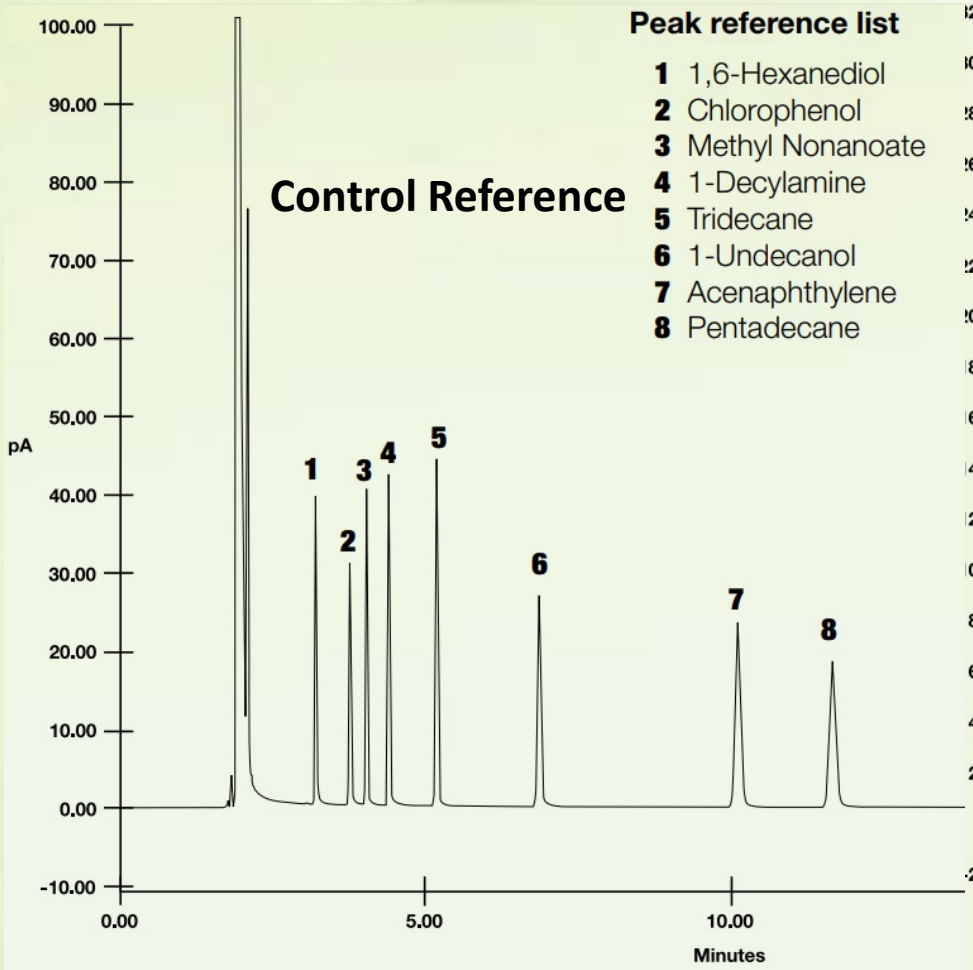


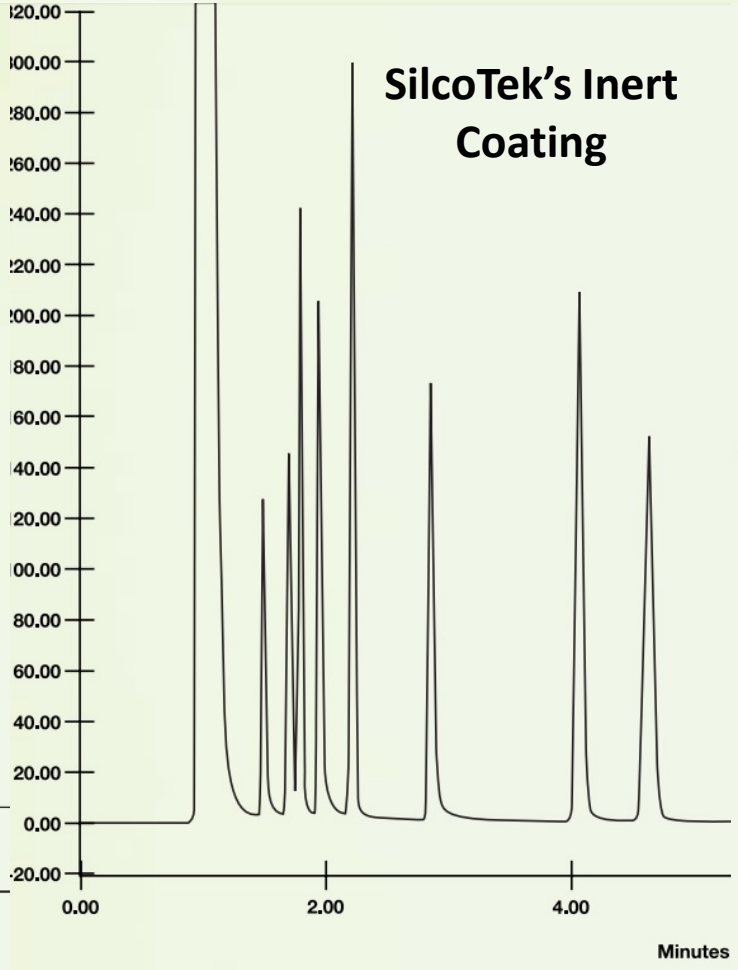
Inertness prevents loss of analytes

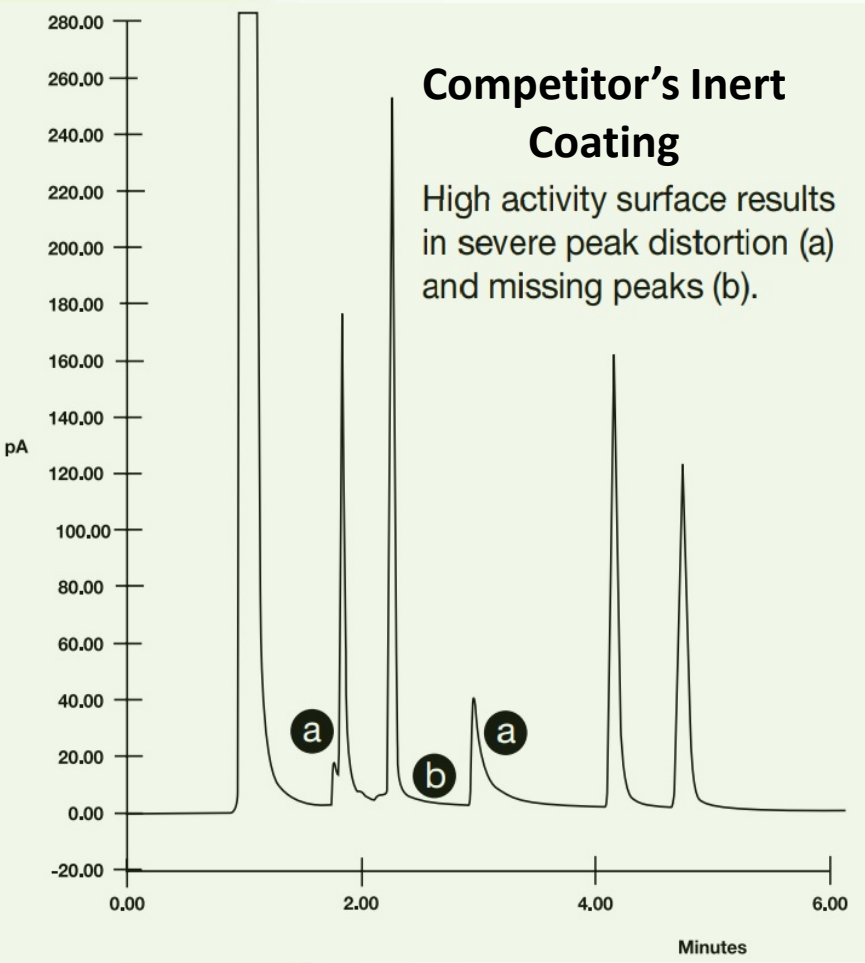


	0:00	25 hours	50 hours	75 hours
Stainless Steel	100	19	10	4
Dursan	100	98	96.6	98.3

Inert analytical flow path example





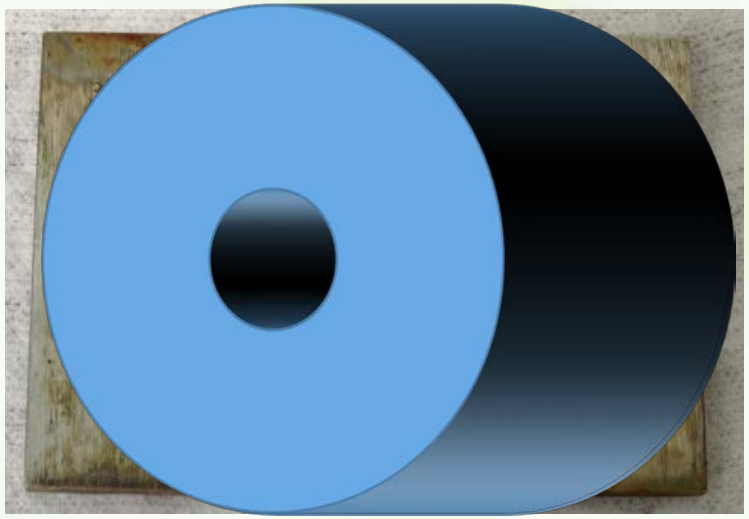


Current inert flow paths for HPLC

Material	Benefits	New issues
Titanium	Bio-inert	Much more brittle than steel Corrosion issue to consider
PEEK	Bio-inert Flexible Lightweight and easily cut	Swelling causes back pressure Temperature limitations Solvent limitations
Ceramics	Robust Chemically inert	Brittle Can be expensive

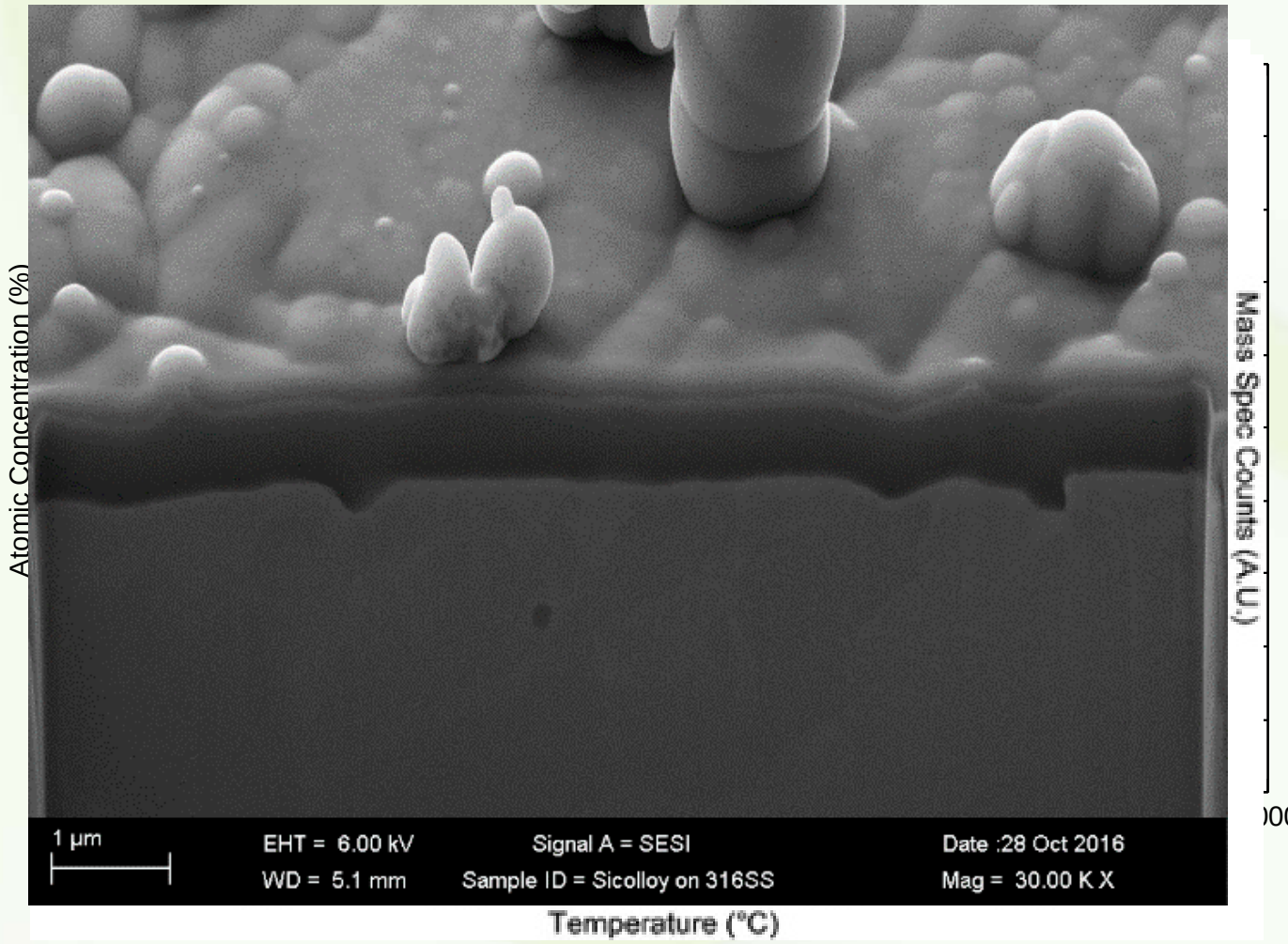
Stainless steel and PEEK issues

- Stainless steel issues:
  - Acid corrosion (Halogenated solvents – HCl, HBr)
  - Highly reactive toward chelating agents
  - Protein Fouling/carryover
- PEEK issues:
  - Temperature limitations (Tg = 148°C)
  - Halogenated solvent damage
  - THF, Acetone, and other organic solvents cause swelling
    - Increases pressures and potential for delamination



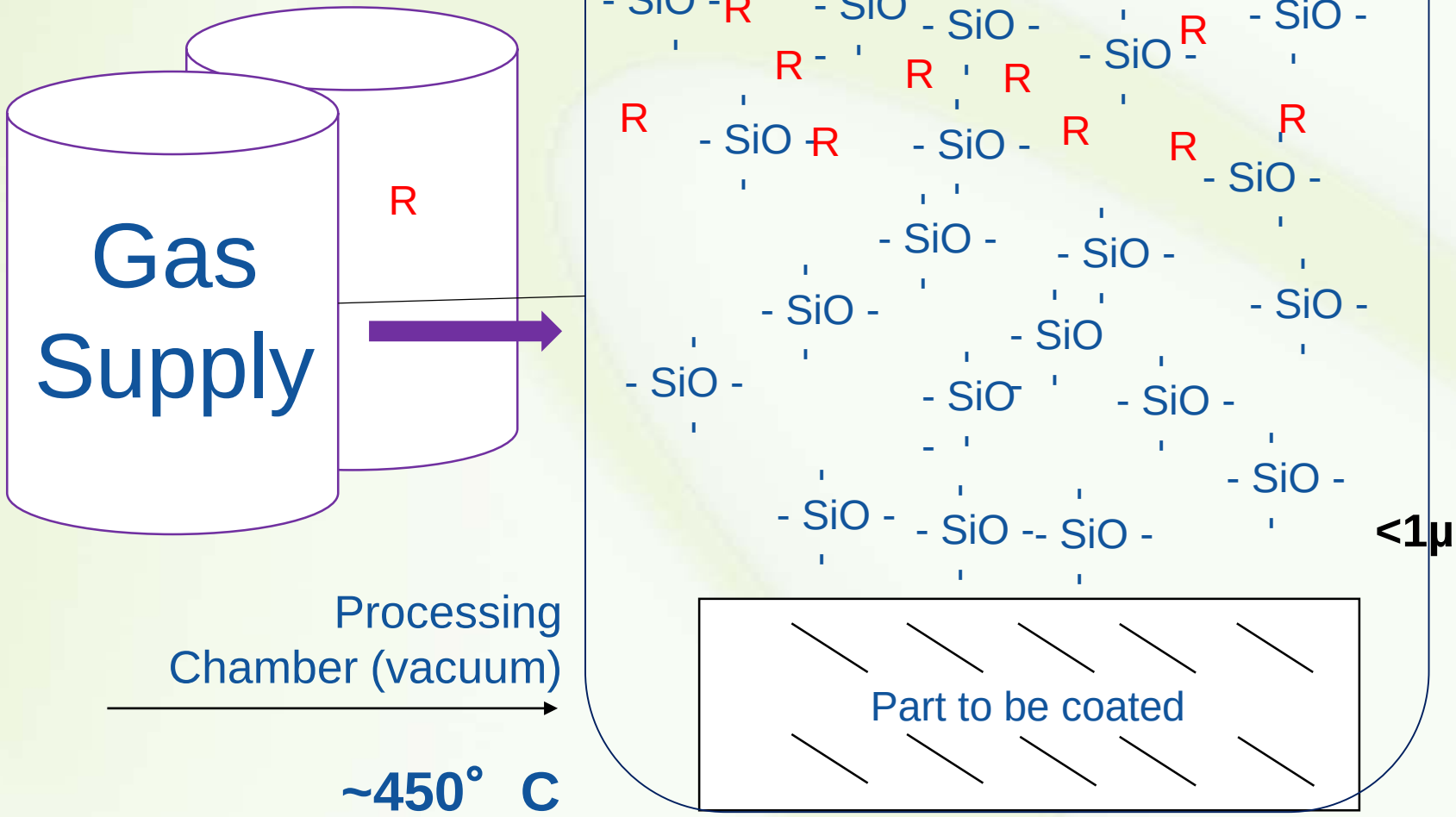
SilcoTek's solution: Dursan®

- Functionalized silica-like coating
- Highly corrosion resistant
- Stable up 450°C
- Provides a barrier between your solvent/analyte, and the substrate
  - Can be applied directly onto existing parts
  - <1 µm thick so will not affect tolerances



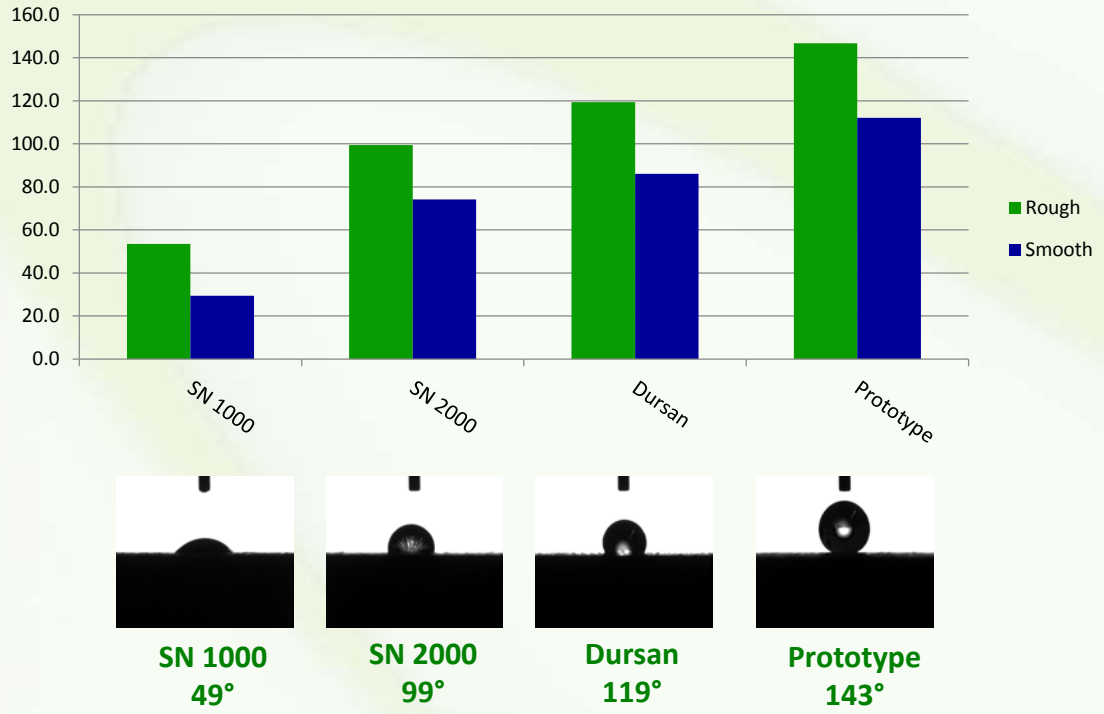
The CVD Coating Process

- Silane based CVD coatings
- Most metal alloys and ceramics can be coated
- Provides an inert barrier between substrate and flow path
- Surface chemistry can be altered by varying R.



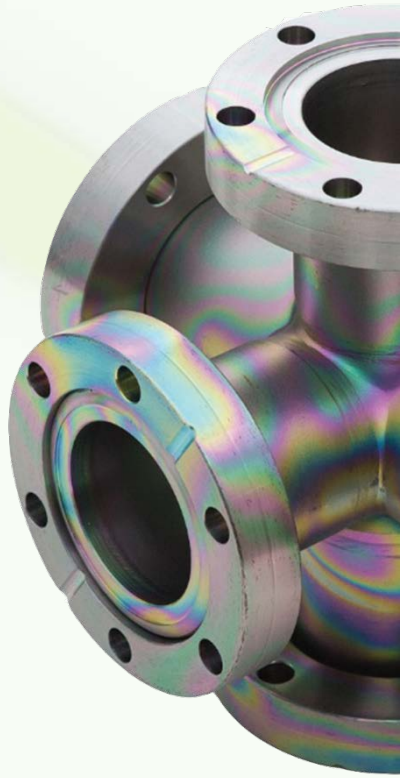
Surface Properties

- Chemically inert / non-reactive
- Non-stick, high release
  - Hydrophobic
  - Oleophobic
  - Anti-fouling
- Oxidation resistant
- Corrosion resistant
- Dielectric or semi-conductive
- Stable at temperatures <450° C



Advantages of CVD Coatings

- Non-line-of-sight deposition; uniformly treats 3D, high aspect ratio part geometries
- Molecular adhesion to base substrate. Won't flake nor delaminate.
- Scalable, versatile, and highly reproducible



Coating Capabilities

- We apply CVD coatings to:
  - Tubing: as small as 0.10mm ID
    - Internal coating, 600m ++ lengths if coiled
  - Frits, valve components (wetted), manifolds, etc.
  - Entire analytical flow path
  - Parts can be bent or flexed without flaking*

Substrates that coat well

- 300 and 400 grade stainless steel
- Titanium
- Ceramics
- Glass
- Inconel®
- Hastelloy®
- Tantalum

Substrates with issues

- 5000 series aluminum
- Pure Nickel / Nickel plating
- Copper
- Monel®
- Brass
- Gold and Silver plated components
- Magnesium
- Elastomers / plastics

Materials properties of Dursan®

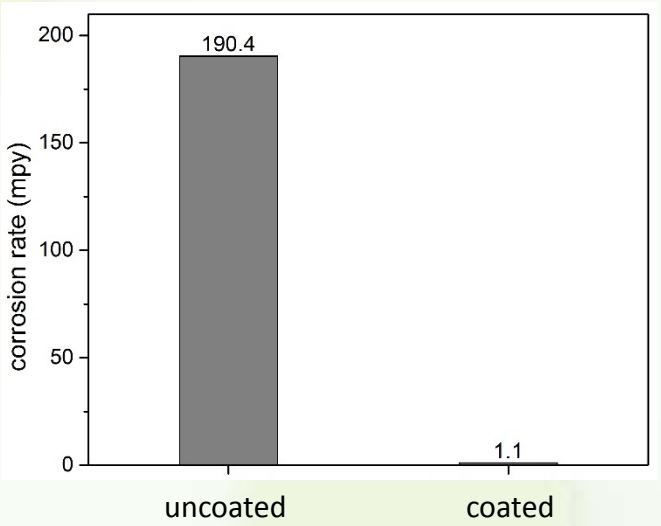
- High temperature: Stable up to 450°C
- Usable in wide pH range: 0-14
- Molecularly bound to the substrate: Good adhesion
- Wear: 2x more resistant than 316 Stainless steel
- Inert to most chemicals

Corrosion resistance in acid

- ASTM G31 guidelines
- 20% (6M) HCl room temperature immersion 24 hours
- Over 170x improvement with coating

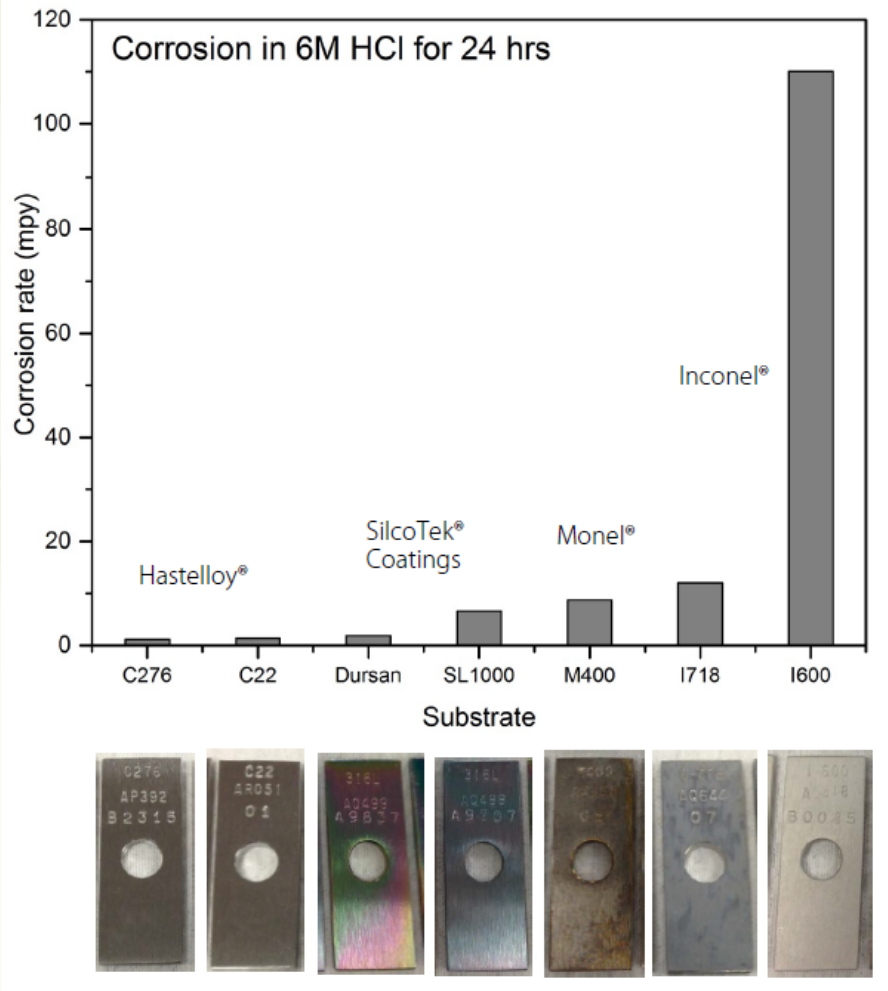
uncoated

coated



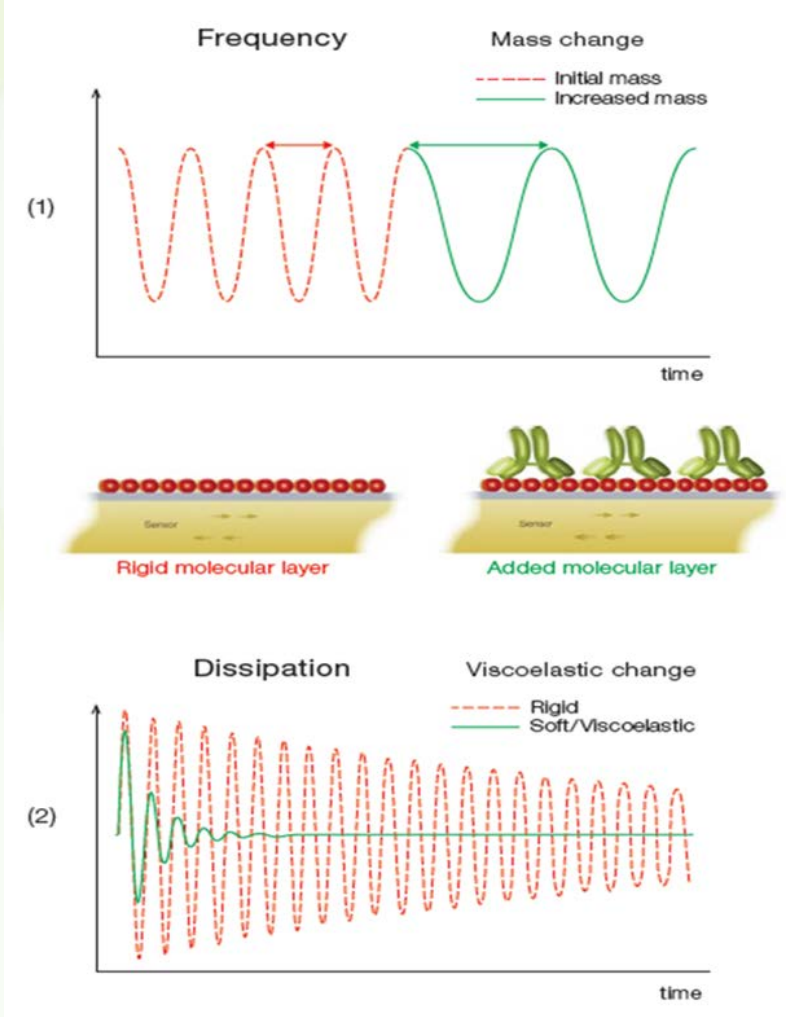
Additional corrosion resistance

Corrosive media	Bare Stainless Steel (MPY)	Dursan coated steel (MPY)	Improvement multiplier
6M HCl @ 50°C	3116.1	23.5	133x
Concentrated H_2SO_4	78.45	0.15	523x
48% HBr	2.05	0.29	7x
Bleach	1.70	0.10	17x
Concentrated H_3PO_4 @ 80°C	2.14	0.53	4x
Concentrated Nitric Acid	Unaffected	Unaffected	-

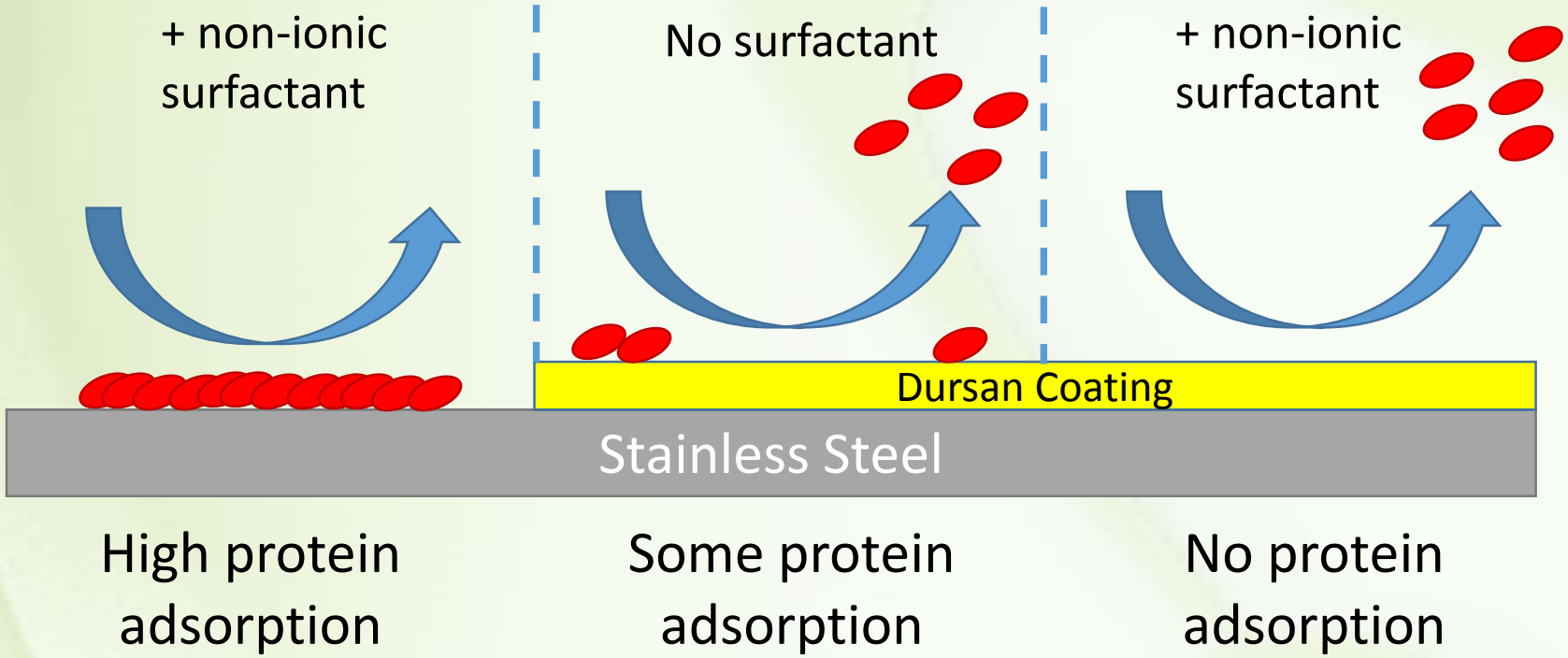


Bio-Inertness: Non-specific protein adsorption studies

- Collaborative study between Abbott Laboratories and SilcoTek on protein adsorption
- QCM-D with a thin layer of 316L SS was coated with Dursan
- Protein solutions were flowed over the sensor and the frequency was monitored over time



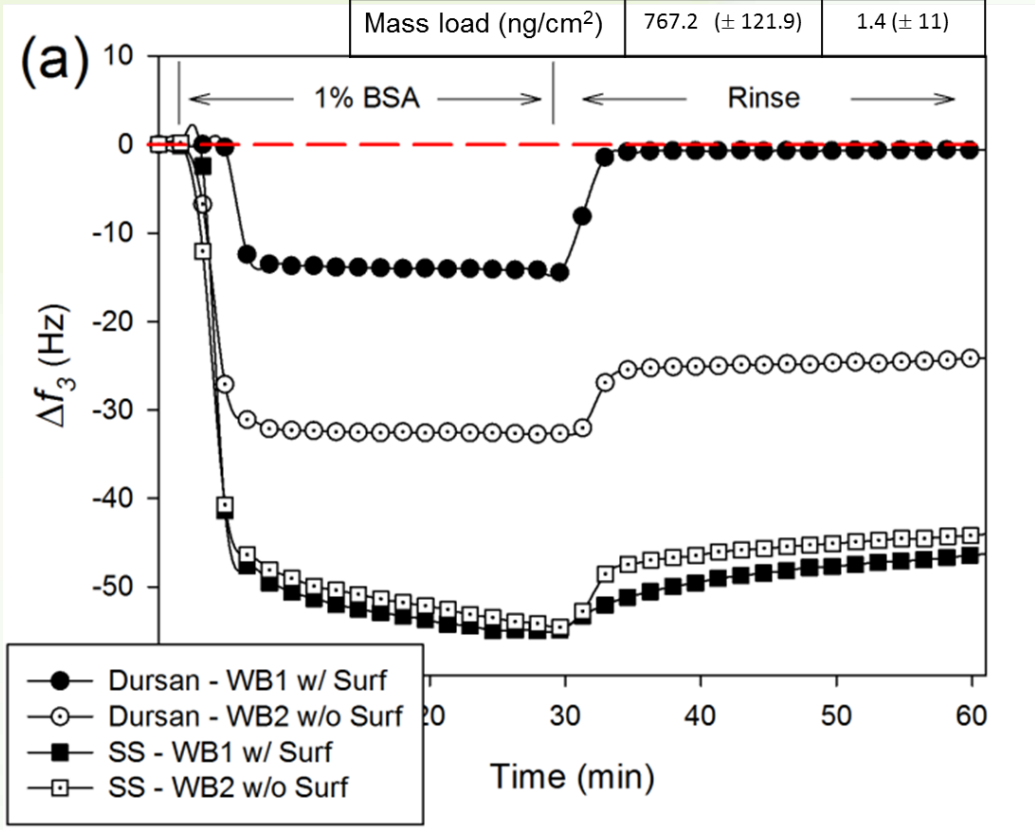
Dursan coating reduces protein adsorption and facilitates removal



Effect of Dursan® coating and surfactant

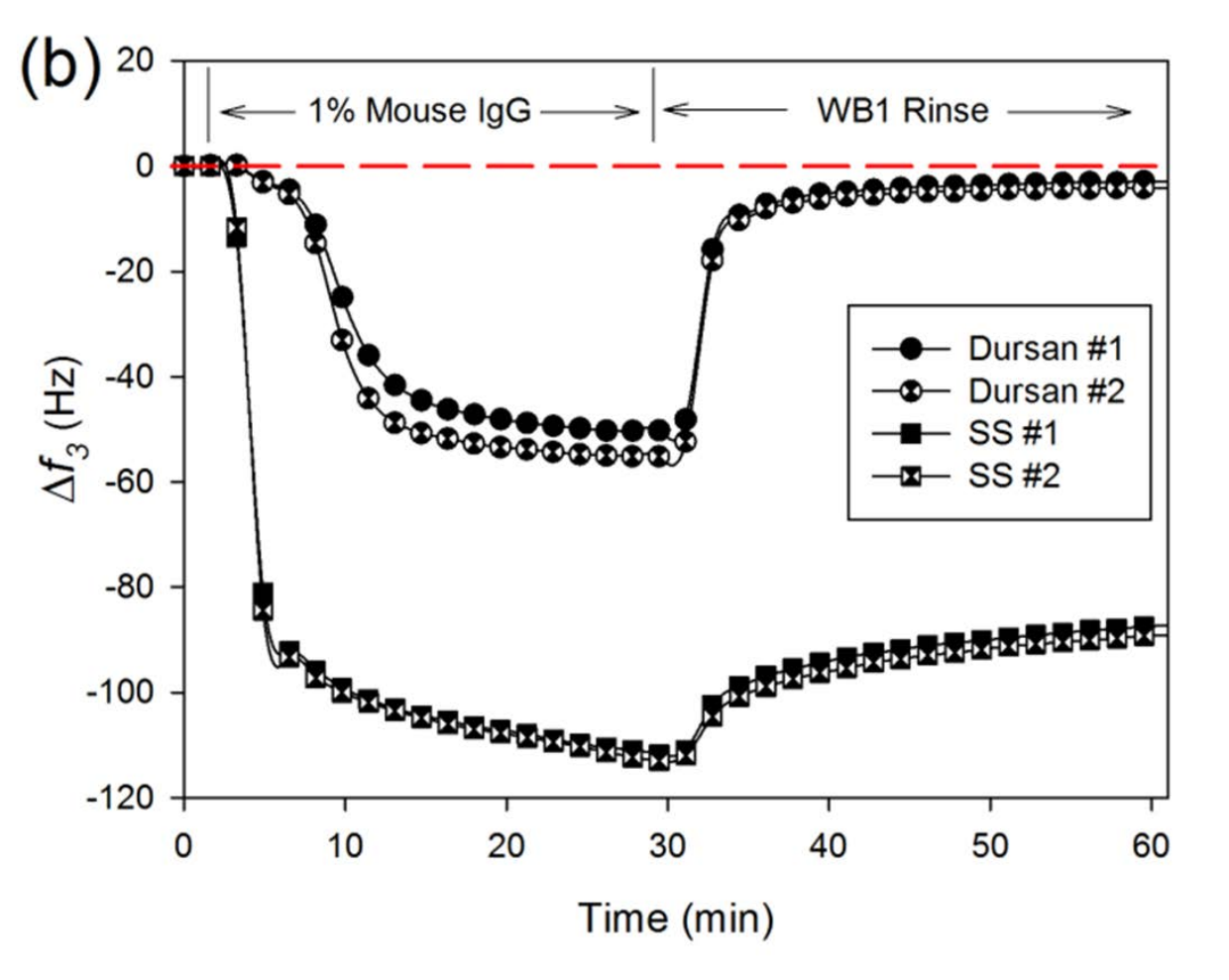
- Bovine serum albumin was tested with Brij 35 surfactant
- A rinse and use of non-ionic surfactant, all protein was removed
- Surfactant had no effect on the bare stainless steel

Surface	Bare SS	Dursan
Mass load (ng/cm^2)	767.2 (± 121.9)	1.4 (± 11)



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

Mouse immunoglobulins do not stick



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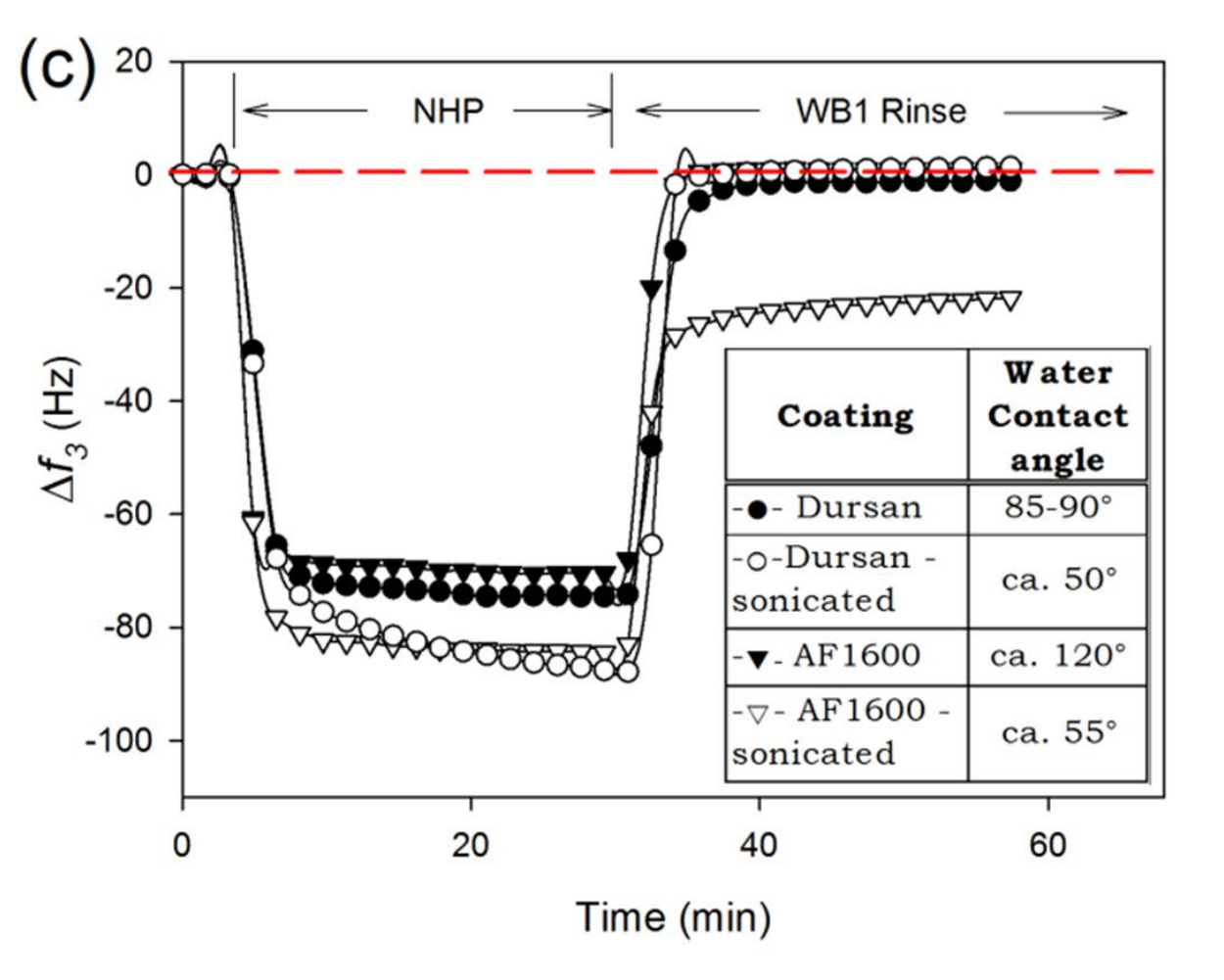
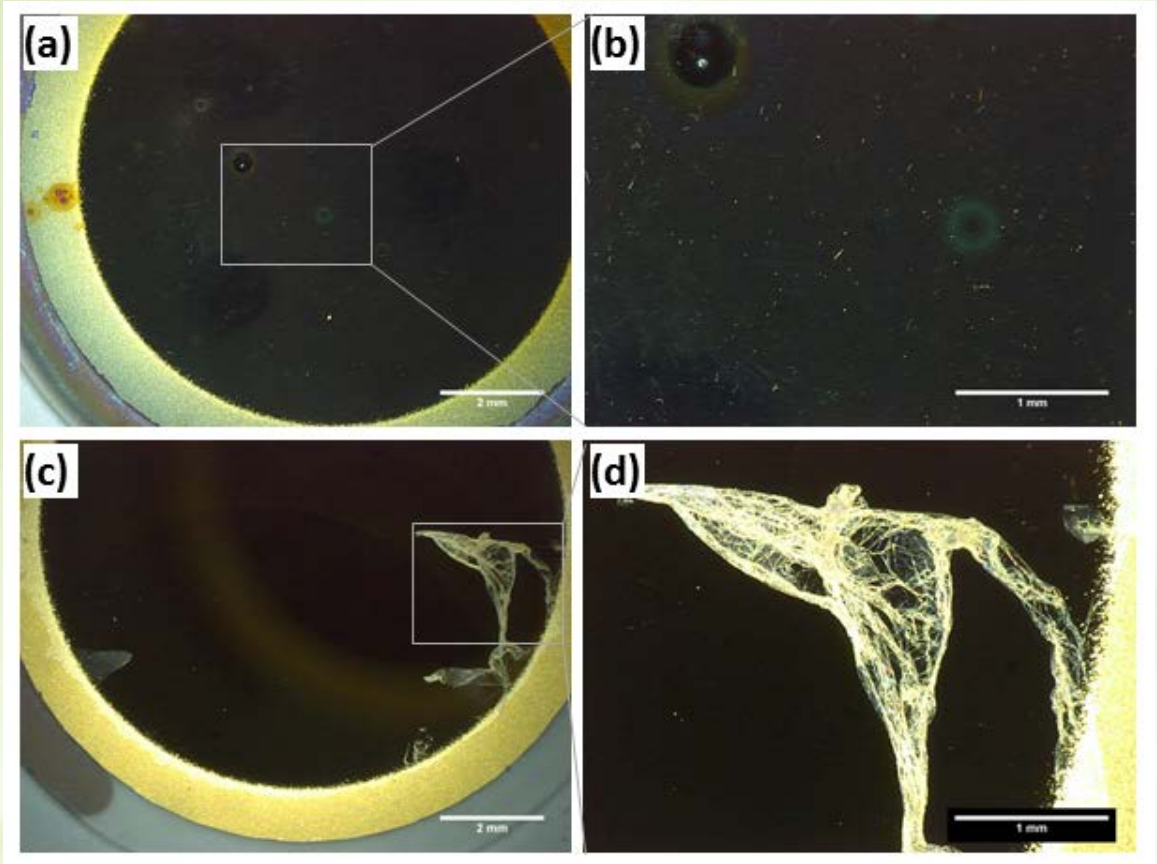
Performs as well as fluoroplastic in non-sonicated environment



Dursan® remains attached to the steel surface after sonication.

30x 100x



Dursan

AF1600

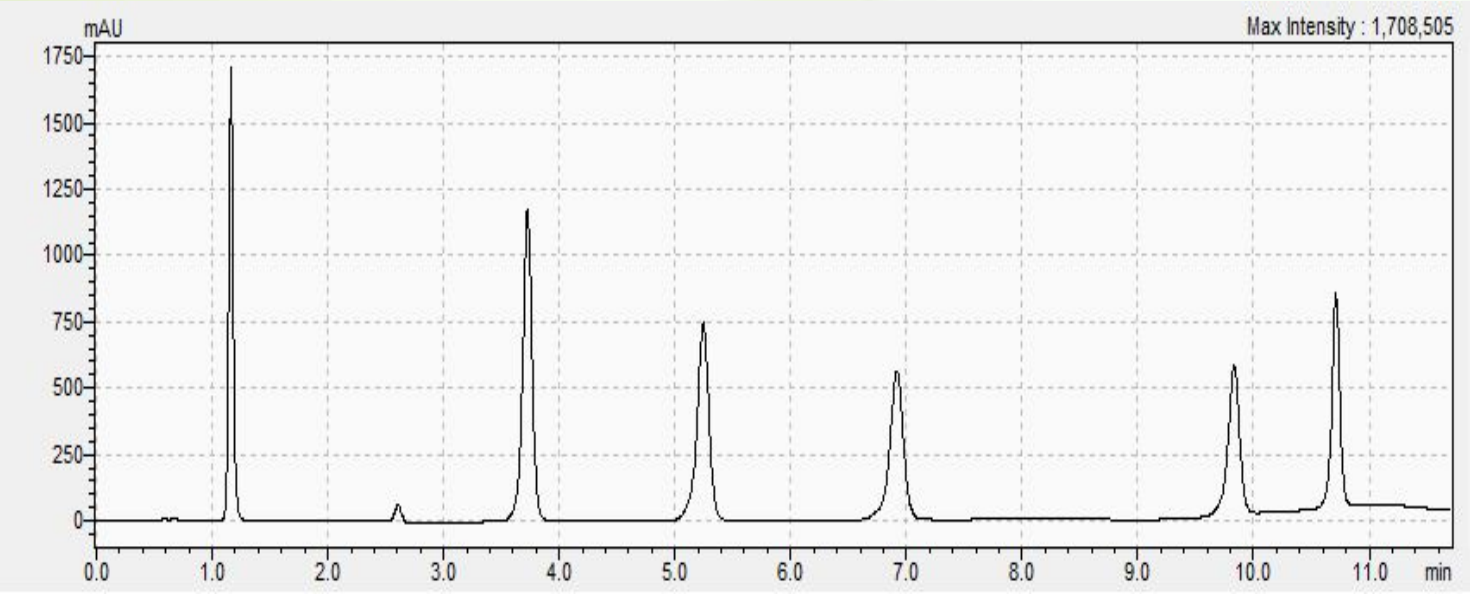
Vaidya, S.V.; Yuan, M.; Narvaez, A.R.; Daghfal, D.; Mattzela, J.; Smith, D. Appl. Surf. Sci. 2016, 364, 896-908.

Summary of Dursan® benefits

- Conformal coating that can be applied to columns, frits, tubing, pumps, sampling needles, etc.
- Molecularly bound to the surface and will not delaminate
- More wear resistant than stainless steel and PEEK
- Protects the substrate from corrosive attack
- Provides a bio-inert surface to decrease the amount of proteins that adsorb to the surface

Dursan® shows no detrimental effects during routine analysis

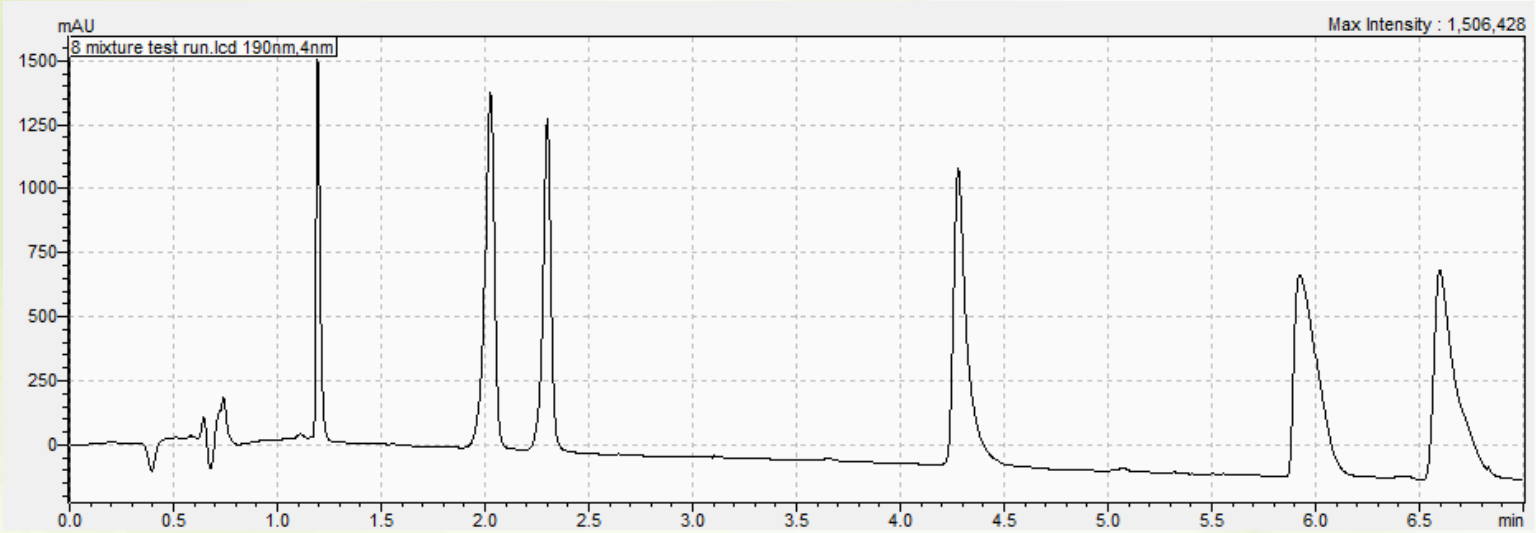
DNPH-derivatized aldehydes and ketones
C18 end-capped
2.1mm x 150mm x 5 µm
Dursan-coated stainless steel column



Chromatogram of 100ppm standard mix. The peak identities and elution times are as follows: methylparaben-1.19min, propanal-DNPH-3.75min, butanal-DNPH-5.21min, pentanal-DNPH-6.88min, hexanal-DNPH-9.82min, and 4-octylphenol-10.72min.

Dursan® shows no detrimental effects during routine analysis

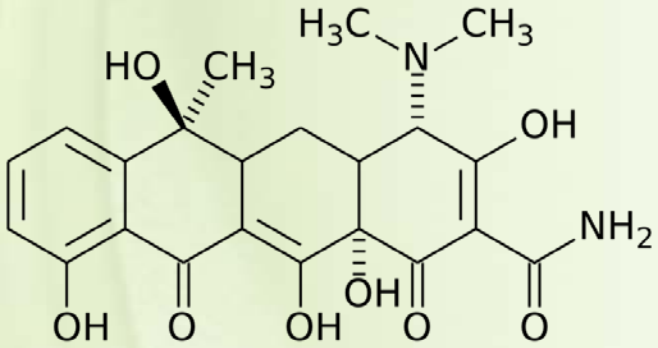
Drug Mixture
CN end-capped
2.1mm x 150mm x 5 µm
Dursan-coated stainless steel column



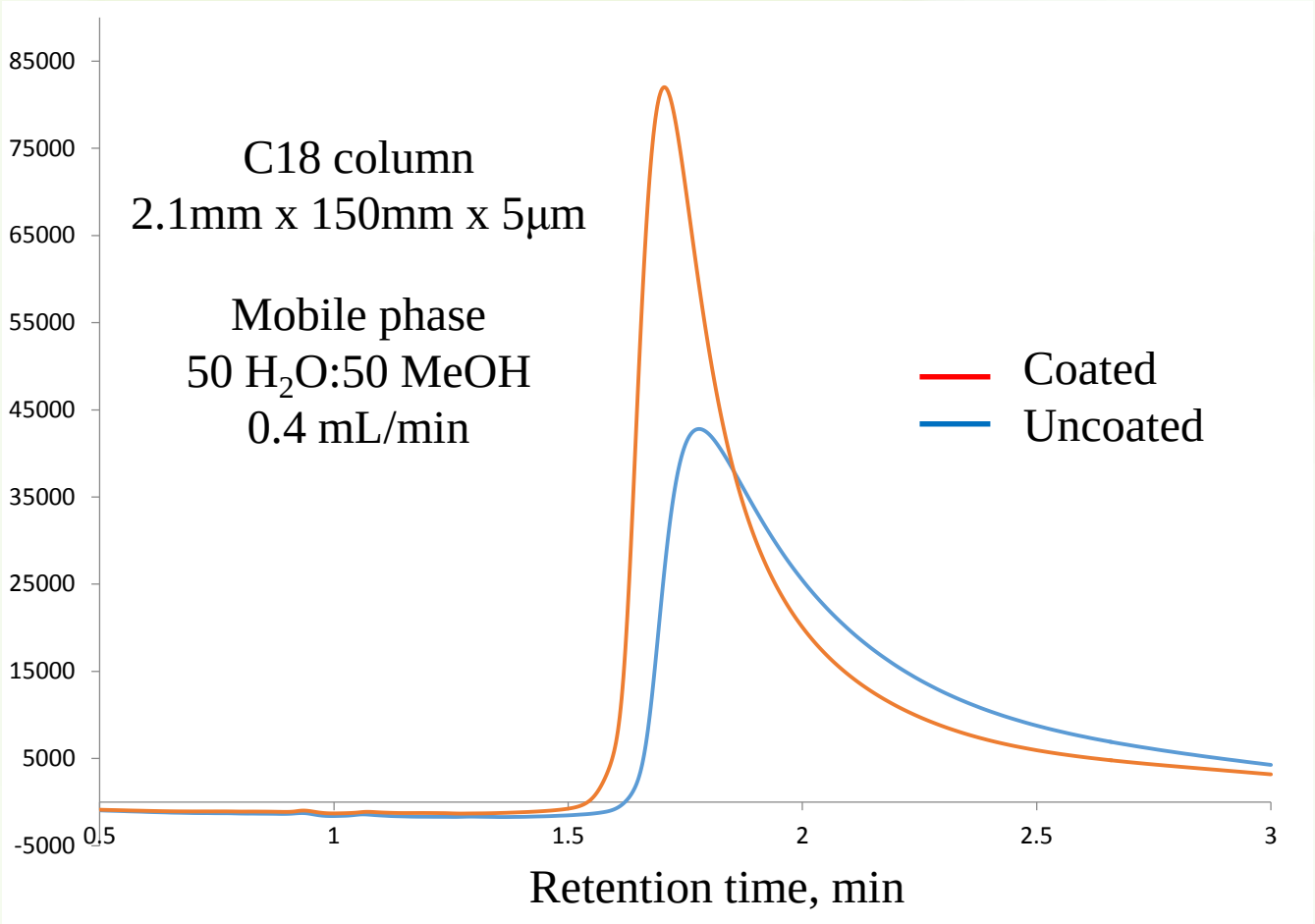
Chromatogram of 100ppm standard mix. The peak identities and elution times are as follows: uracil-1.21min, acetaminophen-2.05min, caffeine-2.30min, ibuprofen-4.32min, amitriptyline-5.90min, and doxepin-6.65min.

Chelating agent chromatogram

- Tetracycline has a number of potential chelating groups



- Dursan® coated column shows improvement in peak shape.



Dursan® coated HPLC columns: Customer testimonials

"The group that was using the coated column to isolate and purify their sensitive novel compounds have completed their study. The LC work was critical as it allowed further characterization of the compounds they synthesized."

"We have tried the treated tubes on two different column phases and we are seeing an increase in efficiency and improved peak shape."

"The coated columns have so far passed all tests bravely. The chromatographic separation in standard samples remains unchanged; in the case of biomolecules the results were, as expected, much better than in normal steel columns, but also better than in the case of pure PEEK columns."

Conclusion

- Dursan® can provide a bio-inert coating to all as-built stainless steel components
- Increased corrosion resistance without the possibility of swelling and delamination due to various solvents
- Decreased non-specific protein and chelating agent adsorption
- Column lifetime measurements and performance in extreme environment studies are planned