

Discussion of SilcoTek's coating technology in HPLC applications

Overview of discussion



- SilcoTek Corporation 101
 - Who are we and what do we do?
- Chemical and physical properties of Dursan[®]
 - Corrosion resistance
 - Protein anti-fouling properties
- Dursan® in use for "sticky" molecules
 - Sialylated N-Glycans
 - Small molecule drugs



SilcoTek Corporation Key Facts



 Founded in 2009 (currently 60 employees) as a CVD coatings service provider

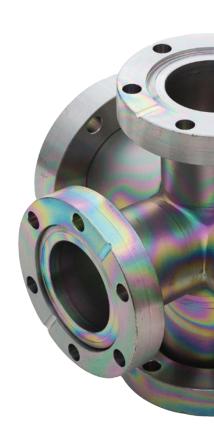
 Vision: To have the world's best coating technology and service, as told by our customers

 Purpose: To solve our customers' toughest material problems, help them beat the competition, and take their innovations and products to the next level

Advantages of CVD Coatings

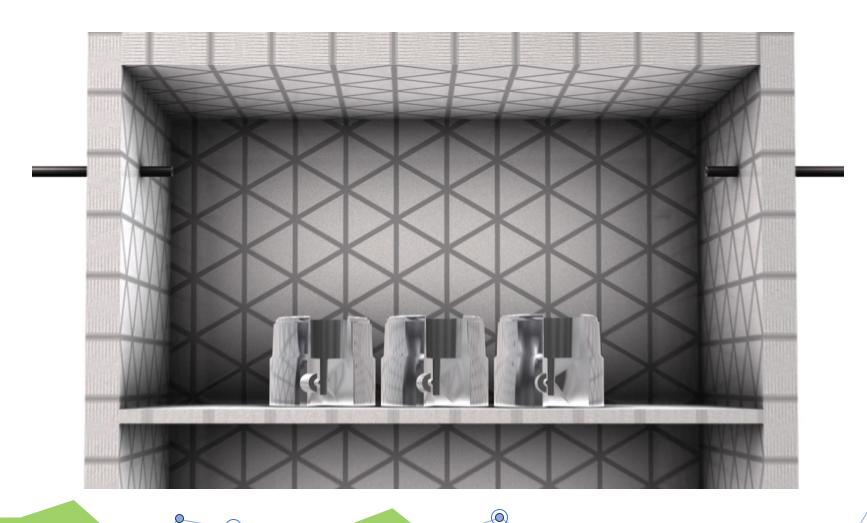


- Non-line-of-sight deposition; uniformly treats 3D, high aspect ratio part geometries
- Molecular adhesion to base substrate. Won't flake nor delaminate.
- Scalable, versatile, and highly reproducible



The CVD process



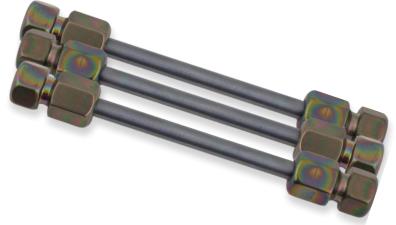


Our HPLC coating of choice: Dursan®

SilcoTek

- Non-line of sight coating (can coat frits)
- 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
- USP Class VI compliant

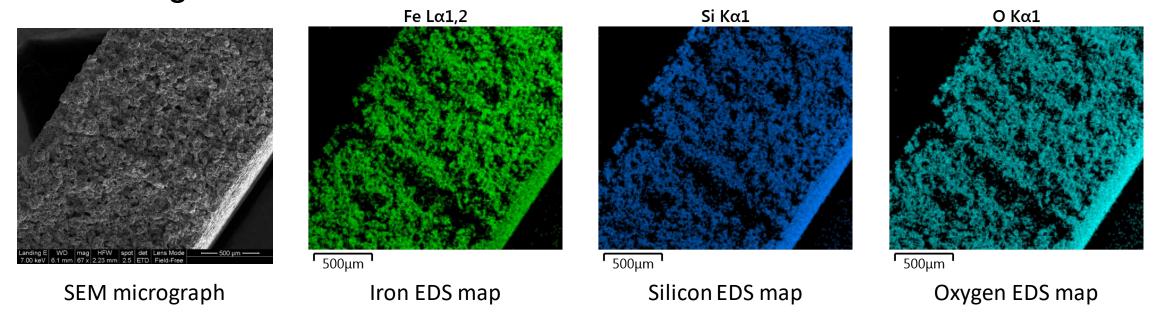




Example of non line of sight coating:



Cross section of a 2µm nominal pore size frit after Dursan® coating:



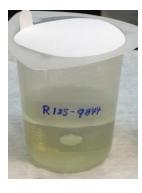
Corrosion resistance in harsh conditions



Corrosive media	Bare Stainless Steel (MPY)	Dursan coated steel (MPY)	Improvement multiplier
6M HCl @ room temp	190.4	1.1	170x
6M HCl @ 50°C	3116.1	23.5	133x
Concentrated H ₂ SO ₄	78.45	0.15	523x
48% HBr	2.05	0.29	7x
Bleach	1.70	0.10	17x
Concentrated H ₃ PO ₄ @ 80°C	2.14	0.53	4x
2% TFA	No corrosion, change in CA	Unaffected	-









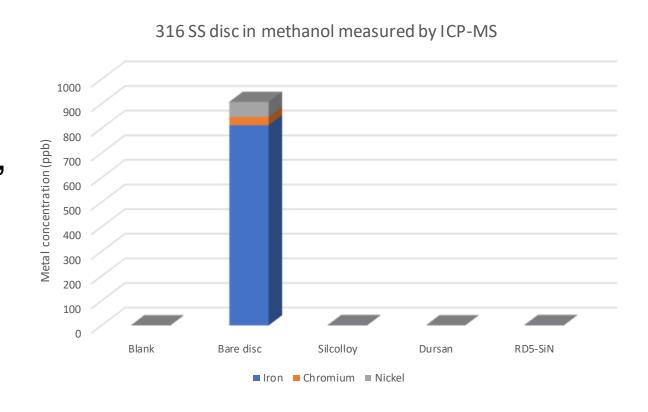
uncoated

coated



Protection against organic solvents

- Frits were left to soak in methanol for 1 month
- Results showed significantly higher levels of iron, chromium, and nickel in the uncoated frit container (908 ppb total metal content)
- Coated frits were all ± 0.5 ppb total metal of the blank

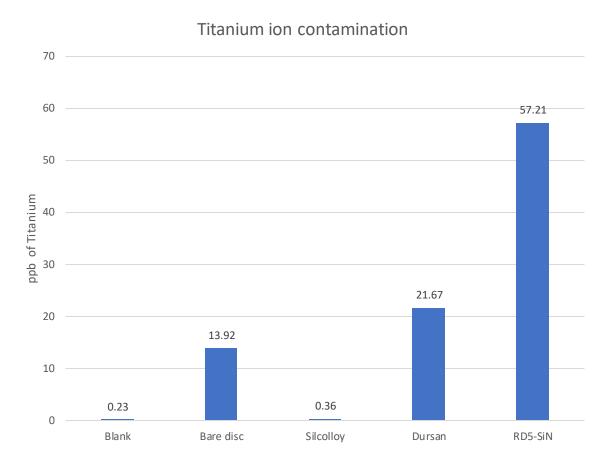


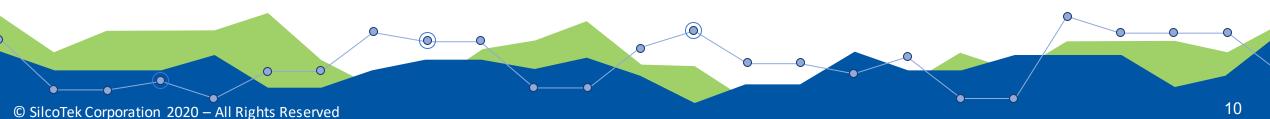




Coated titanium shows a different story:

- Silcolloy coating seems to work well where others fail
- Dursan and RD5-SiN both have an oxidation step in the process, and titanium is very sensitive to high temperature oxidation where Silcolloy (an amorphous silicon coating) is an oxidative barrier and protects the titanium





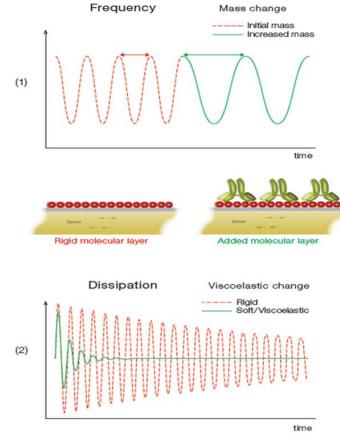
Bio-Inertness: Non-specific protein adsorption studies



 Collaborative study between Abbott Laboratories and SilcoTek on protein adsorption

 QCM-D with a thin layer of 316L SS was coated with Dursan

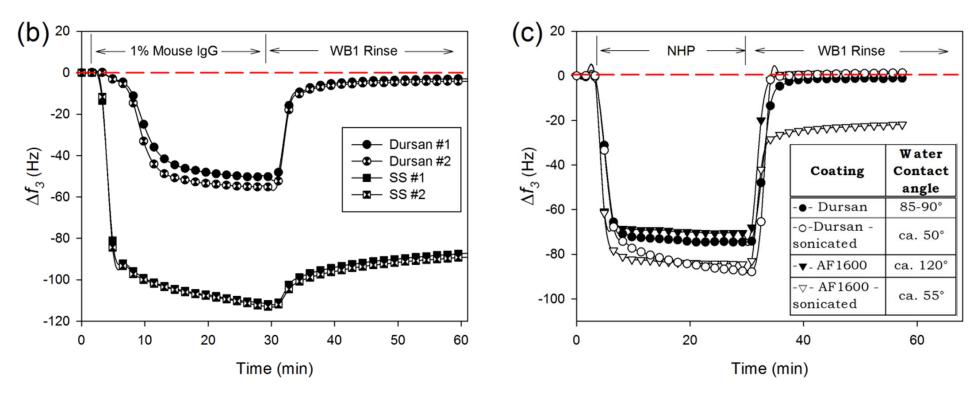
 Protein solutions were flowed over the sensor and the frequency was monitored over time



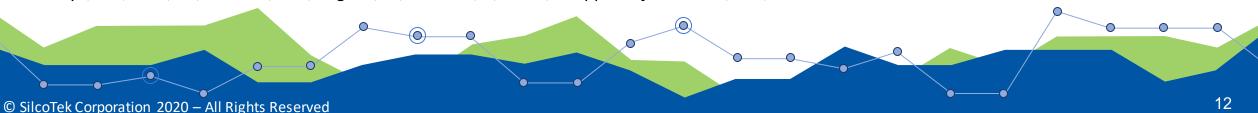
Vaidya, S.V.; Yuan, M.; Narvaez, A.R.; Daghfal, D.; Mattzela, J.; Smith, D. *Appl. Surf. Sci.* **2016**, *364*, 896-908.

Mouse immunoglobulins and normal human plasma adsorption





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

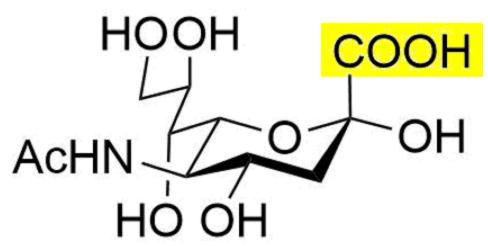


Analysis of N-Glycans



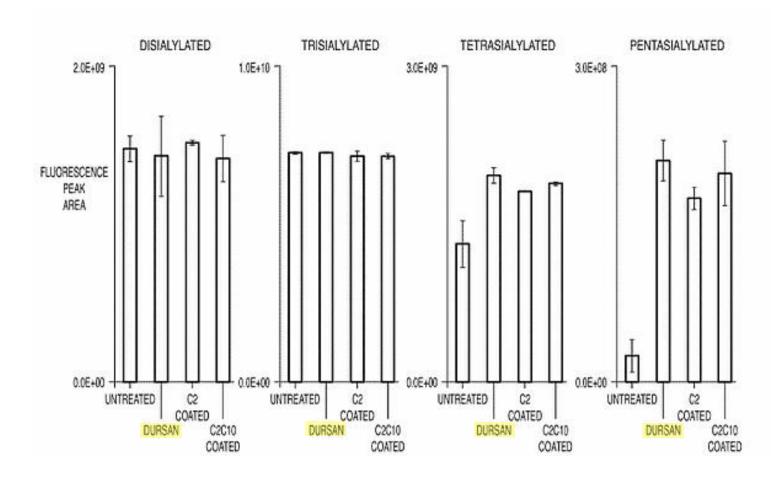
- Deglycosylation of a protein is standard in protein and biologic analysis
- Sialylated glycans contain carboxylic acids that have weak affinity toward metals

N-Acetylneuraminic acid:



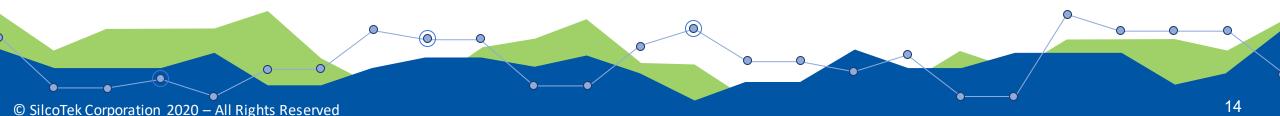
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Analysis of multiple sialic acid residues:



Lauber, Matthew A., et al. "Use of vapor deposition coated flow paths for improved chromatography of metal interacting analytes." U.S. Patent Application No. 16/133,089.

This patent has been harshly rejected by the patent office, but provides some great data on what coatings can do in HPLC equipment

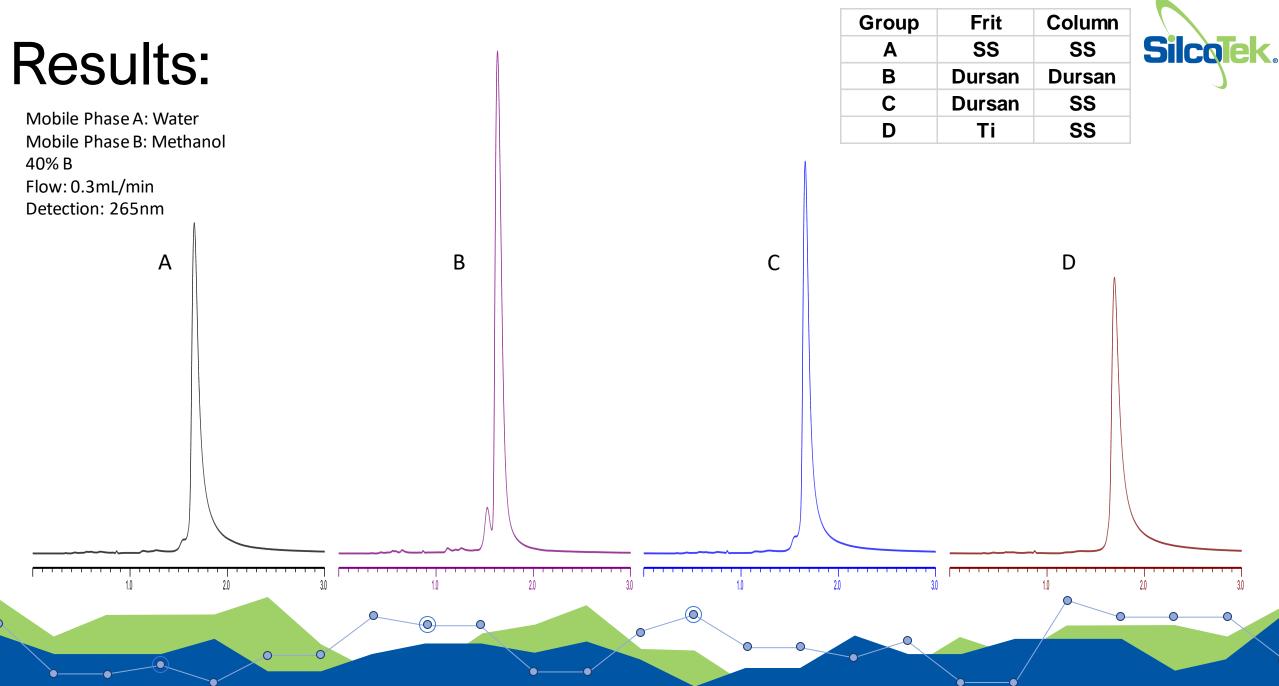


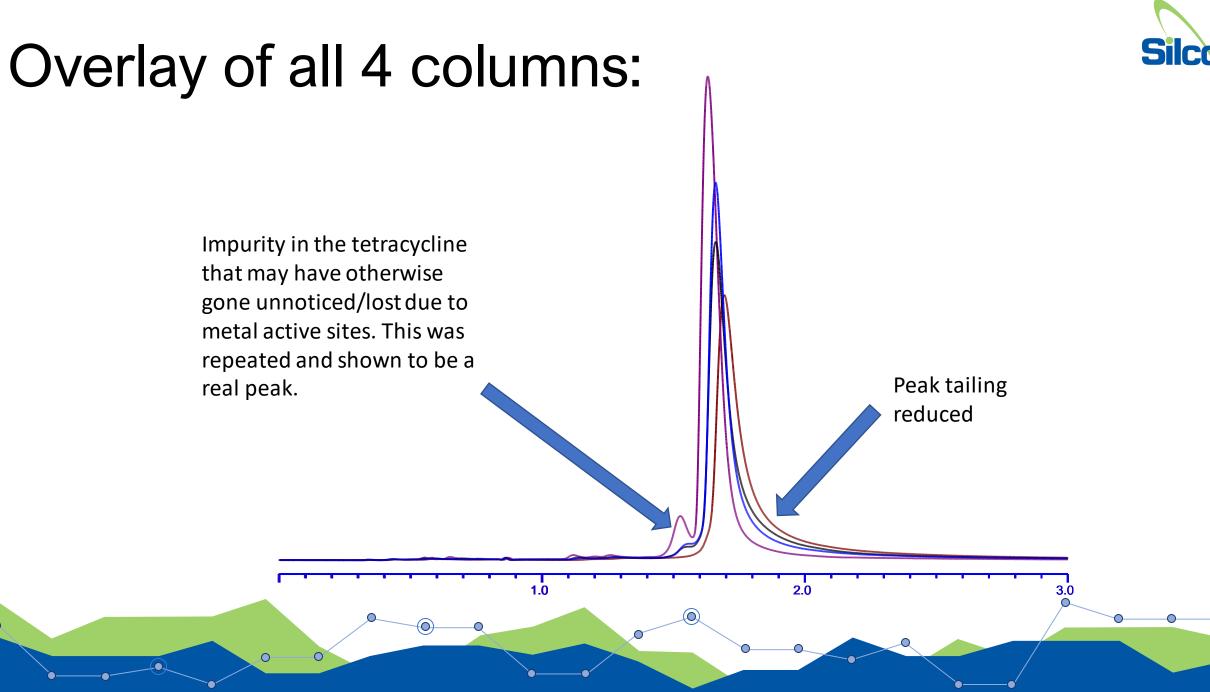
Tetracycline



- Tetracycline is an antibiotic, commonly used for acne and skin infections
- The molecule has numerous chelating groups that bind readily to metal sites
- Dursan can make the steel column more inert toward metal loving molecules like tetracycline

Molecular structure of tetracycline



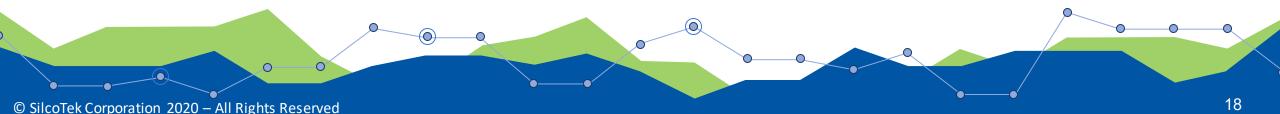


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2-pyridinol-1-oxide



- Ciclopirox is an antifungal agent typically used in topical fingernail and toenail infections
- 2-pyridinol-1-oxide is the chelating part of this antifungal agent
 - It is a very powerful metal chelating agent
- The chromatograms show significant loss of signal due to metal interactions in the separation
 - This highlights the interaction that the column wall has with the analyte and there is a need for column coating as this interaction is not negligible.





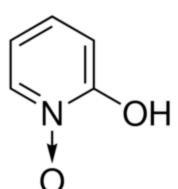
Mobile phase: Water no buffer

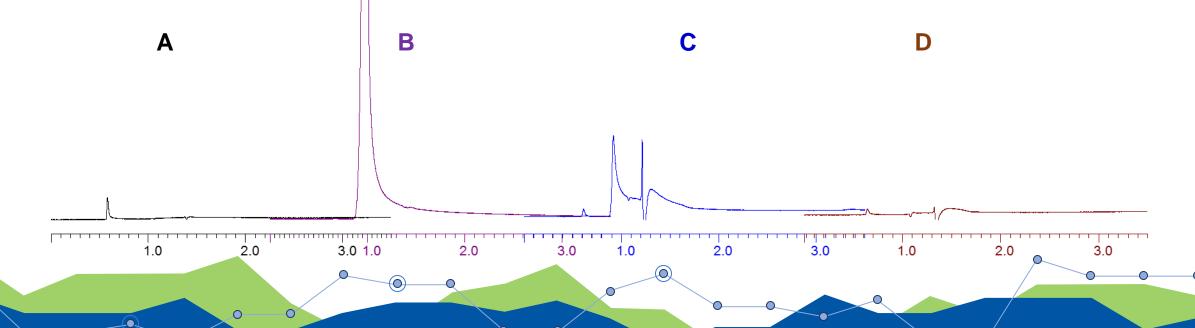
Flow: 0.3mL/min Detection: 230nm

100 X 2.1

Group	Frit	Column
Α	SS	SS
В	Dursan	Dursan
С	Dursan	SS
D	Ti	SS







SilcoTek.

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 while still having the robustness of stainless steel
- No need for priming or passivation. A protective, barrier layer allows for reliable, reproducible data.

