COVALENT METROLOGY

Welcome

covalentmetrology.com

COVALENT METROLOGY

SOLID SURFACE ZETA POTENTIAL: INDUSTRIAL APPLICATIONS, CHALLENGES, AND SOLUTIONS

GUEST SPEAKER:

Dr. Christine Körner
Product Specialist,
Materials Characterization

June 25, 2020 11am PDT

COVALENT METROLOGY

Anton Paar

Covalent Metrology

- **Founded 2016**
- Testing, measurement & characterization **Platform**
- **30 team members** (13 PhDs)
- **9,500 ft² lab in Sunnyvale, CA**
- **1-Stop-Shop Source for Answers**
  - 30 instruments in-house
  - 6 partnerships with instrument manufacturers
  - 11 partner labs
  - 6 corporate “tool shares”
- **More than 300 customers** (80% repeat)

Covalent Instrument Partners

Anton Paar

COVALENT METROLOGY

- **Partnership established December 2019**
- **Silicon Valley demo showroom at Covalent**
- **Several Anton Paar instruments installed:**
  - Tosca 400 — Atomic Force Microscope
  - MCR 302 — Modular Compact Rheometer
  - MCR 702 — Rheometer + DMA
  - SurPASS 3™— Electrokinetic Analyzer *(solid surface zeta potential)*
  - PSA 1190 — Particle Size Analyzer
  - Litesizer™ 500 — Light Scattering Nanoparticle Analysis

Other Partners

Rigaku

PVA TePla

J.A. Woollam

KEYENCE

OXFORD INSTRUMENTS

ASYLUM RESEARCH

Covalent Technical Groups and Organization

COVALENT METROLOGY

hello@covalentmetrology.com 408-498-4611

Electron & Scanning Probe Microscopy

SEM / TEM, AFM, EDS, EELS, Nanoindentation, Acoustic Microscopy

Surface Analysis

XPS, Auger, TOF-SIMS, d-SIMS

Optical Microscopy & Spectroscopy

Laser Confocal, White Light, Chromatic Aberration, Spectral Ellipsometry, UV-Vis-Nir Spectroscopy

Materials / PC Board Failure Analysis

Cross Sections, Inspections, Dye & Pry Testing

X-Ray Characterization

XRD / XRR, XRF, µCT

Chemical Analysis

ICPMS, GCMS, FTIR, Raman Spectroscopy, NMR

Nanoparticle Analysis

DLS, PSA, Particle Zeta Potential

Bulk Properties Characterization

DSC, TGA, DMA, Rheometry, Tensile Testing, Pencil Test, Surface Zeta Potential

Introducing

Dr. Christine Körner

Dr. Christine Körner earned a PhD in Physical Chemistry from Graz University of Technology, Austria, before joining Anton Paar in 2011 as a product specialist for material characterization. She has nearly 10 years of experience with zeta potential analysis and is now the international product manager for solid surface charge instrumentation at the Anton Paar headquarters in Austria.

Anton Paar

SOLID SURFACE ZETA POTENTIAL

Industrial Applications, Challenges and Solutions

Dr. Christine Körner
Anton Paar GmbH, Graz, Austria

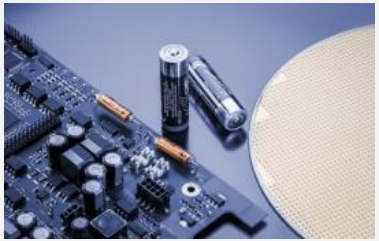
Agenda

- Introduction to solid surface zeta potential
- Applications of solid surface zeta potential:
  - Membrane technology
  - Life sciences
  - Semiconductor industry

Zeta potential – key applications

Membranes
Wastewater

Pharma & Life Science
Biomaterials, Medical Devices, Cosmetics



Electronics
Batteries, Semiconductor

Petroleum Industry
Rocks, Oil

Chemicals
Polymers & Plastic, Latex, Paints & Inks

Food & Beverage
Filtration, Packaging

Zeta potential analysis

3.8 nm — 100 µm

Litesizer 500
Electrophoretic Light Scattering

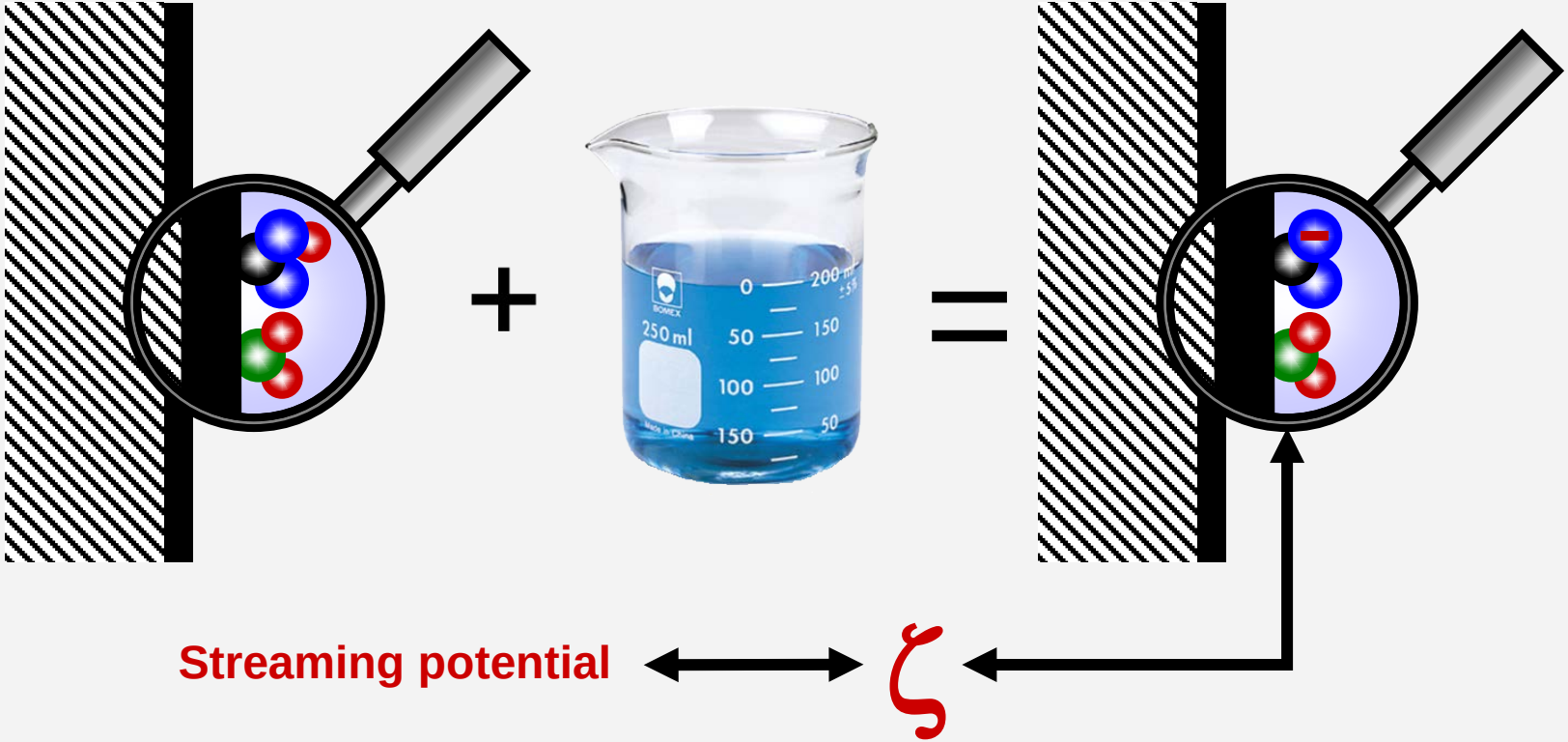
25 µm — unlimited

SurPASS 3
Streaming Potential

Introduction to solid surface zeta potential

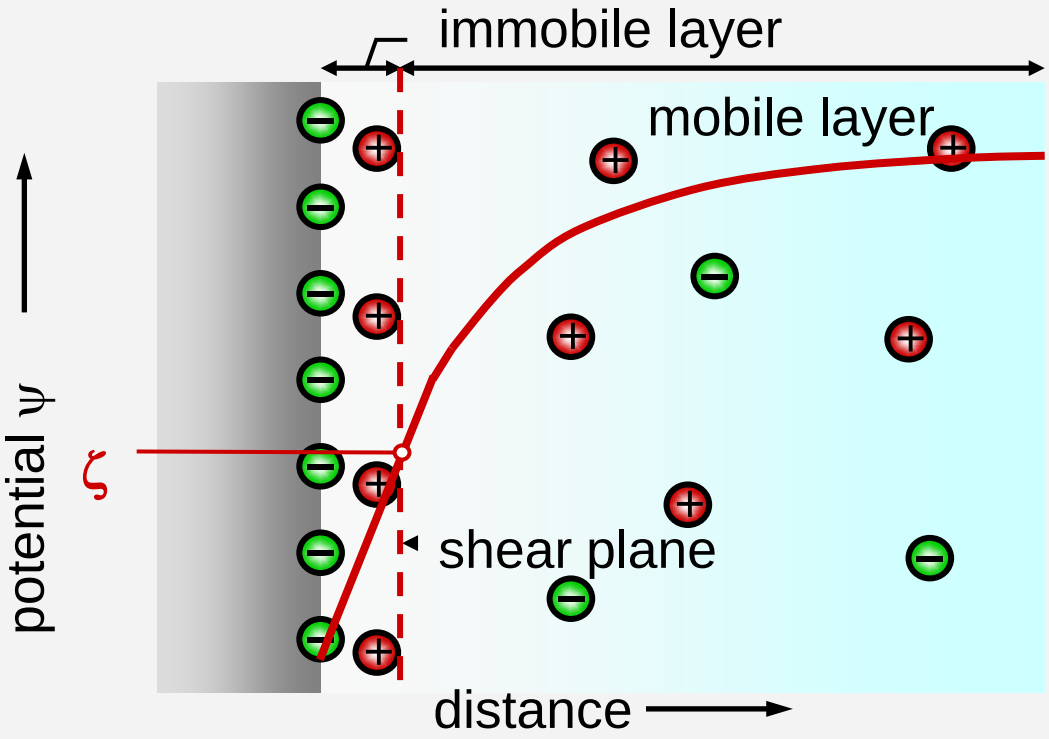
Anton Paar

Analysis of surface functional groups

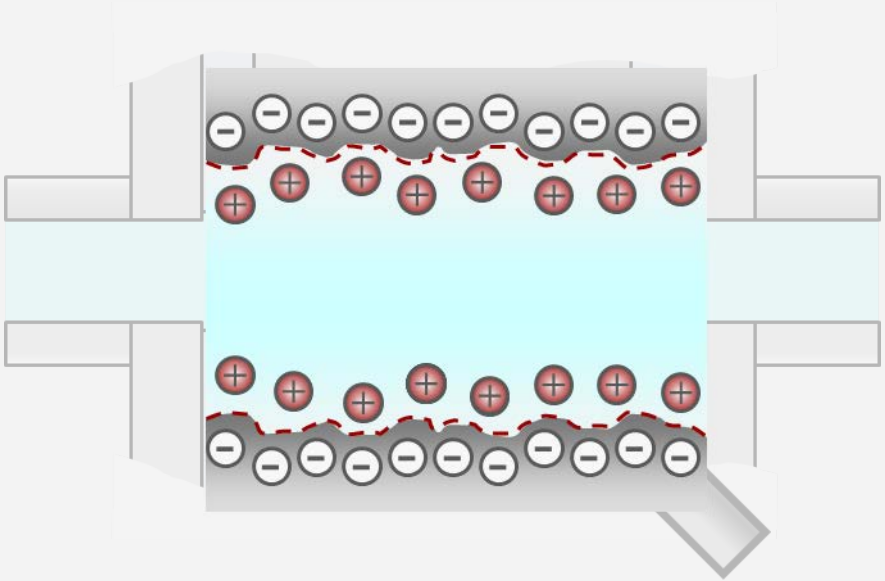


Anton Paar

Electric double layer

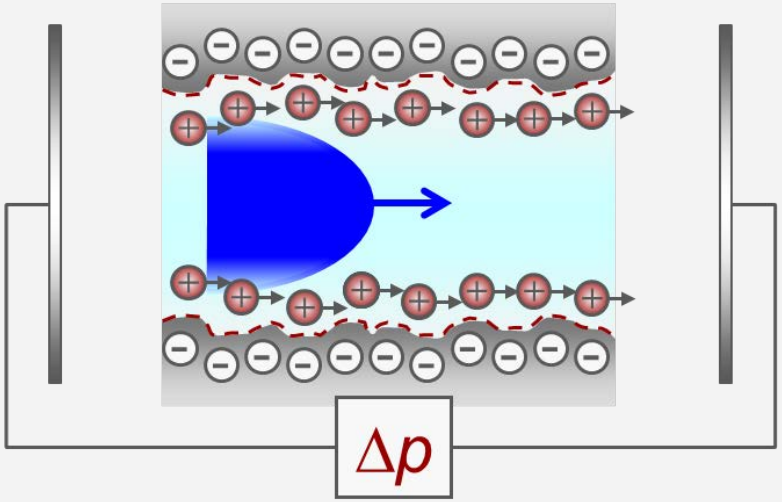


Streaming potential



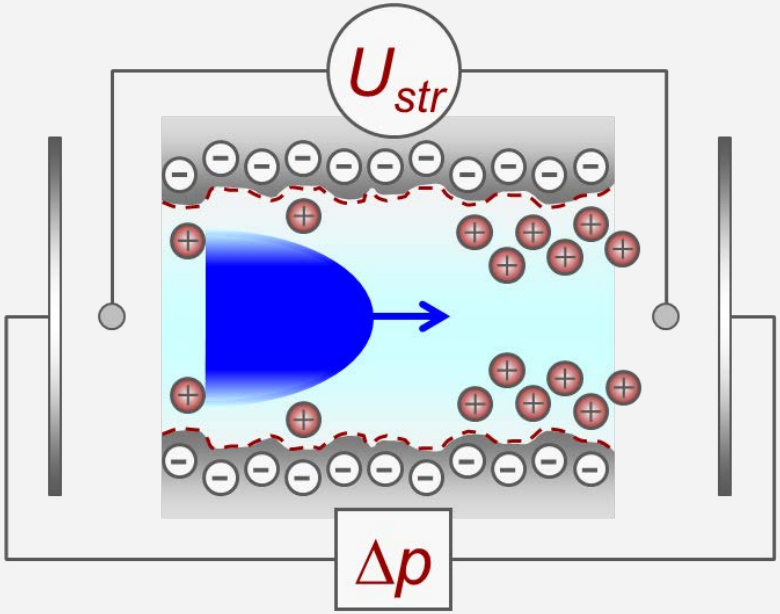
– Solid sample arranged to create a capillary channel

Streaming potential



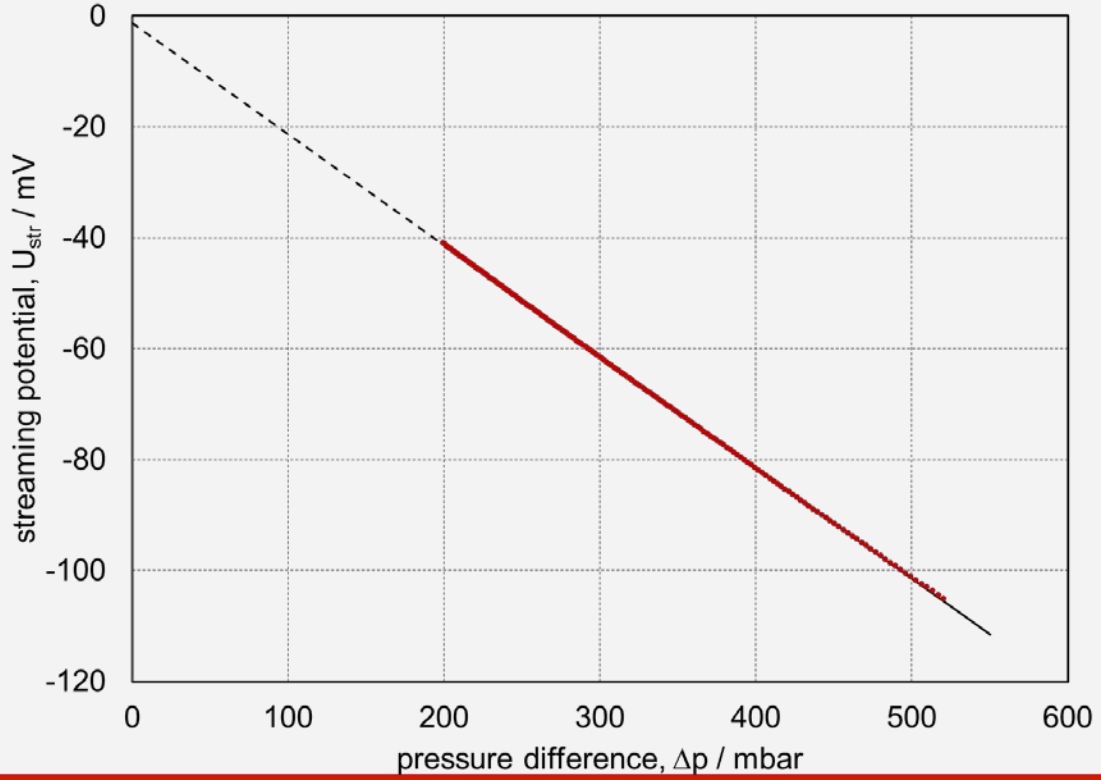
- Solid sample arranged to create a capillary channel
- Pressure gradient provokes liquid flow
- Charge distribution at solid | liquid interface distorted

Streaming potential



- Solid sample arranged to create a capillary channel
- Pressure gradient provokes liquid flow
- Charge distribution at solid | liquid interface distorted
- Streaming potential (d.c. voltage) generated along the flow channel
- $U_{str}/\Delta p$ related to surface zeta potential

Streaming potential → zeta potential

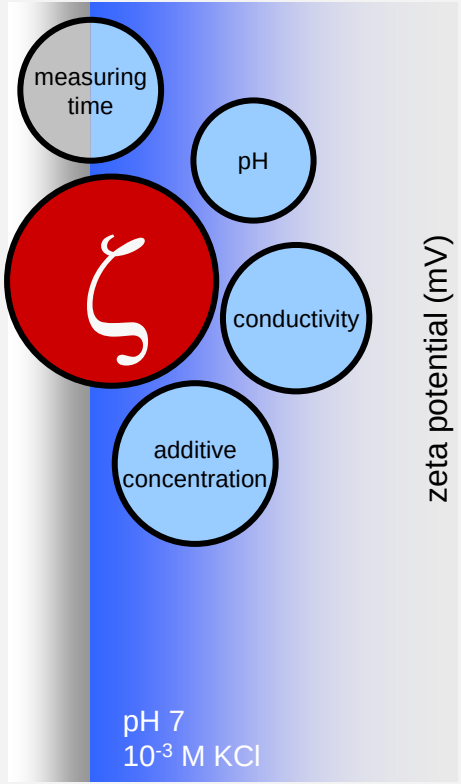


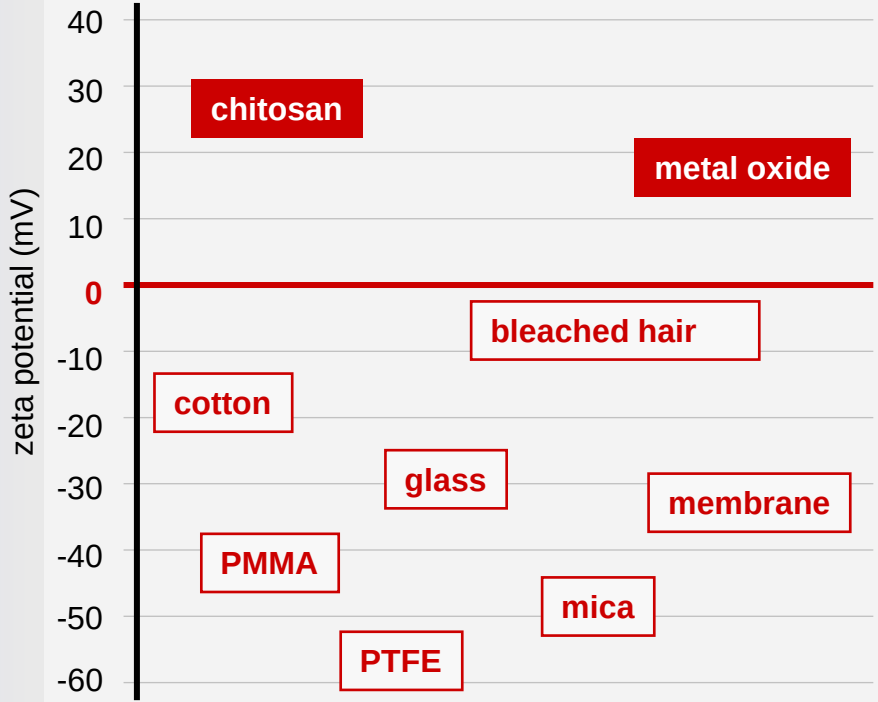
$$\zeta = \frac{dU_{str}}{d\Delta p} \times \frac{\eta}{\varepsilon \times \varepsilon_0} \times \kappa_B$$

$$\zeta = \frac{dI_{str}}{d\Delta p} \times \frac{\eta}{\varepsilon \times \varepsilon_0} \times \frac{L}{A}$$

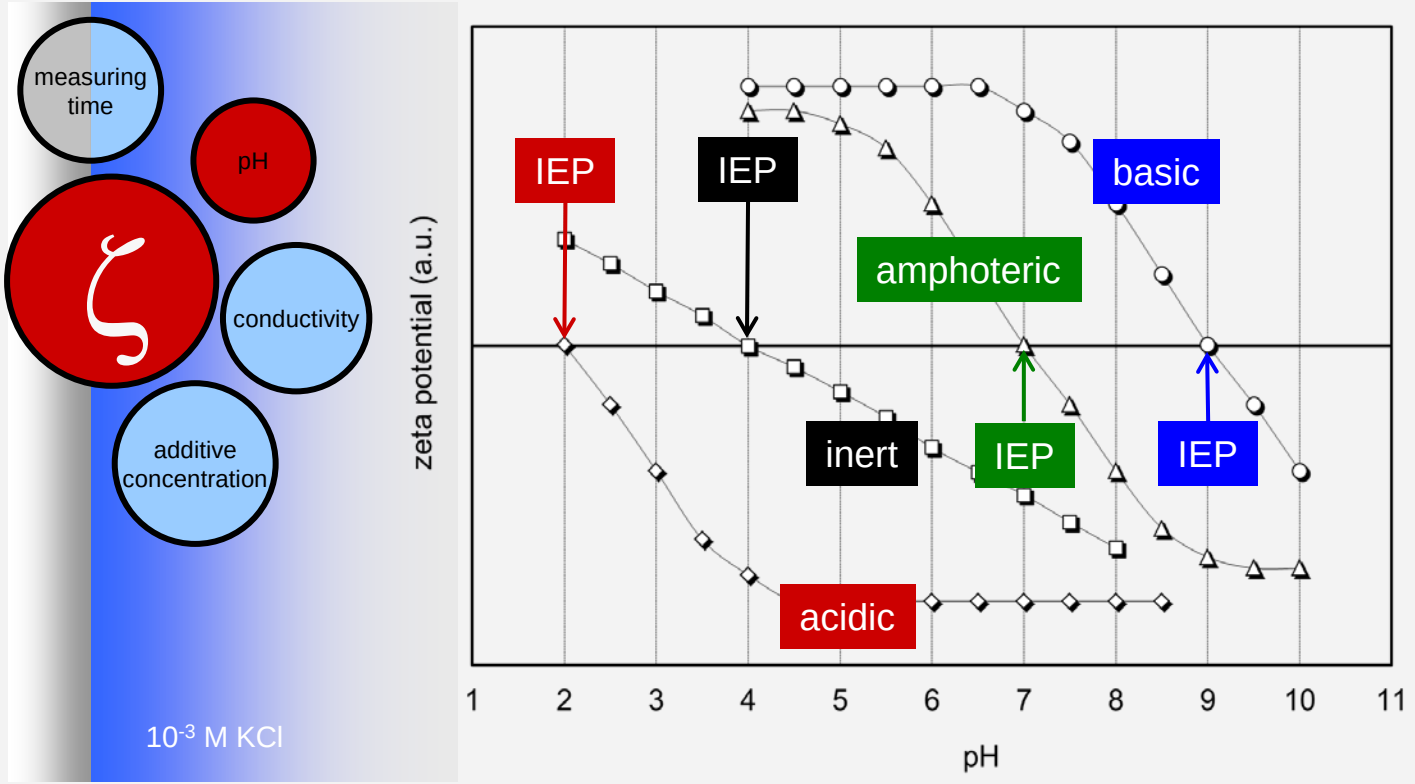
U_{str}	streaming potential
I_{str}	streaming current
Δp	pressure difference
η	electrolyte viscosity
$\varepsilon \times \varepsilon_0$	dielectric permittivity
κ_B	electrolyte conductivity
L/A	cell constant

Surface chemistry – single point zeta potential

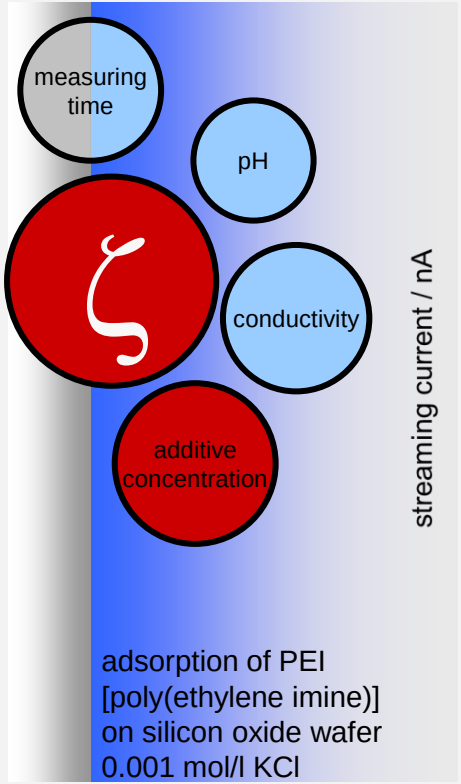


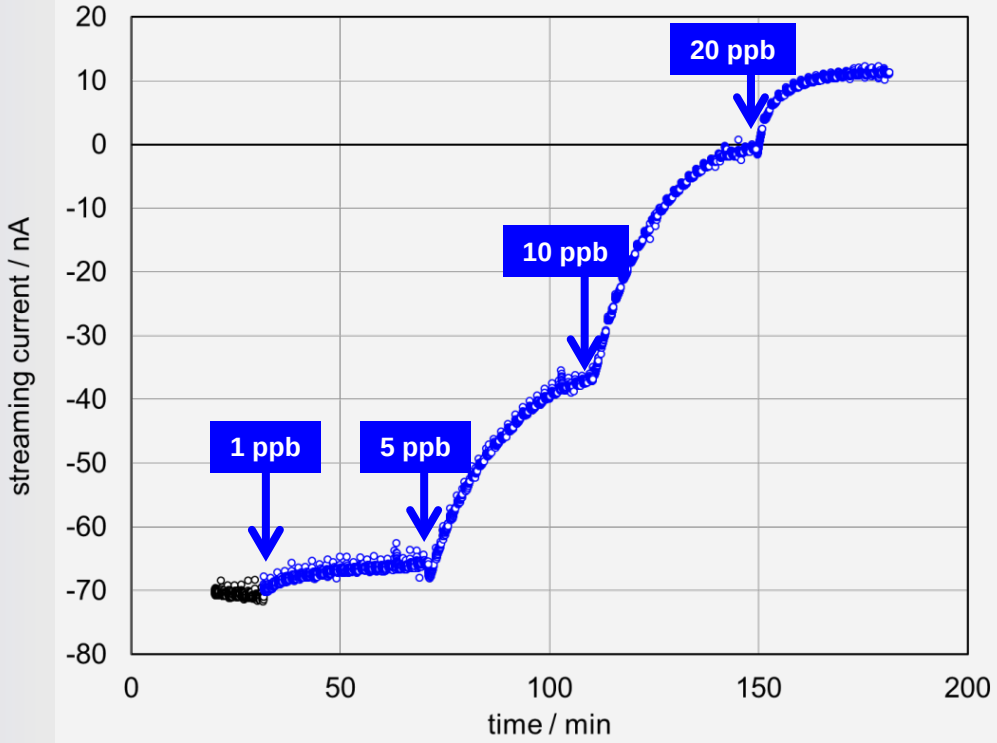


Surface chemistry – pH dependence

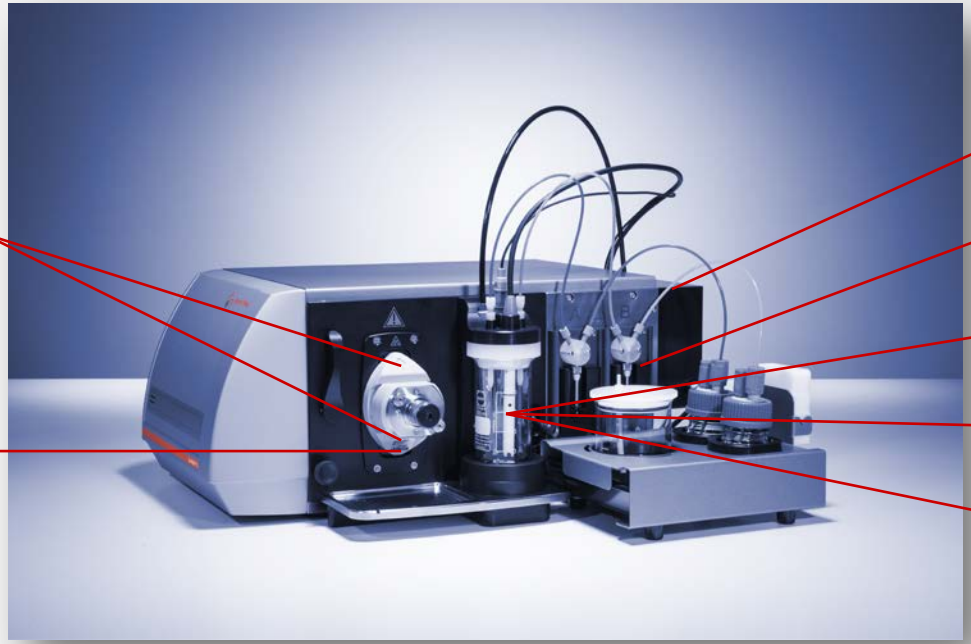


Surface chemistry – additive concentration





SurPASS 3 instrument



Ag | AgCl electrodes:
streaming potential
streaming current
cell resistance

measuring cell

external pressure control

titration unit

pH electrode

conductivity probe

temperature sensor

SurPASS 3 measuring cells

Adjustable Gap Cell

Adjustable Gap Cell for Disks

Clamping Cell

Cylindrical Cell

MC for Ceramic Membranes

MC for Flexible Tubing

MC for Soft Contact Lenses

MC for Hollow Fiber Membranes

MC for 1 ml Pre-Filled Syringes

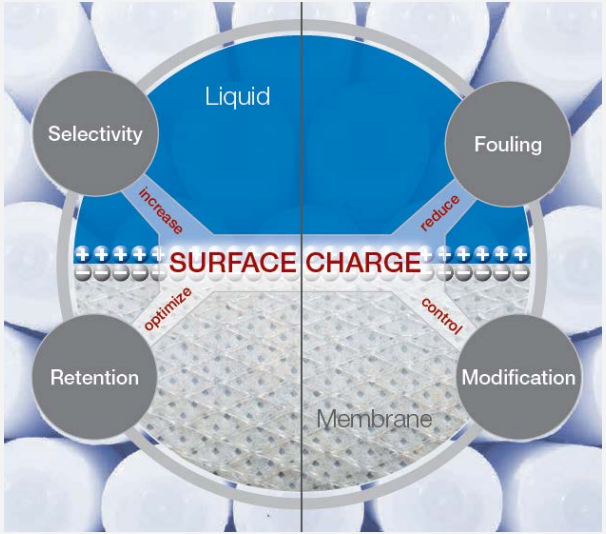
MC for Core Samples

Application of solid surface zeta potential studies: Membrane technology

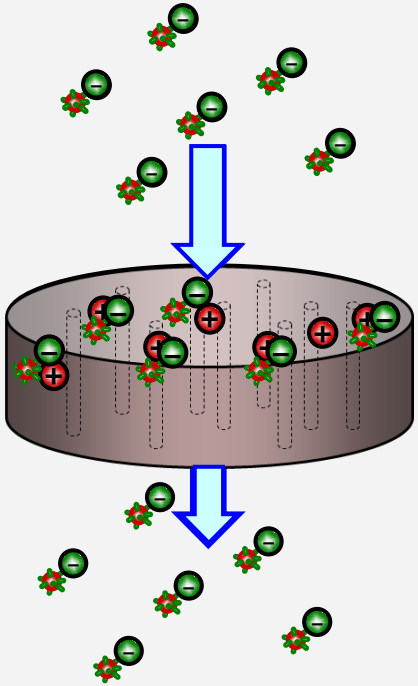
Zeta potential applications in membrane characterization

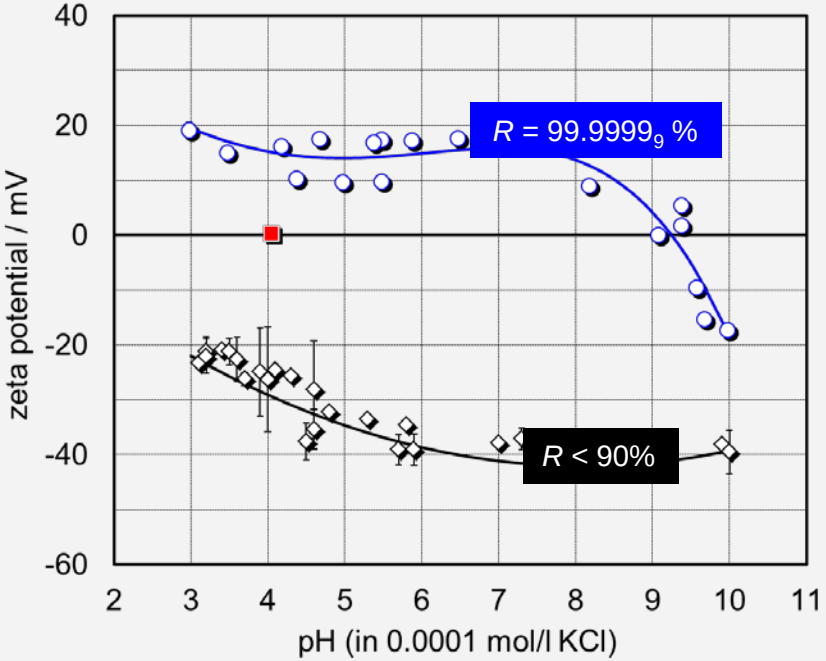
- Surface modification | treatment:
  Surface charge at the membrane-water interface

- Membrane fouling:
  Electrostatic interaction of membranes with solutes



Surface modification: Ceramic filter for virus retention

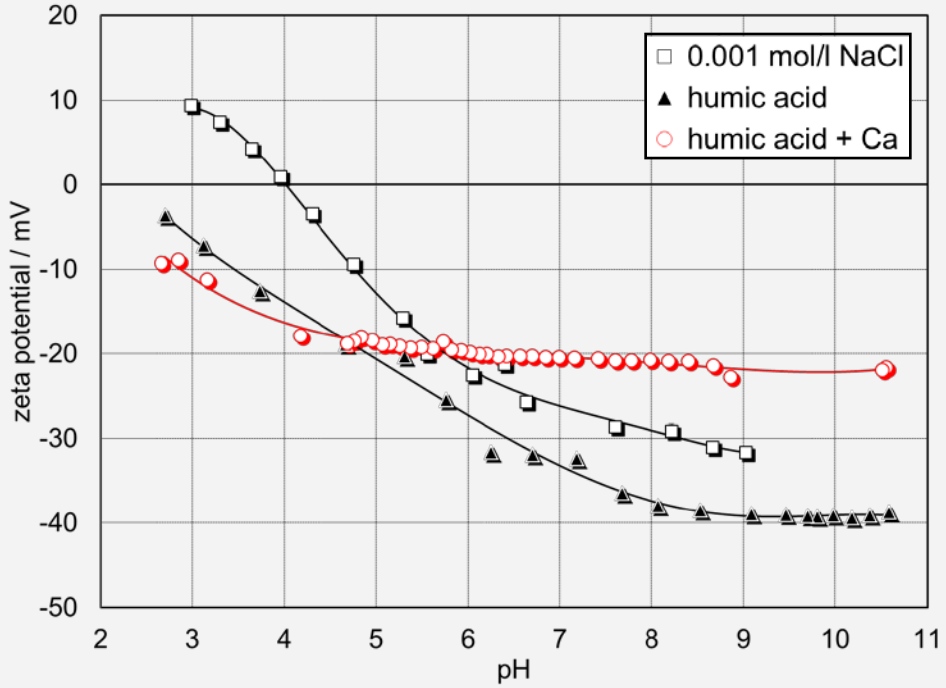




◇ DE filter ■ IEP virus ○ $Zr(OH_x)$, 250 °C

M. Wegmann et al., Water Res. 42 (2008) 1726

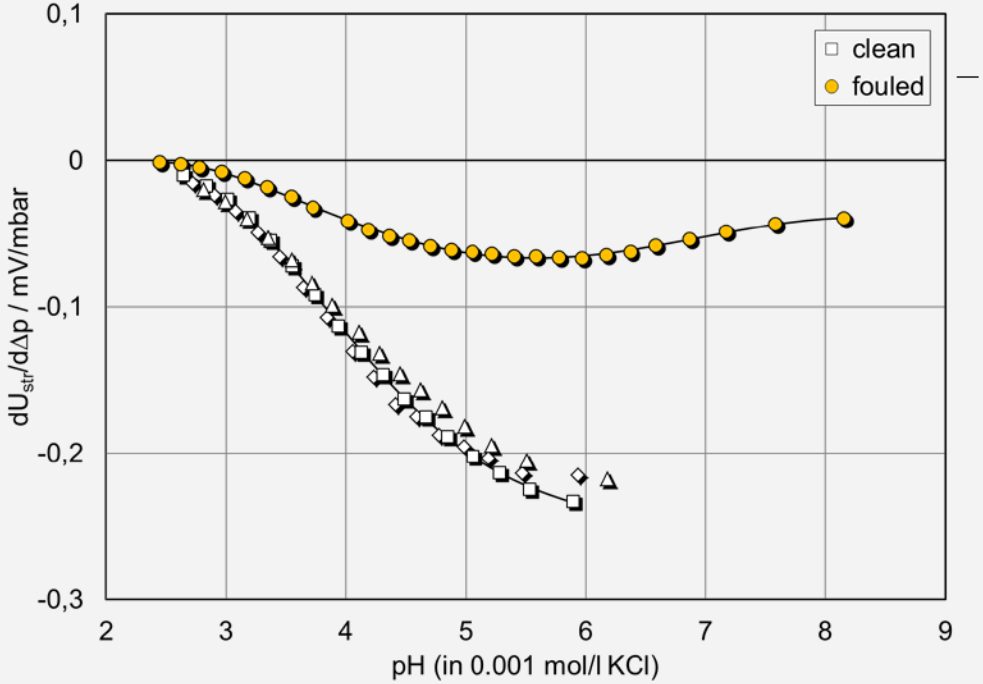
Membrane fouling: RO membrane



- BW30 brackish water RO membrane
- Effect of fouling

I. Owusu-Agyeman et al., J. Membr. Sci. 528 (2017) 82

Membrane fouling: HF membrane



– PES HF membrane
0.05 µm pore size

Carstensen et al., Bioresour. Technol. 137 (2013) 179

Application of solid surface zeta potential studies: Life sciences

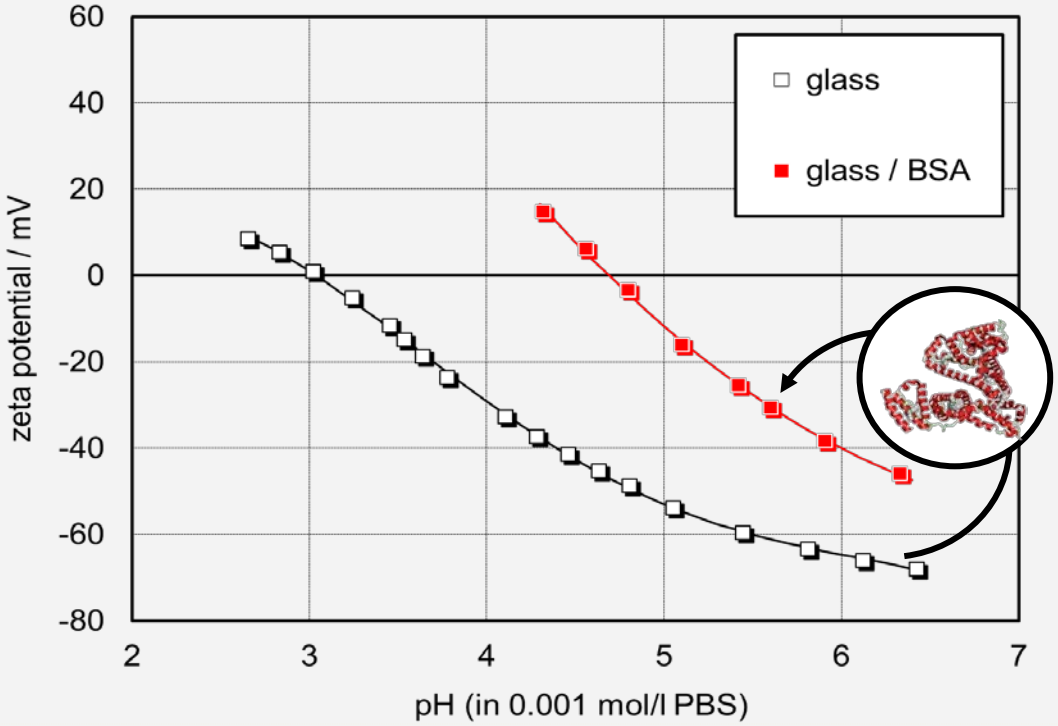
Zeta potential applications in biomaterial and life sciences

- Interaction of implant materials with proteins:
  Surface charge at the biomaterial-buffer interface
  Time-resolved adsorption kinetics

- Inner surface characterization of 1 ml pre-filled syringes

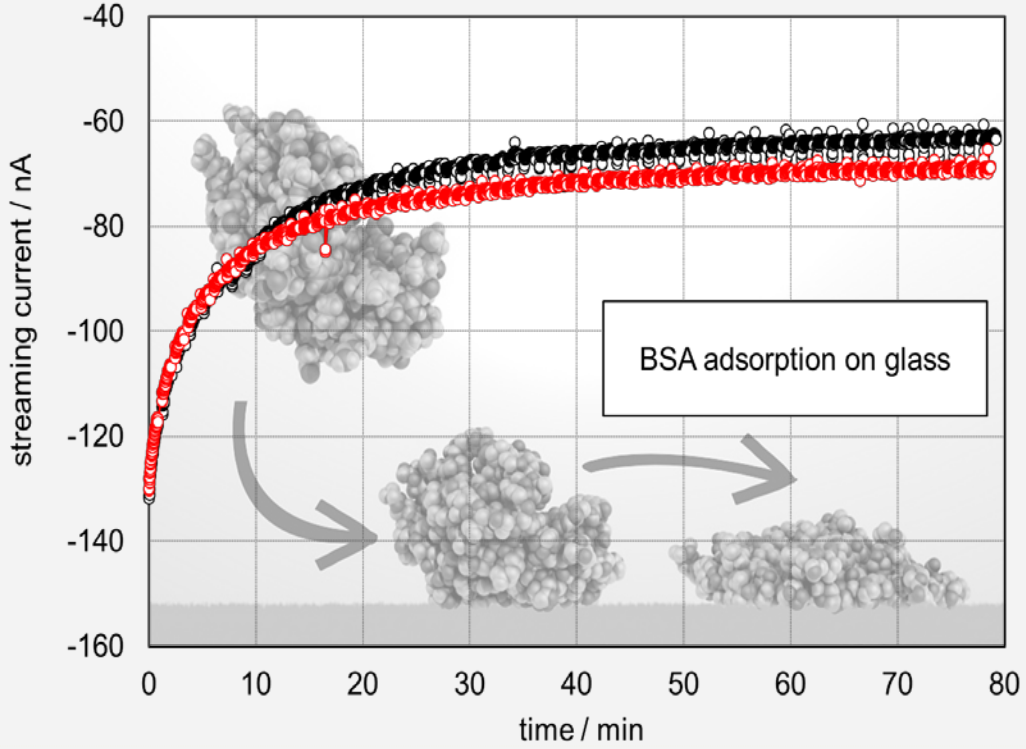
- Hair care:
  Rate of adsorption | desorption of shampoo and conditioner

Glass – protein interaction



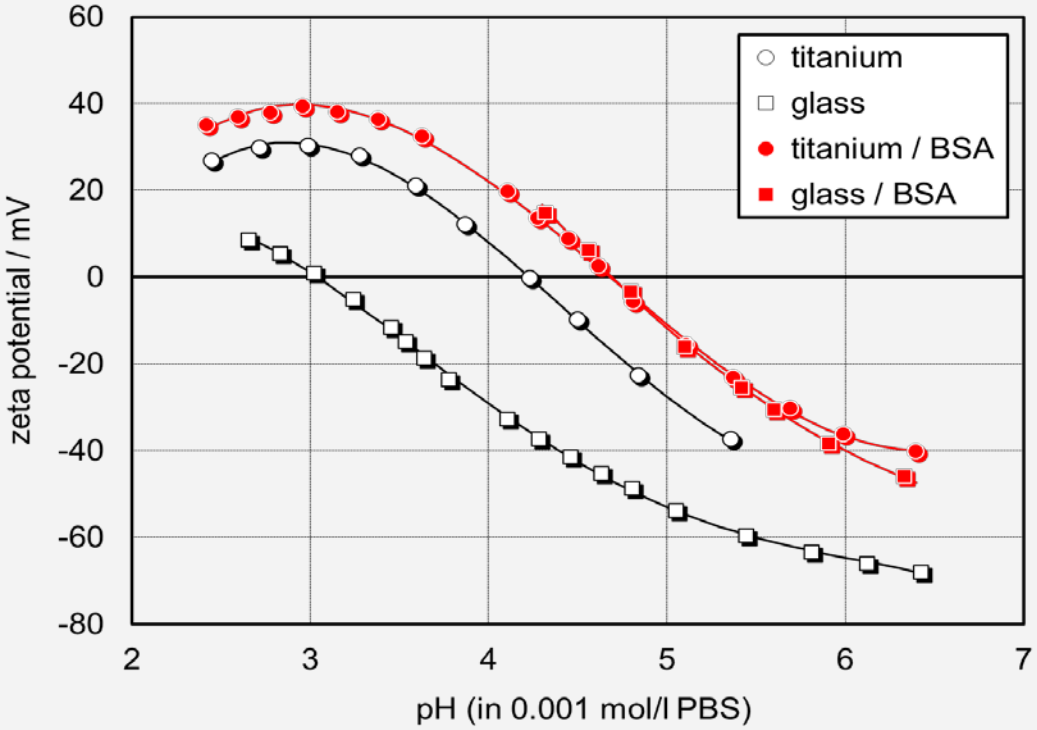
- Adsorption of BSA (bovine serum albumin) on glass
- Isoelectric point (IEP) of glass | BSA matches IEP of BSA in solution

Protein adsorption kinetics



- Adsorption kinetics of BSA (bovine serum albumin) on glass
- 0.2 mg/ml BSA
- 0.001 mol/l PBS, pH 6.5

Protein adsorption



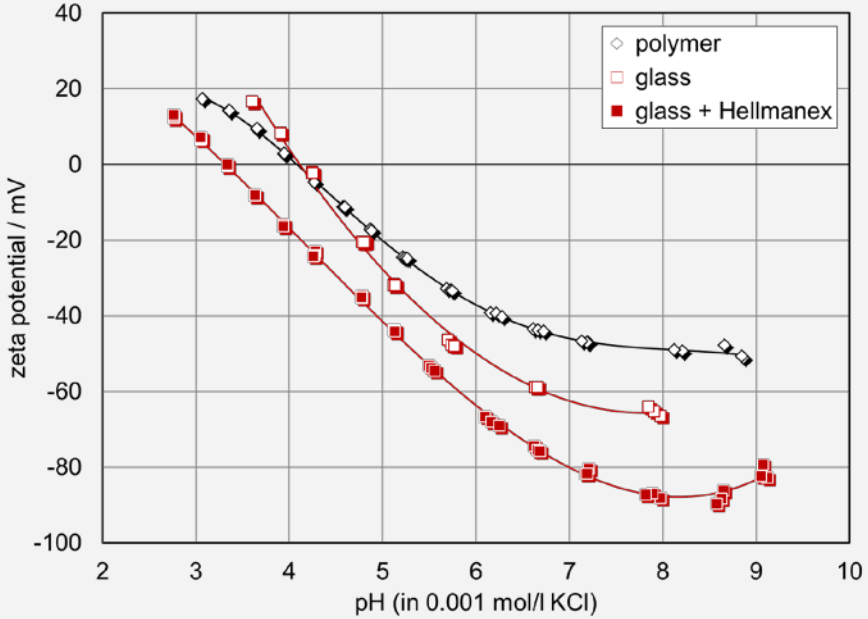
- Adsorption of BSA (bovine serum albumin) on glass
- Isoelectric point (IEP) of glass | BSA matches IEP of BSA in solution
- Same pH dependence of ζ for titanium (reference sample) after adsorption of BSA
- BSA shows non-selective adsorption

Inner surface characterization of 1 ml pre-filled syringes

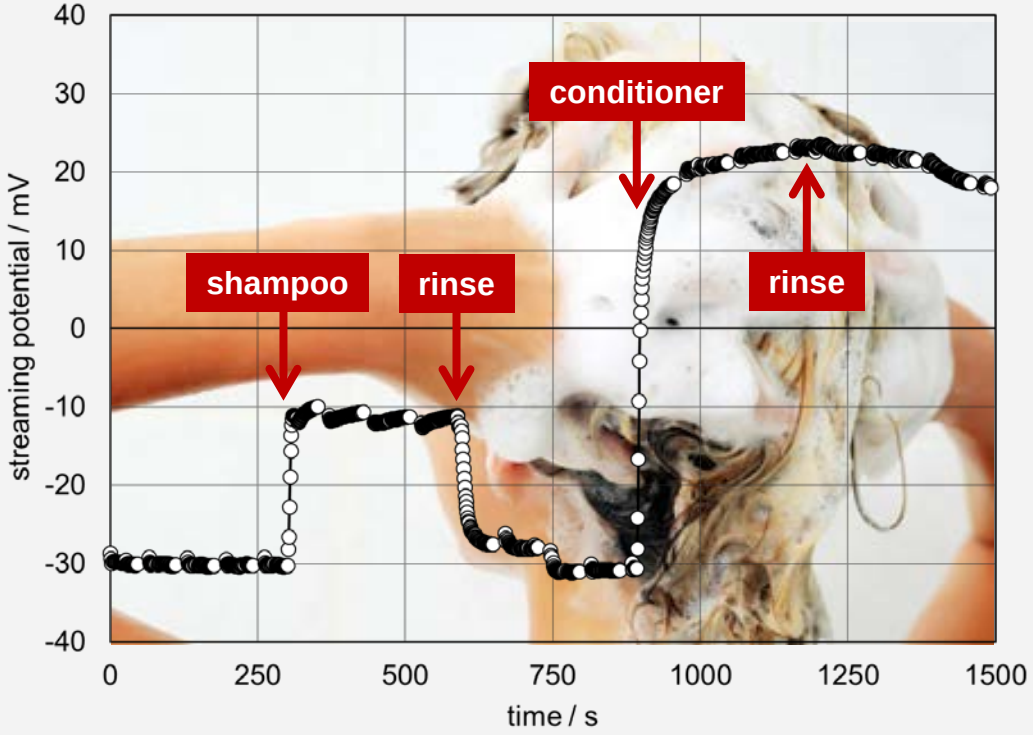
Polymer vs. glass syringe

IEP: pH (ζ = 0 mV)

Findings:
- IEP at pH 4 for both surfaces
- Hellmanex cleaning exposes the clean glass surface



Hair care



The effect of shampoo and conditioner on the surface charge of hair fibers:

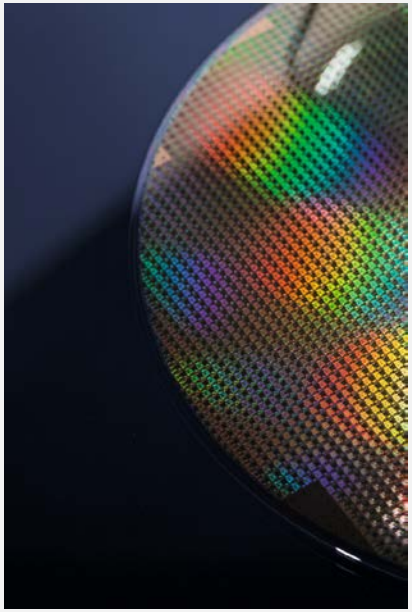
- Real-time studies
- Reversible vs. permanent adsorption

Application of solid surface zeta potential studies: Semiconductor industry

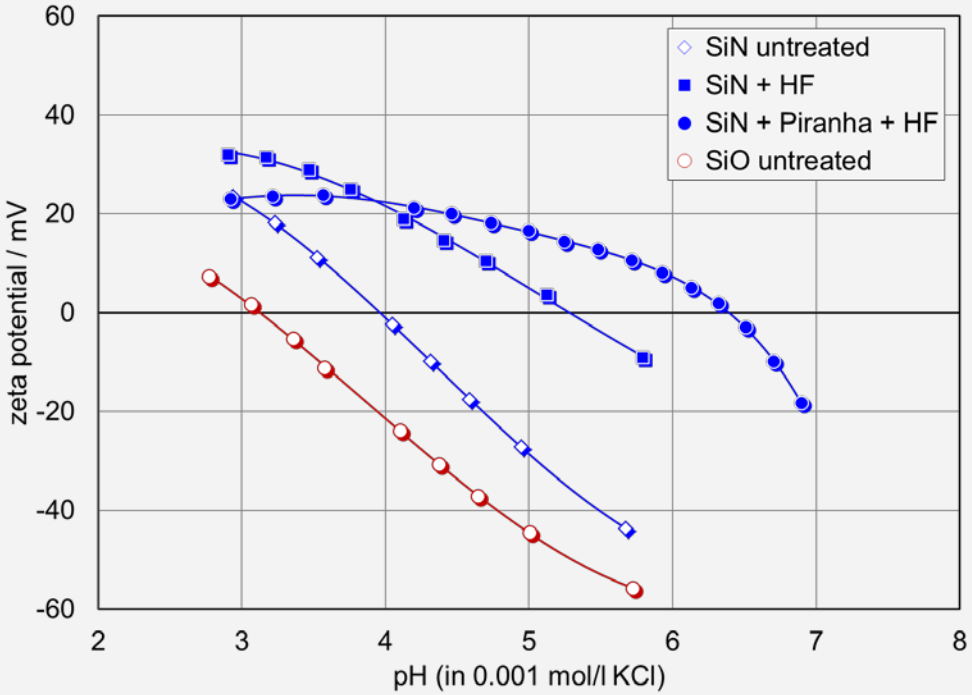
Zeta potential applications in semiconductor wafer processing

- Wafer cleaning efficiency:
  Effect of cleaning | etching strategies for silicon nitride

- Chemical Mechanical Polishing (CMP):
  Effect of wafer-particle interaction
  Tuning the CMP process conditions



Wafer cleaning efficiency



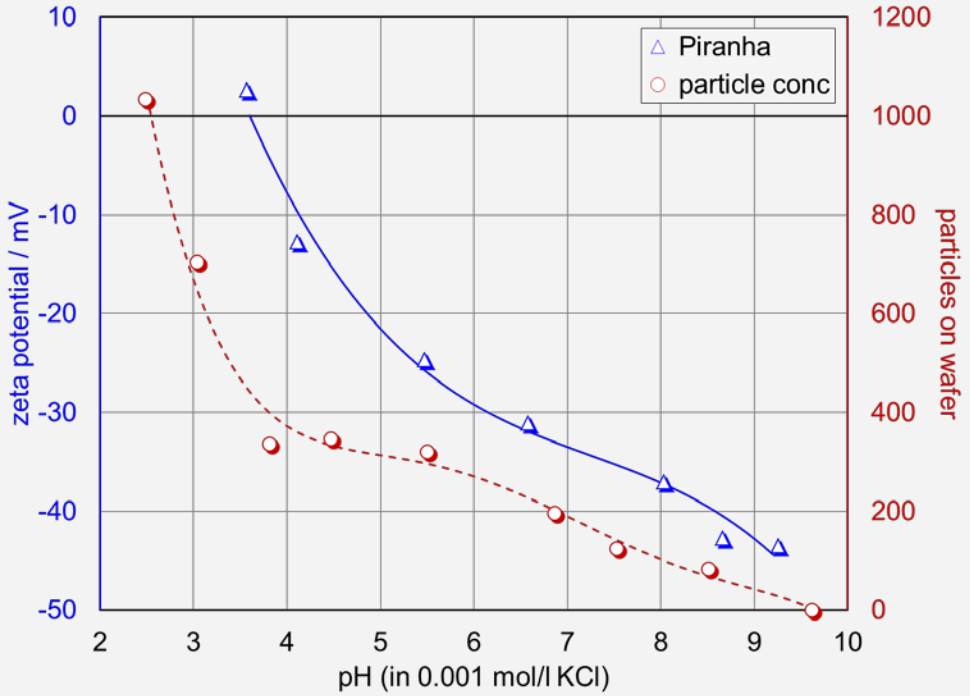
SiN ..... silicon nitride wafer

SiO ..... silicon oxide wafer

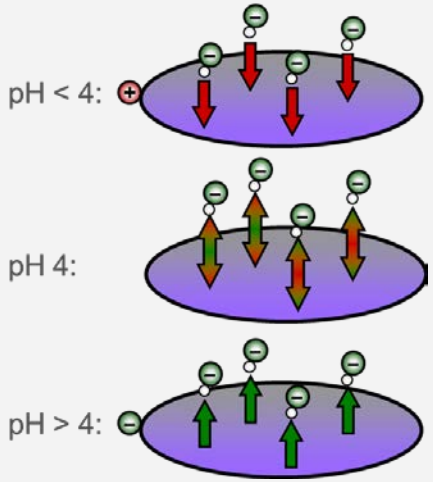
Piranha: H_2SO_4:H_2O_2 = 3:1, oxidative removal of organic contaminant

HF: removal of silicon oxide

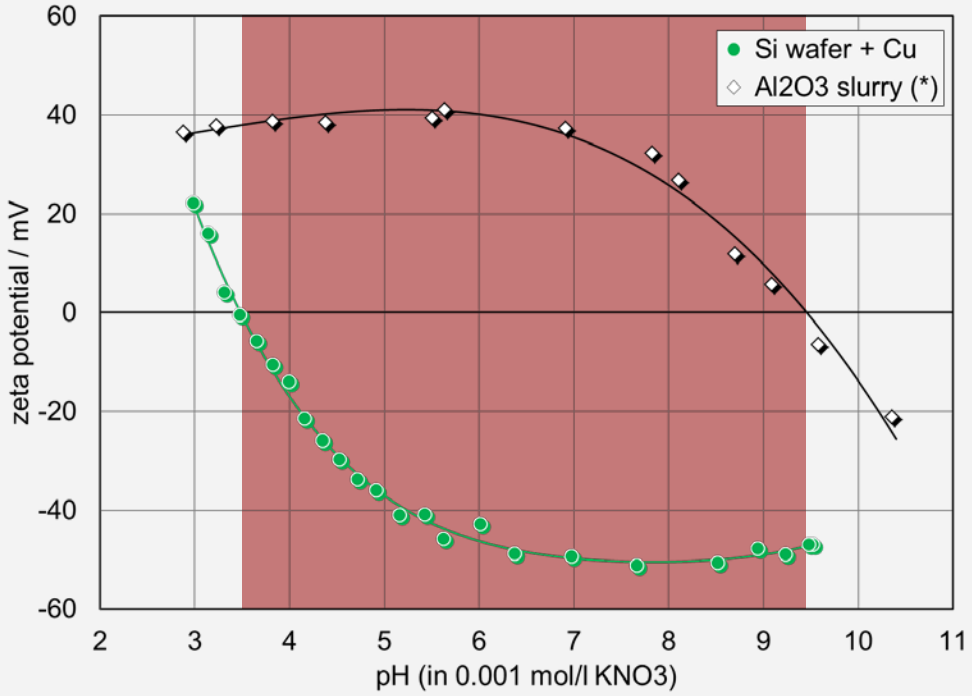
Particle deposition on Si_3N_4 wafer



Contamination of wafer surface with poly(styrene) particles (IEP < 3)



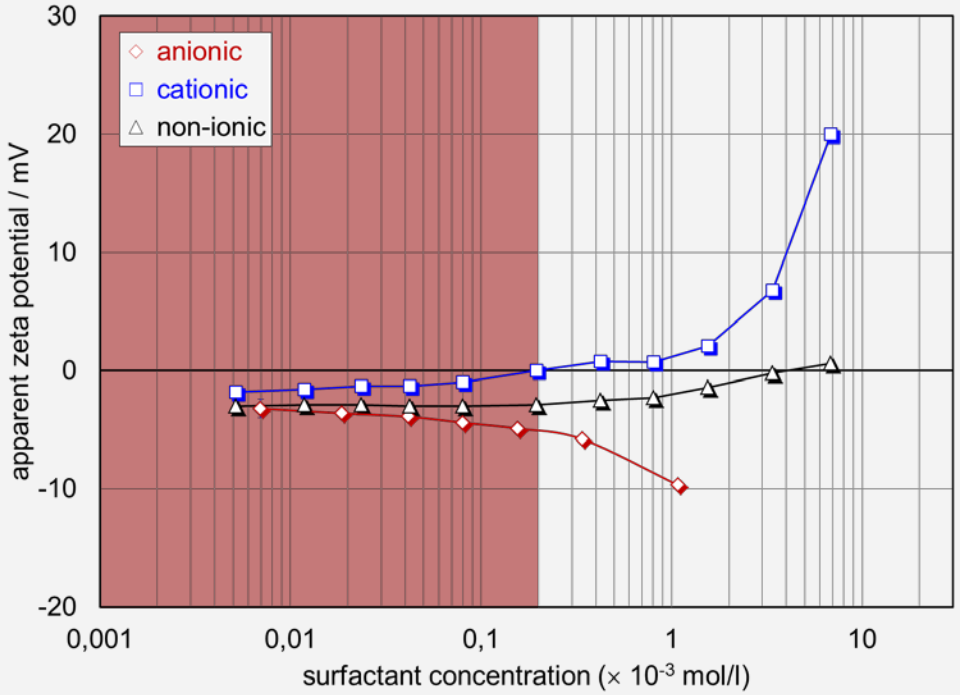
Chemical Mechanical Polishing



- CMP process uses abrasive particles (e.g., Al_2O_3 or SiO_2 slurries)
- After CMP, particles must not adhere to the wafer surface
- Electrostatic repulsion requires equally charged wafer and particle surfaces

(* measured by ELS)

Chemical Mechanical Polishing



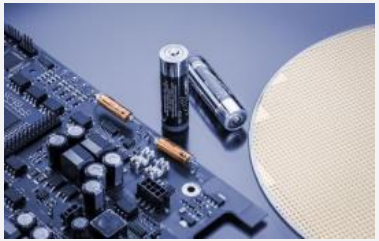
– Addition of cationic surfactant to CMP slurry changes sign of wafer surface charge

anionic: sodium dodecyl sulphate
cationic: dodecyl trimethyl ammonium bromide
non-ionic: polyethylene glycol ether
Si wafer + Cu, pH 5.5

Zeta potential – key applications

Membranes
Wastewater

Pharma & Life Science
Biomaterials, Medical Devices, Cosmetics



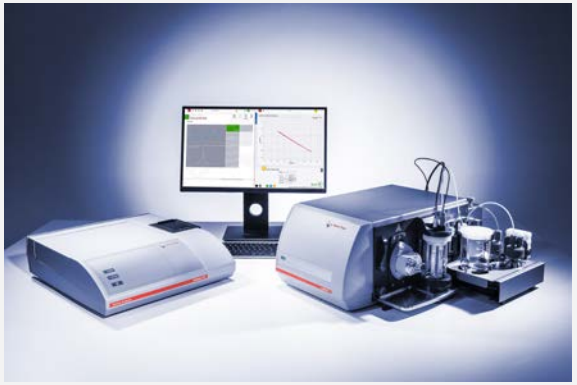
Electronics
Batteries, Semiconductor

Petroleum Industry
Rocks, Oil

Chemicals
Polymers & Plastic, Latex, Paints & Inks

Food & Beverage
Filtration, Packaging

Thank you!



Interested in learning more about zeta potential?

Join our webinars on

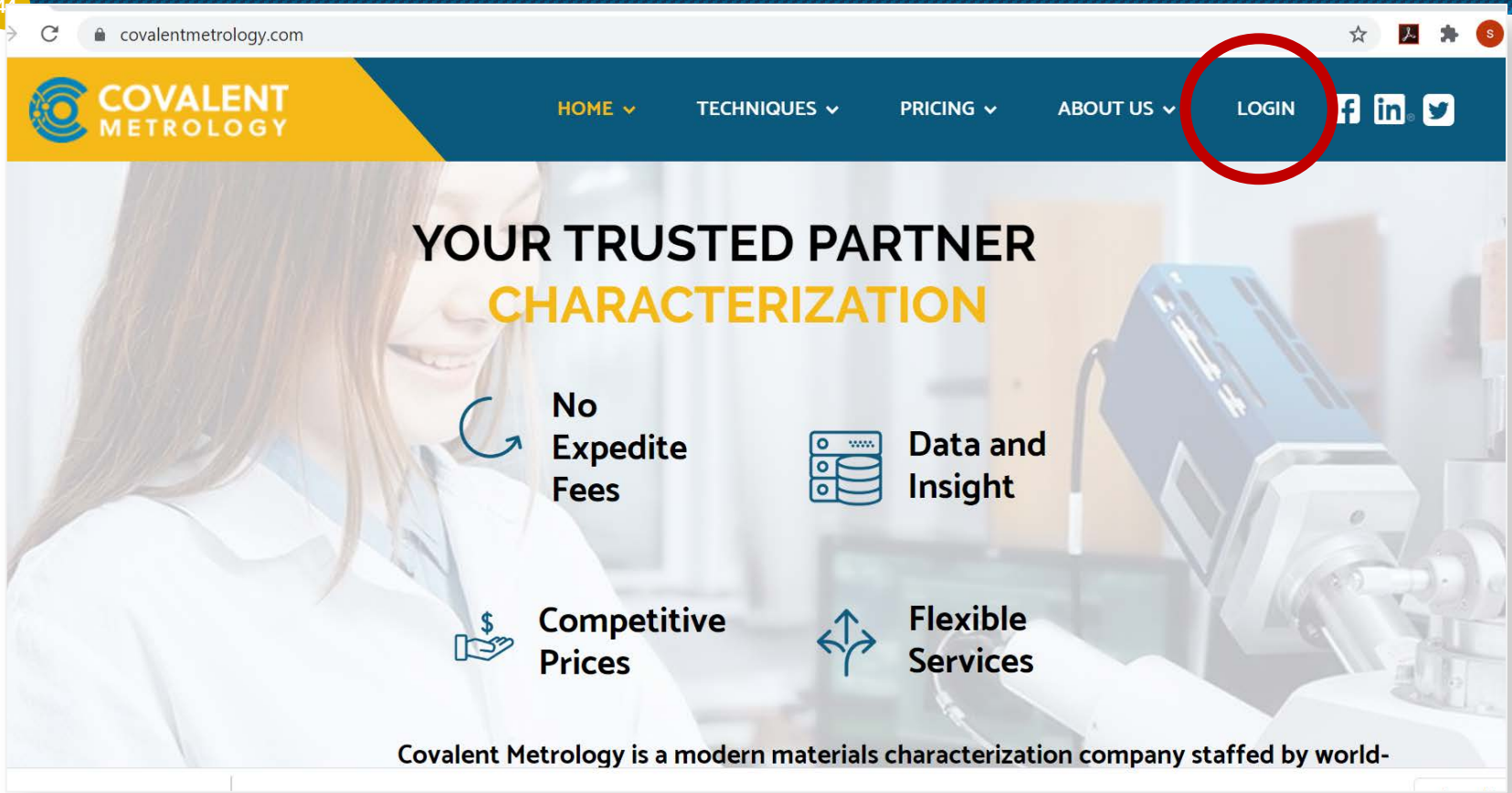
- food and food packaging – July 1, 8 am PDT
- ceramics – July 8, 8 am PDT
- semiconductor applications – July 15, 8 am PDT
- membrane technology – July 22, 8 am PDT

Next Episode

Please join us at the next
Covalent Academy

July 9, 2020
11am PDT

Covalent Community



Covalent Community

Data Portal

Customer Access to Data & Community Content

The DATA PORTAL is used by Customers and Lab Partners for uploading and downloading data. It requires two-factor authentication and advanced password protection. Data Portal users have complete access through their home page on the portal to all Community content, and do not require a separate Community account.

Covalent Community

All Other Users

The COVALENT COMMUNITY PORTAL requires password entry. It contains webinar and other metrology and characterization-related content that we believe would be useful and educational for the materials science innovation community. It does not provide access to any customer data and should only be used by individuals that are not Covalent customers or lab partners.

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Thank you

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