



**Anton Paar**

# IMPROVE YOUR CHROMATOGRAPHY WITH REFRACTIVE INDEX

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27.08.2020



# 3400+ EMPLOYEES

72 % MEN, 28 % WOMEN, INCLUDING 2.5 % APPRENTICES



Established in  
**1922**



Headquarters  
in **Graz / Austria**



Owned by the charitable  
**Santner Foundation**



**95 %**  
export rate



**20 % investment**  
in research & development



**All critical components**  
manufactured in-house

**8**

Producing  
subsidiaries

**31**

Sales  
subsidiaries

**60**

Distribution  
partners

**170**

Measurement  
solutions

# Agenda

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- GPC calibration process & the role played by  $dn/dc$  values
- How a refractometer can improve the GPC calibration process
- Requirements and the specifications your refractometer must meet

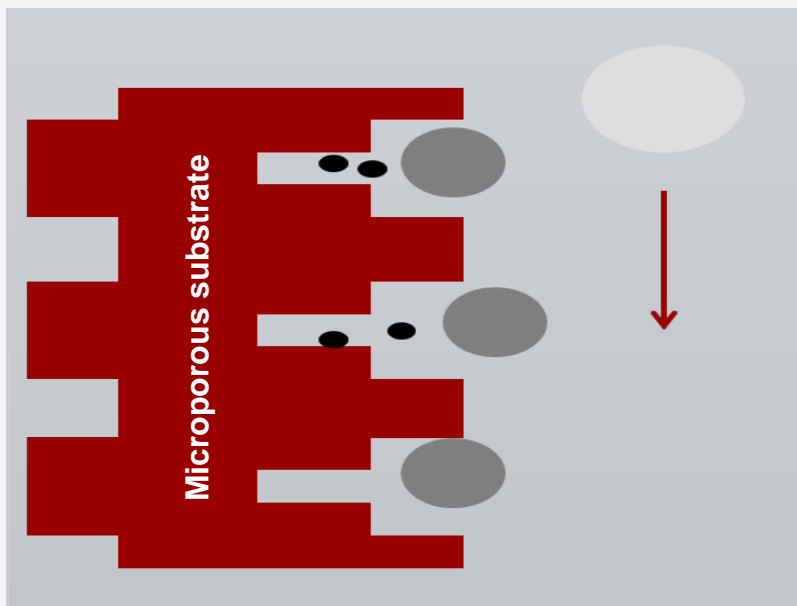
# GEL PERMEATION CHROMATOGRAPHY CALIBRATION PROCESS

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# GPC calibration process & the role played by $dn/dc$ values

## Introduction and principle

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- Liquid chromatography technique
- Polymer or molecule size distribution
- Dispersity and molecular weight
- Separation based on hydrodynamic volumes
- Often used for polymer solutions and also in food or pharmaceutical applications

# GPC calibration process & the role played by $dn/dc$ values

Instrumentation, material & method

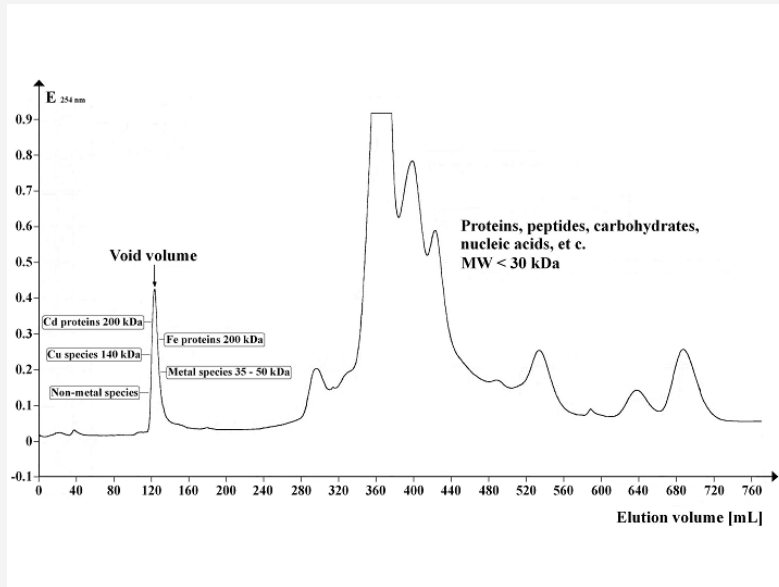
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- Instrumentation similar to liquid chromatography
- **Gel:** stationary phase e.g. agarose gel
- **Column:** Filled with microporous packaging
- **Eluent:** mobile phase (solvent for polymers) e.g. tetrahydrofuran (THF)
- **Detector:** UV; refractive index or differential refractometer

# Gel permeation chromatography calibration process

Instrumentation, material & method



- Refractive index detector → detection of concentration differences  

$$RI_{\text{Output}} = K_{RI} * \frac{dn}{dc} * \text{concentration} * \text{inj. Volume}$$

- Light scattering → detection of molecular weight  

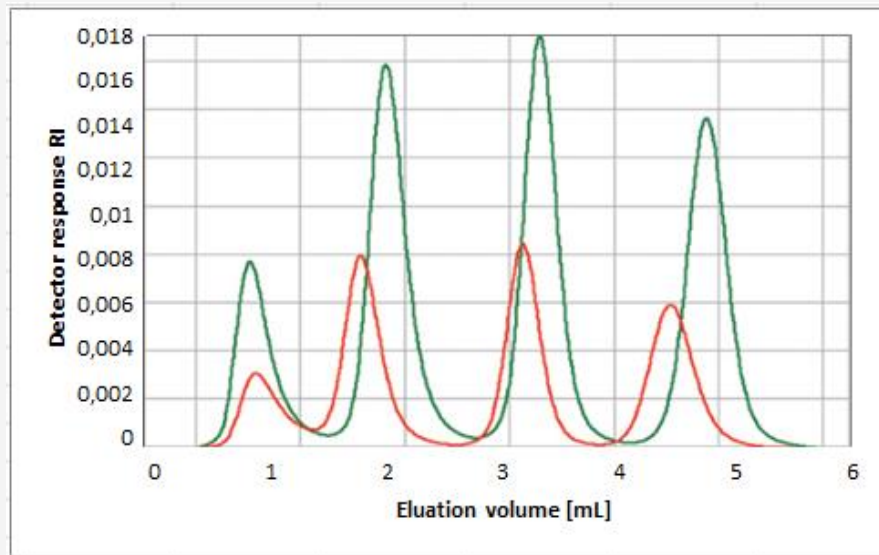
$$LS_{\text{Output}} = K_{LS} * Mw * \left(\frac{dn}{dc}\right)^2 * \text{concentration} * \text{inj. Volume}$$

$dn/dc$  – specific refractive index increment – is the important parameter



# GPC calibration process & the role played by $dn/dc$ values

## Calibration process



- Use of different polymer standards
- Typical standard is polystyrene or PMMA in THF
- Retention period and molecular size is used for calibration curve
- Use of literature data for  $dn/dc$ , online or benchtop refractometer

# GPC calibration process & the role played by dn/dc values

What's in for you?



- **Save 20% of your time** for a second analysis round due to wrong calibration
  - **Avoid complaints** due to faulty results
  - **Prove your calibration process** by testing all your standards with RI
- **Good measurement starts with the calibration**

# HOW A REFRACTOMETER CAN IMPROVE THE GPC CALIBRATION PROCESS

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## Refractive index

The refractive index is expressed as the ratio of the speed of light in the vacuum ( $v_{\text{Vacuum}}$ ) relative to the speed of light in the medium ( $v_{\text{Medium}}$ )

$$n_{\text{Substance}} = \frac{v_{\text{Vacuum}}}{v_{\text{Medium}}}$$

### Standard conditions:

- 20 °C or 25 °C
- 1013 mbar
- 50 % relative humidity
- 589 nm, sodium D- line

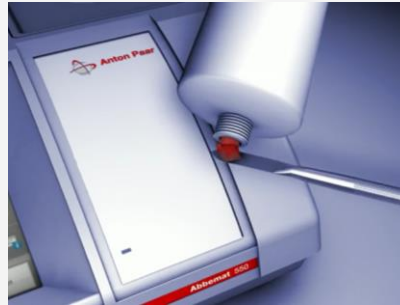


# Refractive Index Measurement

## Which substances can be measured?

- Liquids
- Pastes
- Soft Matter / Polymers
- Solids
- Turbid, colored and non-transparent samples

## Powders can not be measured

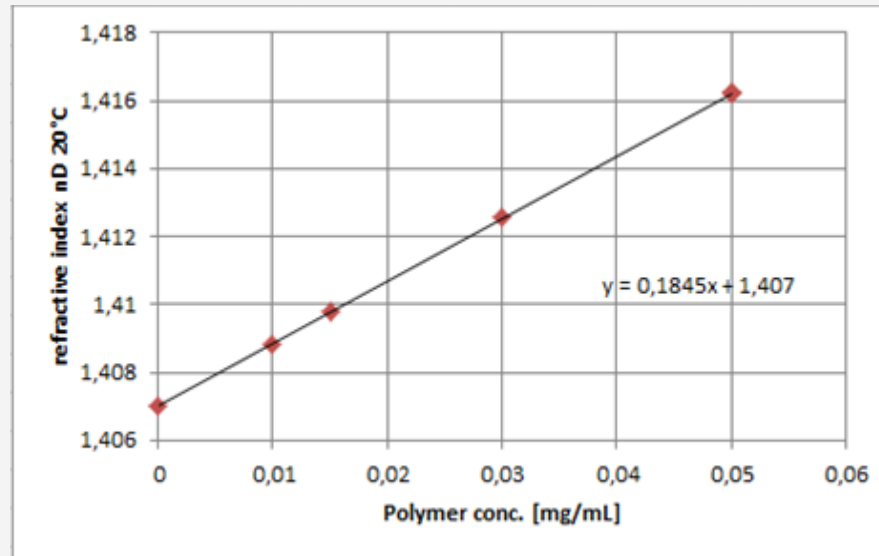


# How a refractometer can improve the GPC calibration process

Prove your calibration

- Prove  $dn/dc$  of your polymer solutions e.g. PS in THF
- Apply your standard solutions to the refractometer
- Calculate the linear function and get the  $dn/dc$  value

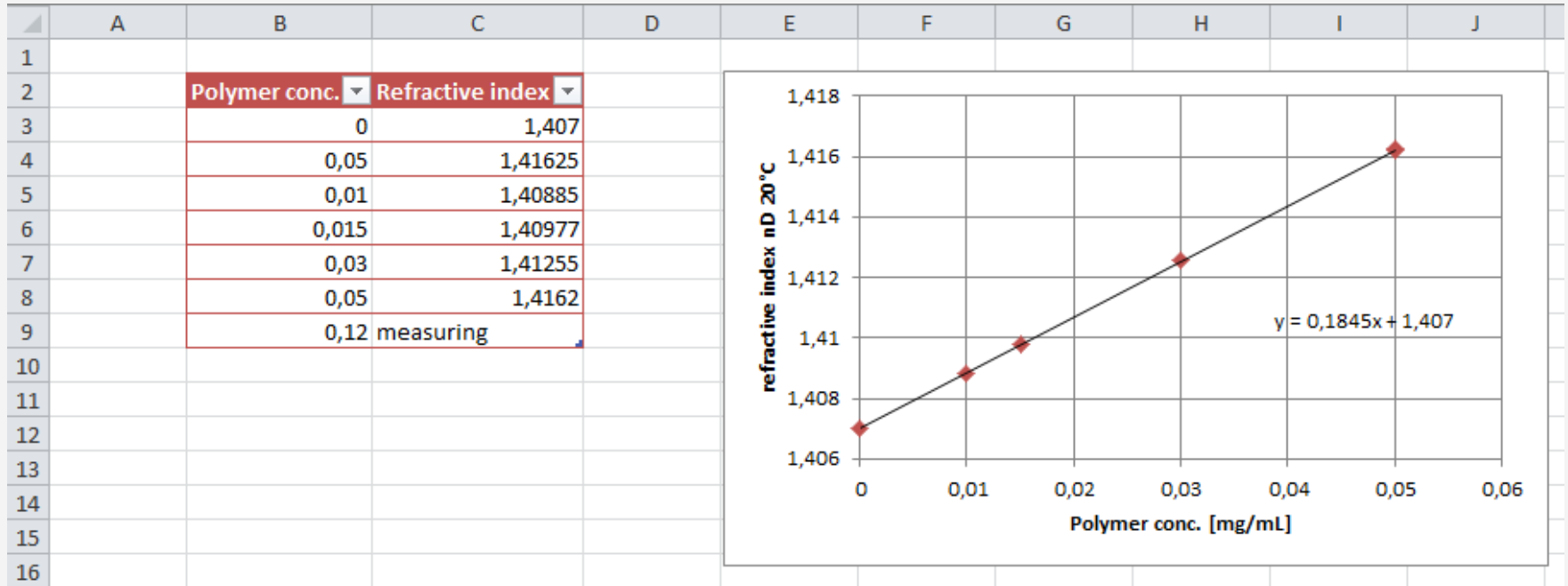
→ **high accuracy necessary**



Correlation of the refractive index (20 °C, 633.1 nm) with the polymer solutions and the corresponding polynomial fit.

# Waiting for your sample

In-built application know-how



# Live Demo of Abbemat



# Influence of Wavelength on dn/dc

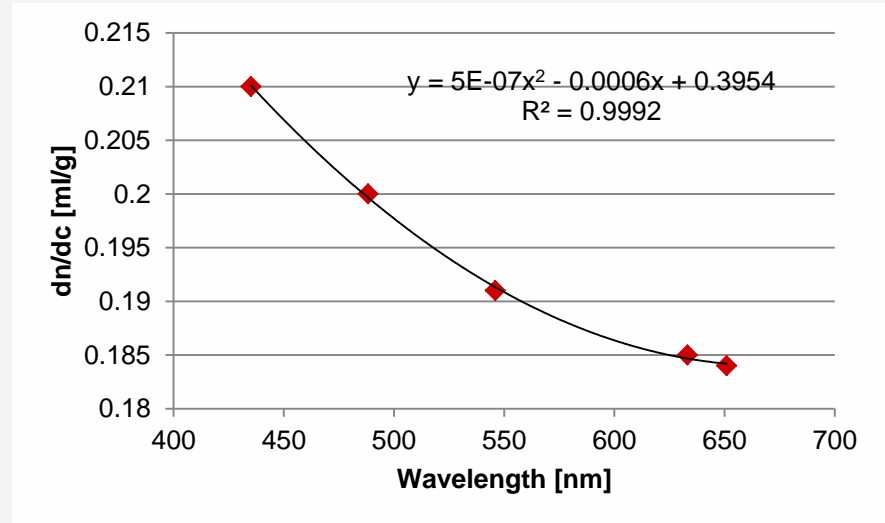
## Dispersion

Phenomenon: Different refraction of the spectral components of a light depending on wavelength

## Abbe number

- Measure of the material's dispersion characteristic
- Used as a specification for polymers and other transparent substances
- Measuring the refractive index value at a variation of wavelengths

→ Influence on dn/dc is not negligibly (up to 10% between 430nm and 650nm)



Influence the wavelength on dn/dc (20 °C) of PS in THF

# Refractive Index Applications for Polymes

## Applications & Solution

### Refractive index as a quality parameter for polymer films

- Substance (material constant)
- Optical density (polymerization degree, chain length, etc.)

### Optical matching with substrate

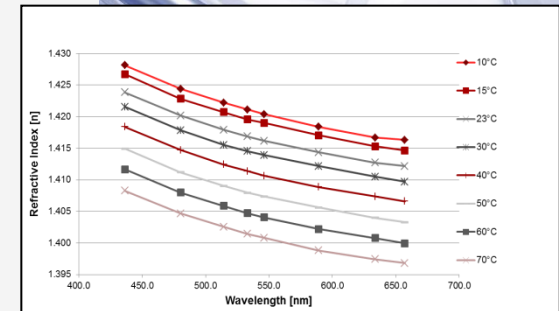
- Should be similar to avoid reflections and visibility of layers

### Dispersion determination by multi-wavelength

- Avoid chromatic abbreviation (e.g. for screens)

### Refractive index for particle size analysis by DLS

- Refractive index of particles is necessary for precise results



# REQUIREMENTS AND THE SPECIFICATIONS YOUR REFRACTOMETER MUST MEET

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# Requirements and the specifications your refractometer must meet

## Specifications

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### Refractive index

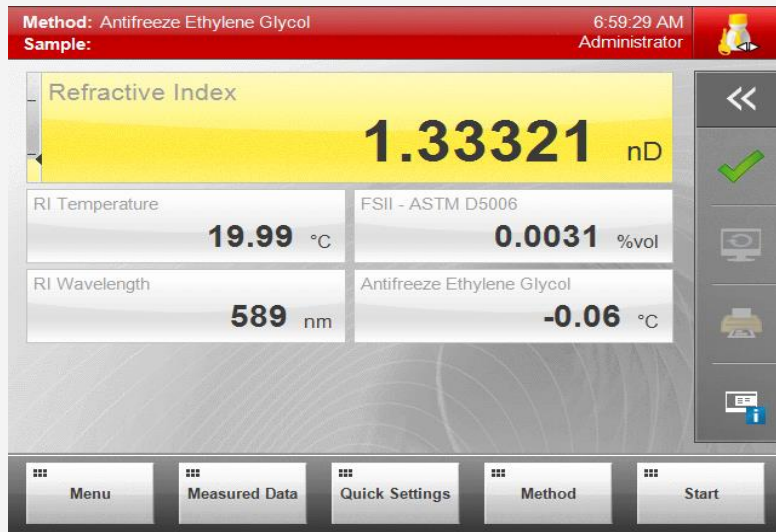
- Accuracy: 0.00002nD
- Range: 1.26 – 1.60nD

### Temperature

- Accuracy 0.05°C
- Range: 20°C or 25°C

# Readings at a glance

## Visual QC-Mode



- Immediate visual pass- or fail feedback
- Acceptance range for sample defined in methods
- Objective pass- / fail judgment independent of user
- Avoidance of human errors

## Reliable results within seconds

1, 2, 3, Result!



- Generally no sample prep required
- Fast measuring times (based on powerful Peltier temp. control)
- Stable readings within 4 sec<sup>1)</sup> (with time-saving temperature-corrected measurements)
- Clean with one swipe (due to small surface area of sample well)

1) Tested on sucrose solutions

# Calibration Safe & Easy

## Guided checks & adjustments



- Guided checks & adjustments minimizing trainings and errors
- Checks compliant with GxP requirements
- Recorded adjustment history for full traceability
- Reference standards traceable to PTB<sup>2)</sup> or GUM<sup>3)</sup>

2) Physikalisch-Technische Bundesanstalt (Germany's Metrological Inst.)

3) Central Office of Measures (Poland's Metrological Inst.)

# Portfolio Refractometers



Heavy Duty

Abbemat 450



Abbemat 650



Abbemat MW



Advanced  
Performance  
Plus

Abbemat 350



Abbemat 550

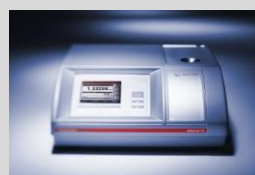


Entry Level  
3X00 and  
Performance

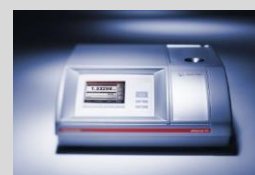
Abbemat 3X00



Abbemat 300



Abbemat 500





# Further Refractive Index Applications for Polymes

## Refractive index as a quality parameter for polymer films

- Substance (material constant)
- Optical density (polymerization degree, chain length, etc.)

## Optical matching with substrate

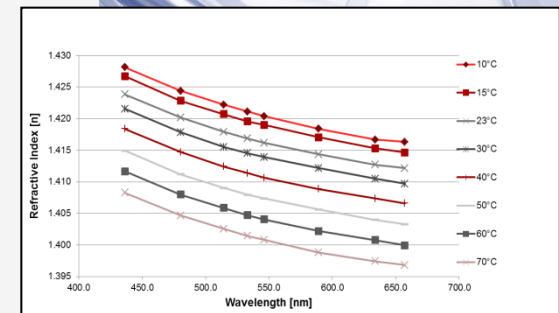
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## Summary

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### **Gel permeation chromatography calibration process**

- Standards with different polymer sizes are used e.g. polystyrene in THF
- Save 20% of your time, avoid faulty results and prove your calibration

### **How a refractometer can improve the GPC calibration process**

- Prove your  $dn/dc$  results and improve your calibration

### **Requirements and the specifications your refractometer must meet**

- 1.26 – 1.60 refractive index range, with 0.00002 accuracy
- In-built application know-how, visual QC mode and guided checks

# THANK YOU FOR YOUR ATTENTION!

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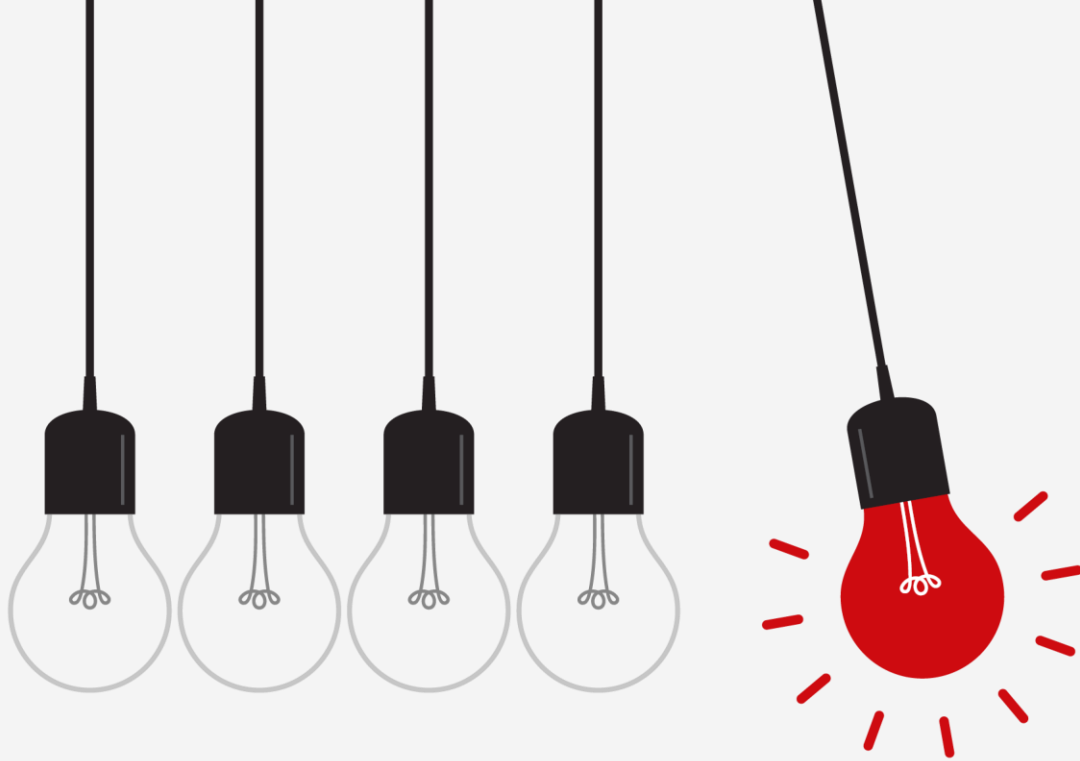
 Upcoming

**Weak acids, strong bases - Replace  
Titration with Refractive Index  
Measurements within Seconds**

**2020-10-08**

09:00 - 09:30 (CET UTC+02)

[Read more »](#)



# Your questions.

[www.anton-paar.com](http://www.anton-paar.com)