



Anton Paar

CONSIDERATIONS FOR HIGH RESOLUTION AND SENSITIVITY XRD MEASUREMENTS

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Topics

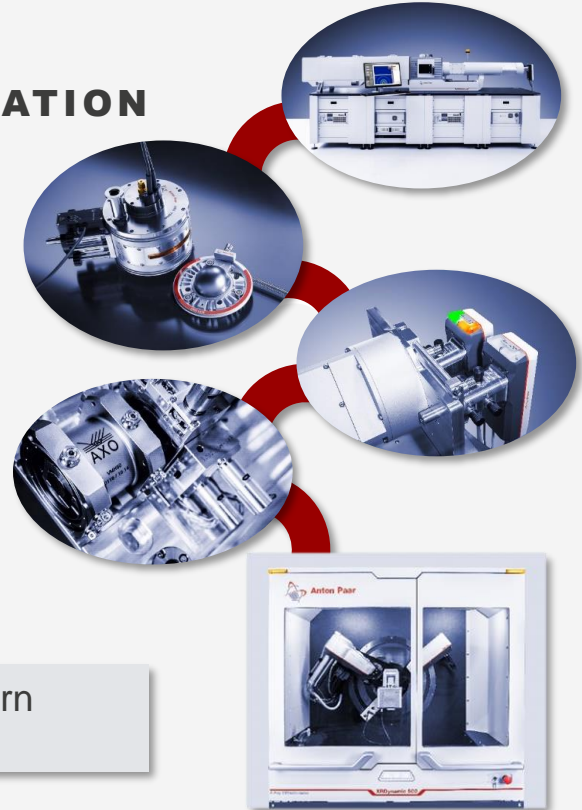
- X-ray to understand the diffractogram ($K\alpha_1$, $K\alpha_2$, $K\beta$, Bremsstrahlung)
- The optics collimating a line to multiple “points”
- Effect of optical components and measurement settings
- Goniometer effects
- Best practices for optics and goniometer
- Examples

X-RAY ANALYSIS AT ANTON PAAR

DECADES OF EXPERIENCE & A SPIRIT OF INNOVATION

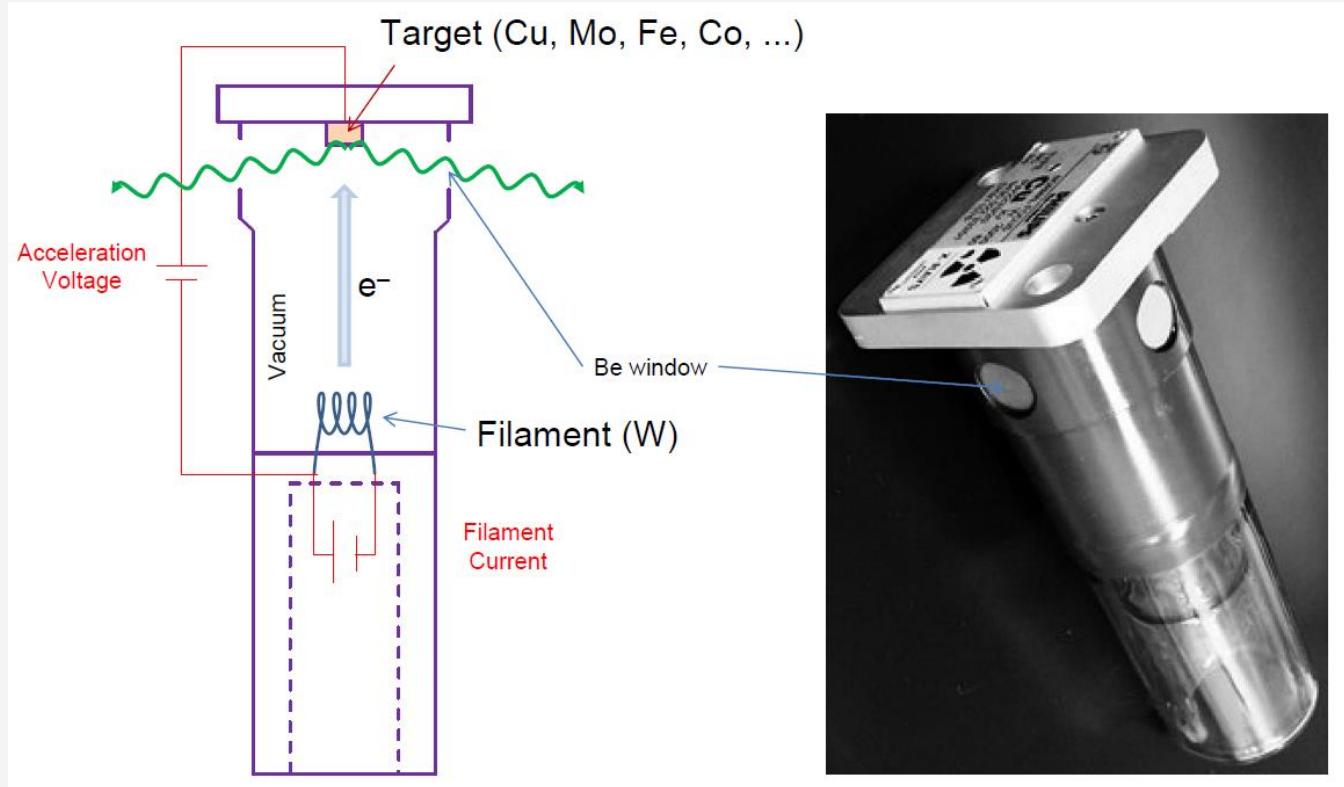
- **SAXS/WAXS/GISAXS:** a pioneer in SAXS (Kratky camera, 1957) and one of the current technology leaders
- **Non-ambient XRD:** the market leader in non-ambient XRD attachments since 1964 with the broadest product portfolio
- **X-ray sources:** independent supplier of high-performance sealed-tube and high-brilliance microfocus X-ray sources
- **X-ray optics:** decades of experience in high-precision multilayer and single crystal X-ray optics from AXO DRESDEN (an Anton Paar company)

XRDynamic 500: the logical next step to successfully deliver a modern automated powder X-ray diffractometer



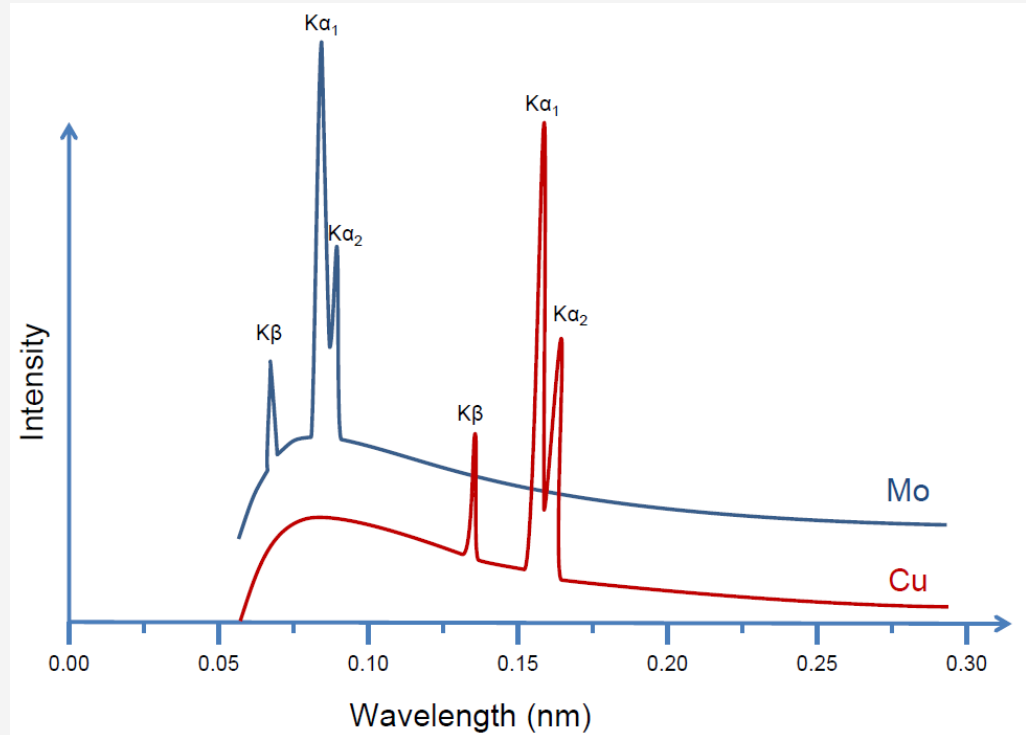
X-RAY EMISSION

- Sealed tube
- Line or point
- Wavelength



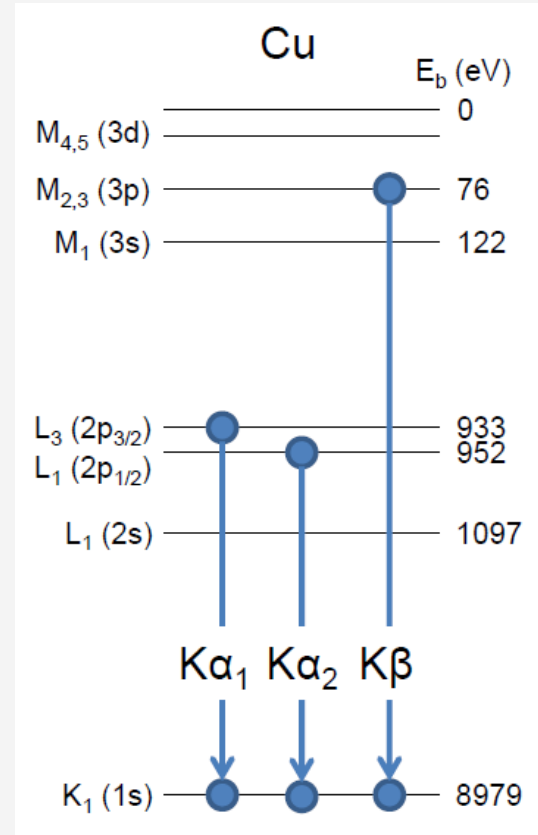
BREAKING RADIATION

- Bremsstrahlung
- Electron transition



MONOCHROMATIC?

- Easier analysis
- Less peak (potential overlap)



PERIODIC TABLE

> Fluorescence & filters

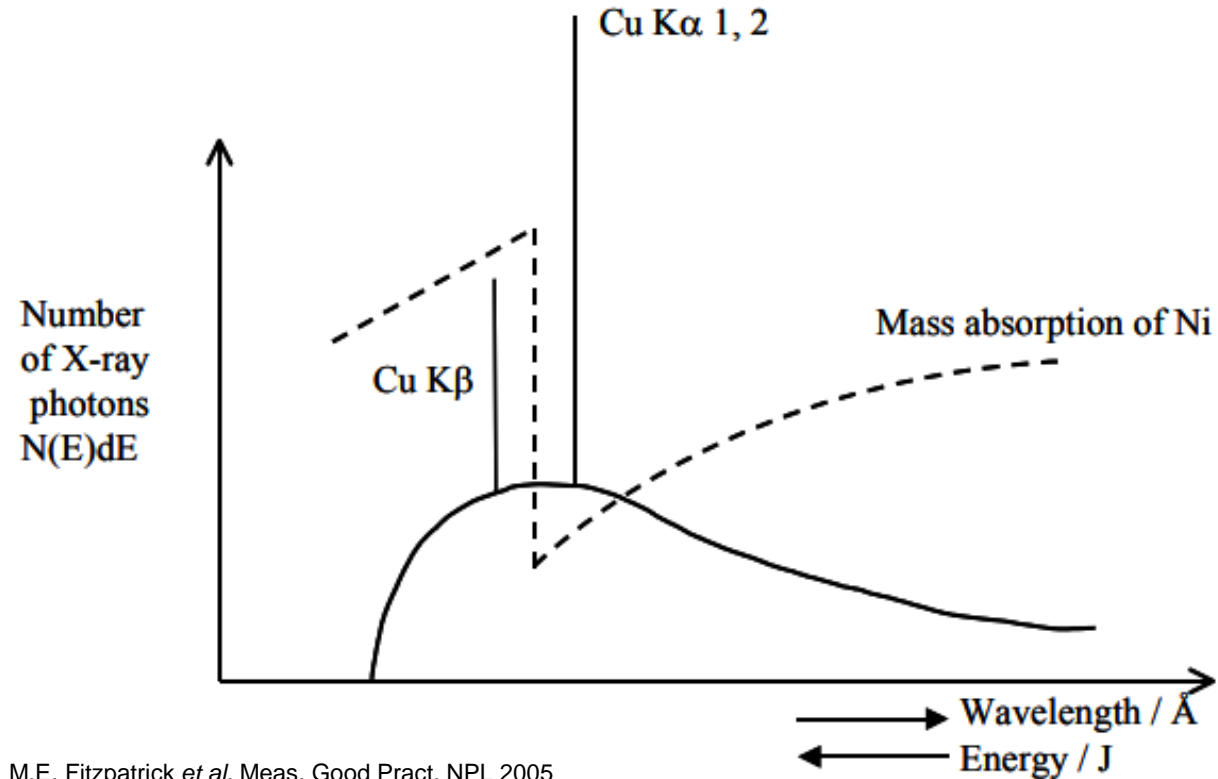
> Scattering signal

Group Period	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	1 H																	2 He
2	3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
3	11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
4	19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
5	37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
6	55 Cs	56 Ba	* 71 Lu	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
7	87 Fr	88 Ra	* 103 Lr	104 Rf	105 Db	106 Sg	107 Bh	108 Hs	109 Mt	110 Ds	111 Rg	112 Cn	113 Nh	114 Fl	115 Mc	116 Lv	117 Ts	118 Og
			* 57 La	58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb		
			* 89 Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No		

PERIODIC TABLE

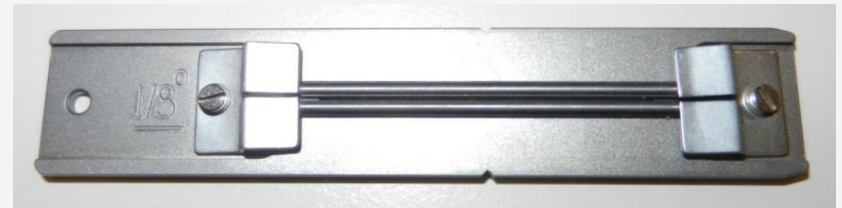
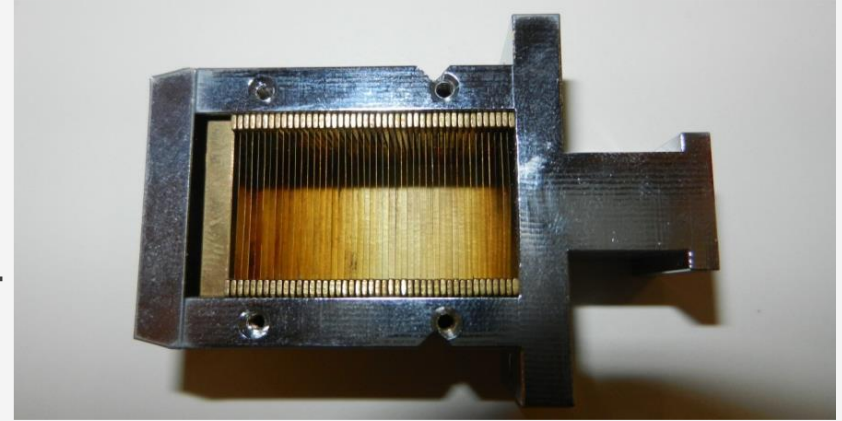
> Fluorescence & filters

> Scattering signal



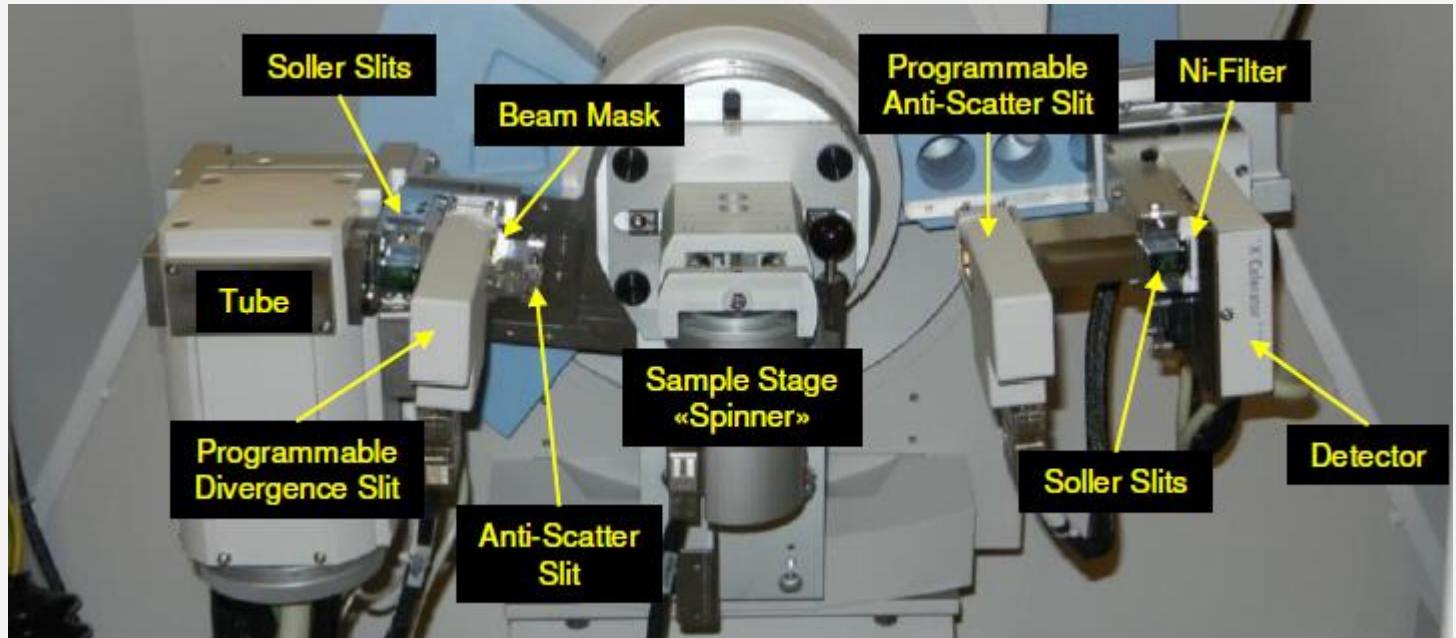
CONSIDERATIONS

- Point source
- $K\alpha_1$ only
- -----synchrotron-----or else-----
- Line source
 - Soller slits
 - Beam Mask
- Divergence
 - Divergence slit
- Chromaticity
 - Filters



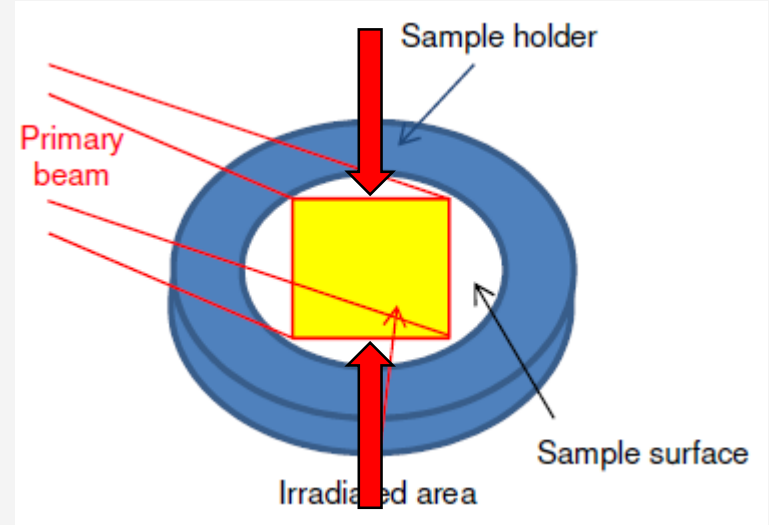
OPTICS

➤ It's not that simple!



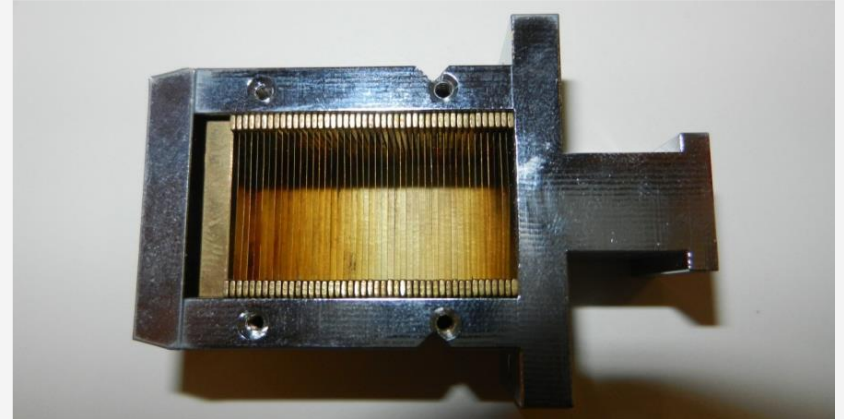
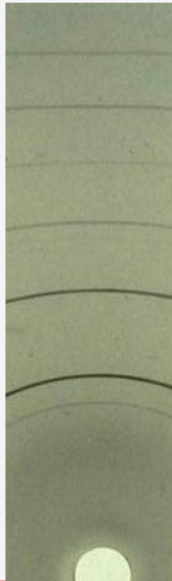
MASK

- › Width of the beam
- › Discrete values for disks
- › What about bulk surfaces?



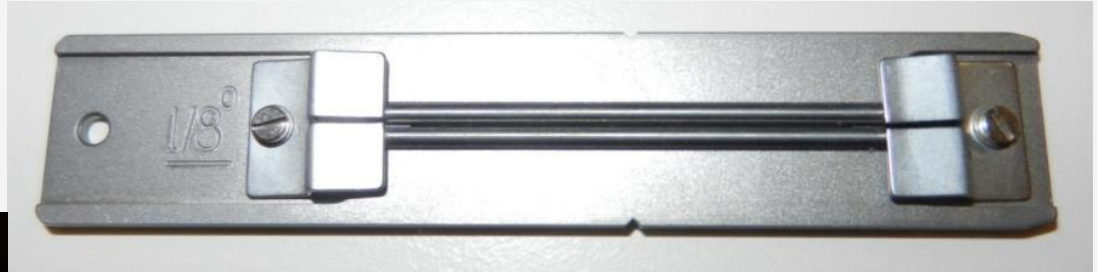
SOLLER

- Making a line into many "points"
- Lower angles more affected



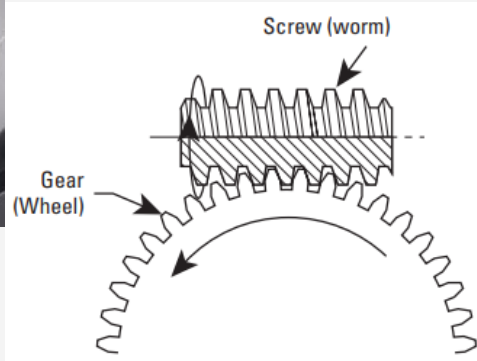
DIVERGENCE / AS

➤ Discrete values



GONIOMETER

➤ Conventional Goniometers (worm screw)



HIGH RESOLUTION **OR** HIGH SIGNAL-TO-NOISE

Large radius gives high resolution

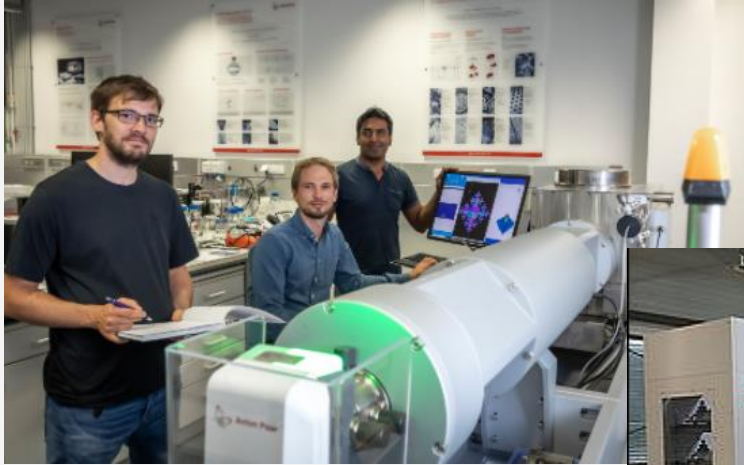
Large radius gives noise from air scattering

Small radius has better Signal-to-Noise

Small radius has poor resolution

Let's design a new, revolutionary XRD

Anton Paar's first XRD



CLEAN SLATE

➤ Let's re-invent XRD and start with the goniometer:

Its product was invented in 1957, but still sells for hundreds or thousands of dollars. The product has been manufactured in Massachusetts consistently since around 1960. And it currently sits on the surface of Mars, on the moon, in the International Space Station, and in operating rooms and factories here on earth.



Mars; Curiosity Rover

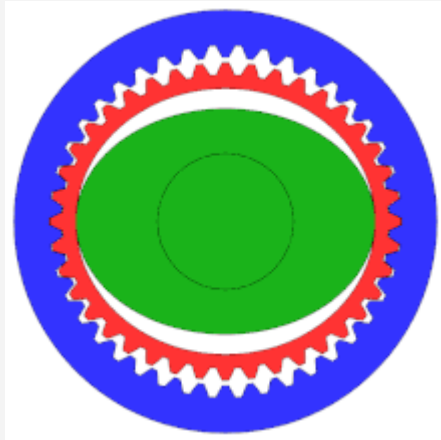
Moon Rover; Apollo 15,16 & 17

XRDynamic 500 goniometer Anton Paar

A next-generation goniometer

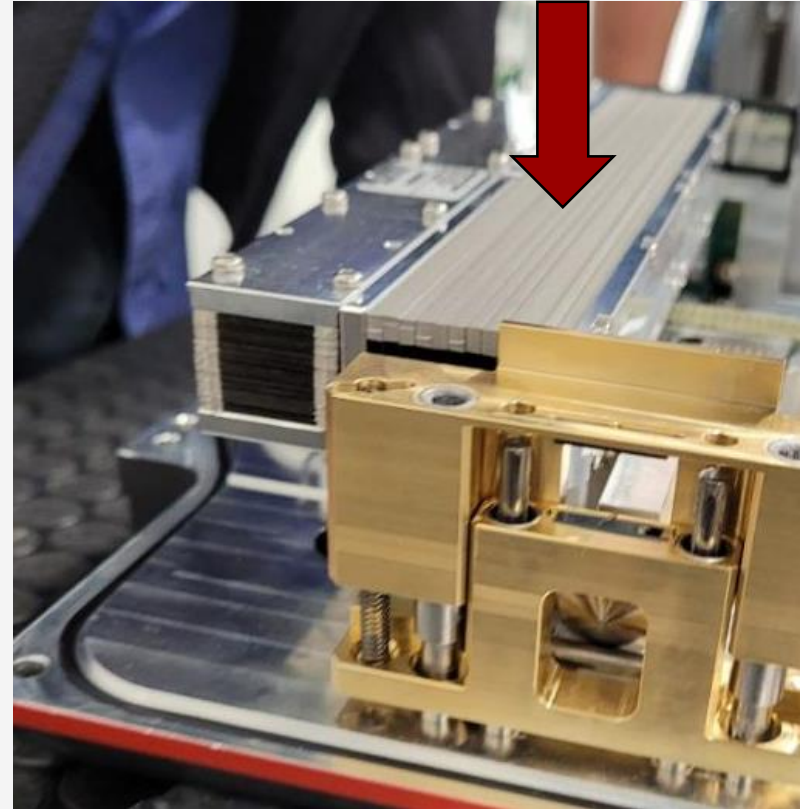
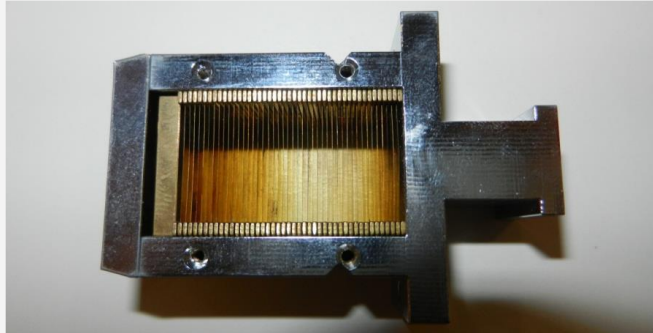
Advantages:

- High precision and linearity
- No backlash / hysteresis
- No counterweights
- Robust and maintenance-free



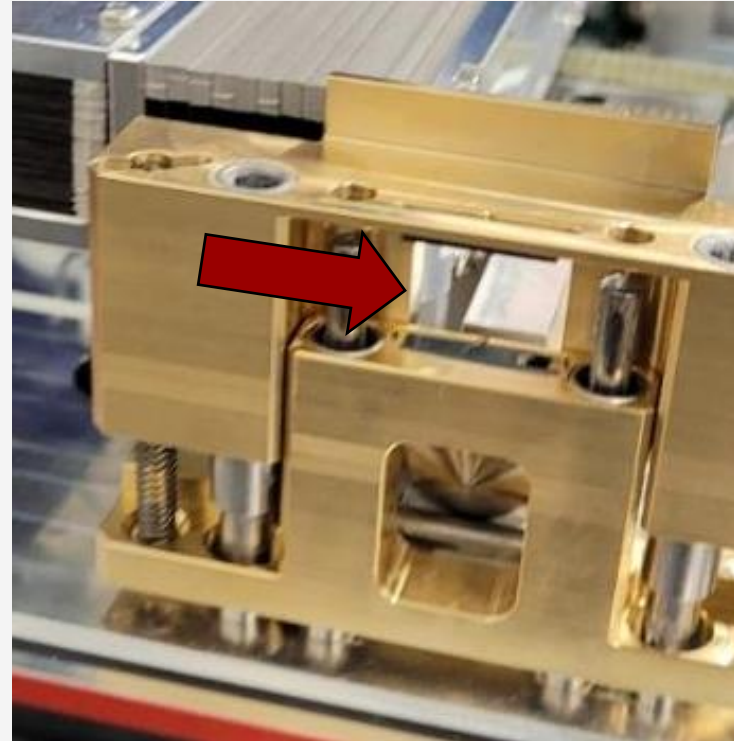
SOLLER

- Longer, less "fins"
- Same or better divergence



DIVERGENCE / AS

➤ Discrete vs continuous



Radius of 400 mm or 360 mm

Largest in class for high resolution

Cu photons remaining after 800 mm : 38%

Cr photons remaining after 800 mm : 4.6%

Conventional solution? Rotating anode! MetalJet!

HIGH RESOLUTION **AND** HIGH SIGNAL-TO-NOISE



First XRD instrument with optics under vacuum

(Patent number : [WO2021078424A1](#) X-RAY DEVICE HAVING MULTIPLE BEAM PATHS)

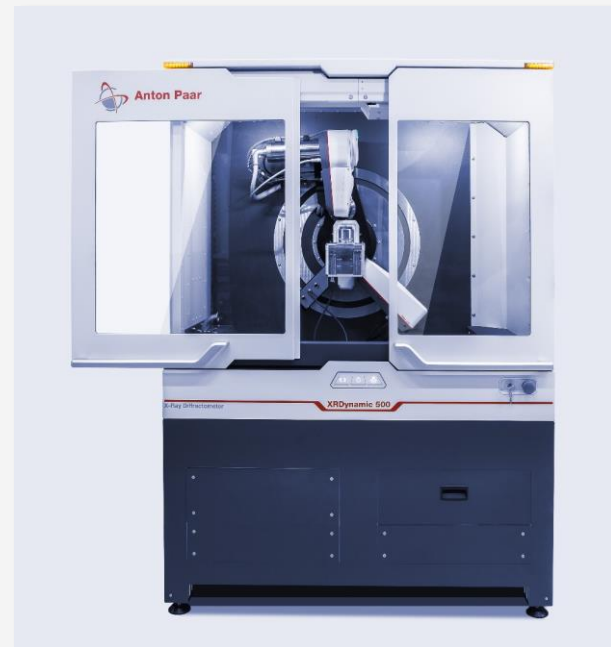
Large radius gives high resolution.

No air scattering leaves more photons – higher intensity peaks.

No air scattering reduces baseline noise – better S/N ratio.

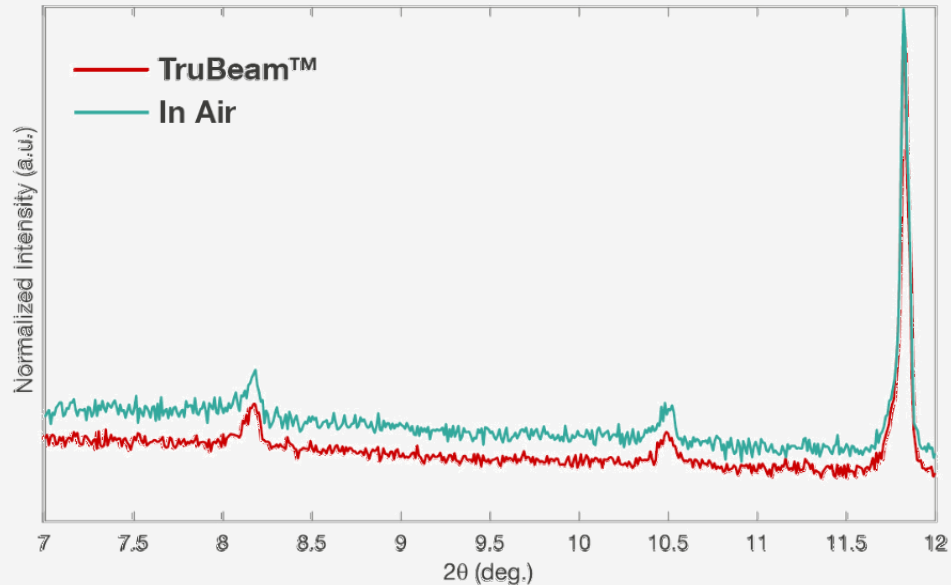
A new class of goniometer

- Strain wave gearing technology – new in XRD
- Guaranteed $\Delta 2\theta < 0.01^\circ$
- Minimum step size = 0.0001°
- **Advantages:**
 - No backlash / hysteresis
 - No counterweights
 - Robust and maintenance-free



TruBeam™ - Evacuated optics units

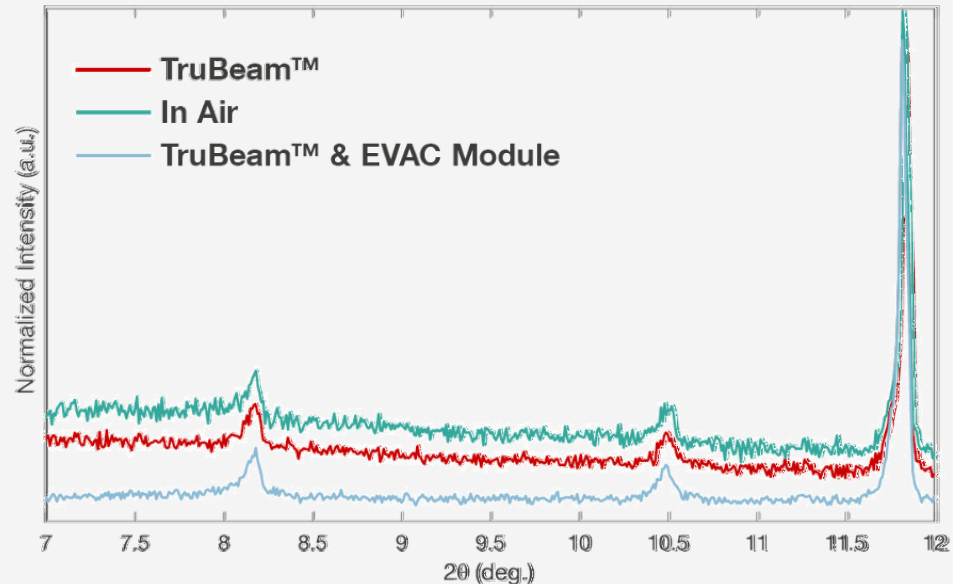
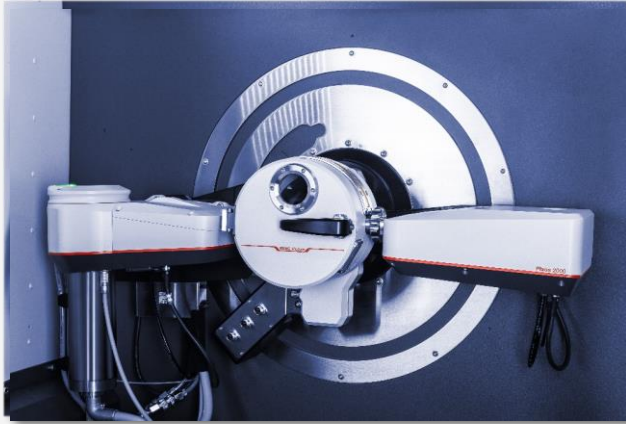
- Evacuated beam path provides outstanding signal-to-noise ratio
- **Sample:** α -Lactose



TruBeam™ - Evacuated optics units

Patent pending on the EVAC module

- Evacuated beam path provides outstanding signal-to-noise ratio
- **Sample:** α -Lactose



TruBeam™ - The core of XRDynamic 500

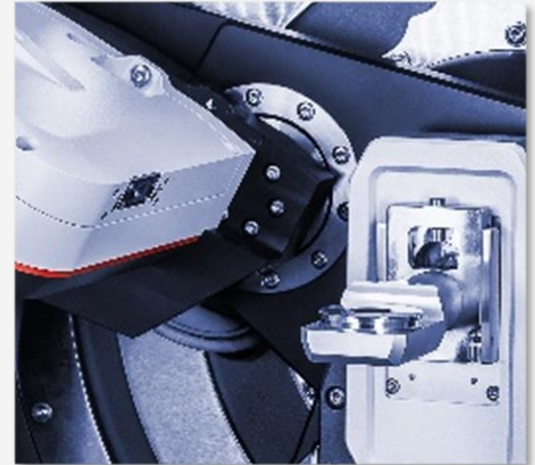
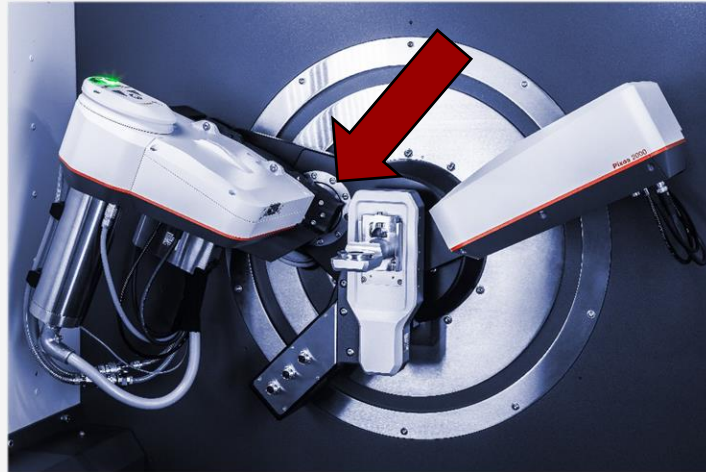
- Change the instrument configuration with a click
- Measure with completely different instrument configurations in one batch
- All components automatically recognized
- **The evacuated optics units of XRDynamic 500 contain:**
 - Optics stack to switch between up to three beam geometries
 - Divergence & anti-scatter slits
 - Soller slits
 - Parallel plate collimator
 - Beam mask
 - Absorbers and $K\beta$ filters



AUTOMATIC ALIGNMENT

➤ First XRD instrument with optics under vacuum and automatic alignment

(Patent number : [WO2021078424A1](#) X-RAY DEVICE HAVING MULTIPLE BEAM PATHS)



Primux 3000: Ceramic sealed-tube X-ray source

The ideal source for XRD/SAXS

- Brilliant line / point focus source with a variety of anode materials available
- Automatic detection of mounted X-ray tube and focus

Target material	Cu, Co, Cr, Mo, Ag (others available on request)
Focal type	Line or point (focus size depending on tube type)
Max. output power	3 kW
Max. output voltage / current	60 kV direct voltage / 50 mA
Cooling	Water cooling

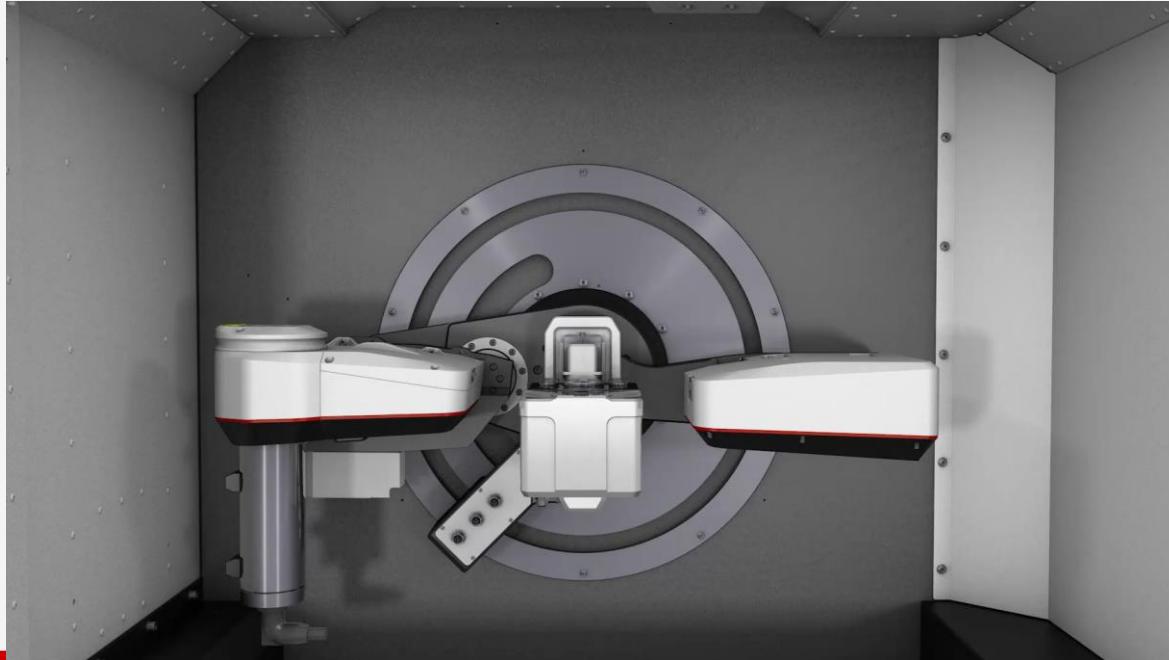


12 minute automatic alignment, easy source exchange

TruBeam™ Concept

The core of XRDynamic 500

TruBeam™ Features



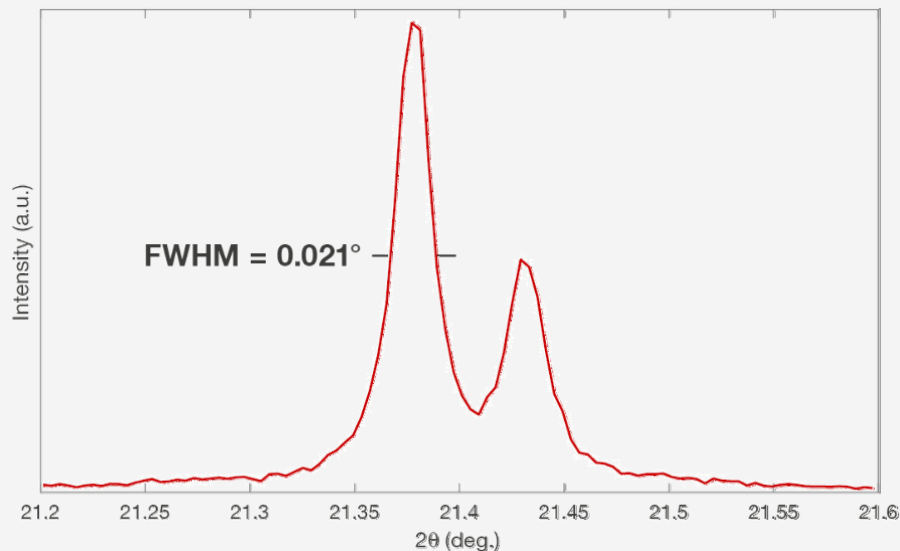
XRDynamic 500 – Driving XRD

- Automated multipurpose powder X-ray diffractometer
- **Applications in:**
 - Powder XRD
 - Grazing incidence diffraction
 - Non-ambient XRD
 - PDF analysis
 - SAXS



TruBeam™ - Unparalleled resolution

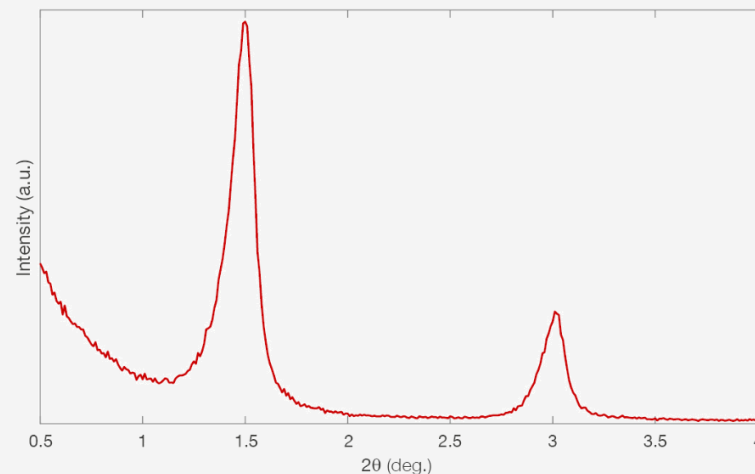
- Large goniometer radius and small detector pixel size combine to give best-in-class resolution
- **Sample:** LaB_6 , **Measurement geometry:** Bragg-Brentano



TruBeam™ Data Quality

Low angle Performance

- Ultra-low background even at low 2θ angles
- Highly symmetric peak profiles in the low angle regime
- No need for absorbers – work with the detector in the direct beam



Sample: Silver behenate

Powerful components

X-ray source

- Primux 3000 ceramic sealed-tube X-ray source
- Cu, Cr, Co, Mo, Ag anodes (others available on request)
- Fast tube exchange or change of focus (line/point)



Optics

- Advanced multilayer optics from AXO DRESDEN fitted in optics stack
- Monochromators, parallel beam and focusing beam mirrors for all wavelengths
- Unique Ni/C multilayers for maximum Cu K β suppression



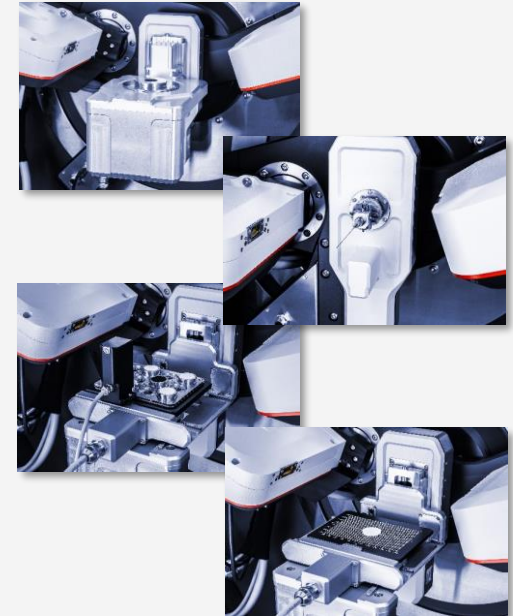
Detectors

- Pixos detectors based on Timepix3 chip from Advacam
- 0D and 1D photon counting detectors
- 55 μm x 55 μm pixel size
- Si or CdTe sensors to cover every wavelength



Sample stages and holders

- Sample stages and holders for every application
- Measurement in reflection and transmission
- Sample changers for up to 12 samples



Sample Stages

Capillary spinner stage

Capillary measurements in transmission

- Allows sample spinning to improve measurement statistics
- Capillary diameters from 0.2 mm to 3 mm
- Suitable for transmission measurements
- Ideal for small sample quantities, X-ray transparent or environmentally sensitive samples
- Includes tools and accessories for capillary alignment



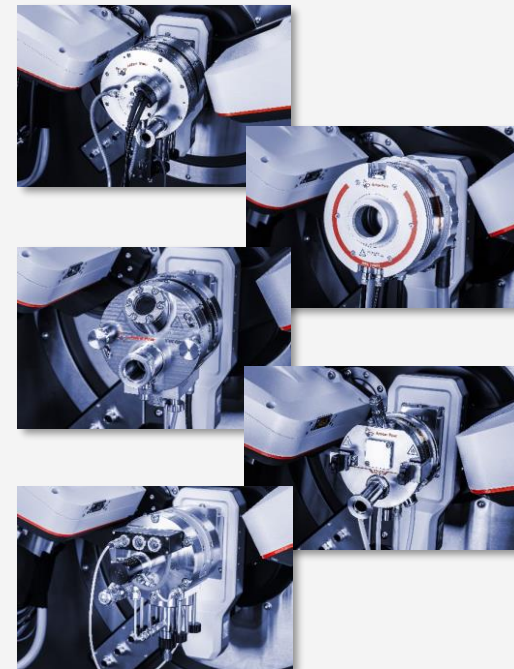
Sample stages – EVAC Module

- Fully evacuated beam path – from source to detector
- Measurement up to 162.5° 2θ in reflection and transmission
- Best possible signal-to-noise ratio
- For XRD, SAXS, PDF analysis
- Obtain SAXS data with quality comparable to a stand-alone instrument



Non-ambient attachments

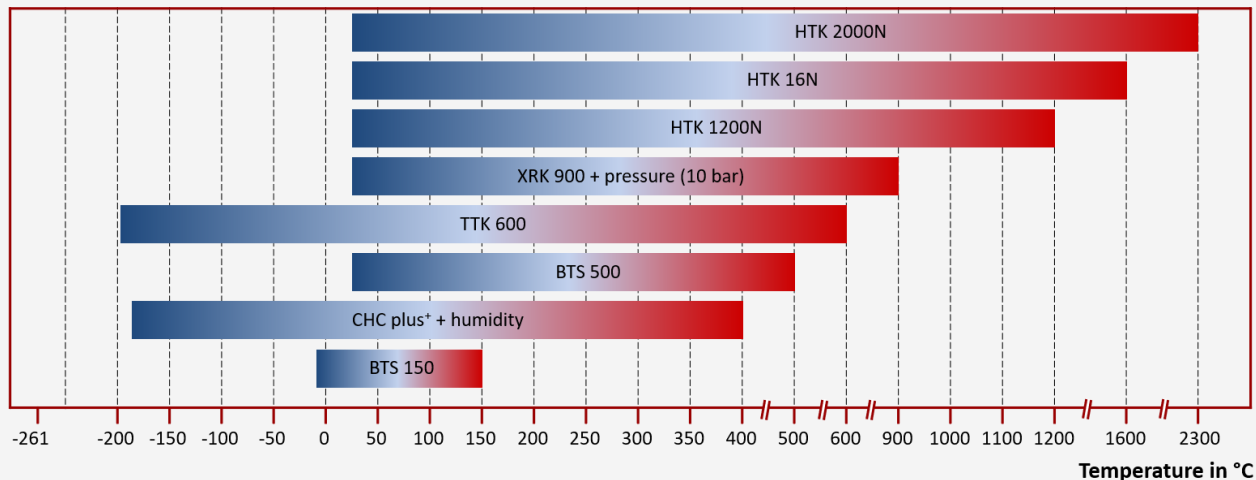
- **Temperature range:** -190 – 2300 °C
- **Relative humidity (%RH):** 5 – 95 %RH (also with temperature)
- **Atmospheres:** Air, vacuum, inert gas, reactive gases
- **Non-ambient concept of XRDynamic 500:**
 - Built-in CCU control unit with stage recognition
 - One control unit for all non-ambient attachments
 - Convenient non-ambient connections in the instrument housing (water, vacuum, compressed air, etc.)



Non-ambient XRD Attachments

Specification range

Covering a huge range of non-ambient conditions



Software



XRDdrive: Instrument control software

- Automatic instrument and sample alignment routines
- Change of instrument configuration with a click
- Easily set up complex measurement batches



XRDanalysis: Data analysis and evaluation software

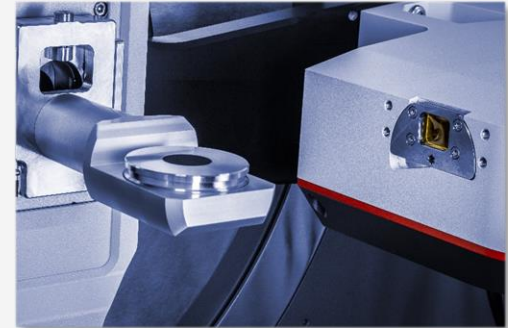
- Background fitting and peak search
- Profile fitting
- Qualitative analysis (e.g. using PDF databases)
- Quantitative analysis (Rietveld refinement)
- Batch analysis



APPLICATIONS OVERVIEW

MEASURE A HUGE VARIETY OF SAMPLE TYPES

- XRDynamic 500 is suitable for powder samples of all types independent of the material class
- Various options to measure solids, foils, fibers, and thin films
- Dedicated SAXS configuration for the measurement of nanostructured and biological samples



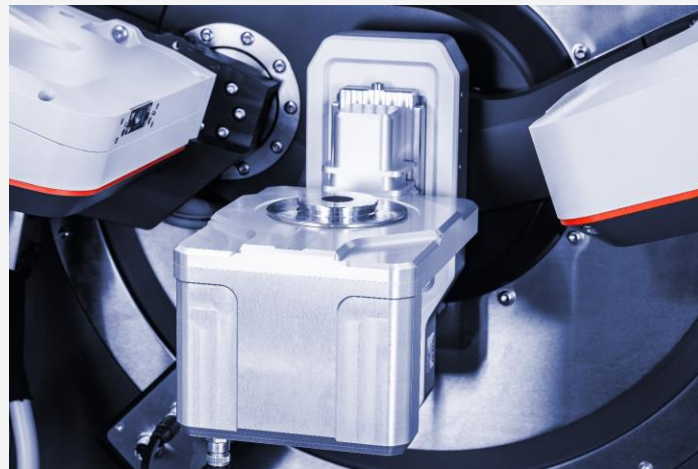
BUILDING MATERIALS

Application example 1: Cement

Cement: Background information

- Main application: Binder material in construction
- Concrete (= cement + sand + gravel + water) is the most commonly used construction material worldwide.
- Most common type of cement: Portland cement
 - About 4bn tonnes produced per year
 - Production (especially calcination of limestone) causes > 5% of global CO₂ emissions.
- Consists of several crystallographic phases.
- XRD applications:
 - Structural analysis of new cements (academic research + R&D)
 - Quality control
 - In-situ (or ex-situ) measurement of phase changes for example during hardening

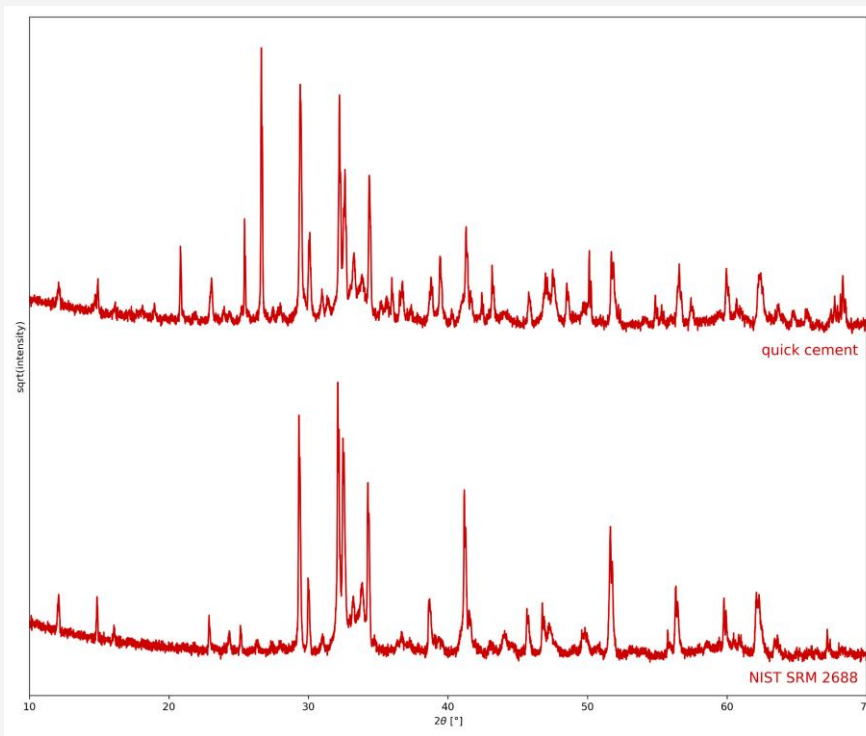
- Sample stage: Sample Spinner
- X-ray tube: Cu
- Optics: Bragg-Brentano with Ni-C monochromator



Samples:

- Sample 1: NIST SRM 2688 Portland Cement Clinker
- Sample 2: Quick cement

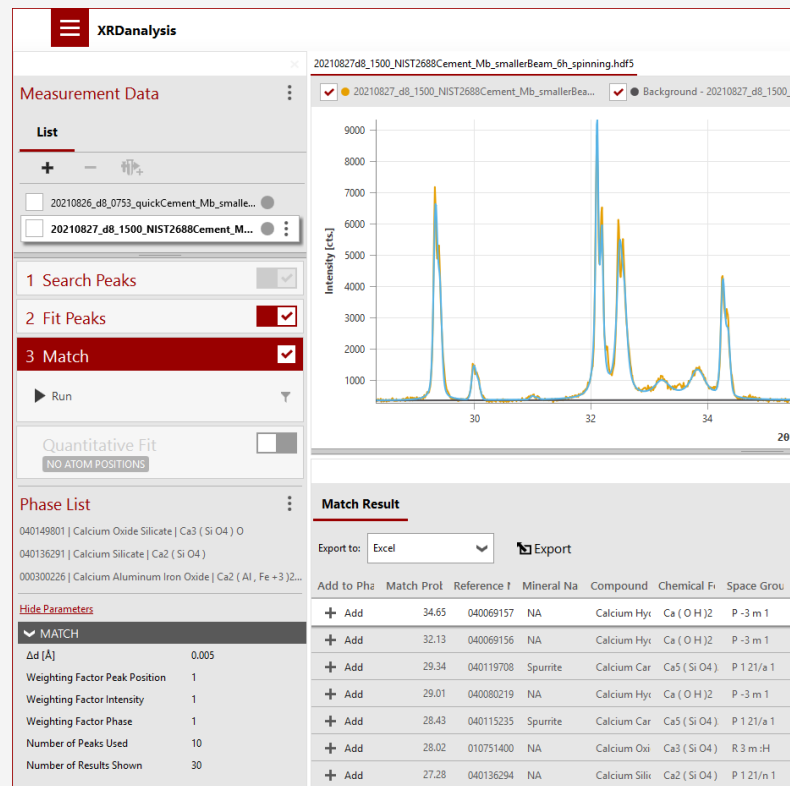
Comparison shows that some phases are the same in both cements, but there are also significant differences.



Cement: Results 2

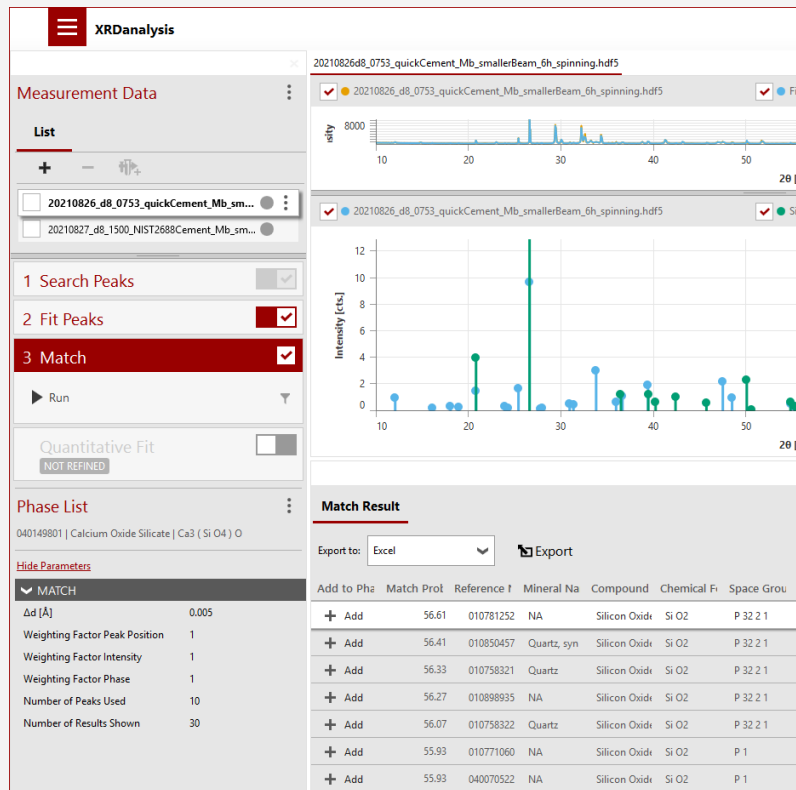
Qualitative phase analysis
with XRDanalysis
("Match") reveals main
phases of Portland
cement:

- Alite ($\text{Ca}_3\text{SiO}_4\text{O}$)
- Belite (Ca_2SiO_4)
- Ferite (calcium
aluminium iron oxide)



Cement: Results 3

On the contrary, quartz is
found in the quick cement:

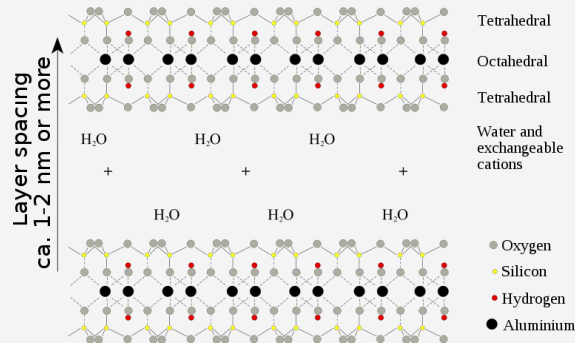


MINERALS

Application example 4: Heating of bentonite

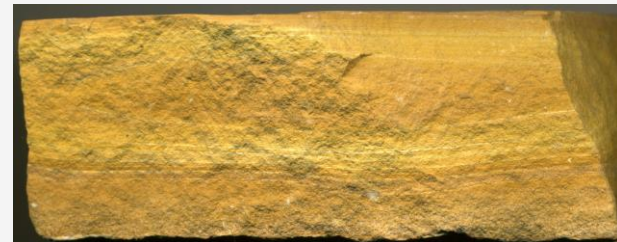
Bentonite: Background information

- Swelling clay
- Formed from volcanic ash
- Consisting of several minerals
- Main phase: montmorillonite
 - Sodium calcium aluminium magnesium silicate hydroxide
 - Layered structure:



Applications:

- Drilling mud
- Foundry sand
- Ceramic
- Nuclear waste barrier
- Pharmaceuticals
- Paint
- Food
- Cat litter



- Sample stage: HTK 1200 N
- X-ray tube: Cu
- Optics: Bragg-Brentano with Ni-C monochromator

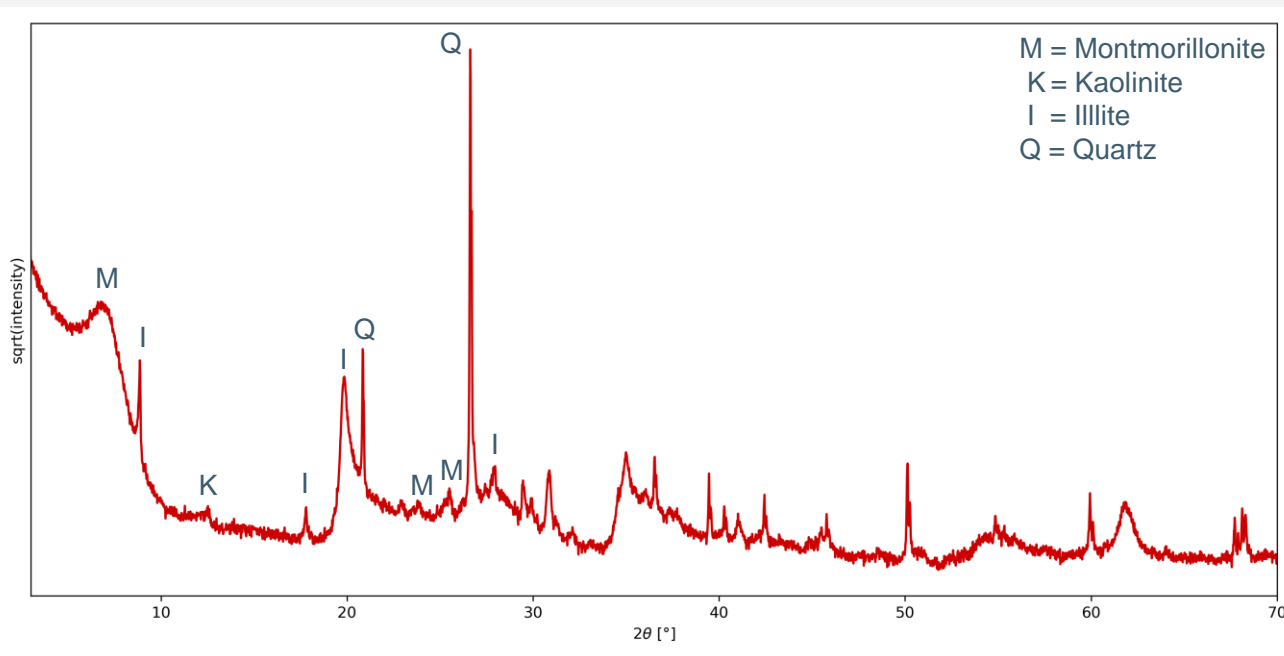


Sample:

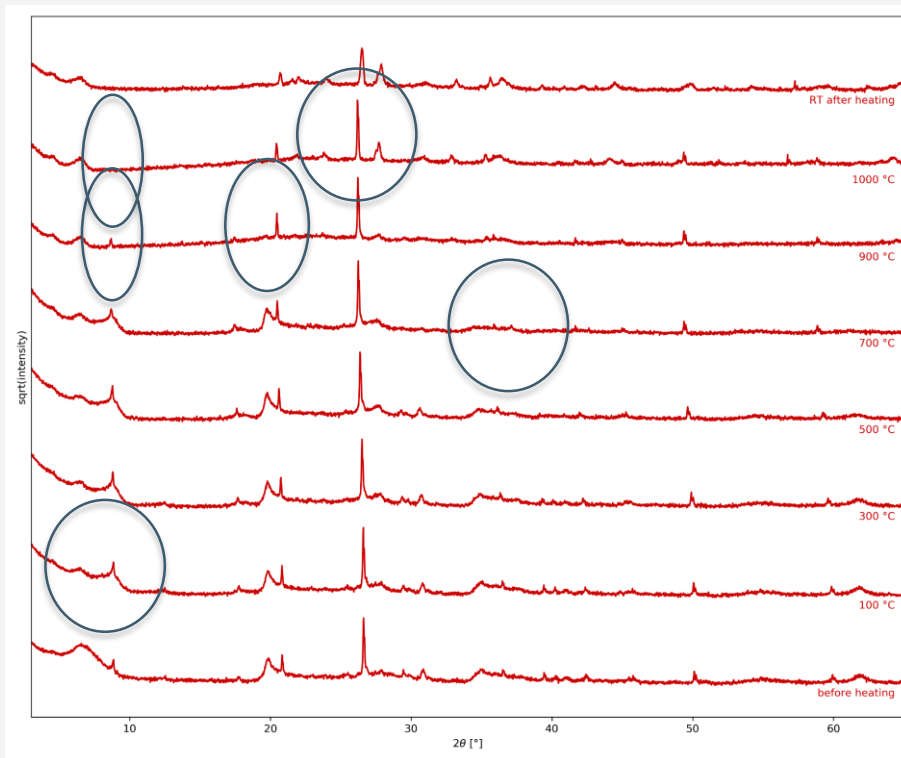
- Bentonite with about 70 % montmorillonite and 6-12 % water.
15 minutes milled to get smaller particles and crystals.

Bentonite: Results

Long ex-situ scan (3 hours) to identify also phases with small peaks.



Bentonite: Results



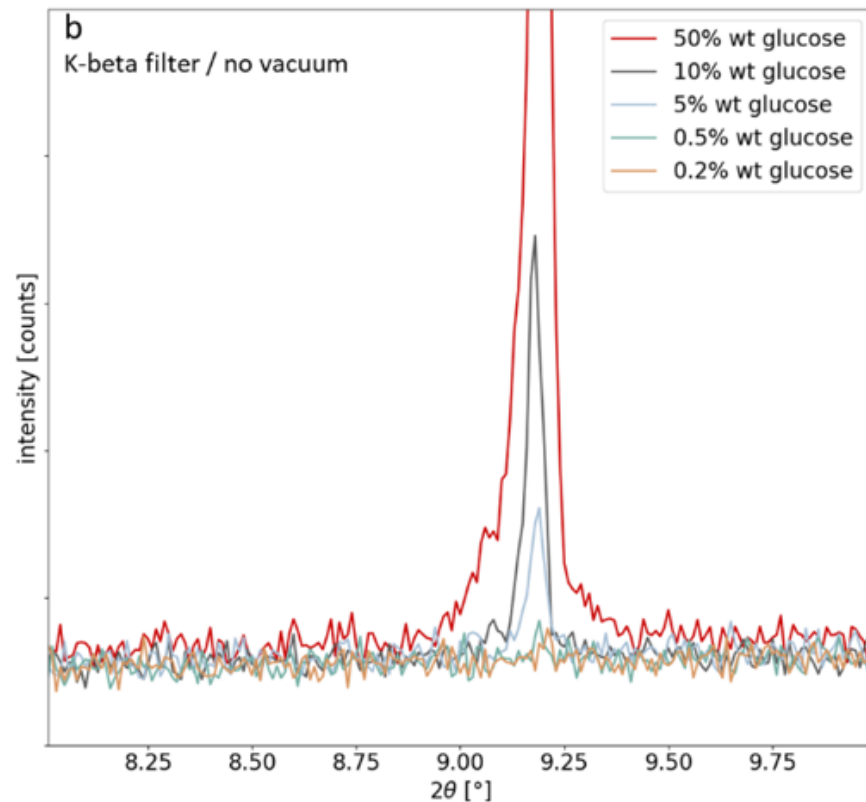
Non-ambient measurement reveals structural changes during heating. Also the colour of the sample changes significantly during heating.



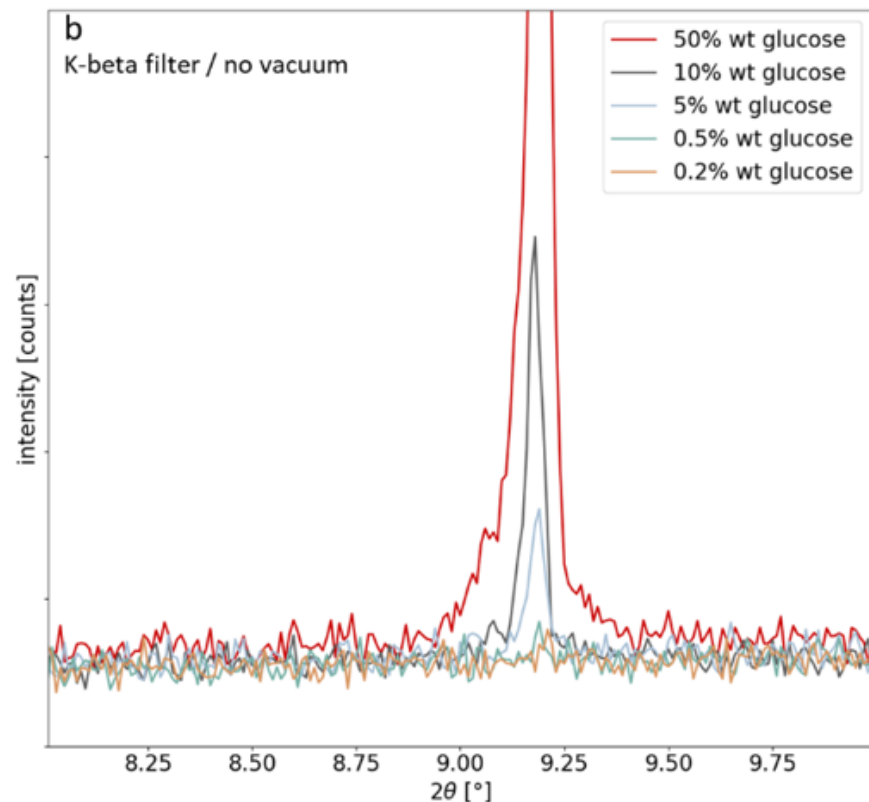
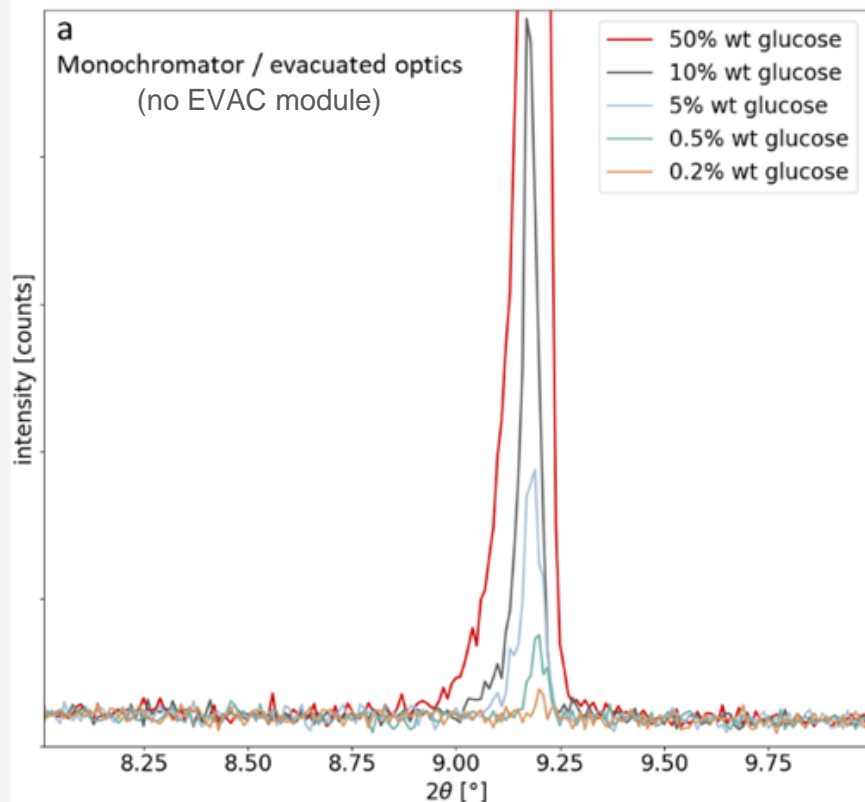
LIMIT OF DETECTION LIMIT OF QUANTIFICATION

Application example 5: Sugar in CaCO_3

Glucose peak – with and without vacuum (Cu)



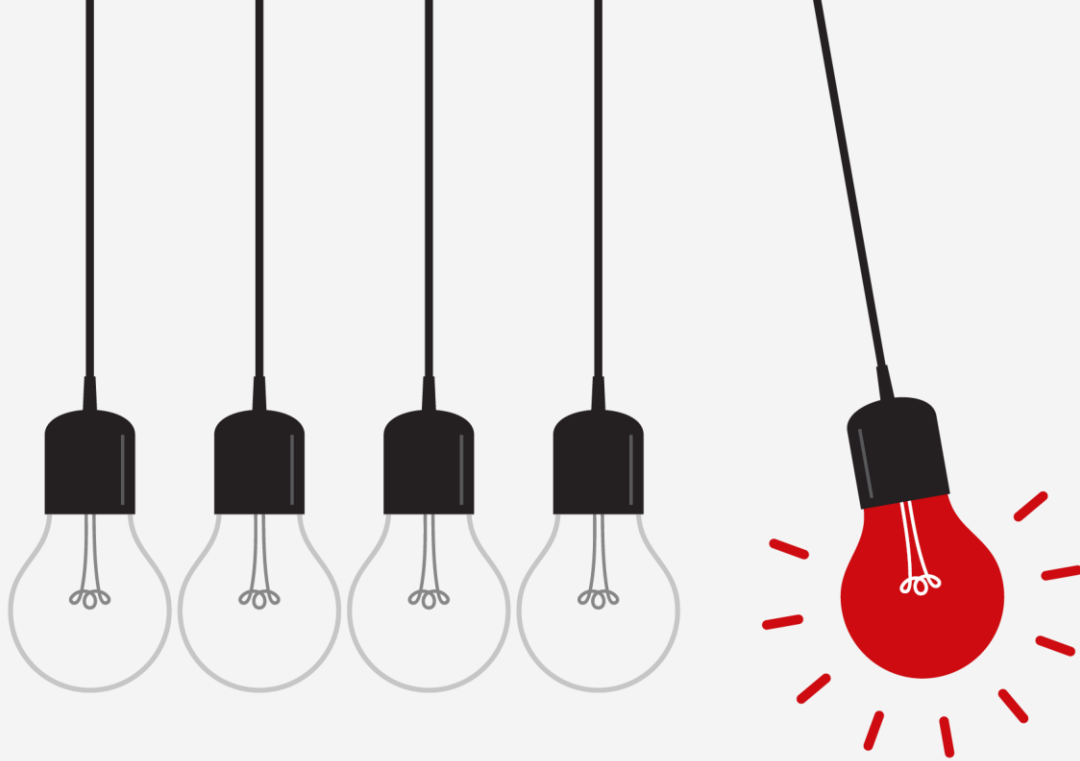
Glucose peak – with and without vacuum (Cu)



Summary

- **XRDynamic 500:** the new automated multipurpose powder X-ray diffractometer from Anton Paar - Driving XRD
- **Intuitive and super-efficient**
 - Automated beam geometry and optics changes
 - Automated instrument and sample alignment routines
- **Best-in-class data quality**
 - Large goniometer radius and small detector pixel size
 - Evacuated optics units with option for fully evacuated beam path from source to detector
- **Maximum flexibility**
 - Instrument set-ups for every application
 - Wide variety of ambient and non-ambient sample stages
 - Optimized solutions for powder XRD, non-ambient XRD, PDF analysis and SAXS





**Thank you for
your attention!**

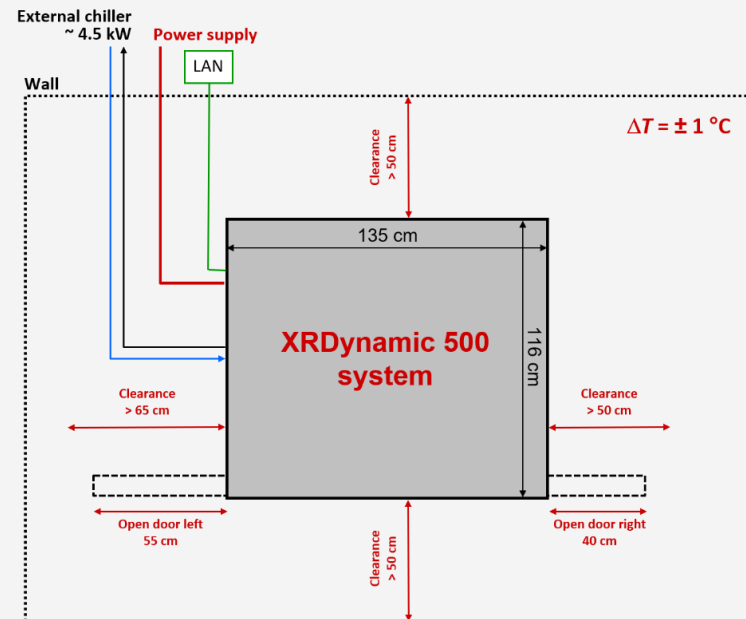
Your questions.

XRDynamic 500 REQUIREMENTS

AMBIENT CONDITIONS

Temperature	20 °C to 25 °C
Temperature variation	< ± 1 °C (within 30 minutes)
Altitude	< 2000 m
Relative humidity	20 %RH to 80 %RH
Dimensions	135 x 116 x 185 (cm; W x D x H) \Rightarrow excl. door opening and 50 cm clearance
Weight	≥ 750 kg (depending on configuration)

\rightarrow For further information refer to “E29IB012EN XRDynamic 500 Installation Site Requirements”





Anton Paar

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