VÅNTEC-500 Area Detector for Pharmaceutical XRD
XRD$^2$ for Pharmaceutical: Reflection System (CS)
XRD$^2$ for Pharmaceutical: Reflection & Transmission (HTS)

US Patent #7,242,745
The most dramatic development in XRD² happens in three critical components and data evaluation algorithms:

- **Source**: required radiation energy, focal spot size and intensity.
- **Optics**: select wavelength, beam profile and divergence.
- **Detector**: collect 2D pattern with correct intensity and position.
- **Data evaluation Algorithm**: Diffraction Vector Approach.
X-ray Source for XRD$^2$:
Incoatec Microsource (I$\mu$S)$^\text{TM}$

- High brilliance
- Low energy: 30 W
- Air-cooled
- Spot size < 100 µm
- Montel mirror
IμS & VÅNTEC-2000 vs. Classic Set-up
Corundum Comparison

IμS & VÅNTEC-2000
45kV, 0.650mA,
0.3mm snout
total counts: 1235K

Single 40mm Göbel Mirror,
45kV, 40mA,
0.3mm collimator
total counts: 78K
**XRD$^2$: Choice of Detectors**

**Sensitivity vs. Count Rate**

Detective Quantum Efficiency (DQE):

- The DQE is a parameter defined as the square of the ratio of the output and input signal-to-noise ratios (SNR).

\[
DQE = \left( \frac{(S / N)_{\text{out}}}{(S / N)_{\text{in}}} \right)^2
\]

- The DQE of a real detector is less than 100% because not every incident x-ray photon is detected, and because there is always some detector noise.

- MiKroGap™ has the best overall performance.

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![Detection Efficiency Graph](image.png)
Detector Technology from MWPC to MikroGap™

- MikroGap™ technology with resistive anode:
  - shortens drift time of ions
  - fast electrons induce charge on readout strips
- Adjusted surface resistance ($10^5 - 10^7 \ \Omega/\text{area}$):
  - high enough to limit discharges
  - low enough to support high count rates

US Patent US 6,340,819 B1
VÅNTEC-500 – Outperforms all previous gaseous detectors

Similar to Hi-Star (MWPC) detector:
- High sensitivity: 80% DQE (detection quantum efficiency) at 8.04 keV radiation
- Energy range: 3-15 keV (good for Cu, Co, Fe and Cr X-ray sources, not recommended for Mo)
- Energy resolution ($\Delta E/E$): 20% at 8.04 keV radiation
- Low background noise: <5 cps/global
- Readout time: real time
- No cooling
- Curved Be-window to reduce parallax
VÅNTEC-500 – Outperforms all previous gaseous detectors

**Advances from MWPC:**

- Tapered front end for high 2θ angle access and space for large samples and sample stages
- Doubled the spatial resolution: The FWHM of the PSF is 200μm
- Two orders of magnitude higher maximum count rate: Global count rate: 1.5Mcps Local count rate: 250kcps per point-like reflection
- Radiation hardness: accidental intensive irradiation without permanent damage
- Maintenance-free: no re-gassing
VÅNTEC-500 – Tapered front for low and high $2\theta$ accessibility

$$2\theta_{range} = 2 \arctan \frac{l}{D}$$

$$2\theta_{max} = \pi - \frac{m + h}{D}$$

Bruker AXS
VÅNTEC-500 – Outperforms all previous gaseous detectors

Detector geometry:
- Be-window opening 140 mm in dia.
- Frame size:
  - 2048 x 2048 pixels
  - 1024 x 1024 pixels
  - 512 x 512 pixels
- Pixel size:
  - 68 µm x 68 µm
  - 136 µm x 136 µm
  - 272 µm x 272 µm
- Detector working distance:
  - 5~30 cm in D8 DISCOVER enclosure
- 2θ range in a single frame:
  - 5 cm 83°
  - 10 cm 56°
  - 15 cm 42°
  - 20 cm 33°
  - 25 cm 27°
  - 30 cm 23°
Hi-resolution with MikroGap™ Technology

- 68 μm pixel size delivers the best spatial resolution

272 μm pixel (512x512)  136 μm pixel (1024x1024)  68 μm pixel (2048x2048)
The New D8 DISCOVER
The New D8 DISCOVER

Patented Door Mechanism

Swing door: wide opening for good accessibility

Sliding door: easy access for sample loading and configuration changes
The D8 DISCOVER with DAVINCI TWIST-TUBE – from line to spot, and back

- Fast and easy switching between line and spot focus
- Focus direction recognition
- No need to disconnect cables
- Compatible with standard tube design
- Bruker AXS proprietary technology
The D8 DISCOVER with DAVINCI
Tool-free mount & component recognition

DAVINCI.SNAP-LOCK
Tool-free change of optics

DAVINCI.MODE
Component recognition
SAXS

- $q_{\text{min}} = 0.025 \text{ Å}^{-1}$
- $d_{\text{max}} = 25\text{nm}$

He beampath, SAXS beamstop, Vantec 500
Configure: GADDS HTS
Vertical theta-theta, Reflection/Transmission

SCD, December 2008
No barrier between 0D/1D/2D
Vertical theta-theta, CEC for microdiffraction/stress/texture, 0D->1D->2D
XRD$^2$: Systems with VÅNTEC-500 Detector
XRD$^2$ & Single Crystals

Laue equation

\[ a \cdot (s - s_0) = h\lambda \]
\[ b \cdot (s - s_0) = k\lambda \]
\[ c \cdot (s - s_0) = l\lambda \]
XRD² & Powders

Bragg law

\[ n\lambda = 2d \sin \theta \]
XRD\textsuperscript{2}: Diffraction pattern with both $\gamma$ and $2\theta$ information

Diffraction vector with $\gamma$

$$H = \frac{s - s_0}{\lambda} = \frac{1}{\lambda} \begin{bmatrix} \cos 2\theta - 1 \\ -\sin 2\theta \sin \gamma \\ -\sin 2\theta \cos \gamma \end{bmatrix}$$
## XRD$^2$: Diffraction vector approach

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<td>Texture Analysis:</td>
<td>Orientation mapping angles; Data collection strategy (scheme)</td>
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<td>Stress Measurement:</td>
<td>Fundamental equation derived by second order tensor transformation; Data collection strategy (scheme)</td>
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<td>Equations for the effective volume calculation at both reflection and transmission modes.</td>
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XRD²: PhaseID Measurement Geometry
XRD$^2$: Single Frame Covering All
XRD$^2$: Frame Merge and Integration
The pole figure angles \((\alpha, \beta)\) can be calculated from the unit vector components by the pole mapping equations:

\[
\alpha = \sin^{-1} \left| h_3 \right| = \cos^{-1} \sqrt{h_1^2 + h_2^2}
\]

\[
\beta = \pm \cos^{-1} \frac{h_1}{\sqrt{h_1^2 + h_2^2}} \quad \begin{cases} 
\beta \geq 0^\circ & \text{if } h_2 \geq 0 \\
\beta < 0^\circ & \text{if } h_2 < 0
\end{cases}
\]
The D8 DISCOVER with DAVINCI

VÅNTEC-500 for texture measurement

- Steel can
- (200) & (110) rings
- Intensity variation during \( \phi \) scan
**XRD²:** Particle size measurement by $2\theta$ & $\gamma$ profile analysis:

![XRD Pattern](image)

- **Incident Beam**
- **Sample**
- **Debye Cone**

$\gamma$

$2\theta$
XRD²: Peak broadening-gold Nanoparticles
**XRD^2**: Particle size and instrument broadening:

The measured profile is a convolution of the functions representing particle-size broadening and instrument broadening.

\[
h(x) = \frac{1}{A} \int g(z) f(x-z) dz
\]

where \( A \) is the area of the \( f(y) \) curve and \( y = x - z \).

**XRD²: Particle size calculation:**

Scherrer equation:

\[ t = \frac{C\lambda}{B \cos \theta} \]

where \( \lambda \) is wavelength (Å), \( B \) is FWHM (radians) corrected for instrument broadening, \( \theta \) is Bragg angle, \( C \) is a crystal shape factor from 0.9~1.

For Gaussian profiles, \( B^2 = U^2 - S^2 \)

while for Cauchy profiles, \( B = U - S \)

where \( B \) is the corrected FWHM for crystallite size calculation by Scherrer equation, and \( U \) and \( S \) are the FWHM’s of the unknown and standard peaks, respectively.
XRD$^2$: Data Collection:

Acetaminophen powder

- The spotty diffraction ring is due to the large crystallites compared to the sampling volume (beam size).
- The number of spots on the ring is determined by crystallite size, instrumental window ($\gamma$-range), multiplicity of the crystal plane, and effective diffraction volume.
XRD²: Particle size measurement by γ profile analysis:

For XRD² in reflection mode, the particle size is given by

\[
d = k \left\{ \frac{p_{hkl} b^2 \arcsin[\cos \theta \sin(\Delta \gamma / 2)]}{2 \mu N_s} \right\}^{1/3}
\]

where \( k \) is the instrumental calibration factor or can be calculated from

\[
k = \left( \frac{3 \beta}{4 \pi} \right)^{1/3}
\]

if the instrument broadening in 2θ direction is known.

For transmission mode with the incident beam perpendicular to the sample surface, the particle size is given by

\[
d = k \left\{ \frac{p_{hkl} b^2 t \arcsin[\cos \theta \sin(\Delta \gamma / 2)]}{N_s} \right\}^{1/3}
\]

where \( k \) is the instrumental calibration factor or

\[
k = \left( \frac{3 \beta}{4 \pi} \right)^{1/3}
\]
XRD$^2$: Particle size measurement by $\gamma$ profile analysis:

- The frame was collected from a SRM660a (LaB6) sample in transmission mode and Cu-K$\alpha$ x-rays.
- The 2D detector (Hi-Star™) is set at 23.75 cm from the instrument center.
- The beam (collimator pinhole) size $b$ is 200 $\mu$m.
- The sample thickness $t$ is 7.0 $\mu$m, based on the calculated $\mu$ of 1138 cm$^{-1}$ and the measured transmission of 0.45.
**XRD²**: Particle size measurement by $\gamma$ profile analysis:

- The $2\theta$-integrated plots ($\gamma$-profiles) of three rings from (100), (110) and (111) planes are displayed.
- The number of crystallites is counted from the number of intersections of the $\gamma$-profile with a threshold line.
- To cancel out the effects of the overall intensity fluctuation (texture, etc.), a 2$^{\text{nd}}$ order polynomial trend line is fitted to each $\gamma$-profile as a threshold line.
- Every two intersections of $\gamma$-profile with the threshold line represents a crystallite.
- New analysis strategy?
- Size distribution?
XRD\textsuperscript{2}: Particle size measurement by $\gamma$ profile analysis:

Calibration Results:

<table>
<thead>
<tr>
<th>(hkl)</th>
<th>$P_{hkl}$</th>
<th>2$\theta$</th>
<th>$\Delta\omega$</th>
<th>$N_s$</th>
<th>k</th>
</tr>
</thead>
<tbody>
<tr>
<td>(100)</td>
<td>6</td>
<td>21.36</td>
<td>38</td>
<td>23</td>
<td>0.1217</td>
</tr>
<tr>
<td>(110)</td>
<td>12</td>
<td>30.38</td>
<td>46</td>
<td>41</td>
<td>0.1106</td>
</tr>
<tr>
<td>(111)</td>
<td>8</td>
<td>37.44</td>
<td>42</td>
<td>38</td>
<td>0.1281</td>
</tr>
</tbody>
</table>

- The average scaling factor $k$ is 0.12 in this calibration. The system can then be used to measure the crystallite size of unknown materials if the data can be collected in approximately the same condition.
- It is always necessary to calibrate the system with a known standard, preferably with a comparable sample geometry and crystallite size.
- For reflection mode, it is critical to have a standard with a comparable linear absorption coefficient so as to have similar penetration.
XRD²: Particle Size Analysis by X-ray Diffraction:

- 2θ profile analysis, including measurement from peak FWHM by Scherrer equation, or profile analysis by Stokes and Wilson, is suitable for particle size below 100 nm.
- γ profile analysis, based on sampling statistics, is suitable for particle size from sub-micrometer to a few millimeters.
- The particle size range of pharmaceutical substances is from sub-micrometer to a few millimeters.
## More About XRD²

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Thank You for Your Attention