Data integration for point detectors

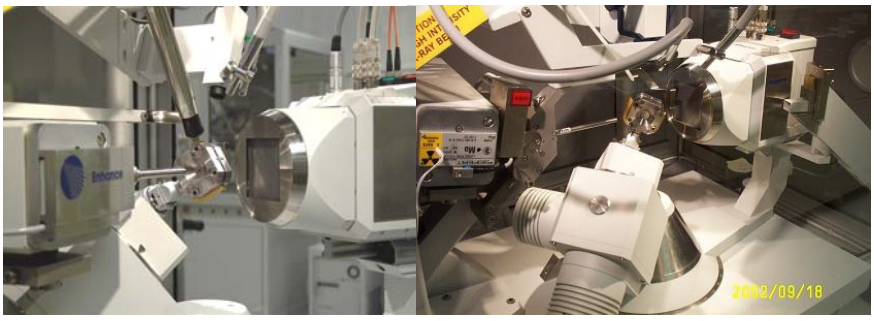
Ross Angel
Virginia Tech Crystallography Laboratory
Blacksburg VA 24060

rangel@vt.edu *www.crystal.vt.edu*

- ***General points about PD vs area D***
- ***General points about PD integration***
- ***Integration with WinIntegrStp***

Determining structures at high pressure

Precise intensity data to measure small changes in structures at high pressures:

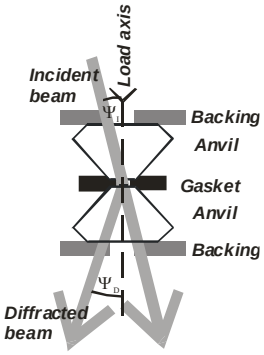


Single-crystal X-ray diffraction.

Point and CCD detectors.

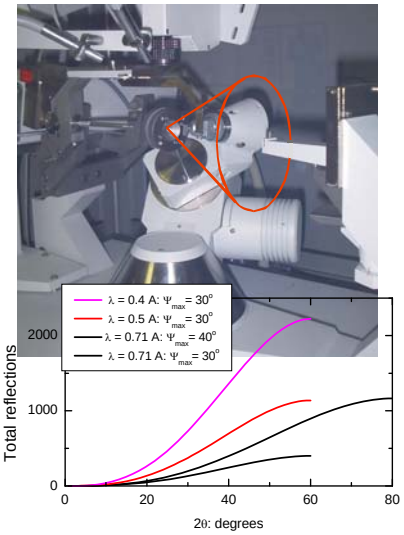
Beamlines and in-house

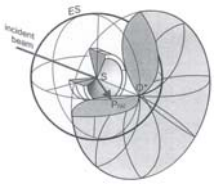
Diamond-anvil geometry



Transmission mode

Limited access



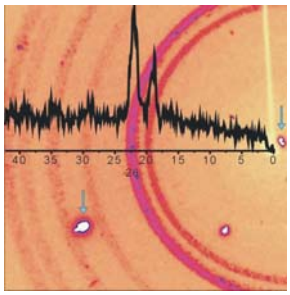


Miletich, RiM volume

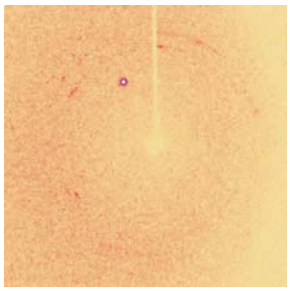
PD is more efficient at collecting data at the edges of accessible space

Reducing background

Everything you see is background!



Conventional DAC with Be seats, steel gasket

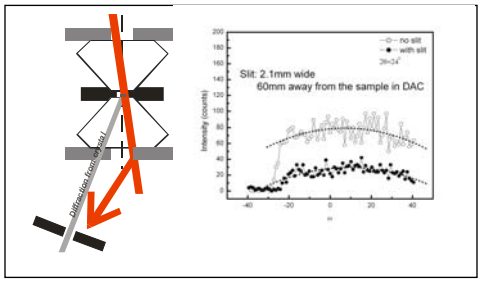


DAC with steel seats and rhenium gasket

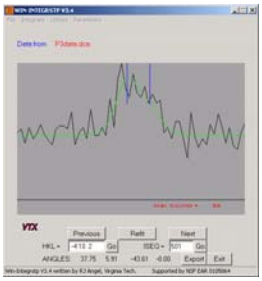
Reducing background

Use a point detector:

- ***Additional collimation***



- ***Optimised scan speed***
- ***Step scans***
- ***Profile fitting***
- ***Recovery of weak data***



Area and Point detectors

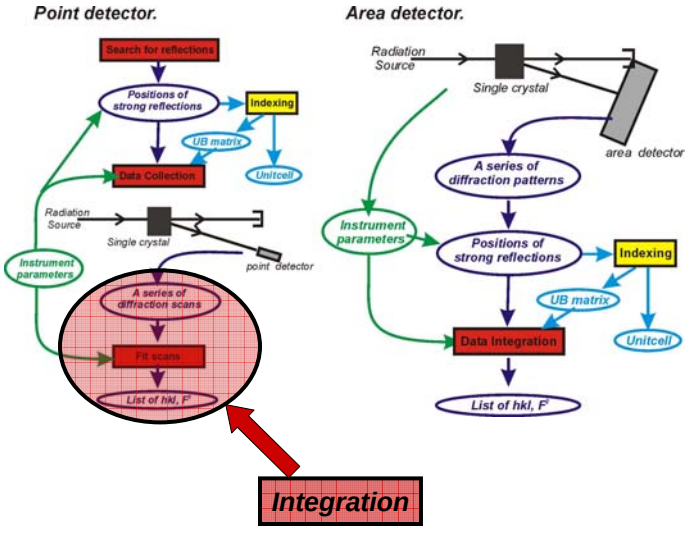
Point detector

- **Accurate peak positions**
  - *Centering the DAC*
  - *Unit-cell parameters*
  - *Equations of State*
- **Small unit cells**
  - *Additional collimation*
  - *Optimised scan speed*
  - *Step scans*
  - *Profile fitting*
  - *Recovery of weak data*

Area

- **Poor peak positions**
  - *Peak hunting*
  - *Diffuse scattering*
- **Large unit cells**
  - *Approx equal intensities*
  - *Equal scan speeds*
  - *Faster data collection*
- ***Twins, multiple crystals***

Intensity data collection



Point detector integration: principles

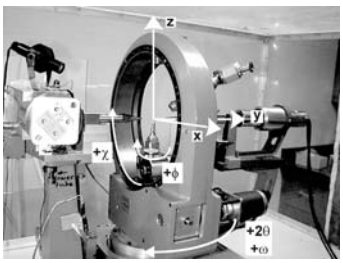
- **Step scan data file**
  - many formats
  - Header info
- **Data for each reflection**
  - intensity as f(ω)
  - scaling info
  - hkl
  - setting angles
  - auxiliary info
    - (stds, nsteps)
    - scan speed

Integrate

- **Integrated intensity file**
  - Few formats
  - SHELX-HKLF4
  - RFINE int
- **Data for each reflection**
  - hkl
  - F^2,$\sigma(F^2)$
  - beam path info

Before starting integration

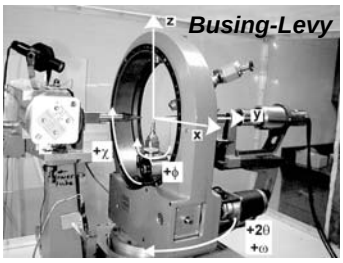
- **Define your instrument**



- Wavelength(s) and intensity ratio
- Monochromator
- Attenuator value
- Diffractometer path lengths (RC, RS)
- Normal peak profile parameters

Defining beam paths for absorption

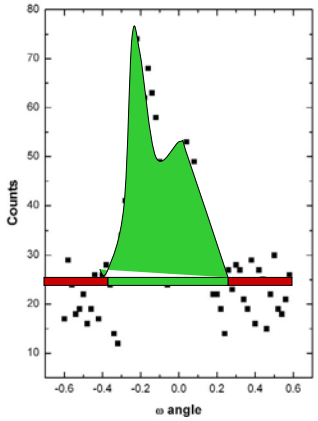
- The definition of the phi-axis system,
  - Axis directions when diffractometer circles at zero



- And you need to know:
  - Type of goniometer (kappa or Eulerian)
  - Circle parities

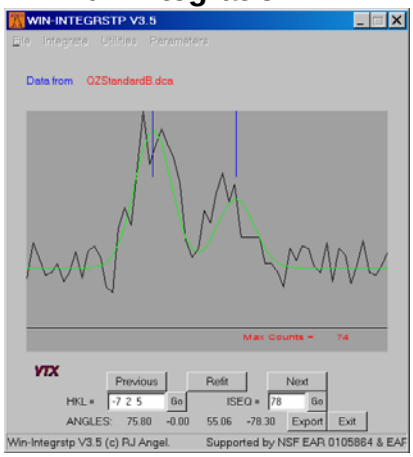
Integration methods

- B-P-B



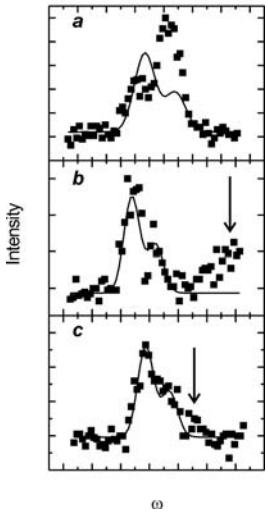
Dynamic backgrounds: Lehman-Larsen

- Full integration



Advantages of full profile integration

- Learn peak shape from strong reflections
- Test of peak shape: is it real?
  - Do not pass bad reflections
- Recover I even with structured background
- More reliable I for weak reflections



Integration Programs

- **Commercial**
  - Built in with instrument software
  - Most do BPB, sometimes with LL
  - Fine for good datasets, not so good for DAC
- **Freeware**
  - Several available by email or web (but developers getting old)
  - Please cite freeware properly (not web site source, but a journal article)
- **WinIntegrStp**
  - Developed from IntegrStp and earlier by Larry Finger
  - Windows version since 2001
  - Current version is 3.5
  - Angel RJ (2003) Automated profile analysis for single-crystal diffraction data. Journal of Applied Crystallography, 36:295-300.

WinIntegrstp features

- **Reads most diffractometer data file formats**
  - Xcalibur, Stoe, Xscans, Phillips febo, Single, CAD4, old Single
  - (does internal conversion)
  - Can 'easily' add new formats if programmer is supplied with good beer!
- **Instrument definition in a text file **.par***
  - Can accommodate any instrument configuration
- **Full learnt-profile fitting**
  - Rejection of bad peaks
  - Recovery of weak reflection data
  - Complex interaction of many controls

WinIntegrStp

- **Download from VTX website**
  - www.crystal.vt.edu
- **Distribution by zip**
  - Manuals and installation instructions
  - Several instrument parameter files
  - Example data file