SI Traceable Diffraction Measurements on the NIST Parallel Beam Diffractometer

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Making XRD Traceable
Parallel Beam Diffractometer Overview

- Interchangeable optics and sample stages
- Vertical axes, concentrically mounted Huber 430 rotation stages
- Heidenhain RON 905 optical encoders on primary axes
- Short and long range encoder calibration
- SI-traceable reference crystals
PBD Schematic

- X-ray source
- Isolation platform
- Nulling Autocollimator
- Specimen
- Encoders
- Mirror & positioner assembly
- Detector
Angular Measurements

• Divide angular domain into two problems:
  • Short-range errors (coherent with encoder features at 100 deg\(^{-1}\)) caused by nonlinearities in the digitizing electronics
  • Long-range errors caused by scale errors in the encoder wheel, eccentricity, etc.
• Avoid use of undocumented internal angular corrections from manufacturer
Short-Period Compensation

- Scan a diffractometer axis at (roughly) uniform speed
- Monitor encoder results as a function of time
- Transform to deviations from linear as a function of angle
- These deviations include screw errors, motor speed variations, all kinds of noise
Deviations have Periodic Structure

Angle Deviation vs. encoder position

Position deviation (arcsec) vs. encoder position (deg)
Example Fourier Spectrum

- Strong peaks at multiples of 100/deg
- Also at motor and gearing frequencies!
- Inset shows how a near collision of gearing and real peak is very well resolved
Extracted Short-period Correction

- Analyze as harmonic series of encoder feature period at 100 features/deg
- Demodulated from 100/deg signal to show coefficients as a function of angle, not correction as a function of angle
Circle Closure Calibration

• Concept
  • compare sums of angles, subject to constraints that full circle is 360°

• Two general methods
  • use polygonal mirror on single stage to provide set of very stable angle offsets
  • use ‘virtual polygon’ and stacked stages and solve for offsets
The Virtual Polygon

\[ \theta_{\text{mirror}} = 2\theta + \omega + \theta_{\text{ring}} \equiv 0 \]
\[ 2\theta_{\text{meas}} + \Delta 2\theta + \omega_{\text{meas}} + \Delta \omega + \theta_{\text{ring}} = 0 \]
\[ 2\theta_{\text{meas}} + \omega_{\text{meas}} + \theta_{\text{ring}} = -\Delta 2\theta - \Delta \omega \]
Details of Virtual Polygon

• For a given ring setting $\theta_{r,n}$ measure a set of angle errors $\{\Delta 2\theta_{n,m} (2\theta_m) + \Delta \omega_{n,m}(-2\theta_m - \theta_{r,n})\}$ for (typically) 36 approximately equally spaced $2\theta$ values and corresponding $\omega$ values which null the autocollimator ($n$ indexes ring, $m$ indexes $2\theta$)

• repeat for at least 3 ring settings, to give enough degrees of freedom to solve for $\Delta 2\theta$, $\Delta \omega$, and the $\theta_r$ associated with each group.

• Do least squares fit for parameters
Circle Closure Results

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Visualization of Error Sum

\[ \omega = 0 \]

\[ 2\theta = 0 \]
Spectrum Measurement (the future...)

- Next Step: measure spectrum through our optics, with fully traceable steps
- use ‘beam walking’ technique in combination with Dectris detector array to map out properties
- requires traceable lattice constants on diffractive optics. These optics are already fabricated.
Beam Walking Experiment

- Measure angular geometry of beam in non-dispersive configuration (top)
- Measure spectrum of beam in dispersive configuration (bottom)
- Depends on fully qualified angle metrology from compensation and circle closure
- Fast, using Dectris Pilatus 2-D detector array