SI Traceable Diffraction Measurements on the NIST Parallel Beam Diffractometer

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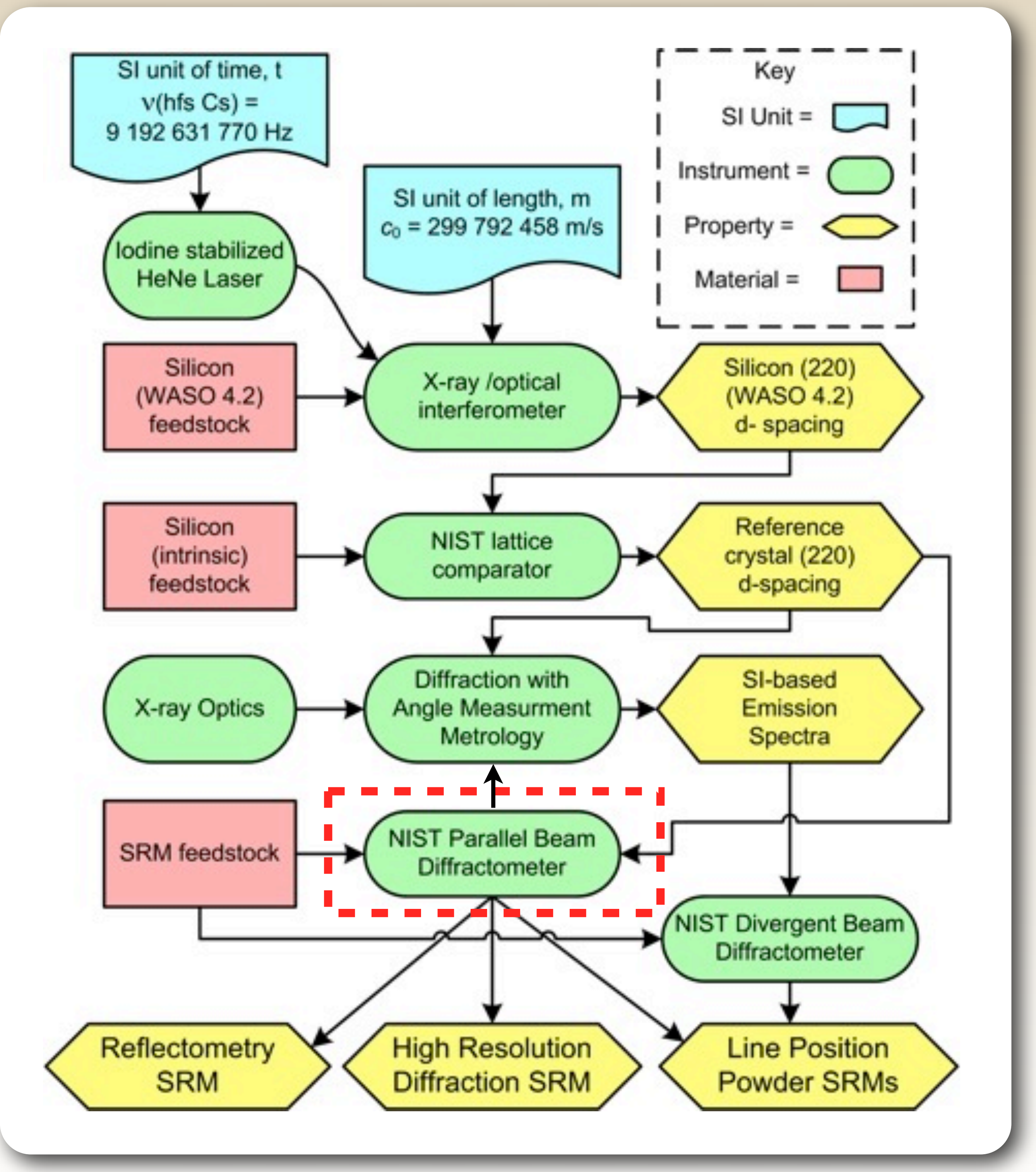
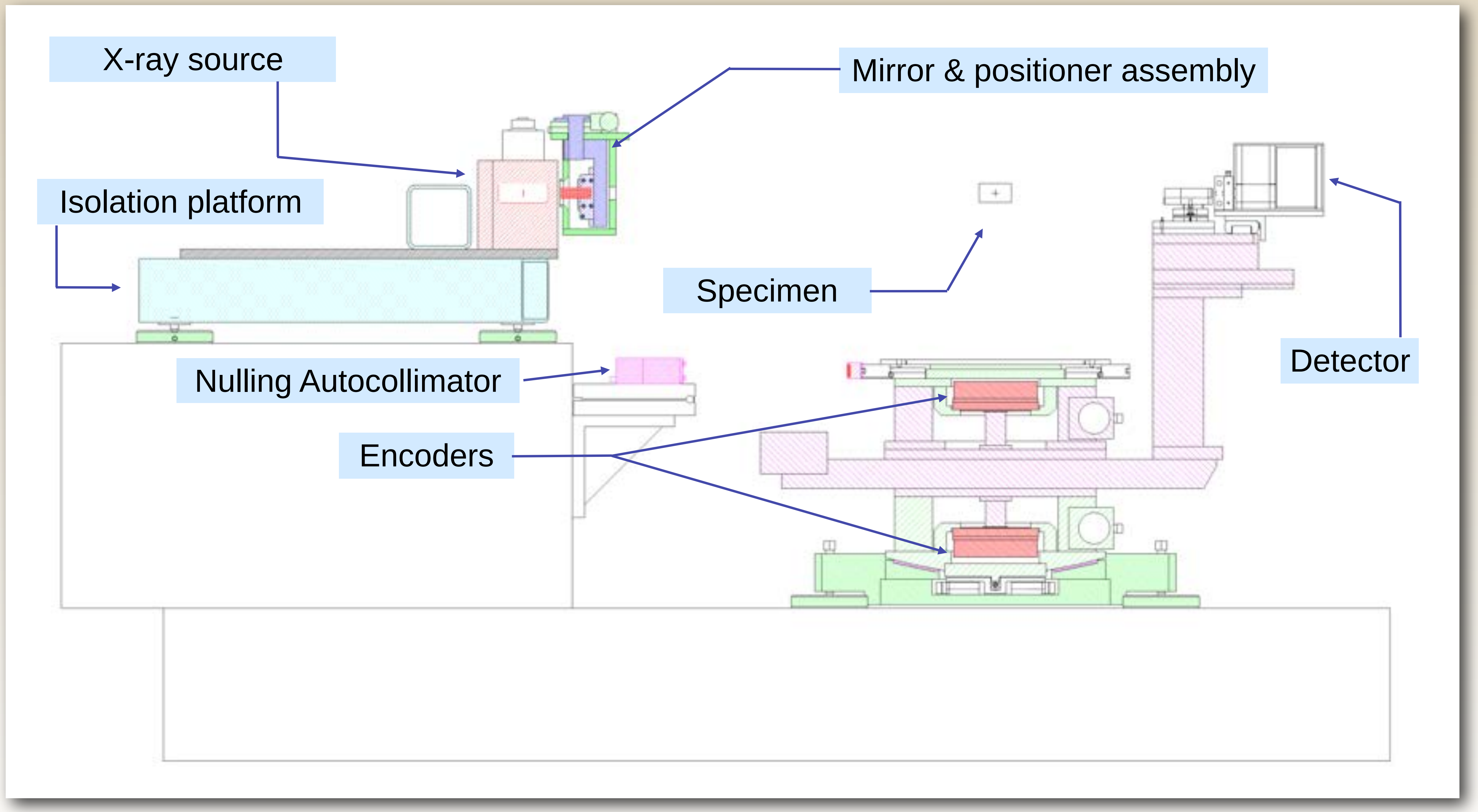
Making XRD Traceable



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



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

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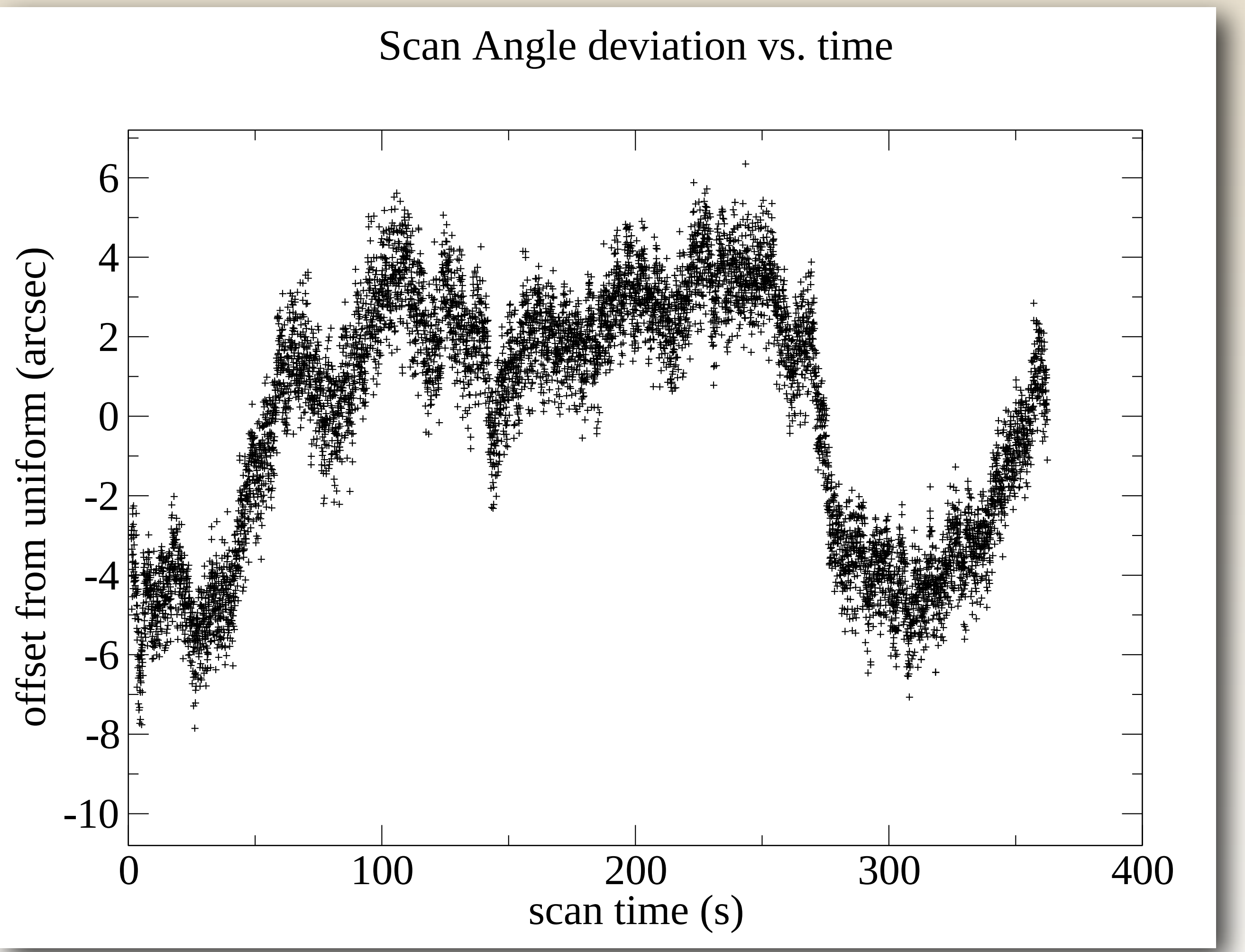
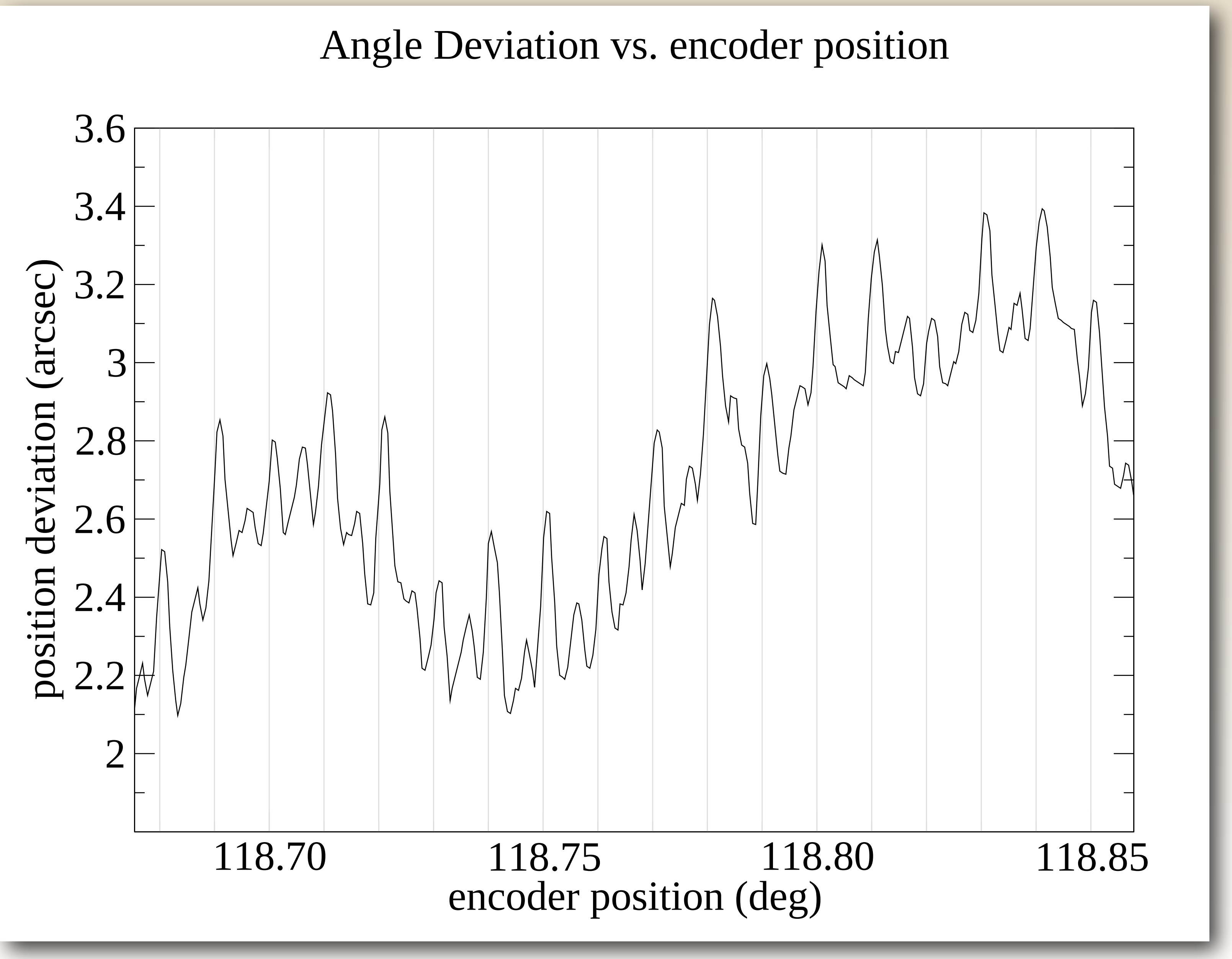
Short-Period Compensation

- 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

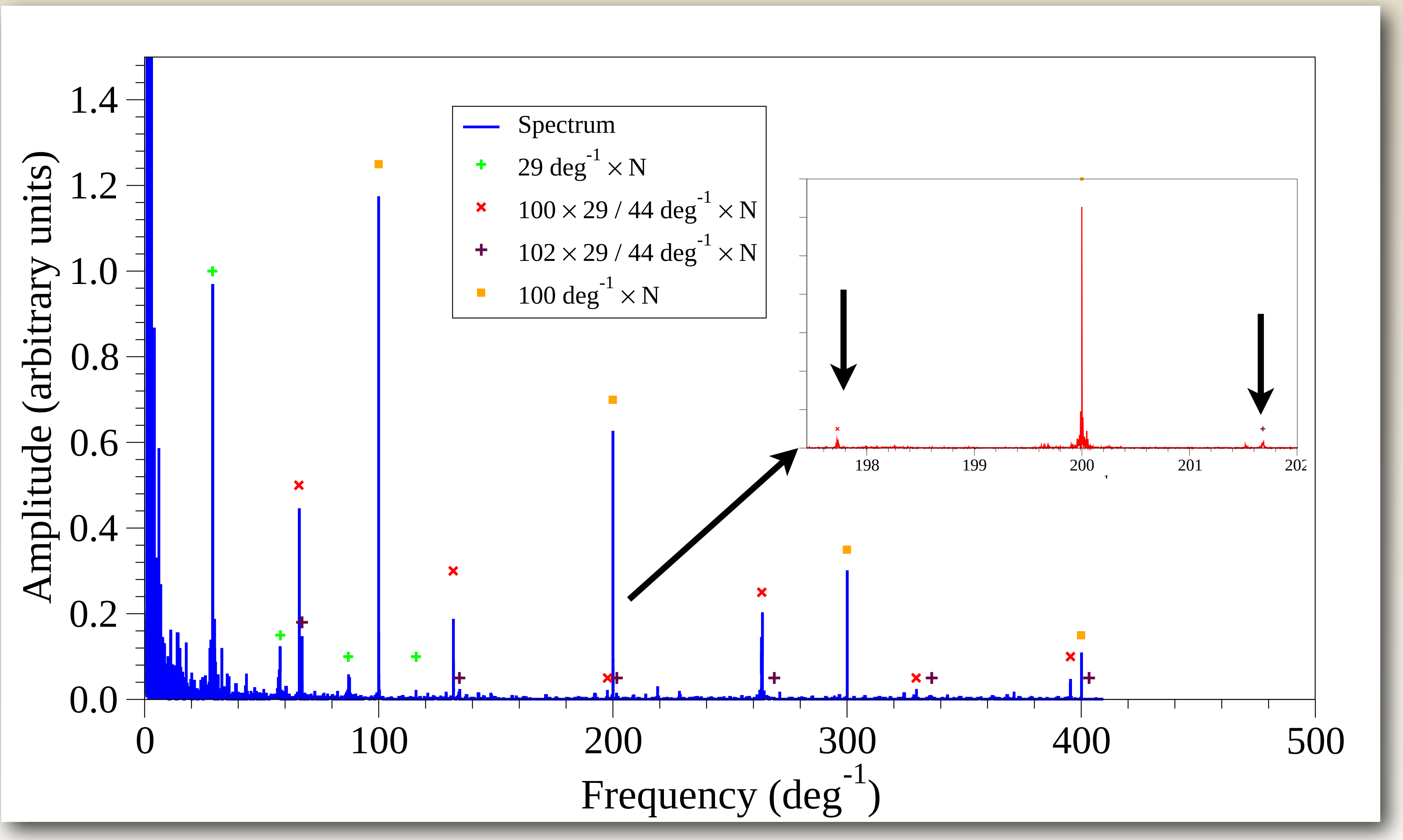


Deviations have Periodic Structure



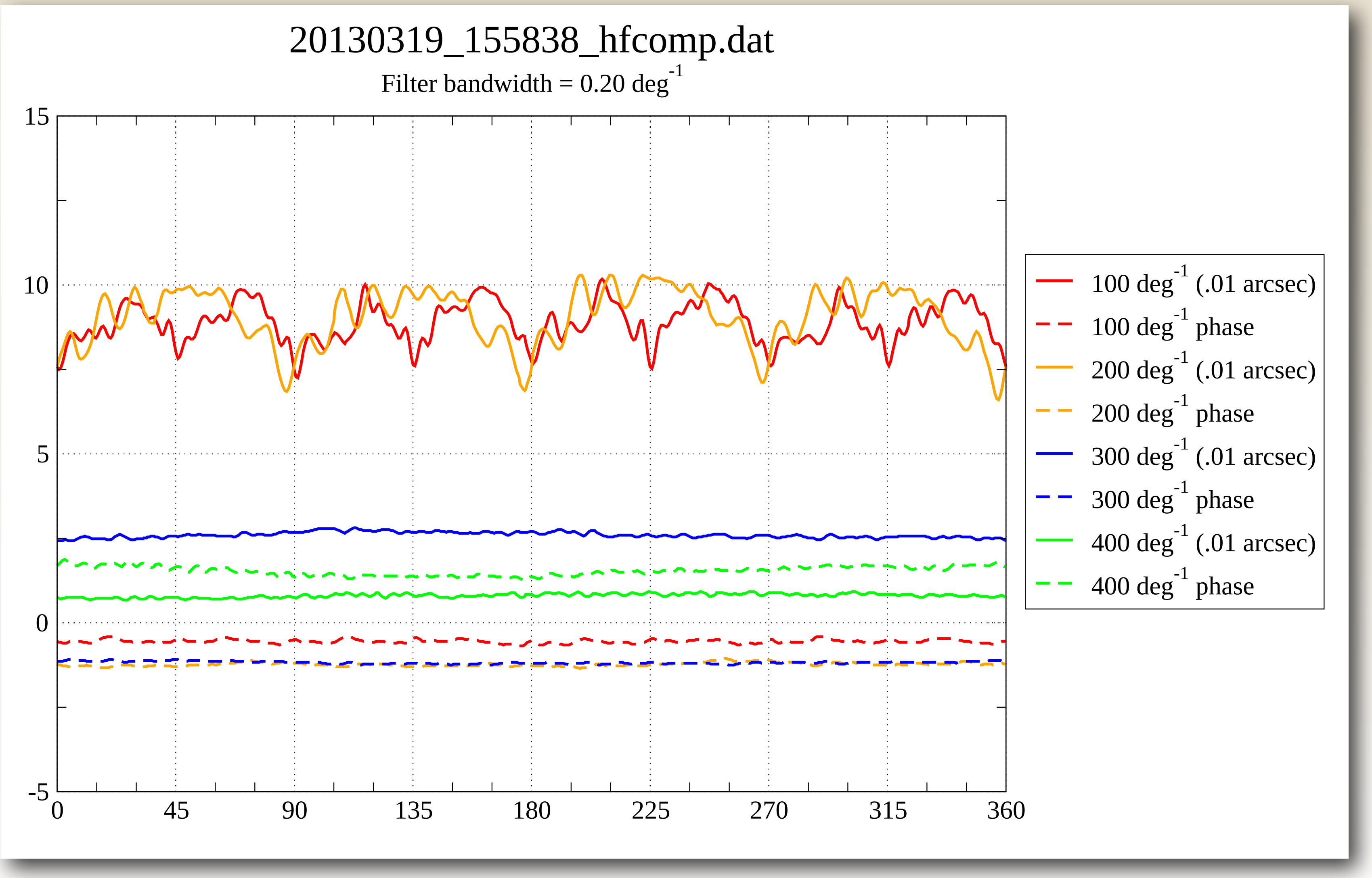
Example Fourier Spectrum

- 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

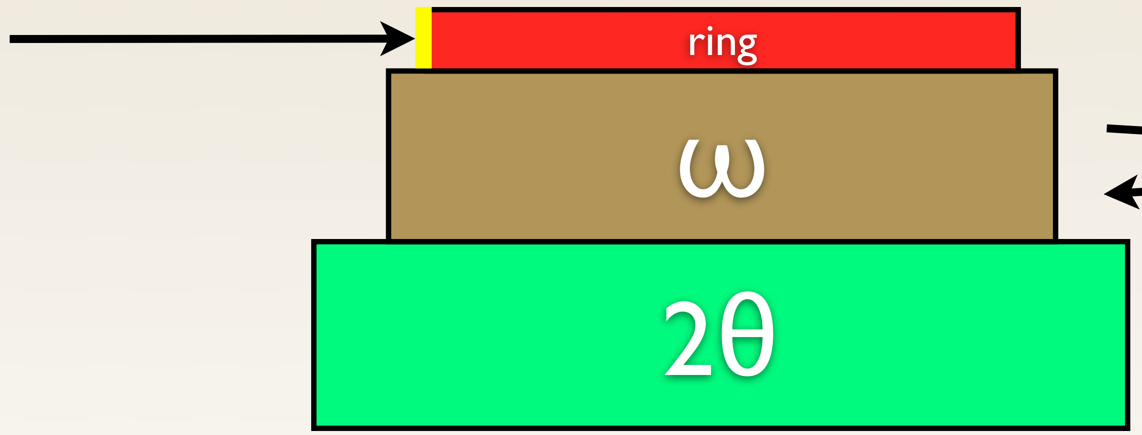
- Concept
  - compare sums of angles, subject to constraints that full circle is 360°
- Two general methods
  - use polygonal mirror on single stage to provide set of very stable angle offsets
  - use 'virtual polygon' and stacked stages and solve for offsets

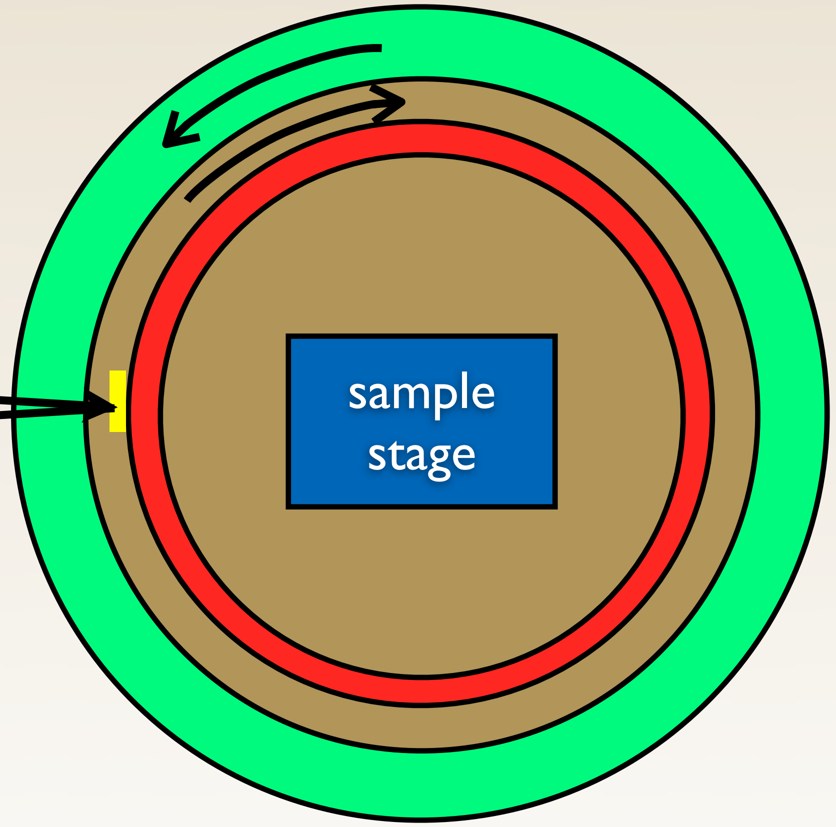
The Virtual Polygon

$$\theta_{mirror} = 2\theta + \omega + \theta_{ring} \equiv 0$$

$$2\theta_{meas} + \Delta 2\theta + \omega_{meas} + \Delta\omega + \theta_{ring} = 0$$

$$2\theta_{meas} + \omega_{meas} + \theta_{ring} = -\Delta 2\theta - \Delta\omega$$

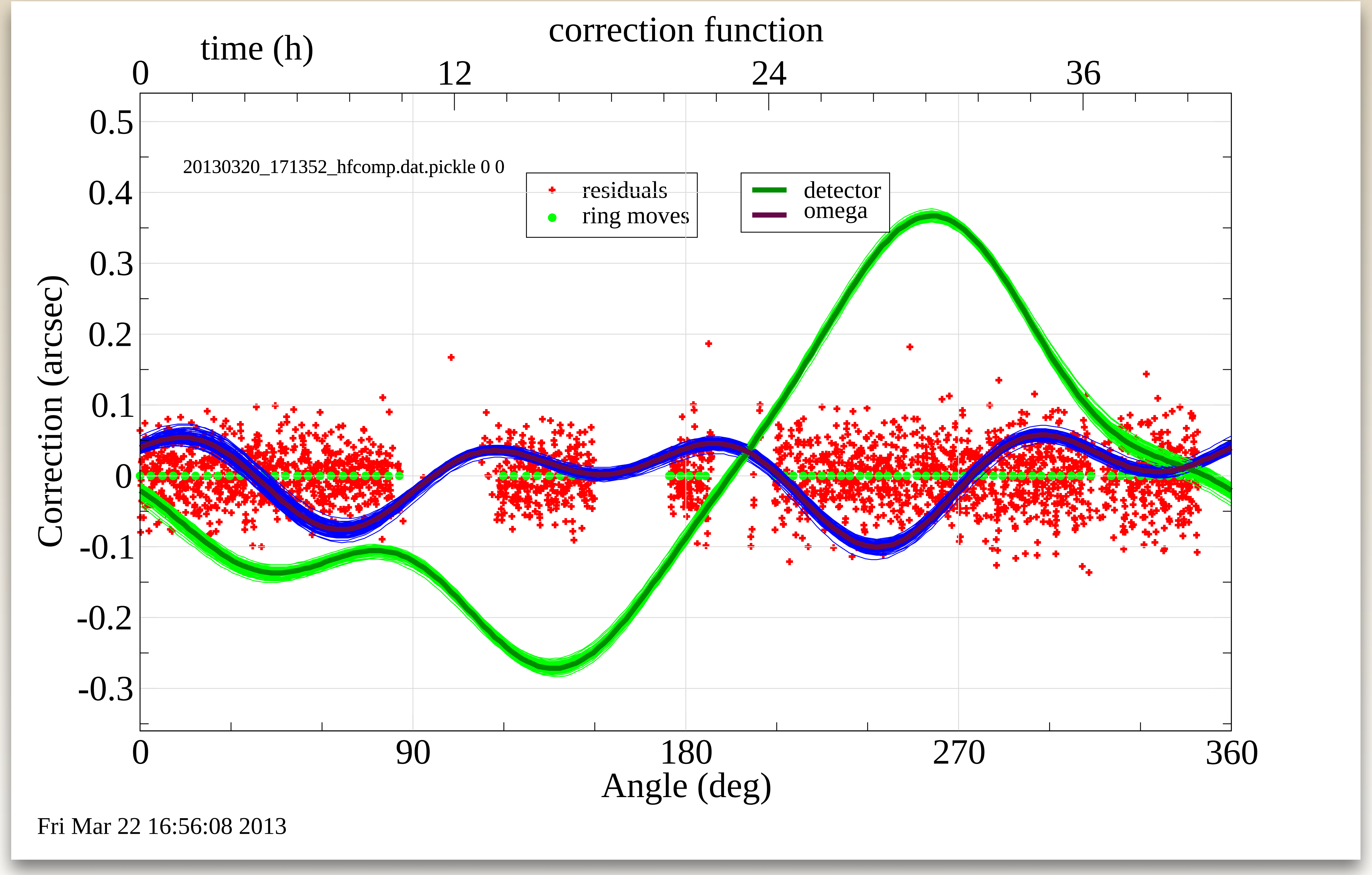




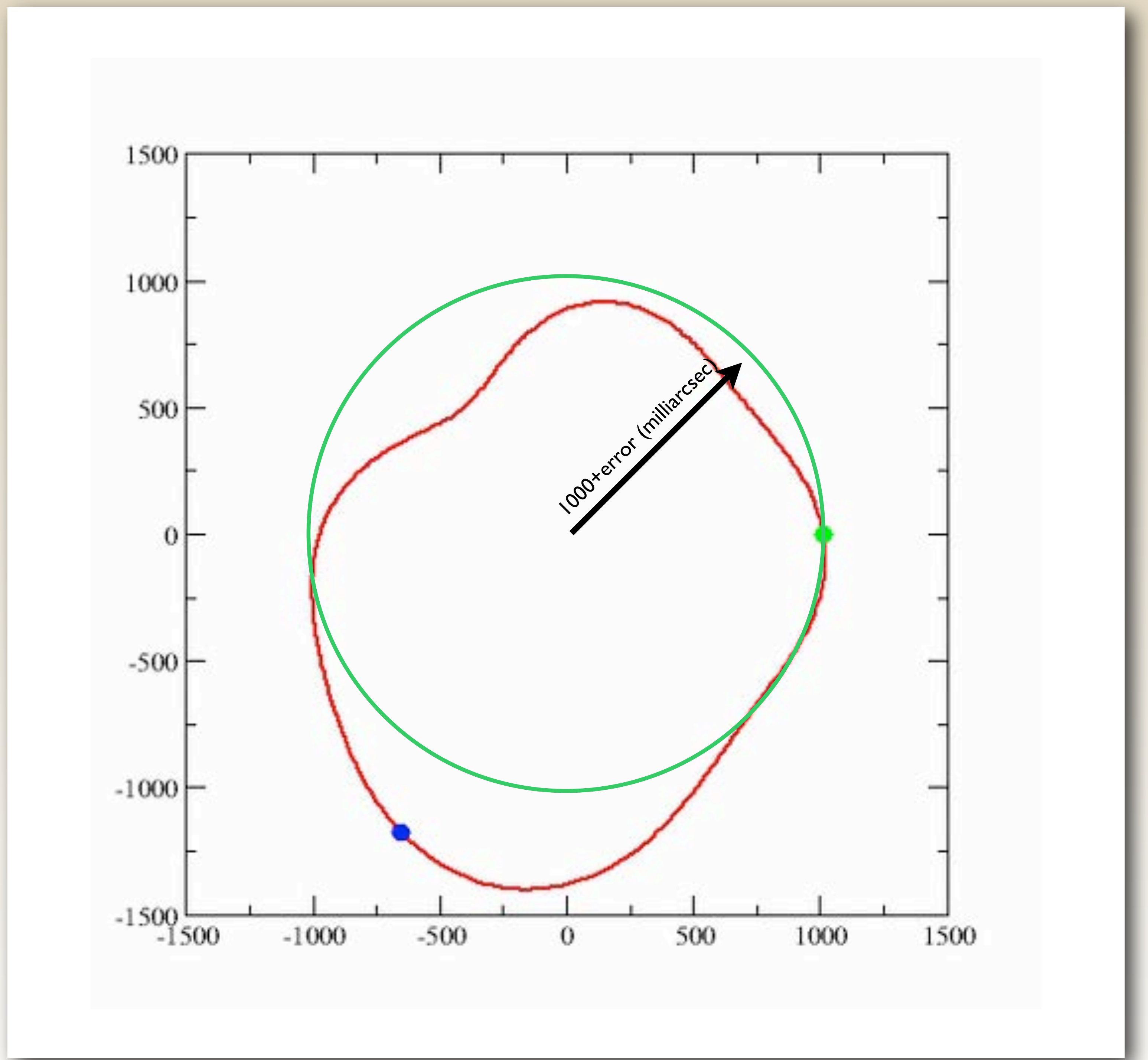
Details of Virtual Polygon

- 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 2θ values and corresponding ω values which null the autocollimator (n indexes ring, m indexes 2θ)
- repeat for at least 3 ring settings, to give enough degrees of freedom to solve for $\Delta 2\theta$, $\Delta\omega$, and the θr associated with each group.
- Do least squares fit for parameters

Circle Closure Results



Visualization of Error Sum



- ω=0
- 2θ=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