

GRAEME WINTER

CCP4 / DIAMOND WORKSHOP 2023

DATA COLLECTION STRATEGIES FOR PHOTON COUNTING DETECTORS

OVERVIEW

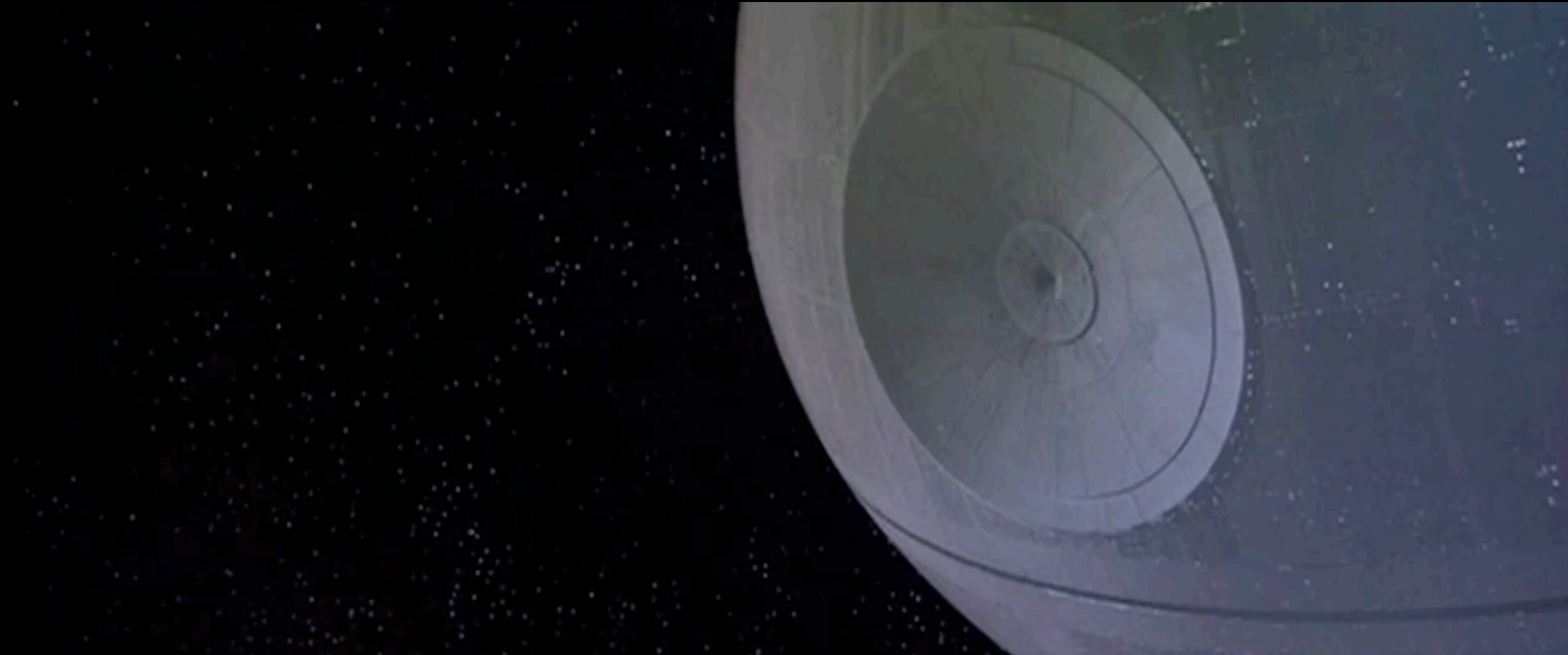
- Before we start advice: if you remember one thing... (and one more thing)
- Data collection strategies
- Detectors
- Photon counting detectors
- Diffraction data collection advice
- Tactical suggestions
- Conclusions

IF YOU ARE GOING TO REMEMBER ONE THING...

Ask your beamline scientist / local contact for advice

“What would you suggest?”

AND IF YOU CAN REMEMBER TWO THINGS



DON'T BE DARTH

DATA COLLECTION STRATEGIES

GOALS FOR DATA COLLECTION

- Measure all the unique reflections
- Measure enough copies that you can assess how well they agree / determine symmetry etc.
- Maximise the “quality” of the signal you record
- Minimise the radiation damage

UNIQUE REFLECTIONS

- Whole volume of reciprocal space “divided by” the crystal symmetry
- For high symmetry crystals can get away with a relatively small rotation to get complete data
- For low symmetry data may need much more
- More measurements of reflections also improves the quality of the average

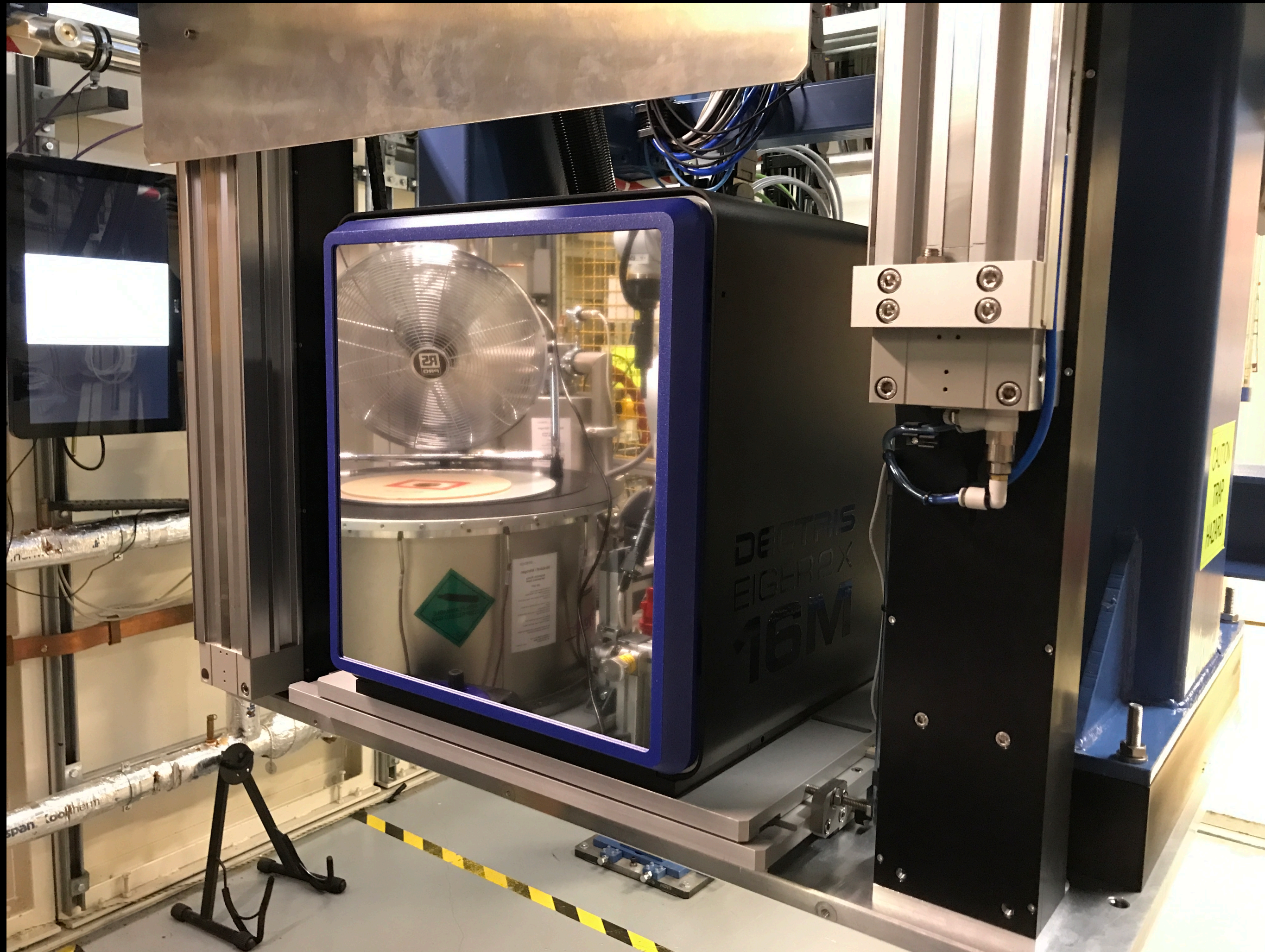
DETECTORS

- Convert X-ray photons into some sort of “intensity value”
- Started off with photographic film (data processing involved chemicals, darkroom)
- Most likely to encounter CCD or photon counting / integrating pixel array detectors
- No detector is perfect but they are very good - knowing the properties will help you to collect good data

COMMON DETECTOR TYPES

- Integrating:
 - Not worried about photons / second, total photons / pixel problem
 - Read-out noise, dark current
 - Potentially slow (CCD) - 0.5s / frame read out time
- Photon counting
 - Limited photons / second rate
 - No read-out noise or dark current
 - Substantially faster than CCD - allows shutterless collection
 - Nuances relating to charge sharing etc.

PHOTON COUNTING DETECTORS



PHOTON COUNTING DETECTORS

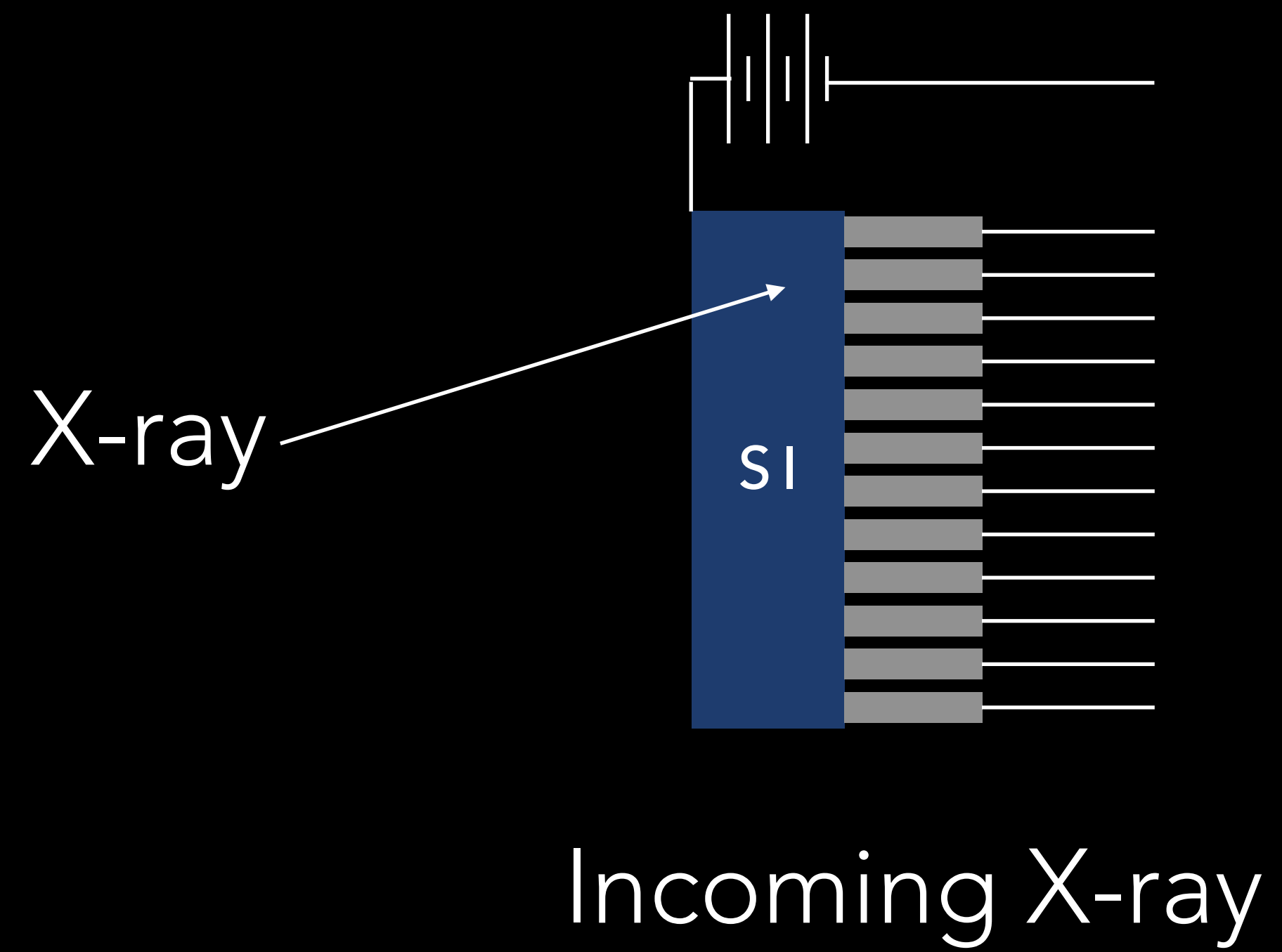
- Direct conversion of photon energy to charge in sensor - derived from sensors from particle physics
- Readout electronics for every pixel (e.g. unlike CCD)
- Enables shutterless data collection as readout is (near-)instant (ms to μ s)
- Dectris PILATUS and EIGER most common

PHOTON COUNTING DETECTORS

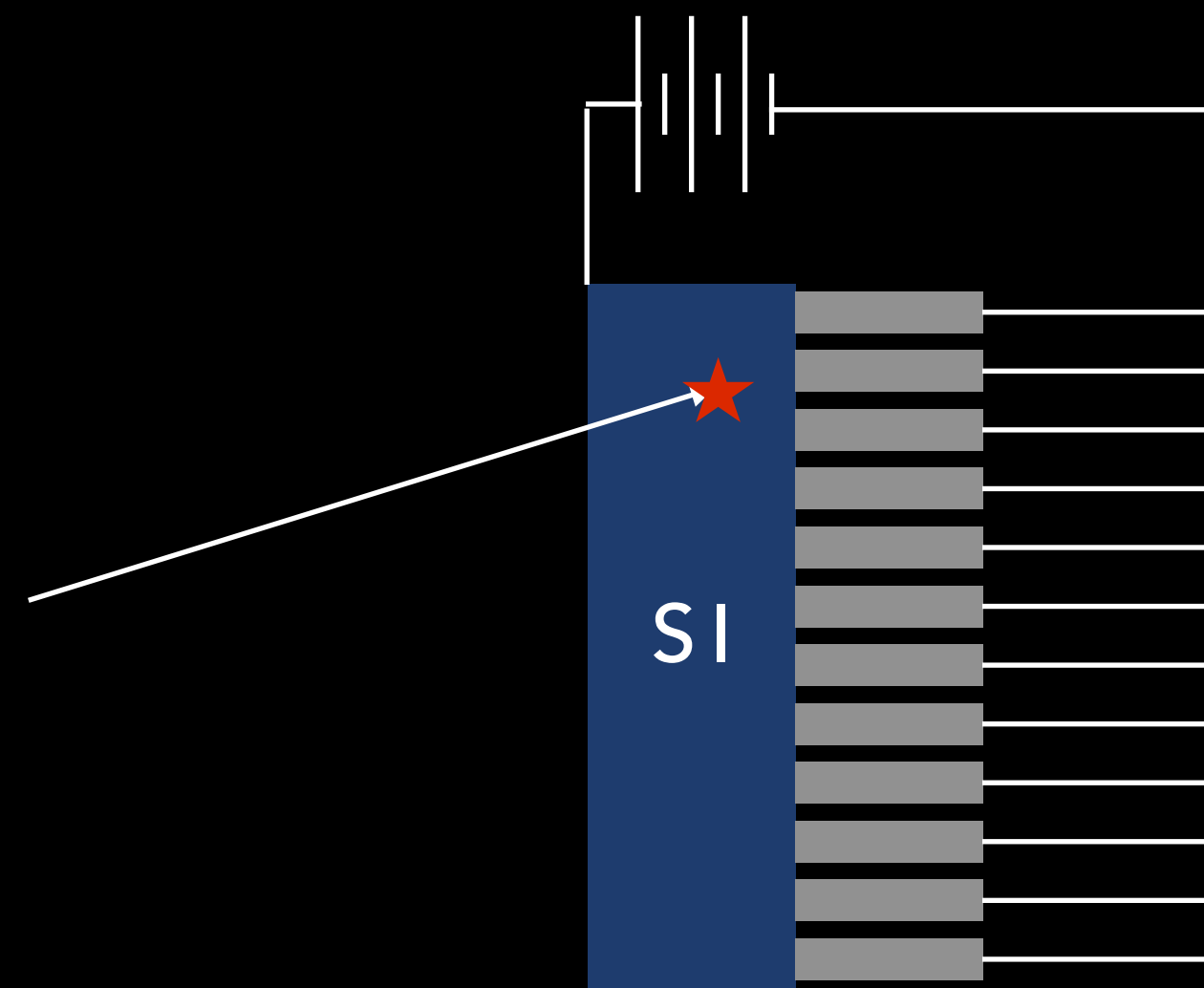


Detector: Si sensor material connected
to readout electronics - large potential
difference across sensor

PHOTON COUNTING DETECTORS



PHOTON COUNTING DETECTORS



X-ray ionizes Si atom

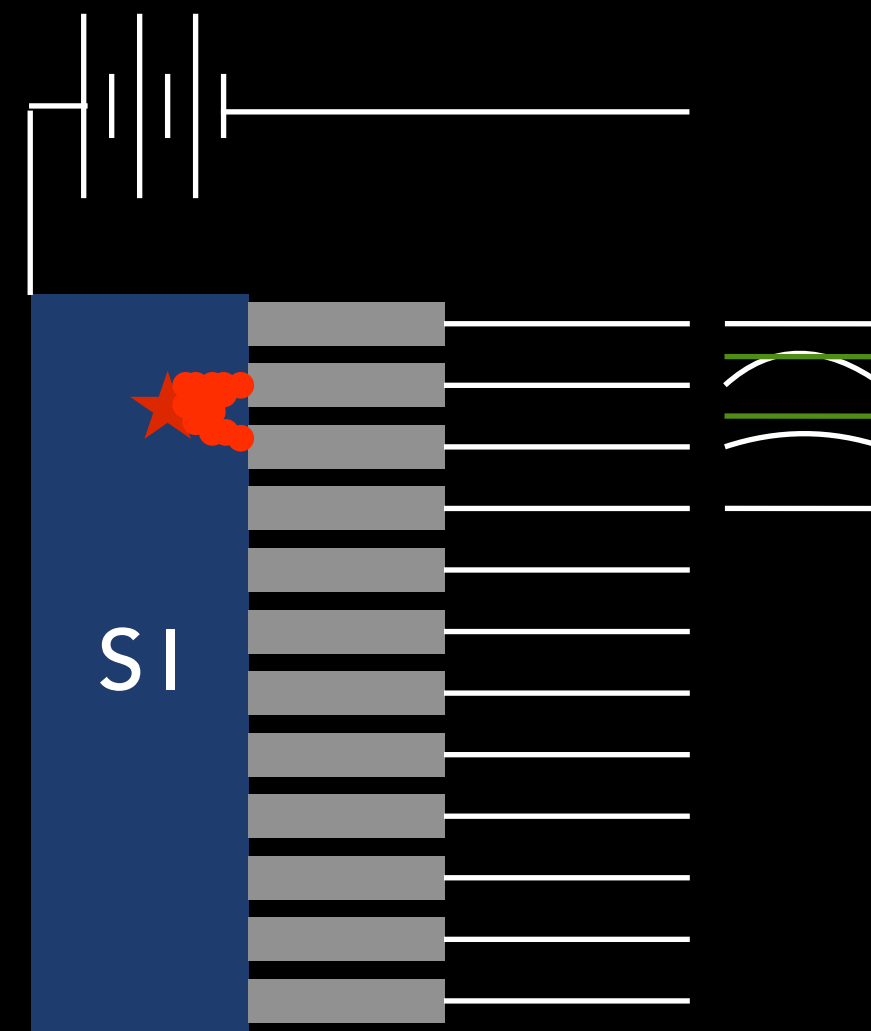
PHOTON COUNTING DETECTORS



e- cascade

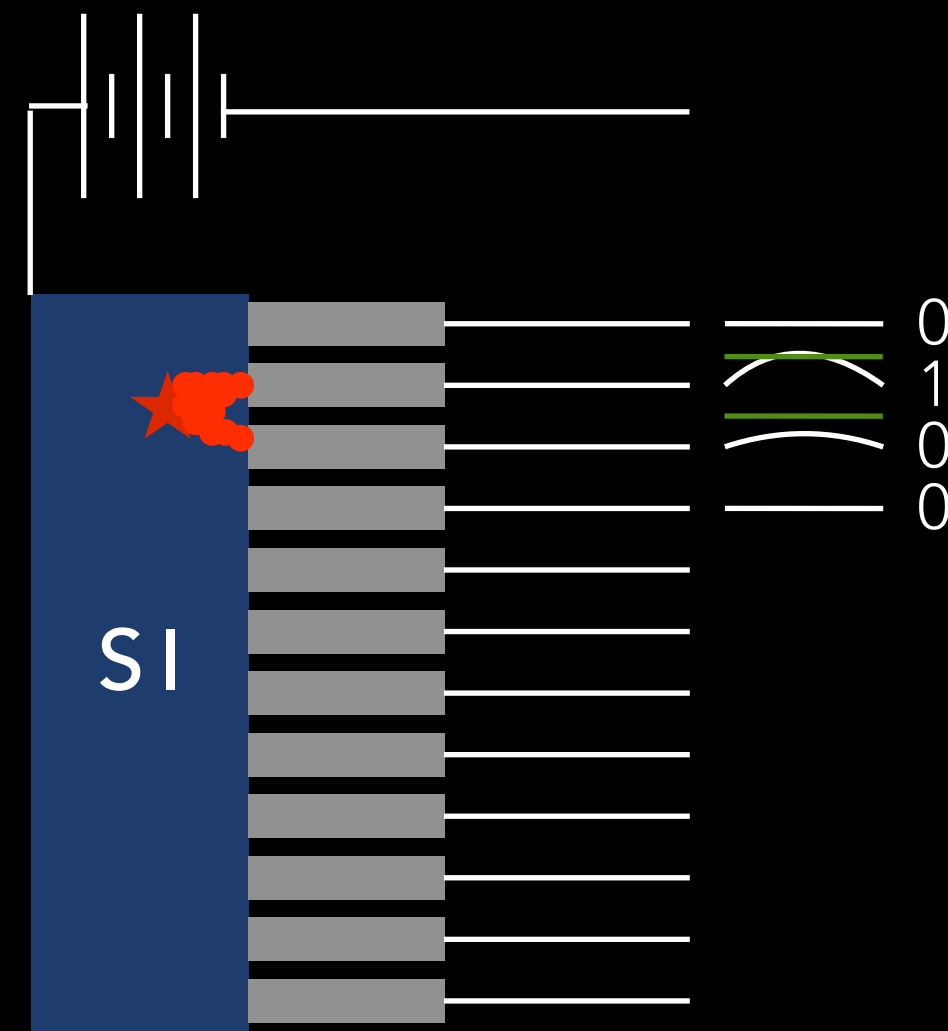
(3500 e-/hole pairs for
12.7 keV photon)

PHOTON COUNTING DETECTORS



Integrated current at
anode \sim charge

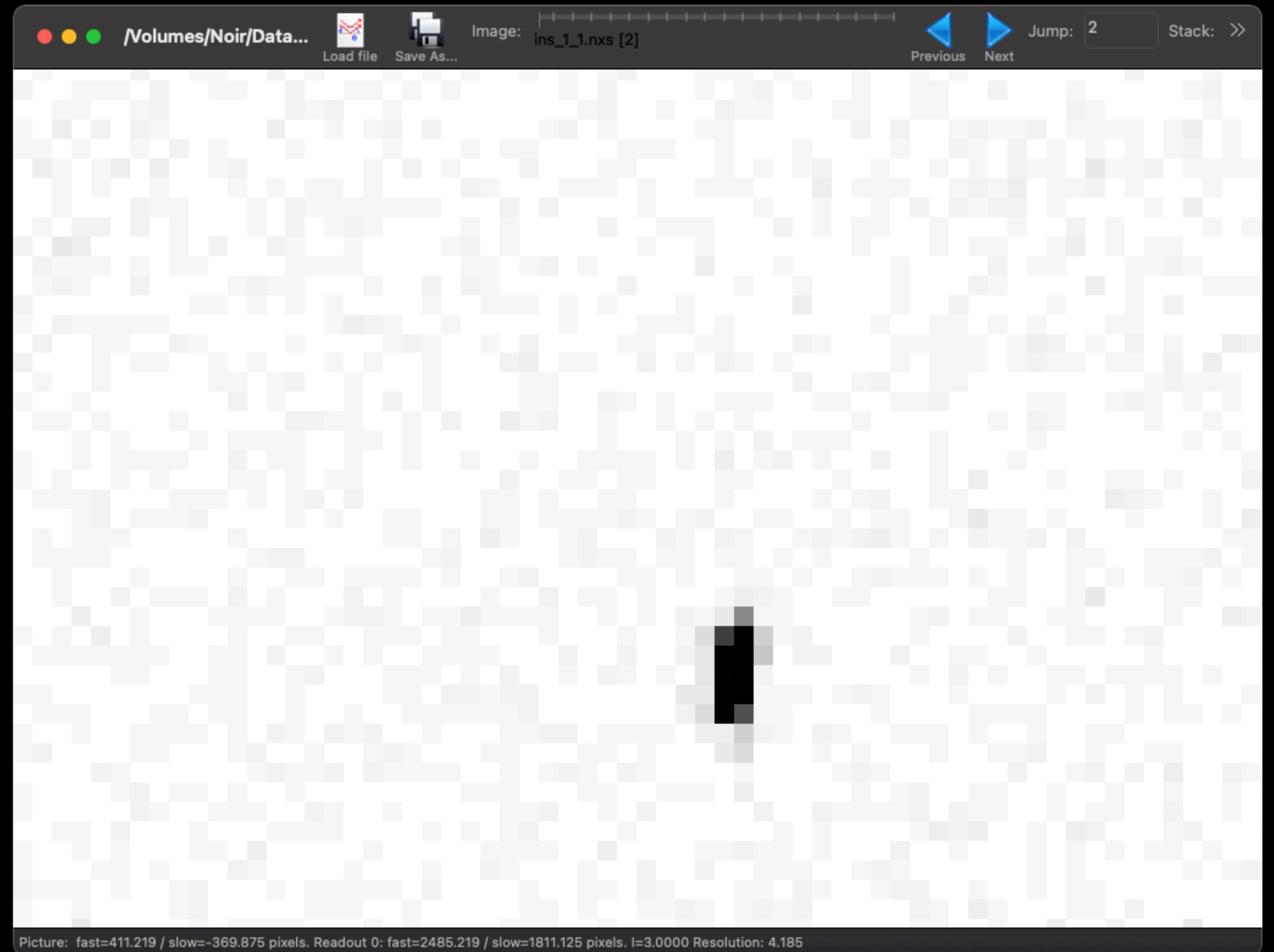
PHOTON COUNTING DETECTORS



Total charge $>$ threshold
(typically 0.5 photon)

PHOTON COUNTING BACKGROUND

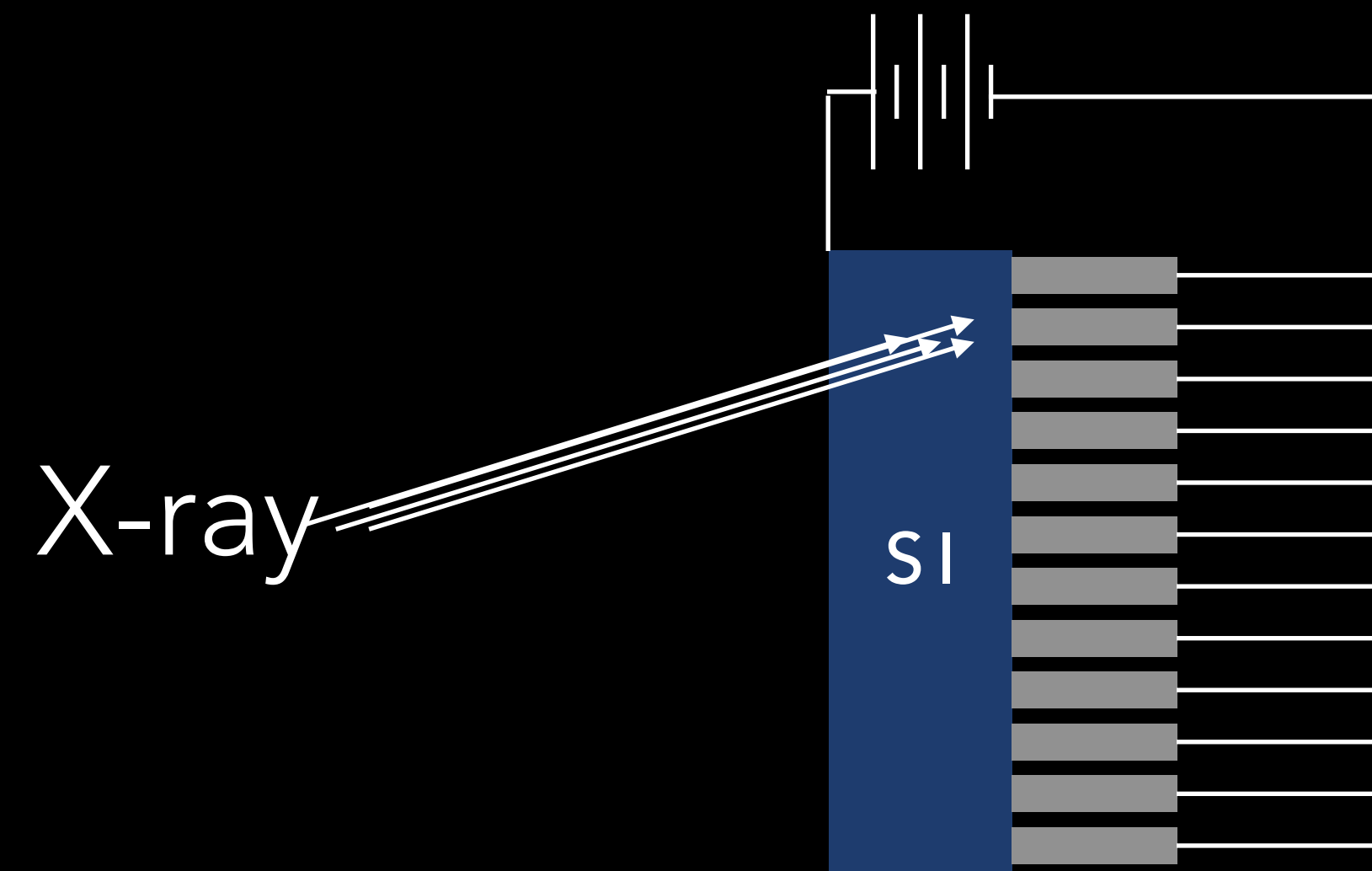
- Thresholding eliminates read-out noise
- Background caused by atoms in beam which are not crystal
- Well mounted samples will have lower background
- Poorly mounted, or in situ, may have high background



DETECTOR PARAMETERS

- Pilatus: 172 μm square pixels, 320 μm to 1mm thick
- Eiger: 75 μm square pixels, typically 450 μm thick
- Material: Si common, good at lower energies, somewhat transparent at high energy, CdTe becoming more common, excellent at high energy (> 20 keV)
- Pixels are very "small and deep" so spots can smear particularly at higher energies as photons pass through the sensor material

PHOTON COUNTING DETECTORS - "PILEUP"



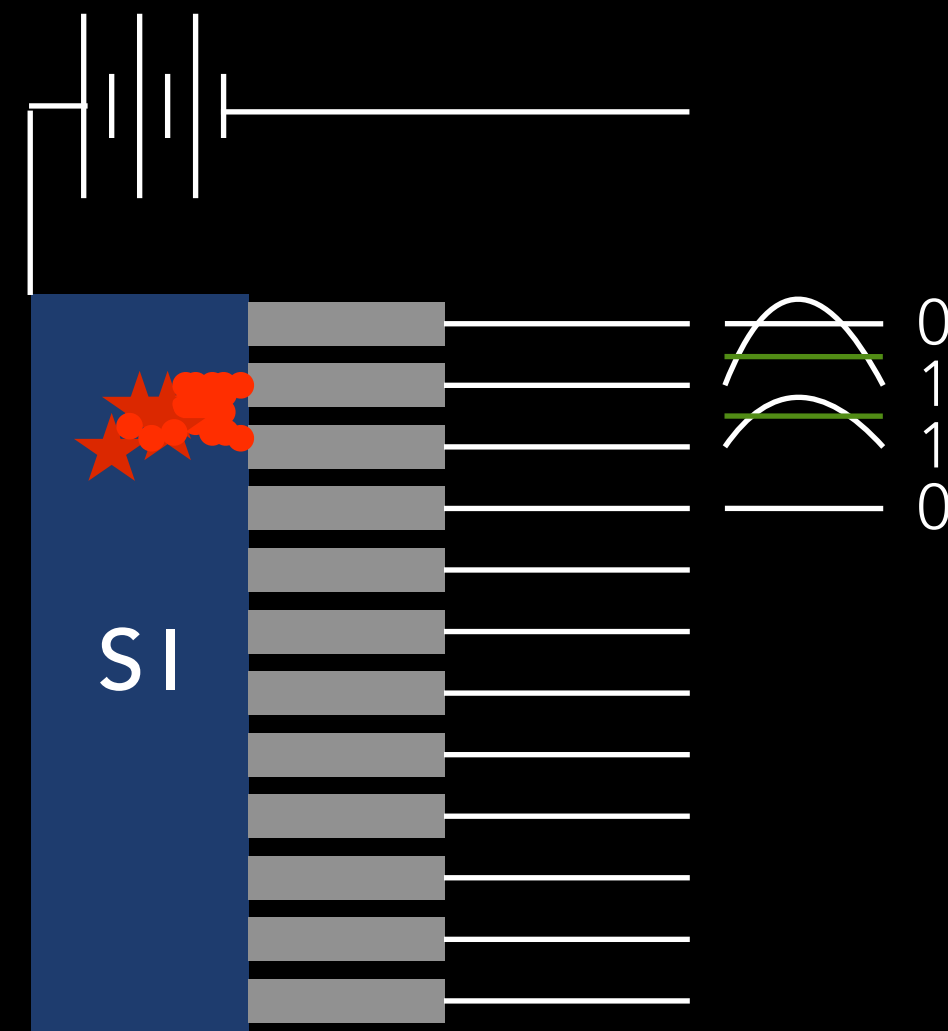
Many photons arrive
within detection interval

PHOTON COUNTING DETECTORS - "PILEUP"



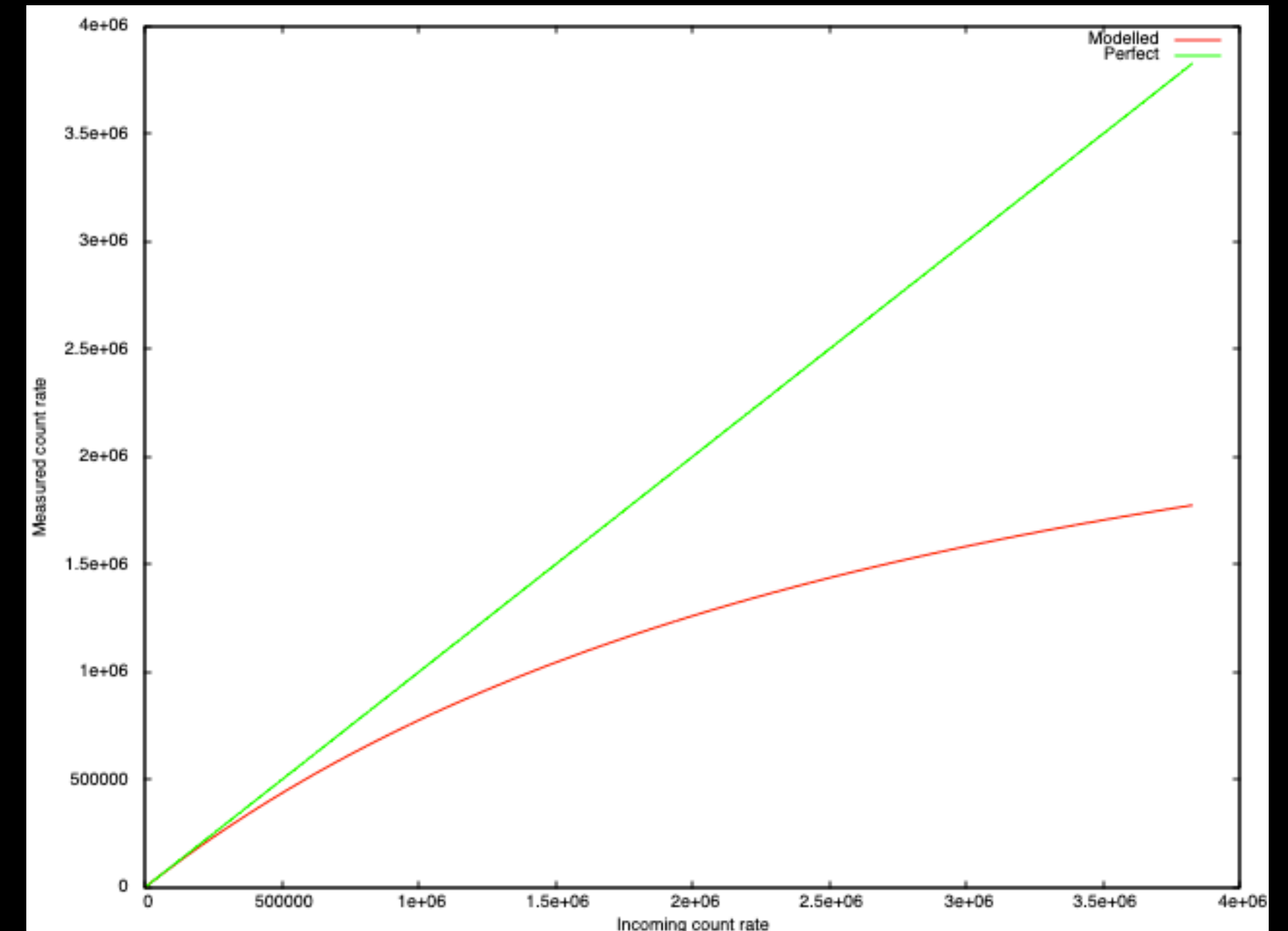
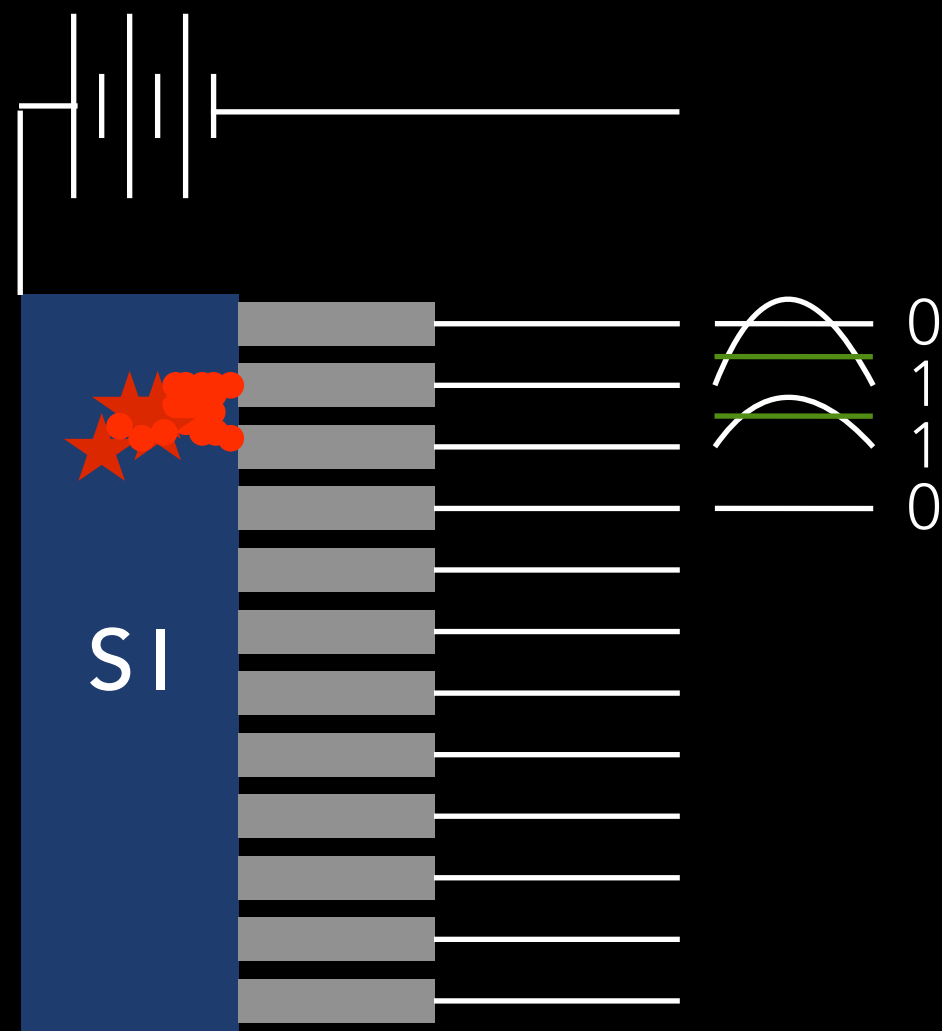
Much ionisation

PHOTON COUNTING DETECTORS - "PILEUP"



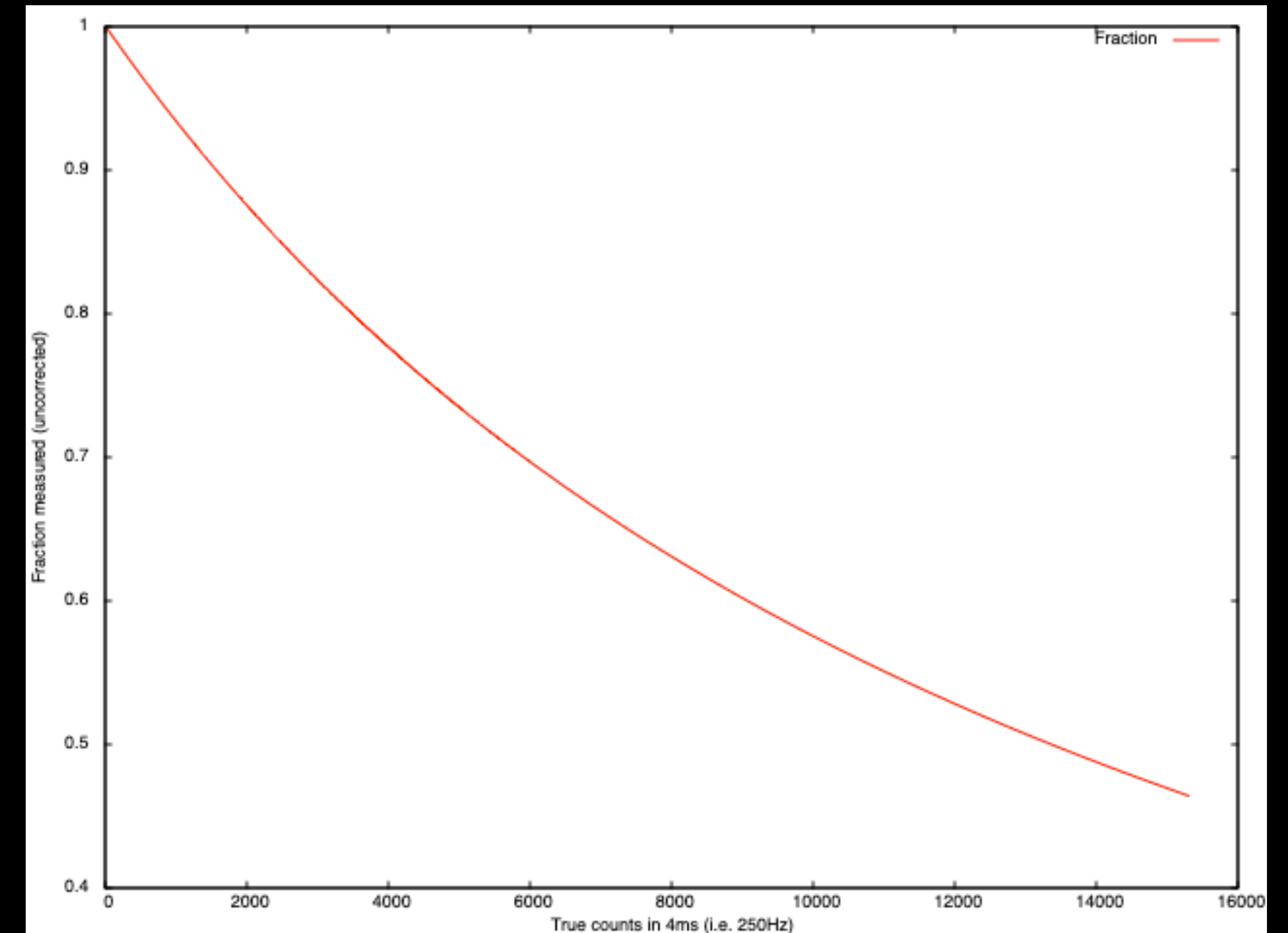
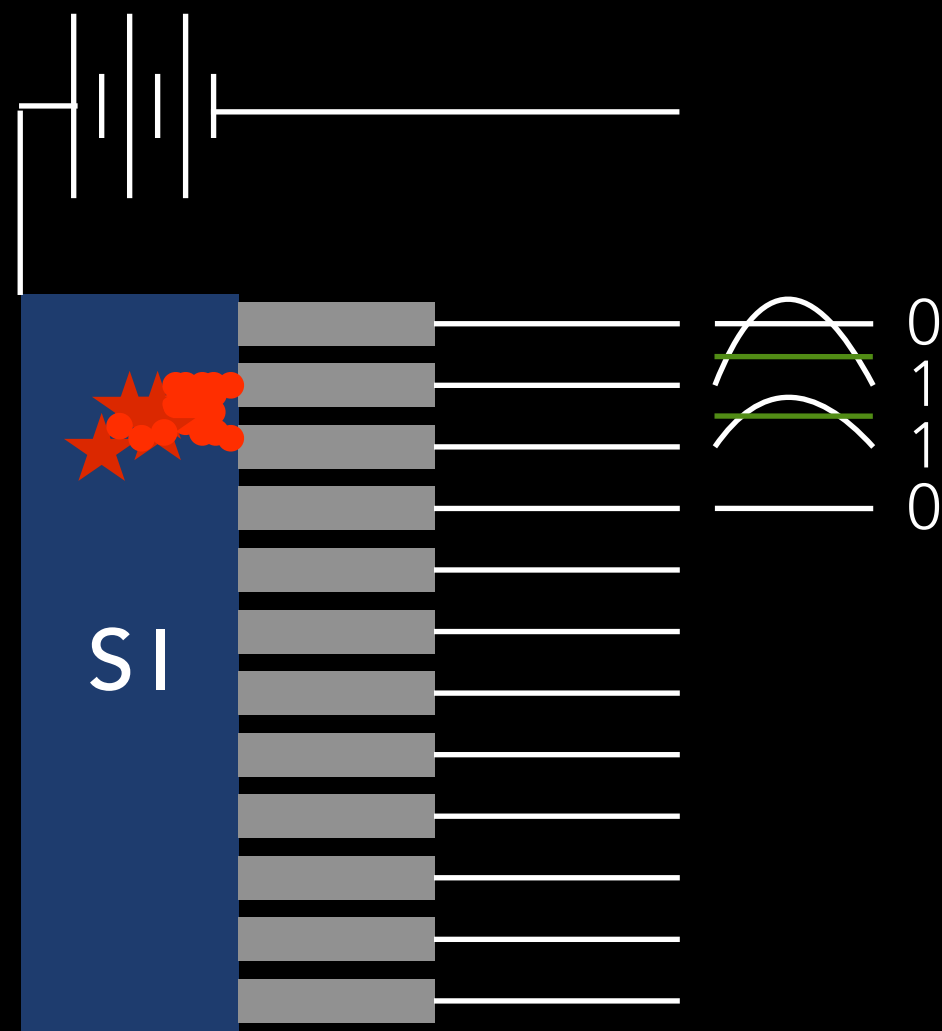
Lots of charge - but still
only passes above
threshold so 1 count

PHOTON COUNTING DETECTORS - "PILEUP"



Incoming vs. uncorrected count rate
(photons / s / pixel)

PHOTON COUNTING DETECTORS - "PILEUP"

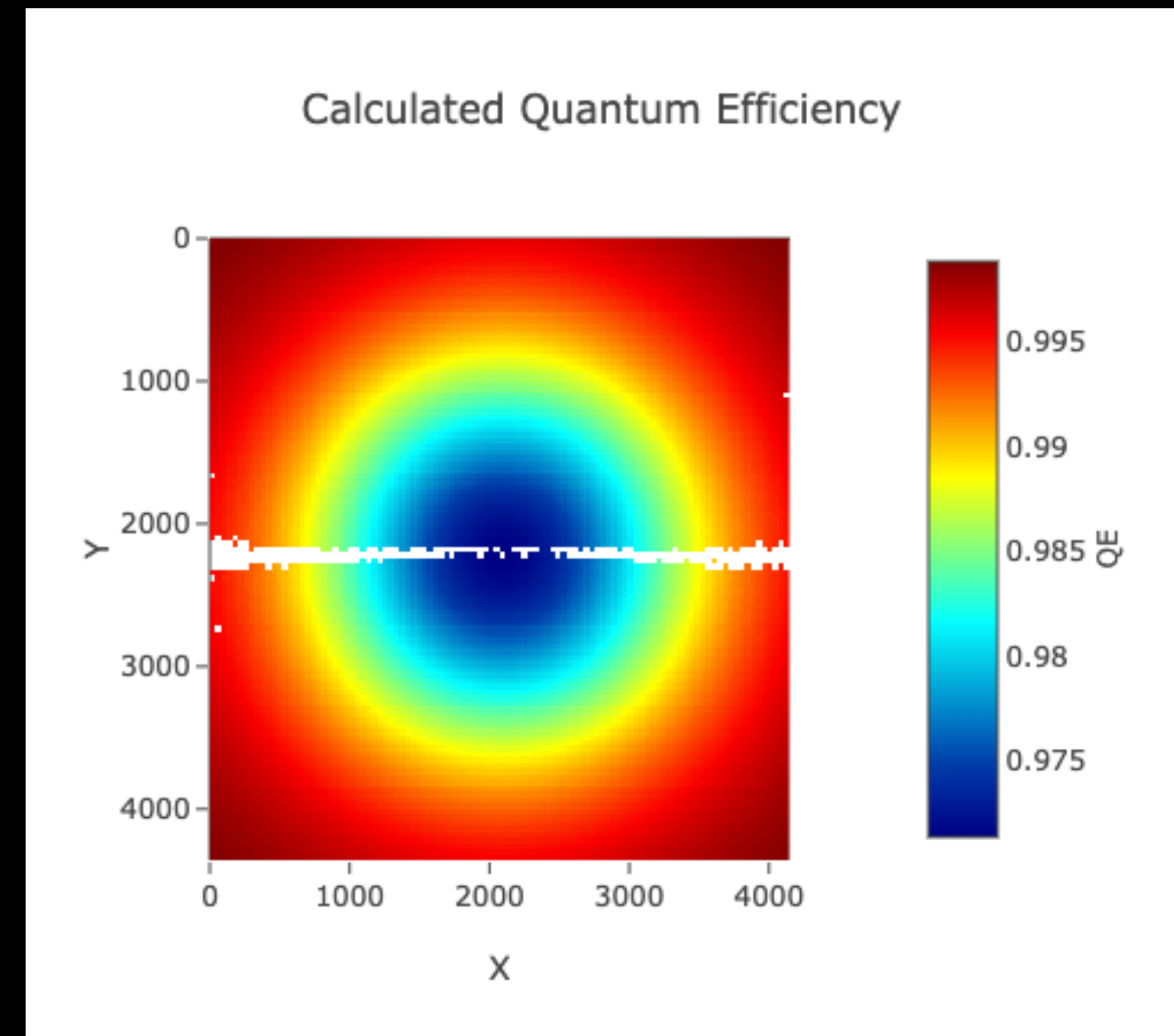
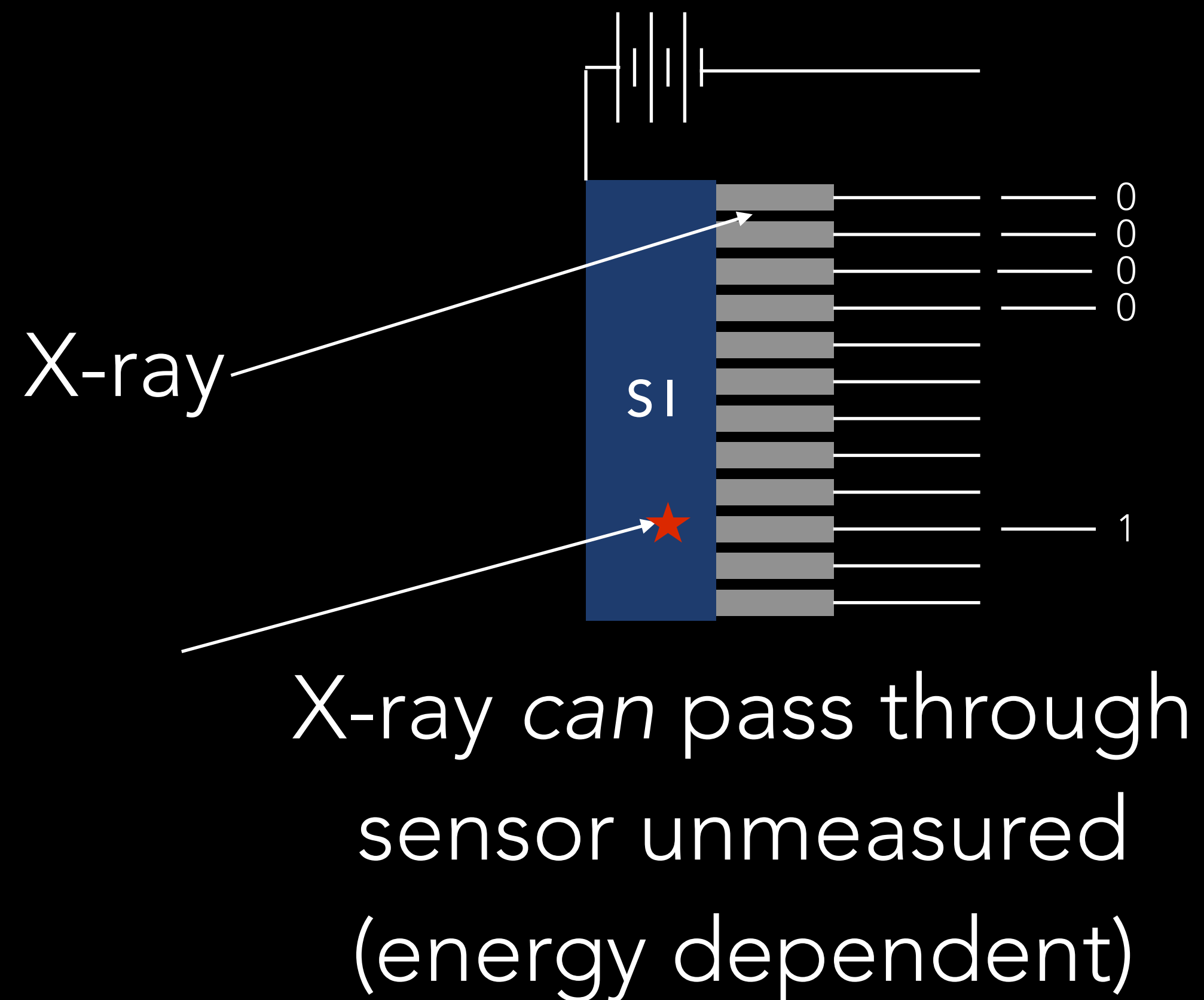


Fraction recorded (uncorrected) vs. pixel count in 4ms

KEY POINTS

- This *is* corrected for but that correction assumes constant count rate in pixel
- If you have finely sliced data, this assumption is probably sound
- If you have recorded data with rotation \gg mosaic spread the assumption is probably rather poor (more on this later)

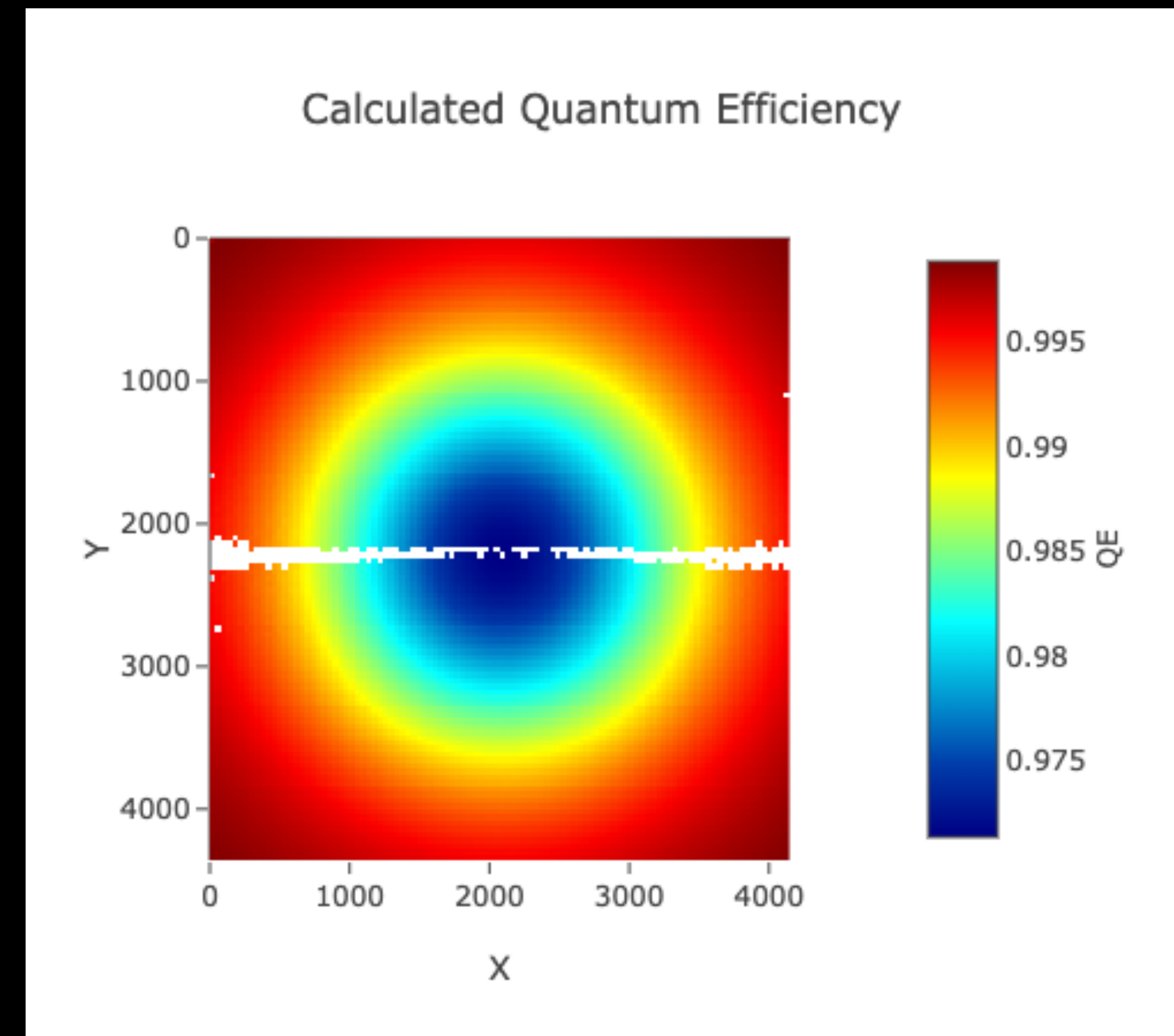
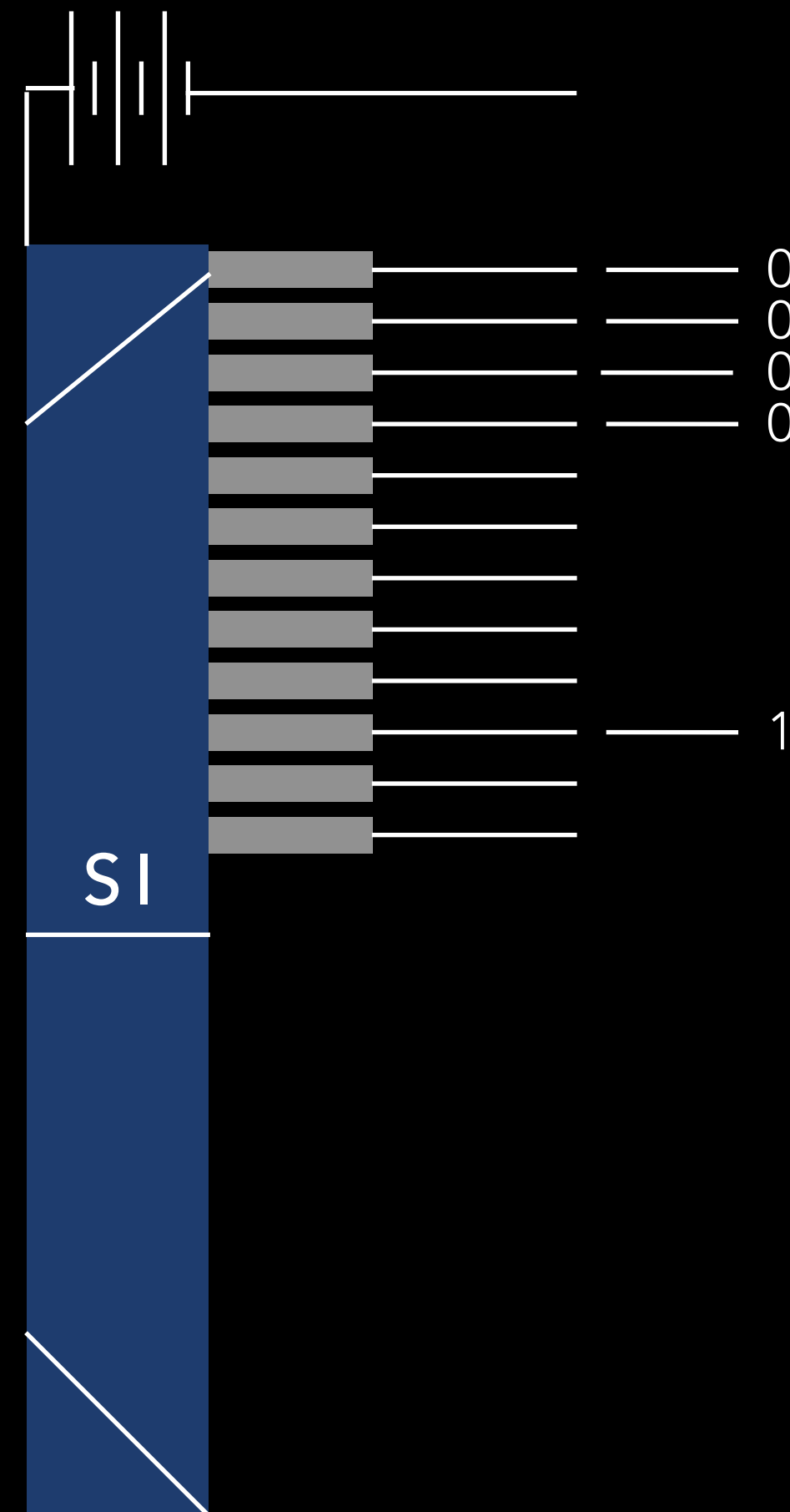
PHOTON COUNTING DETECTORS - EFFICIENCY



i03 Eiger at 10keV

PHOTON COUNTING DETECTORS - EFFICIENCY

Path length through
sensor longer
around edges of
detector than middle



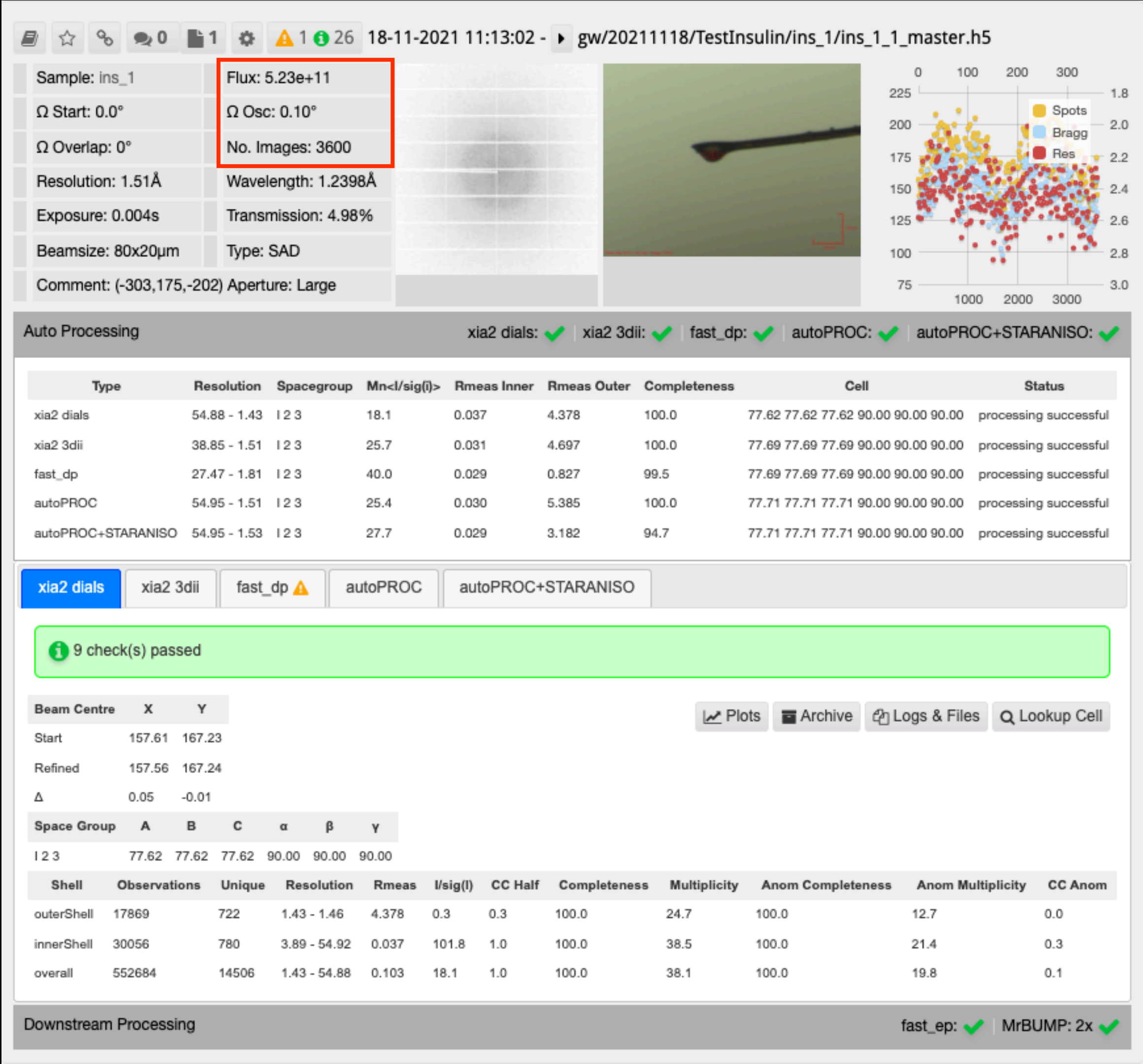
i03 Eiger at 10keV

IDEA #1

- Be aware of saturating the detector
- It is quite hard, but a good crystal on a bright beamline is able to do this
- It is hard to see if this has happened
- For a photon counting detector, too weak is rarely a problem
- Be aware of detector efficiency at higher energies
- Talking to beamline staff a good way to go

DATA INTENSITY / MULTIPLICITY

- The “quality” of data depends on many factors - including the individual spot intensity and number of observations
- Measuring twice as many copies of data half as strong “averages out” to give essentially the same data - with a photon counting detector
- Twice as many, half as strong, measurements also gives opportunity to truncate the data if damage is present



x4
/4

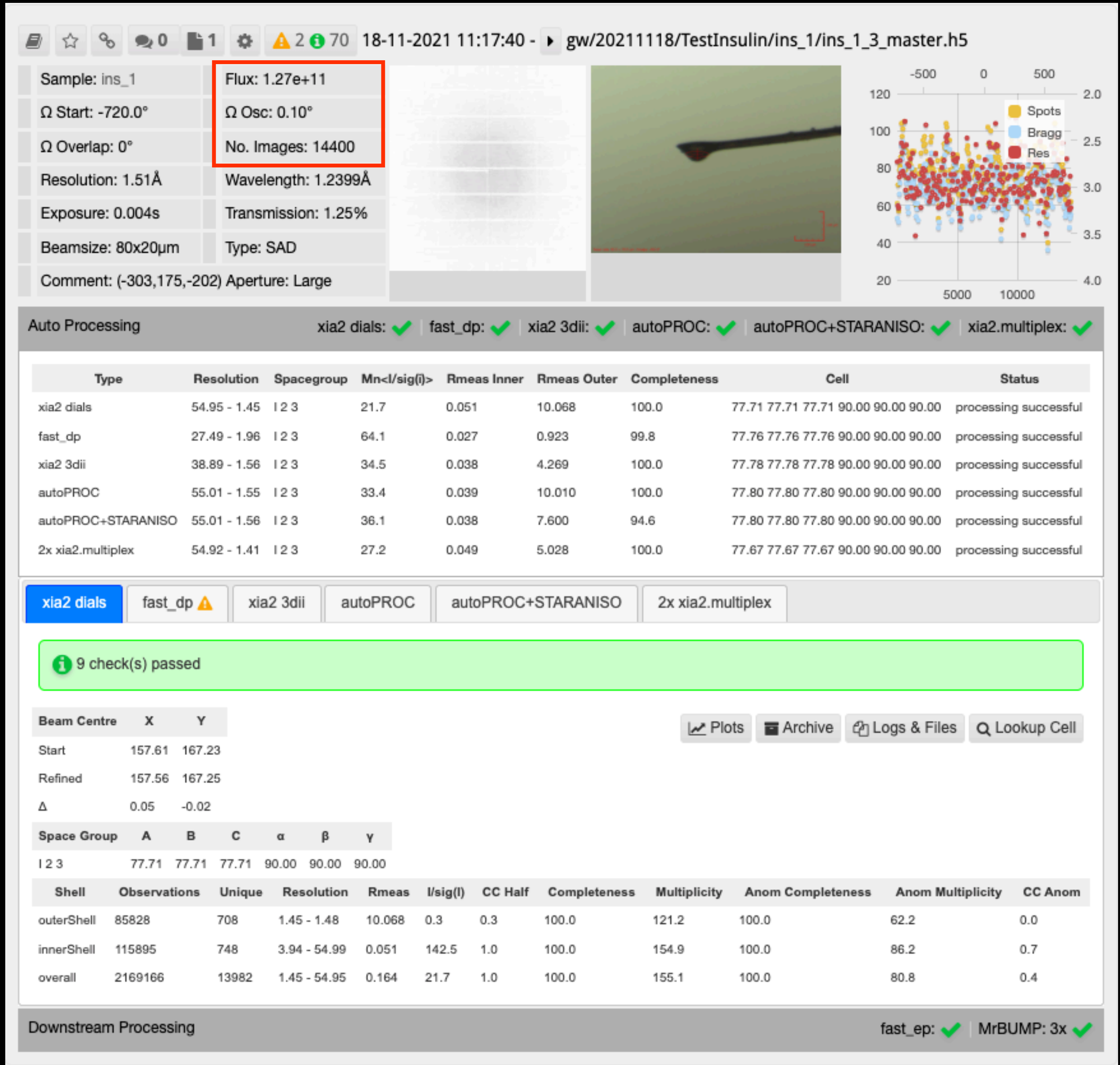
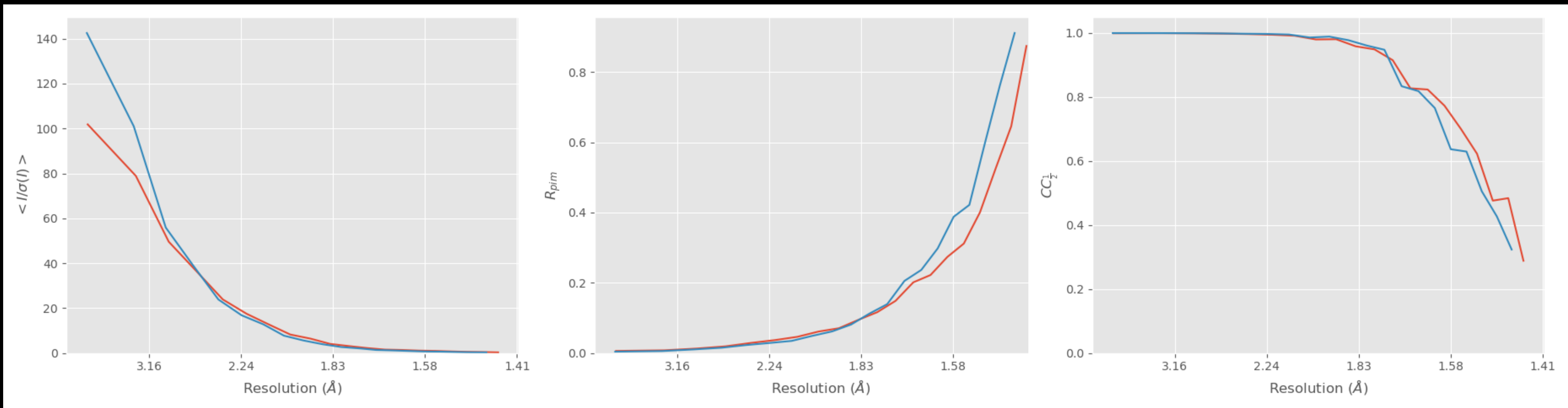
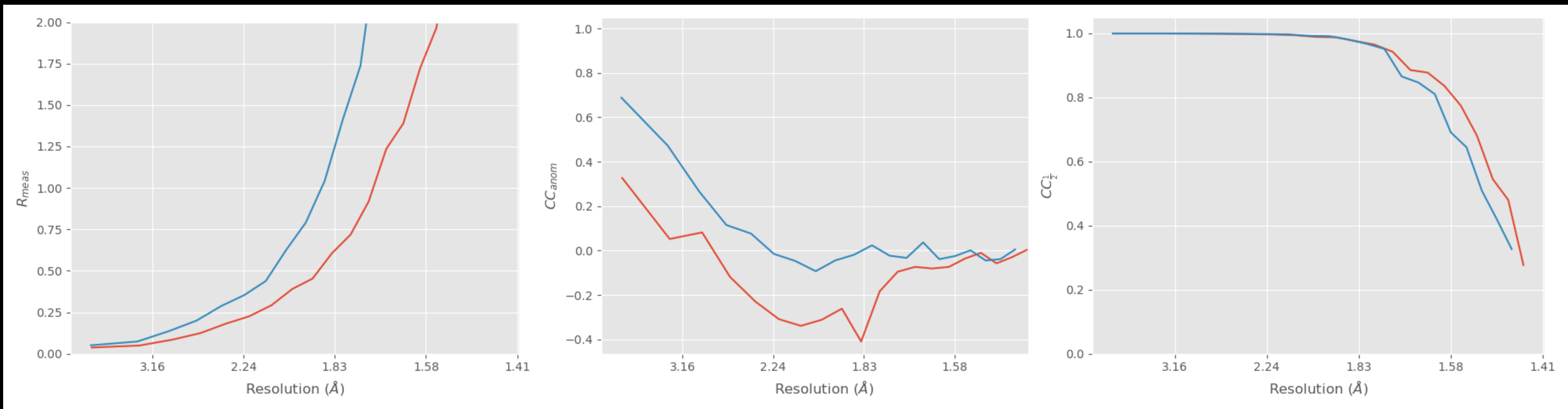


TABLE 1 NOT SO HELPFUL

| Overall | | | |
|--------------------|--------------|----------------|-----------------|
| | Overall | Low resolution | High resolution |
| Resolution (Å) | 54.88 - 1.43 | 54.92 - 3.89 | 1.46 - 1.43 |
| Observations | 552684 | 30056 | 17869 |
| Unique reflections | 14506 | 780 | 722 |
| Multiplicity | 38.1 | 38.5 | 24.7 |
| Completeness | 100.00% | 100.00% | 100.00% |
| Mean I/σ(I) | 18.1 | 101.8 | 0.3 |
| R _{merge} | 0.102 | 0.037 | 4.289 |
| R _{meas} | 0.103 | 0.037 | 4.378 |
| R _{pim} | 0.017 | 0.006 | 0.874 |
| CC _½ | 1.000 | 0.999 | 0.288 |

| Overall | | | |
|--------------------|--------------|----------------|-----------------|
| | Overall | Low resolution | High resolution |
| Resolution (Å) | 54.95 - 1.45 | 54.99 - 3.94 | 1.48 - 1.45 |
| Observations | 2169166 | 115895 | 85828 |
| Unique reflections | 13982 | 748 | 708 |
| Multiplicity | 155.1 | 154.9 | 121.2 |
| Completeness | 100.00% | 100.00% | 100.00% |
| Mean I/σ(I) | 21.7 | 142.5 | 0.3 |
| R _{merge} | 0.164 | 0.051 | 10.026 |
| R _{meas} | 0.164 | 0.051 | 10.068 |
| R _{pim} | 0.013 | 0.004 | 0.911 |
| CC _½ | 1.000 | 1.000 | 0.324 |



IDEA #2

- Measure more data with less transmission (at least until you know your samples well)
- Easy way to achieve this is stepped transmission experiment - start off weak, then 4x dose, 4x dose etc. (4 x should increase $1/\sigma(I)$ by ~ 2)
- Once you know your sample lifetimes, collect more data that are weak to give room for truncation and reduce impact of count rate on data

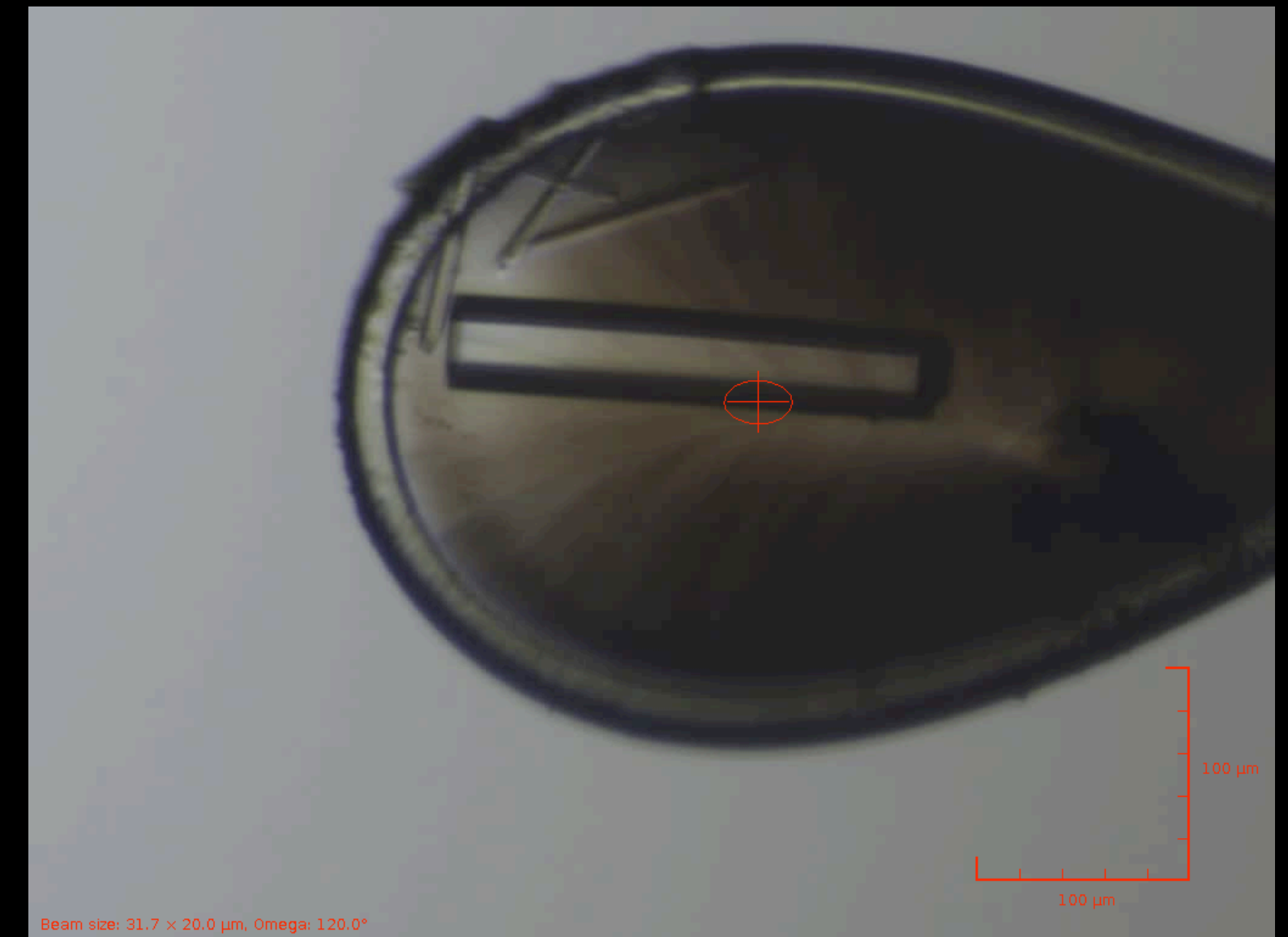
COLLECTION ADVICE

So I am collecting gently, collecting high multiplicity data, what now?

- Background: ideally minimise this - mount sample carefully
- Also collect data carefully - for example sample alignment

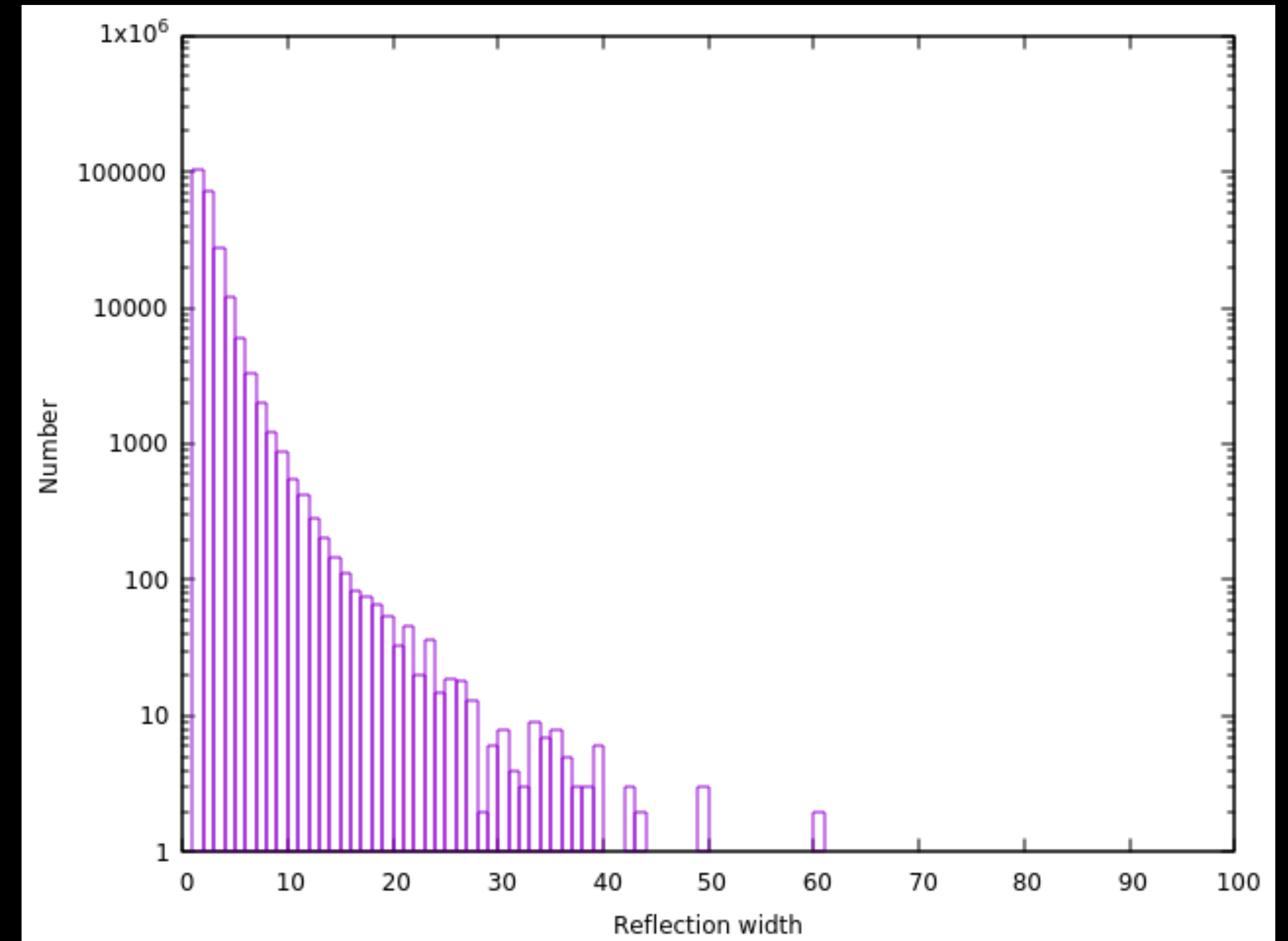
SAMPLE MOUNTING

- Use suitable mount - similar size and ideally shape as sample
- Ideally only one sample
- Less liquid means better cooling (less ice) and lower background



MINIMISE BACKGROUND

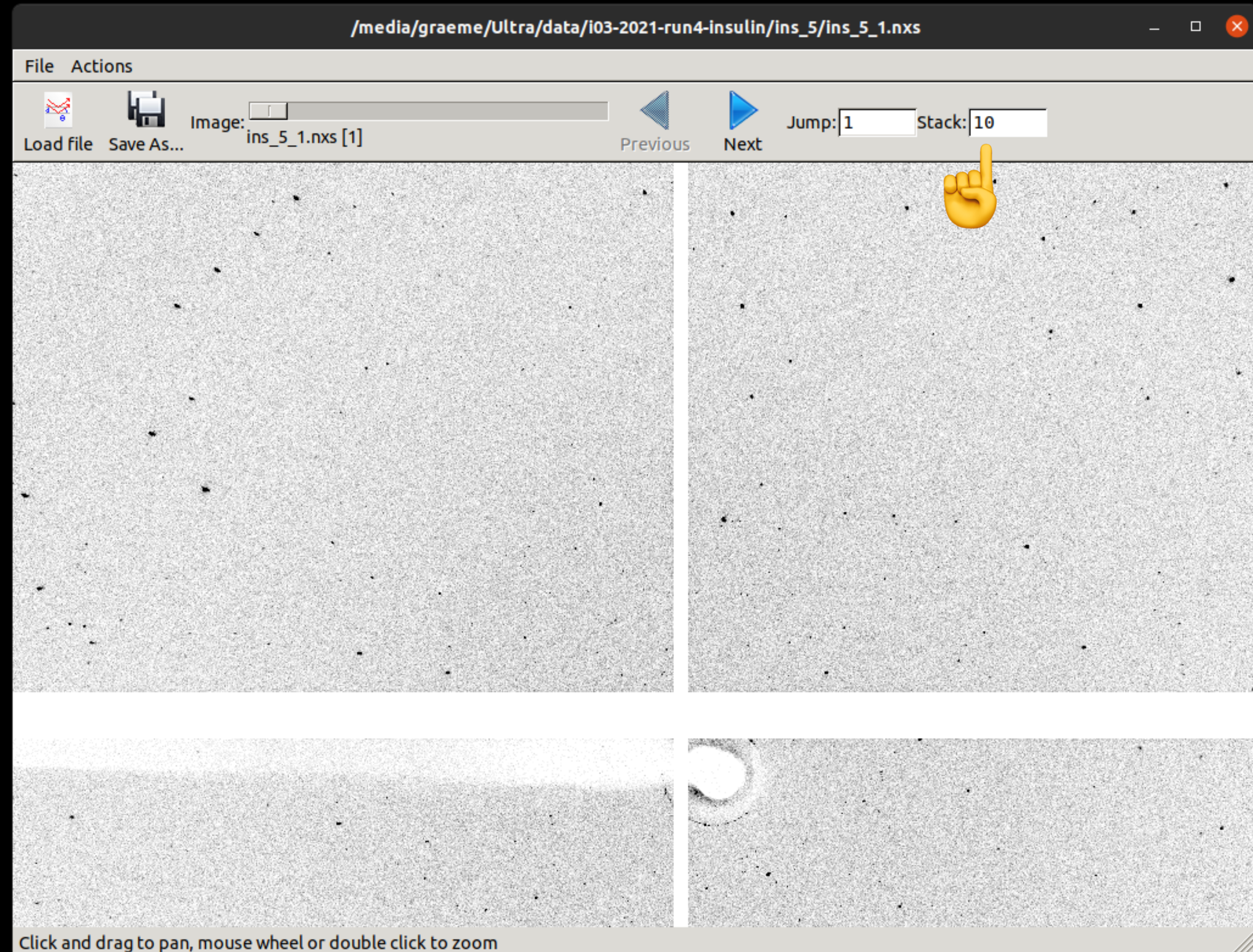
- The background is under the spot
- If we keep the rotation narrow, we have less background “before and after” the spot
- Strong spots taken from spot finding - most are rather narrow even with 0.1° images (median / mean ~ 2 images)
- 1° vs. 0.1° would have 10x background



Histogram of strong spot widths for insulin crystal with 0.1° / frame - most spots 2 pixels wide

LOOKING AT IMAGES

- Some people collect 1° images because “always have done”
- Some because they understand the data that way
- Better to use tools properly -> learn to use image viewers



KNOW YOUR EXPERIMENT

- Collecting data is there to meet experiment goals
- If you know what you are trying to do, you can collect “sub optimal” data which still meets your needs but could be more efficient e.g. wider oscillations
- Keep in mind the count rate correction
- All other things being equal however want rotation width $<$ half mosaic spread

IDEA #3

- Know your experiment goals and collect data to achieve them
- If you don't know anything specific, try to collect 360°+ of low dose finely sliced data
- Learn to look at the data properly

RADIATION DAMAGE

- Typically single biggest problem
- Cannot really be corrected for, or undone - perform experiment carefully
- Usually at workshop have “tricky data sets with added radiation damage”
- The data collection is the last experimental step -> everything later can be repeated etc.
- Where possible avoid radiation damage rather than hoping to correct for it in software

MULTIPLE SAMPLES

- Assumption is your structure / result is indicative of the "truth"
- Combining data from many samples should get you closer to the truth
- Can also help to reduce impact of radiation damage / other experimental effects



IDEA #4

- Radiation damage big problem - may need to combine data from multiple samples to achieve goals
- Carefully collecting more than you need allows options later
- Avoiding radiation damage much easier than correcting for it

CONCLUSIONS

- Ask the beamline staff for advice
- Detectors can be saturated - lots of weak data better than a little very strong data
- Know your experimental goals - learn how your samples behave then apply this knowledge
- Where possible, avoid radiation damage and don't be afraid of combining data from many samples

ACKNOWLEDGEMENTS

- Folks from DECTRIS and elsewhere on writing up how the detectors actually work
- Time on Diamond beamlines to collect example data, characterise detectors
- Samples! Thank you to team at Diamond for the steady supply of good quality test crystals

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