

μ -Diffraction and New Mineral Discoveries at the Canadian Museum of Nature

BY RALPH ROWE





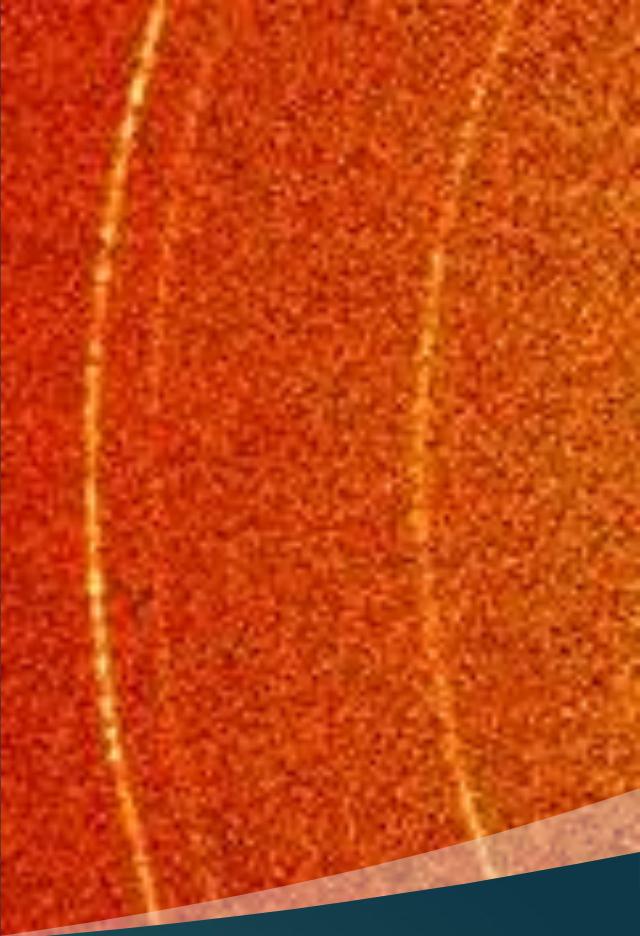
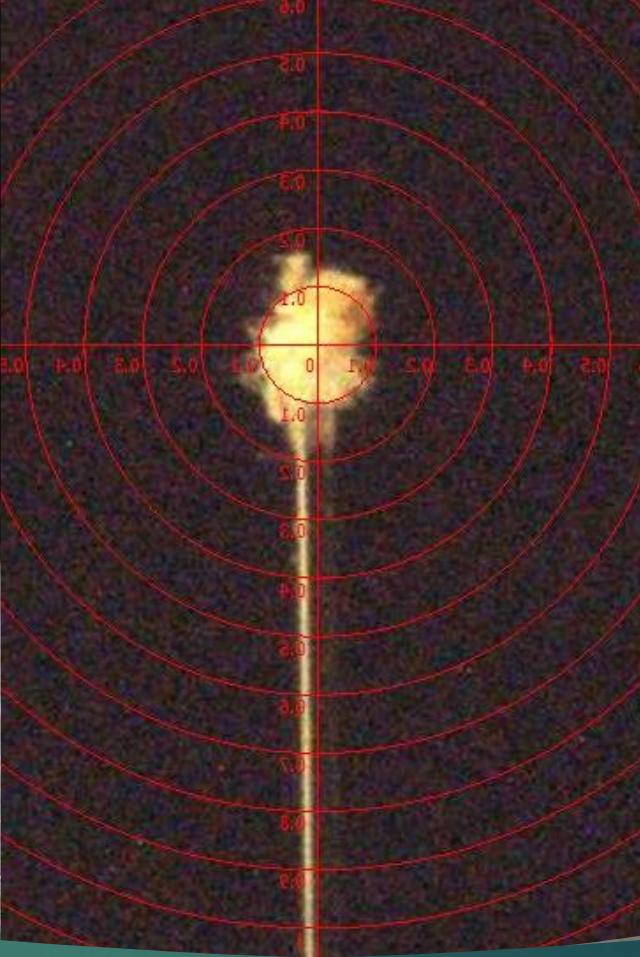
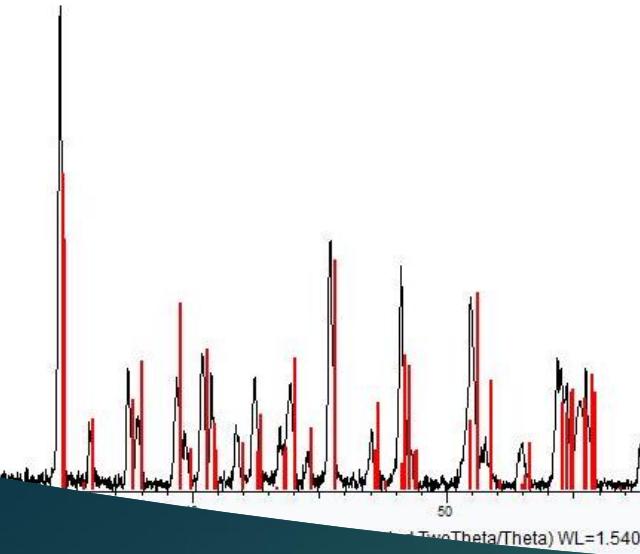
Mineralogy Section:

- ▶ Senior Research Assistants / Lab Techs
 - ▶ Ralph Rowe (XRD)
 - ▶ Glenn Poirier (Microprobe-SEM, partnership with U of Ottawa)
- ▶ Researchers
 - ▶ Dr. Inna Lykova
 - ▶ Dr. Aaron Lussier
 - ▶ Dr. Paula Piilonen
- ▶ Research Associates
 - ▶ Dr. Joel Grice
 - ▶ Robert Gault

Canadian Museum of Nature

X-ray diffraction Lab

- ▶ Equipment
 - ▶ Bruker D8 Discover (2018)
 - ▶ Detector: Dectris Eiger 2R_500K detector
 - ▶ Radiation: Incoatec Cu-microfocus source (1 μ s)
- ▶ Mandate
 - ▶ Mineral identifications for our collections
(labelling, acquisition, etc...)
 - ▶ Support our various research projects
 - ▶ Investigative tool for new mineral discoveries
 - ▶ Detailed data for publications



Micro X-ray Diffraction

Research Material

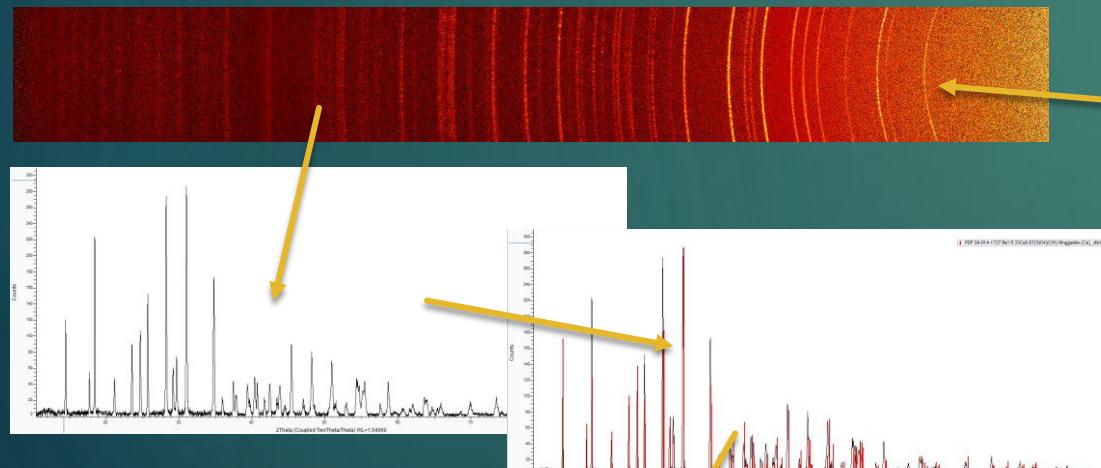
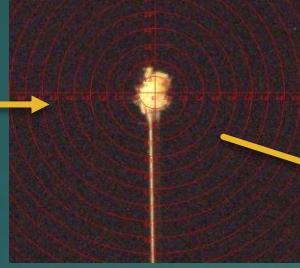
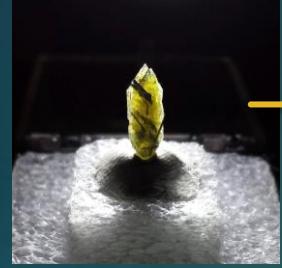
- ▶ Small sample size
- ▶ Rare crystals
- ▶ Unique geochemical environments



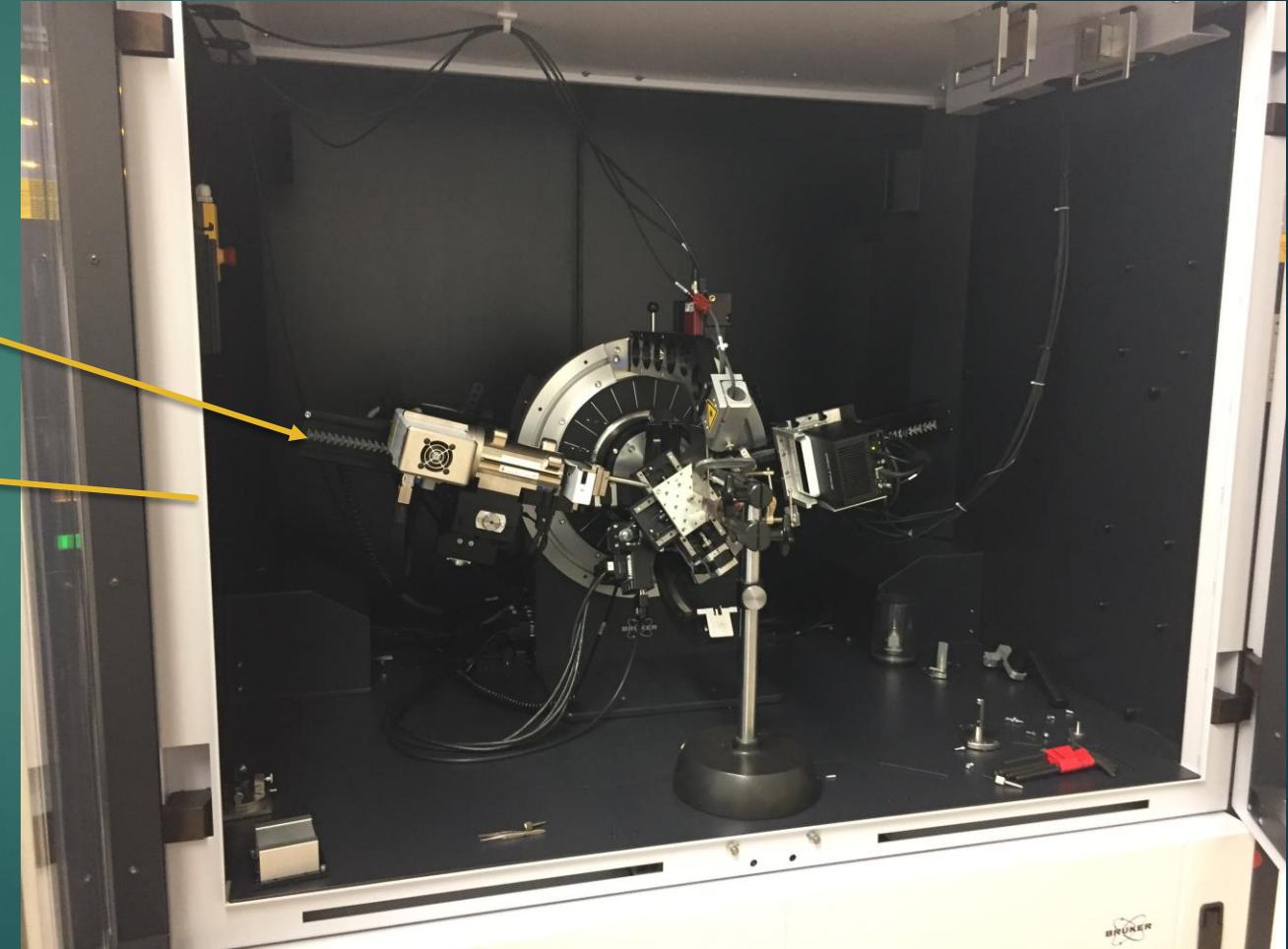


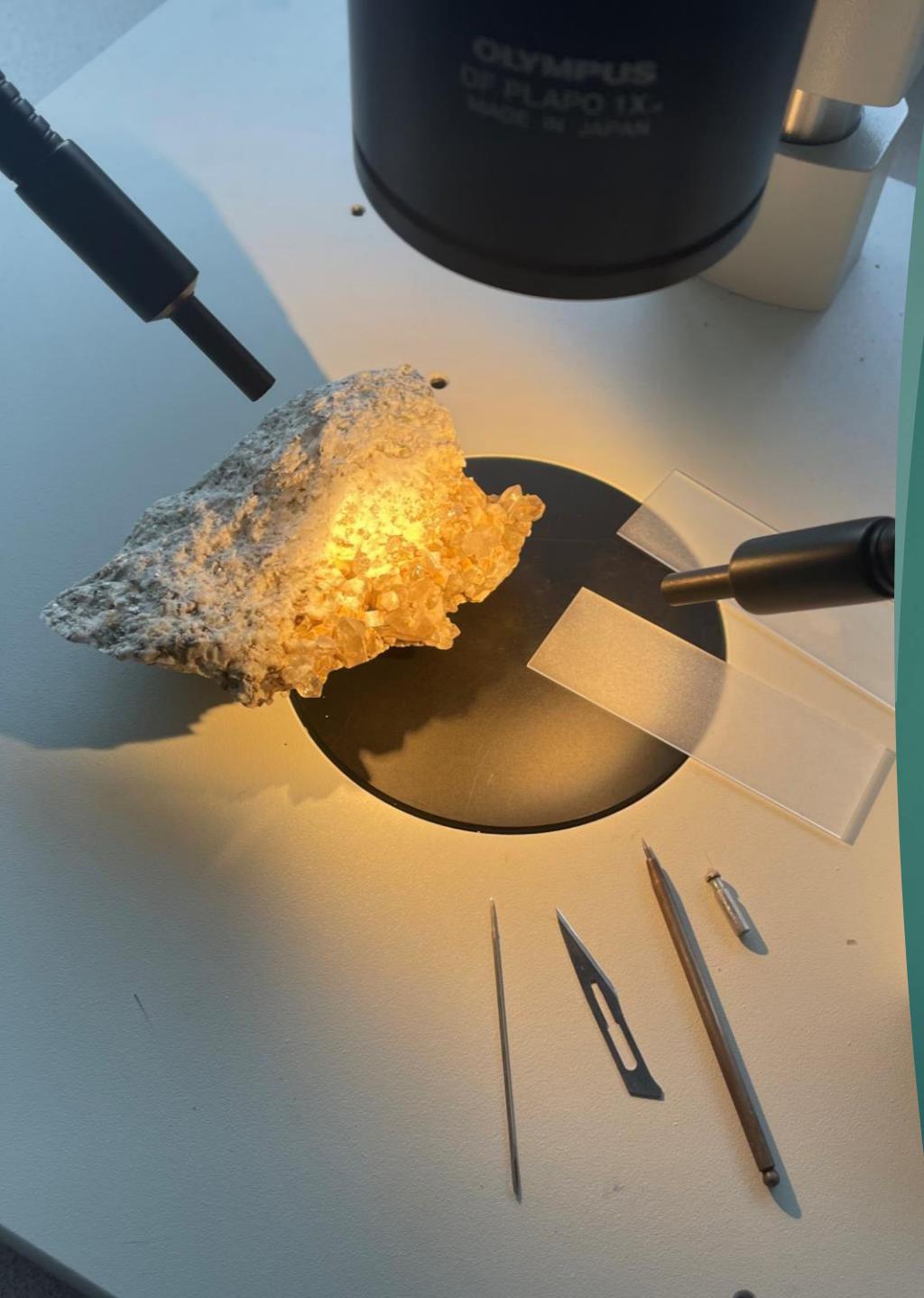
Museum specimens

Mineral Identification



mineral: Hingganite



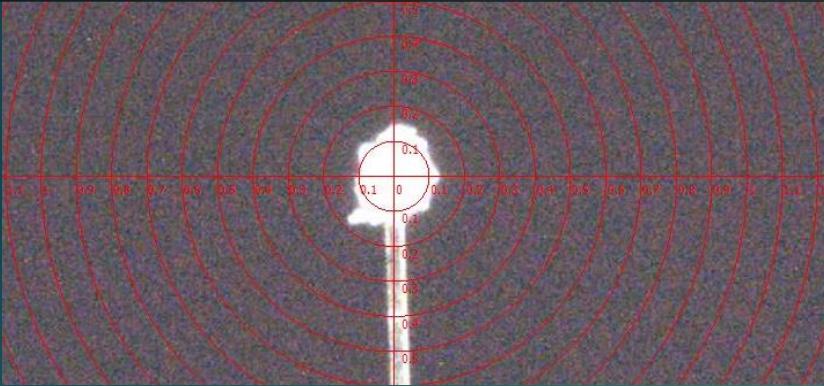


Sampling solutions

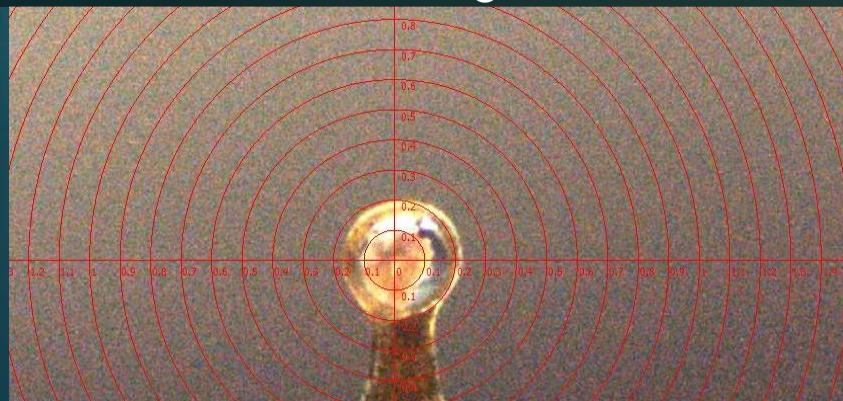
- ▶ Sample the smallest amount!!!
 - ▶ Microscope and a pointy tool!
 - ▶ Frosted slides
- ▶ Ethanol or acetone
 - ▶ Sample transfer
 - ▶ Crystal or fragment splitting

Mounting techniques

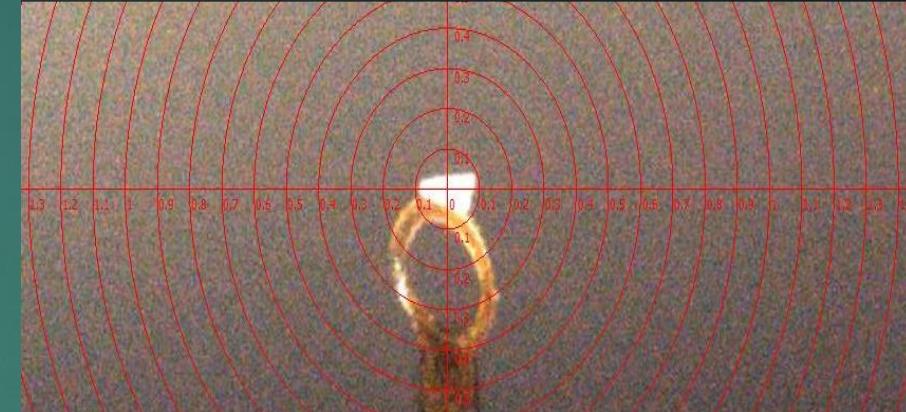
Plenty of material: ~200 µm powder ball



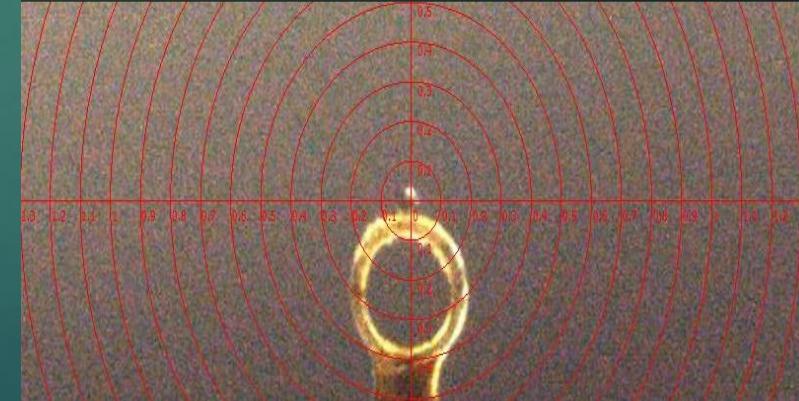
Extremely rare and valuable: use mounting oil



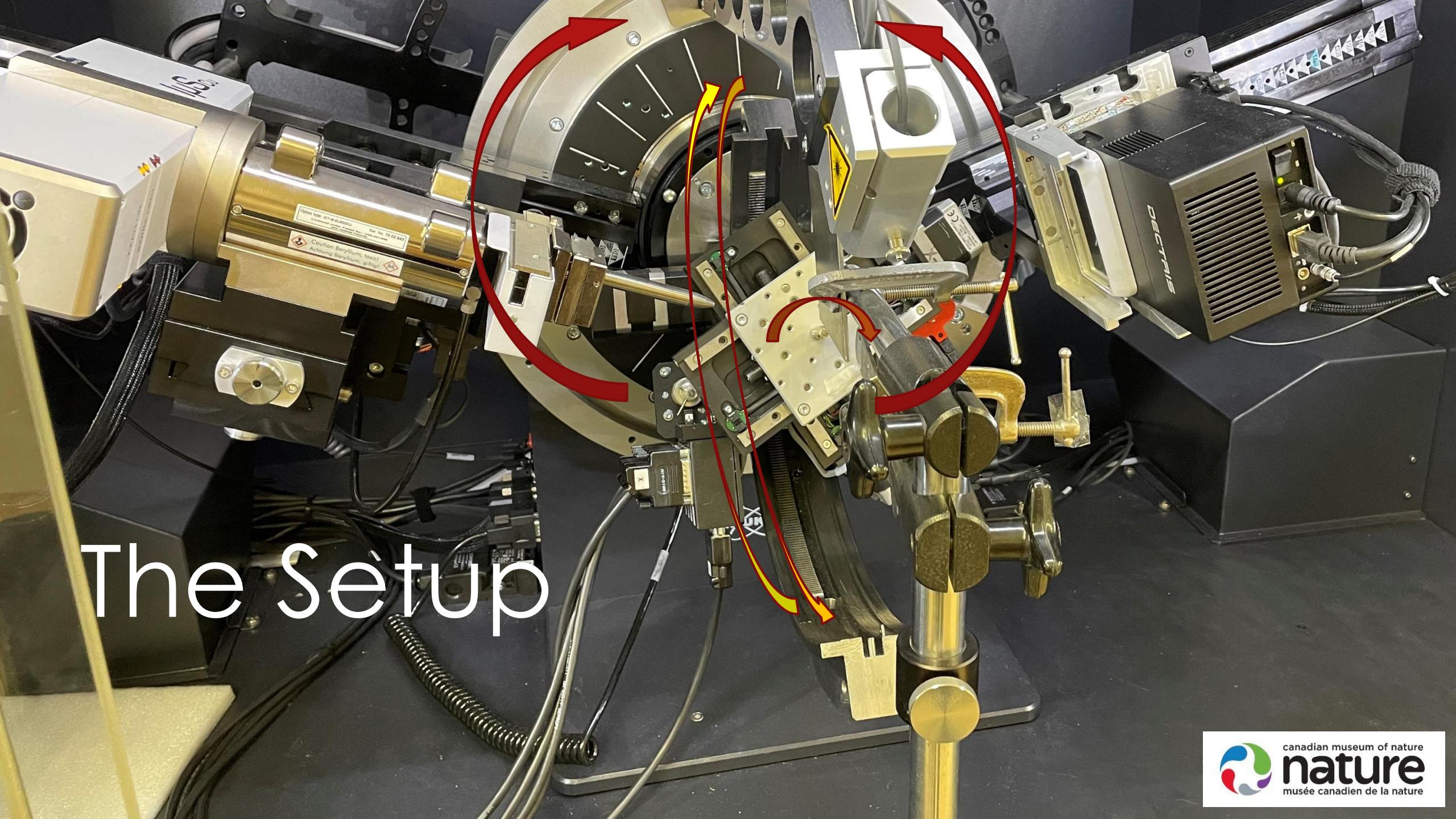
Rare or valuable fragment (research)



Tiny crystal : Very long exposure + smaller collimator



The Setup



Calibration

TRUE QUALITY CONTROL

Calibration Parameters

X [Pixels]	500.01
Y [Pixels]	234.77
Radius [mm]	176.70

Apply And Update Position

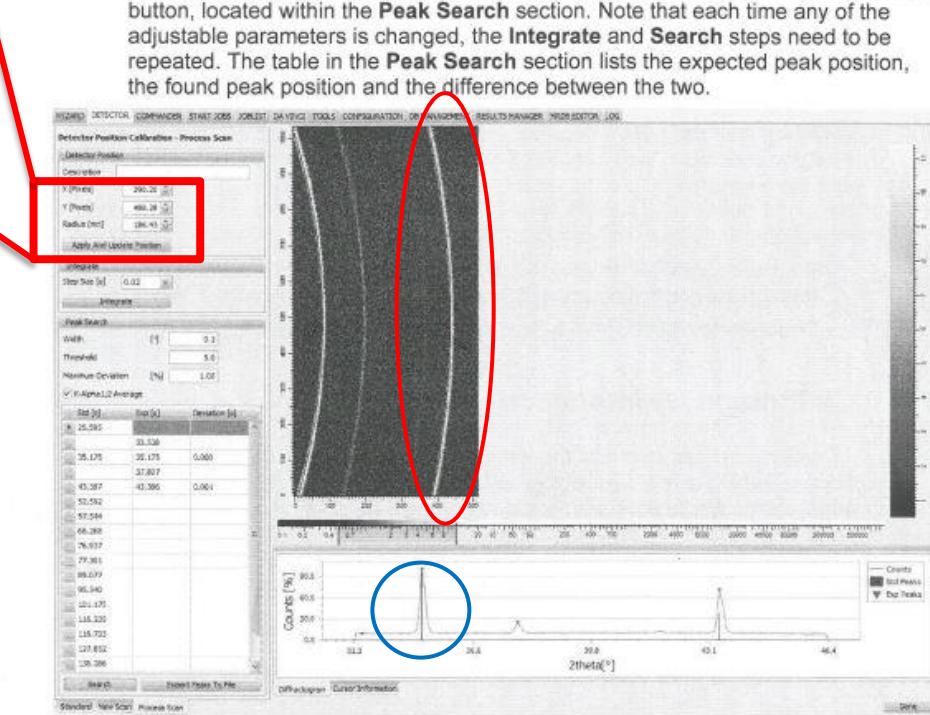


Figure 6.7: Process Scan window of the Position calibration step

Detector Calibration

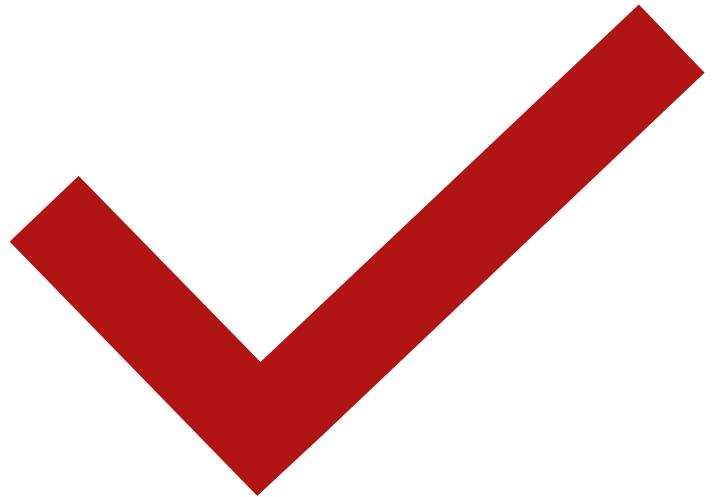
6. In the **Process Scan** step, the acquired diffraction pattern can be integrated and compared to the expected peak positions for the chosen standard sample.
 - An overlay with the expected peak positions for the chosen standard is shown on top of the acquired image. After completing the **Center** step, it should be sufficient to adjust the detector distance to achieve an overlap with the measured data.
 - A precise comparison can be done by looking at the azimuthally averaged radial intensity profile, obtained by pressing the **Integrate** button, followed by the **Search** button, located within the **Peak Search** section. Note that each time any of the adjustable parameters is changed, the **Integrate** and **Search** steps need to be repeated. The table in the **Peak Search** section lists the expected peak position, the found peak position and the difference between the two.

Manufacturer Calibration

- Visual calibration
- Approximation
- Why not use numbers?

Goal of calibration

- ▶ Get data error within desired range
- ▶ Why push for better calibration?
 - ▶ Data useable for search/match mineral identification method
 - ▶ + for reliable cell refinement in Topas
 - ▶ ++ Peak indexing, space group solution, cell-dimension plots
 - ▶ +++ Rietveld structure solution



Calibration obsession

- ▶ The STANDARD
 - ▶ Annealed CaF₂
 - ▶ Standardized against
 - ▶ NBS Si 640a
- ▶ Why 2 standards
 - ▶ Can test the stability through time
 - ▶ Validate calibration

2-Theta Values for Annealed CaF₂ with Cu Radiation

hkl	I	Old Cell			New Cell		
		Ka	Ka1	Ka	Ka1	Ka2	
111	9	28.30	28.28	28.291	28.268	28.33	
220	10	47.06	47.02	47.041	47.001	47.12	
311	4	55.82	55.78	55.804	55.755	55.90	
400	1	68.75	68.68	68.719	68.656	68.84	
331	1	75.94	75.87	75.907	75.835	76.05	
422	2	87.49	87.40	87.455	87.367	87.63	
511	1	94.34	94.25	94.303	94.204	94.50	
440	1	105.96	105.84	105.910	105.788	106.15	
531	1	113.23	113.09	113.177	113.037	113.45	
600	<1	115.74	115.60	115.683	115.536	115.97	
620	1	126.42	126.24	126.345	126.163	126.712	
533	<1	135.50	135.27	135.406	135.182	135.855	

WAVELENGTHS USED:

Ka=1.54184

Ka1=1.54060

Ka2=1.54433

STANDARDIZED against NBS Si (batch no. 460a).

ALL wavelengths, cell edges in Angstroms.
ALL angles in degrees 2-theta.

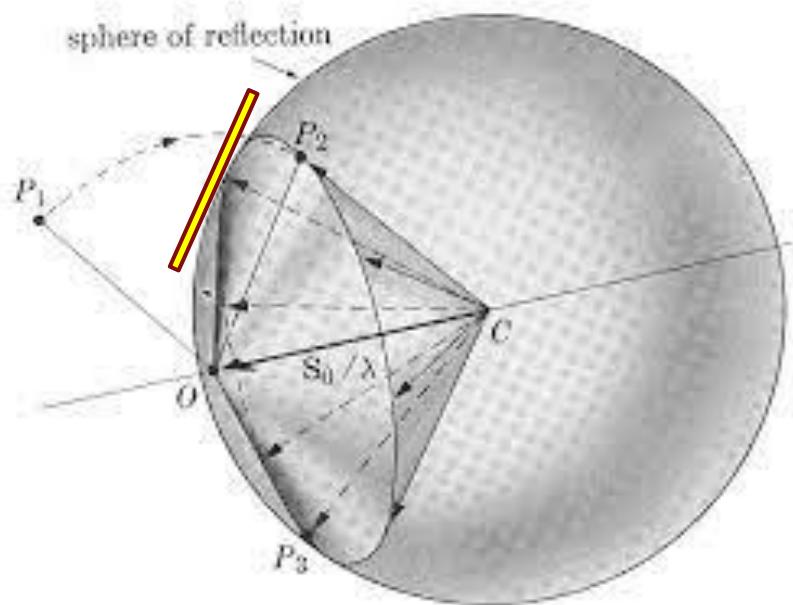
ANNEALING RECIPE:

1. Anneal reagent grade CaF₂ @ 800°C for 1 hour.
2. Grind product thoroughly.
3. Repeat steps 1 & 2 two times.
3. Toast @ 500°C overnight & grind product.

Calibration

(Geometry and parameters)

- ▶ Debye Scherrer geometry
 - ▶ Sample in center of a sphere
 - ▶ Detector on the edge of the sphere
 - ▶ Corrected to give a concave surface fitting on the sphere
- ▶ Calibration parameters
 - ▶ Sample to detector distance (radius of the sphere)
 - ▶ X and Y coordinate of beam center
 - ▶ Relationship between beam and detector



Statistical calibration

- ▶ Goal
 - ▶ Improve on original visual calibration (2002)
 - ▶ +/- 0.05° 2θ tolerance for Celref software
- ▶ How it's done
 - ▶ Peak position error vs location of peak on detector
 - ▶ Investigate the impact
 - ▶ 3 calibration parameters on the error trends
- ▶ The result
 - ▶ Optimal values for all three parameters
 - ▶ limited error fluctuation

New statistical calibration approach for Bruker AXS D8 Discover microdiffractometer with Hi-Star detector using GADDS software

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An additional statistical calibration for the Bruker D8 Discover microdiffractometer is necessary to obtain accurate reproducible 2θ data for cell-refinement work. This new approach uses a graphical mapping method of the 2θ error versus the location of a selected diffraction peak on the detector surface to describe the separate roles of different calibration procedures (rebiasing, flood field, and spatial corrections) and parameters (sample-to-detector distance, x - y center coordinate) in minimizing the error. Optimized parameters are used to obtain the lowest achievable $\Delta 2\theta$ with this setup. Intensity error relative to the position of the diffracted line on the detector was found to be consistent at up to 20% and could not be reduced using any of the investigated techniques and parameters. © 2009 International Centre for Diffraction Data. [DOI: 10.1154/1.3193683]

Key words: diffractometer calibration, microdiffractometer, GADD software, Bruker AXS D8 Discover, two-dimensional detector correction, improved cell-refinement

I. INTRODUCTION

The general belief behind new technology is that it improves the data, but often these improvements focus on processing time and instrument safety of the unit. The general evolution of scientific instruments in the last three decades has been the shift toward digitization of concommitment detectors and acceleration of data collection. This paper will focus on the data collected with a Bruker D8 Discover microdiffractometer using GADDS system with a Hi-Star detector.

Powder cameras have been used in laboratories throughout the last century, but are being replaced by digital diffractometers. The most common powder camera is the Debye-Scherrer camera, which consists of a cylindrical camera body (Zoltai and Stout, 1984) with 35 mm film placed around the interior. This steel cylindrical camera controls most of the geometry and can be precisely machined to give consistent data. These cameras have been used for many years for least squares cell refinements of mineral structures. On the other hand the D8 Discover has many factors controlling the geometry such as (1) sample-to-detector distance, (2) detector center coordinates, (3) detector image correction (unwrapping), and (4) X-ray beam and stage calibration. This geometry is complex since a flat detector surface also introduces challenges in correcting the image for an ideal spherical surface of detection (He *et al.*, 2000).

This paper will test the ability of the D8 Discover to produce accurate peak position and intensity measurements with the user calibration method supplied by the manufacturer. Is it possible that an alternative calibration approach can be used? What are the options available to the user? This approach is from the point of view of a user only and does not intend to change any engineering or programming components of the Bruker D8 Discover system but rather to provide a more refined approach to calibrating the parameters made available by Bruker.

II. BRUKER D8 DISCOVER MICRODIFFRACTOMETER

The Bruker D8 Discover is a microdiffractometer with a General Area Detector Diffraction System (GADDS) equipped with a HI-STAR area detector, a two-dimensional multiwire proportional counter (PWPC) detector. The X-ray source is fixed, while the detector (2θ drive) and the sample holder (ω drive) are on a rotating platform allowing a synchronized rotation (Figure 1). It uses a copper source to generate X-rays and generally runs at 40 kV/40 mA. It is equipped with a graphite monochromator for stronger beam intensity but will not resolve $K\alpha_1$ and $K\alpha_2$ (Bruker AXS, 2001). Two different stages are available for this setup: the fixed-chi and the XYZ. The fixed-chi (54.736°) stage was used for this experiment. Though it can only accommodate a single sample, it has the ability to rotate the sample 360° along φ . The instrument is controlled using windows based GADDS software and an attached controller when in manual mode. Search/match for mineral identification is done using EVA software supplied by Bruker, which has the ICDD PDF-4 database for inorganic compounds.

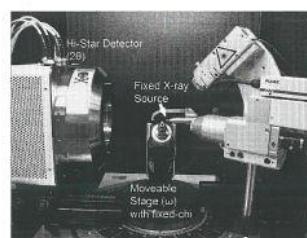
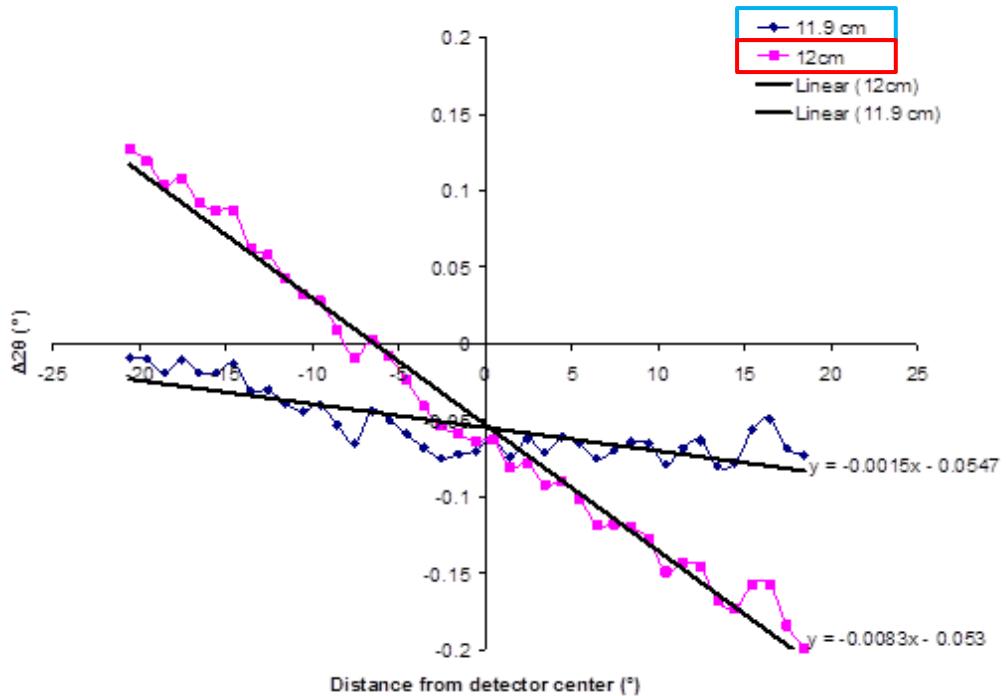


Figure 1. (Color online) The components and geometry of the D8 Discover.

^{a)}Electronic mail: rrowe@mns-nature.ca

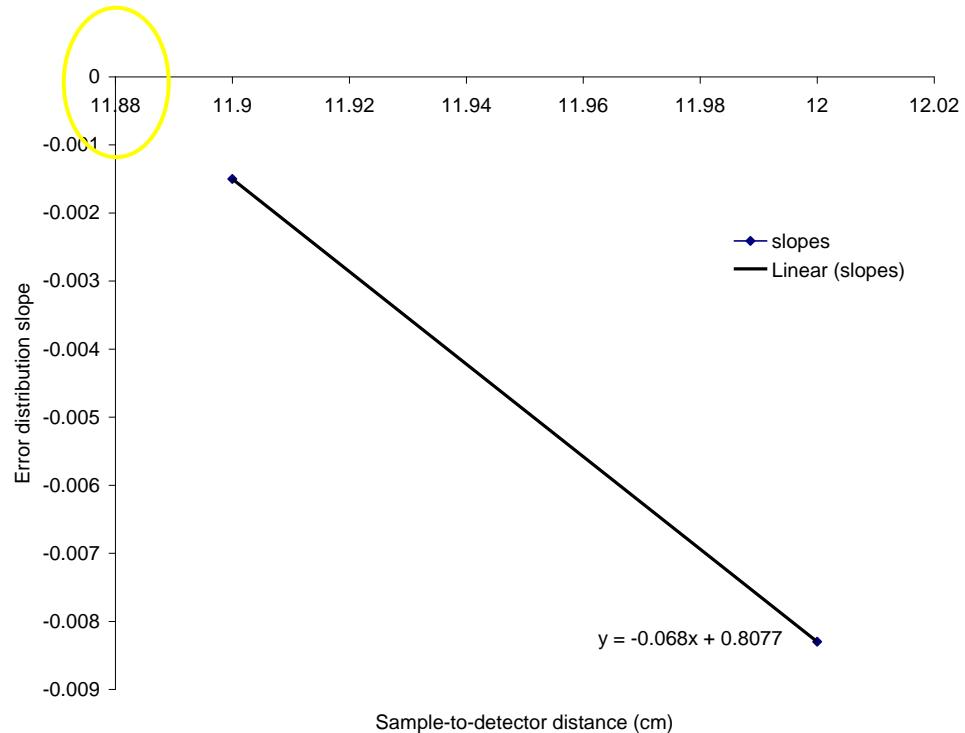
Calculating : true sample to detector distance (S-D)

- ▶ Each data point
 - ▶ compared with the standard peak value
 - ▶ from the CaF_2 standard (47.001°)
- ▶ X-axis
 - ▶ detector width
- ▶ S-D parameter effect
 - ▶ Significant effect on the repeatability



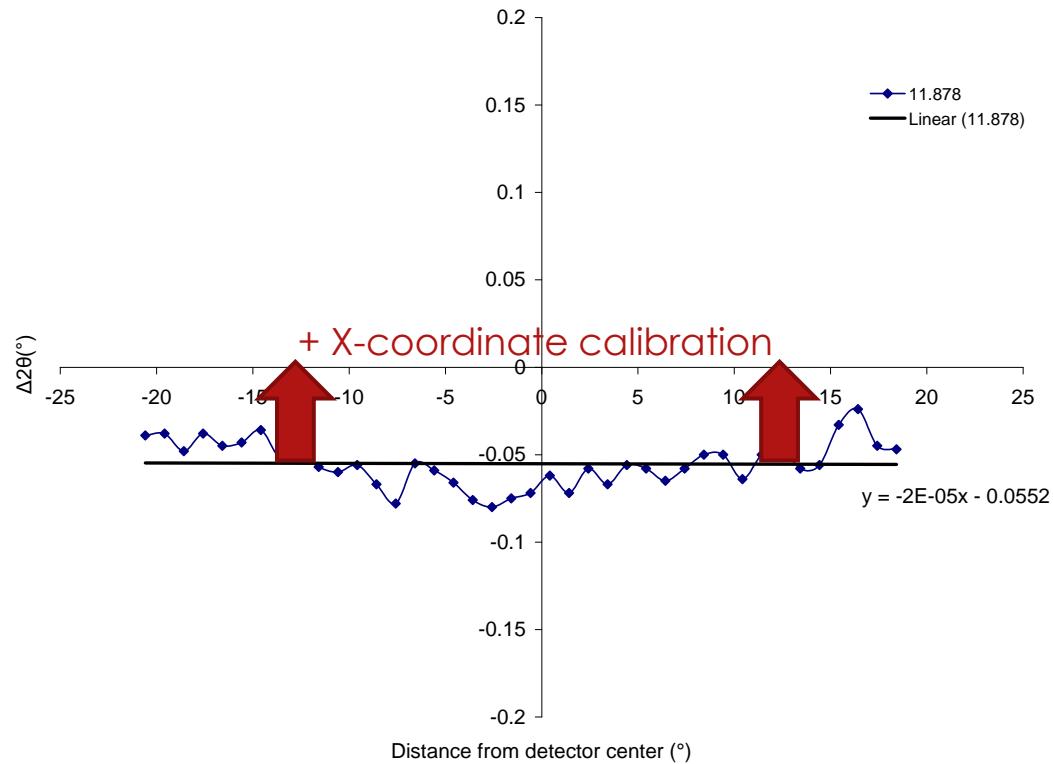
Sample to detector distance (Solution)

- ▶ True distance
 - ▶ → error distribution slope = 0
- ▶ Linear relationship
 - ▶ (S-D vs Slope) solution for true distance
- ▶ Solving x for y = 0
 - ▶ gives an ideal distance **11.88 cm**



New DISTANCE and X-center coordinate

- ▶ New distance
 - ▶ stable measurements
- ▶ X- coordinate
 - ▶ found to move the range up and down
 - ▶ Solving X-coordinate
 - ▶ for a distribution around 0
 - ▶ Ideal calibration obtained
- ▶ Y-coordinate
 - ▶ perpendicular to diffraction arc
 - ▶ Lesser impact

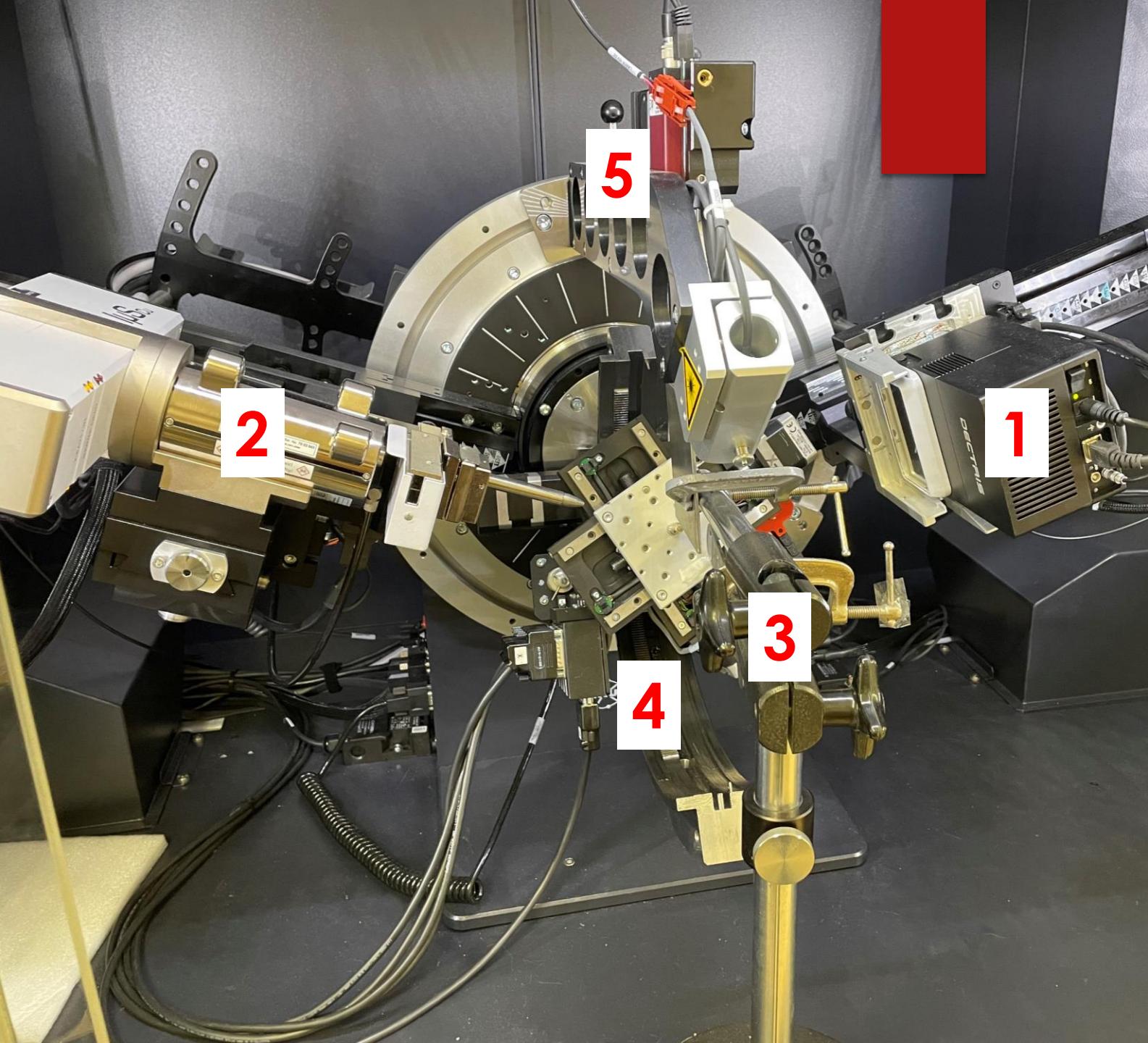




OLD UNIT (2002-2018)

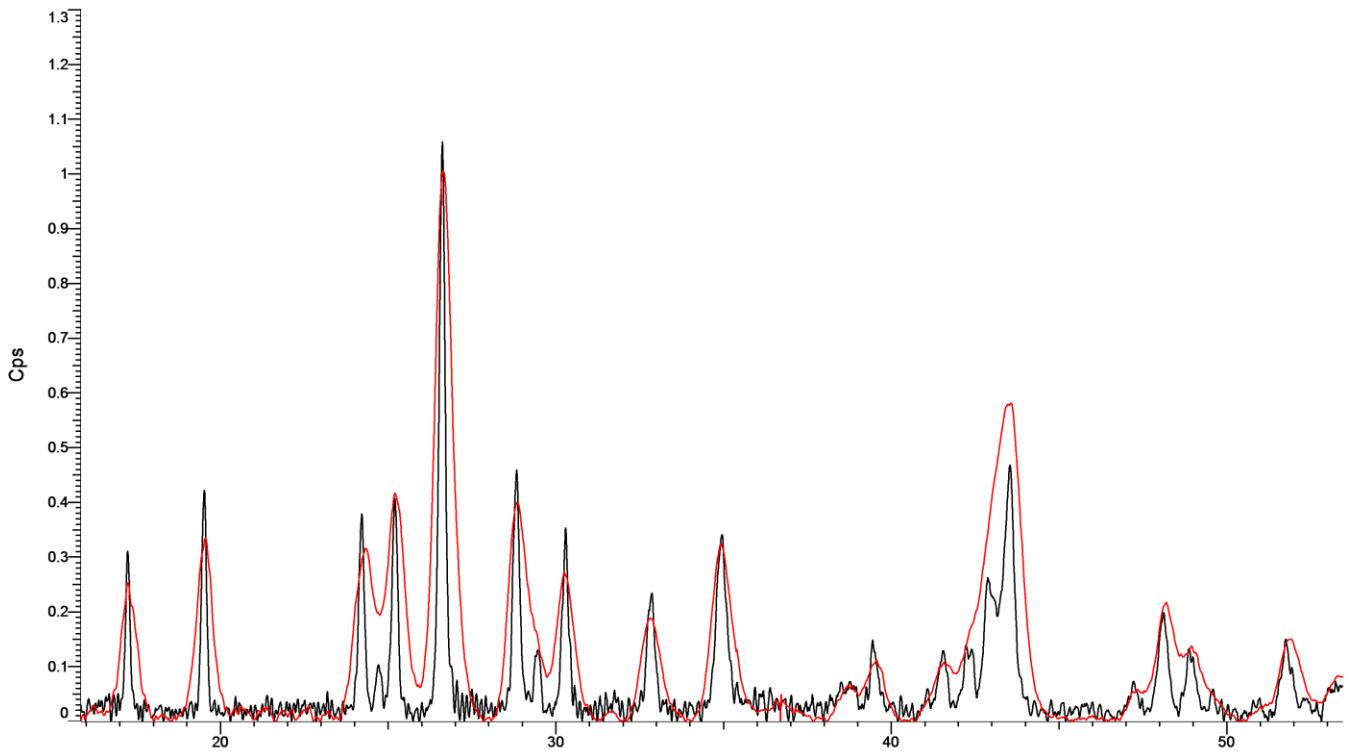
New unit

- ▶ 1. Dectris Eiger 500K detector
- ▶ 2. Indicotec micro-focused Cu source
- ▶ 3. Homemade beam and scatter block
- ▶ 4. Centric Eulerian cradle stage
- ▶ 5. Hi-res alignment camera

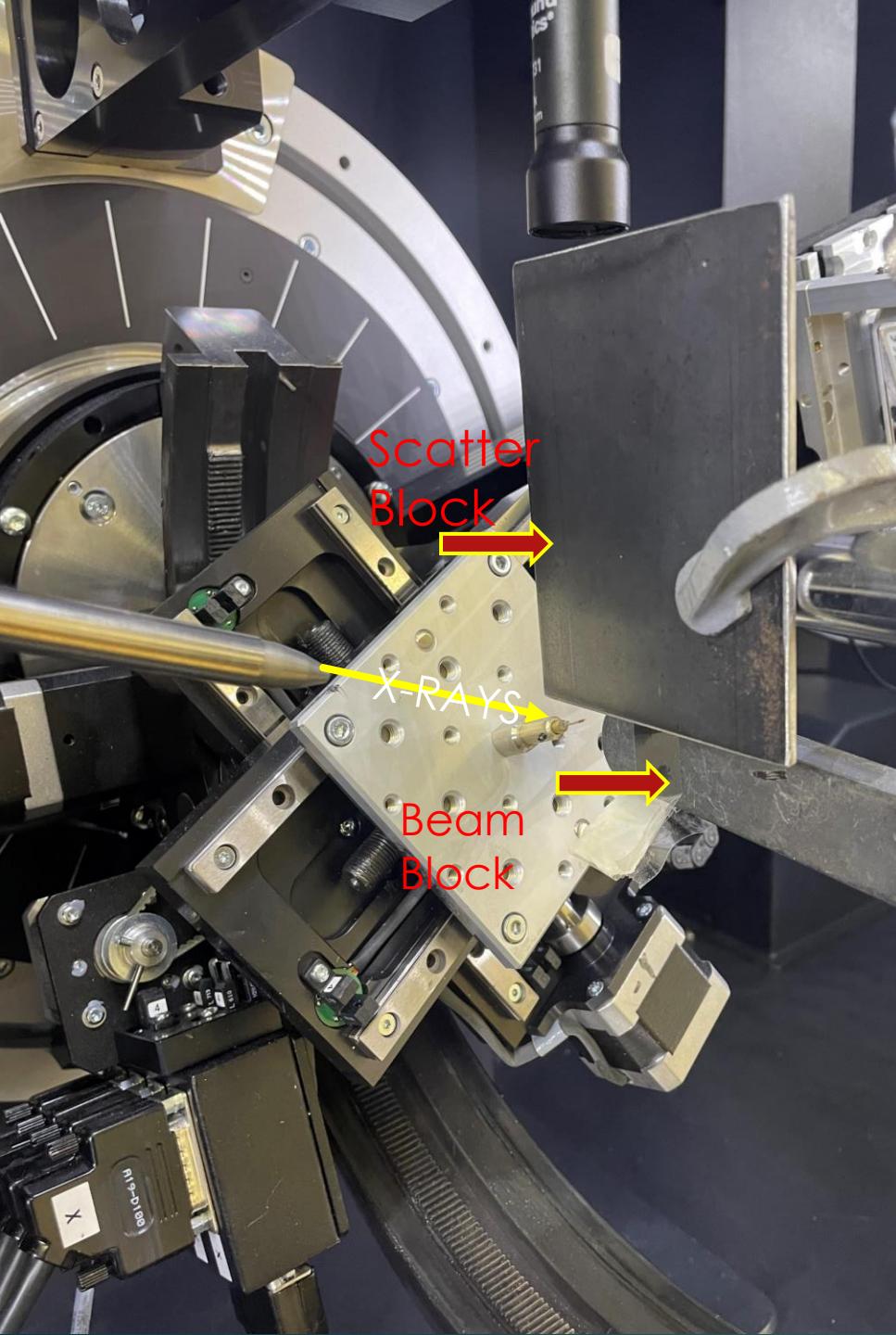


How much better? evolution of the technology

- ▶ Data from Qaqarssukite-(Ce)
 - ▶ My first new mineral
(2006)
 - ▶ $\text{Ba}(\text{Ce,REE})(\text{CO}_3)_2\text{F}$
polymorph



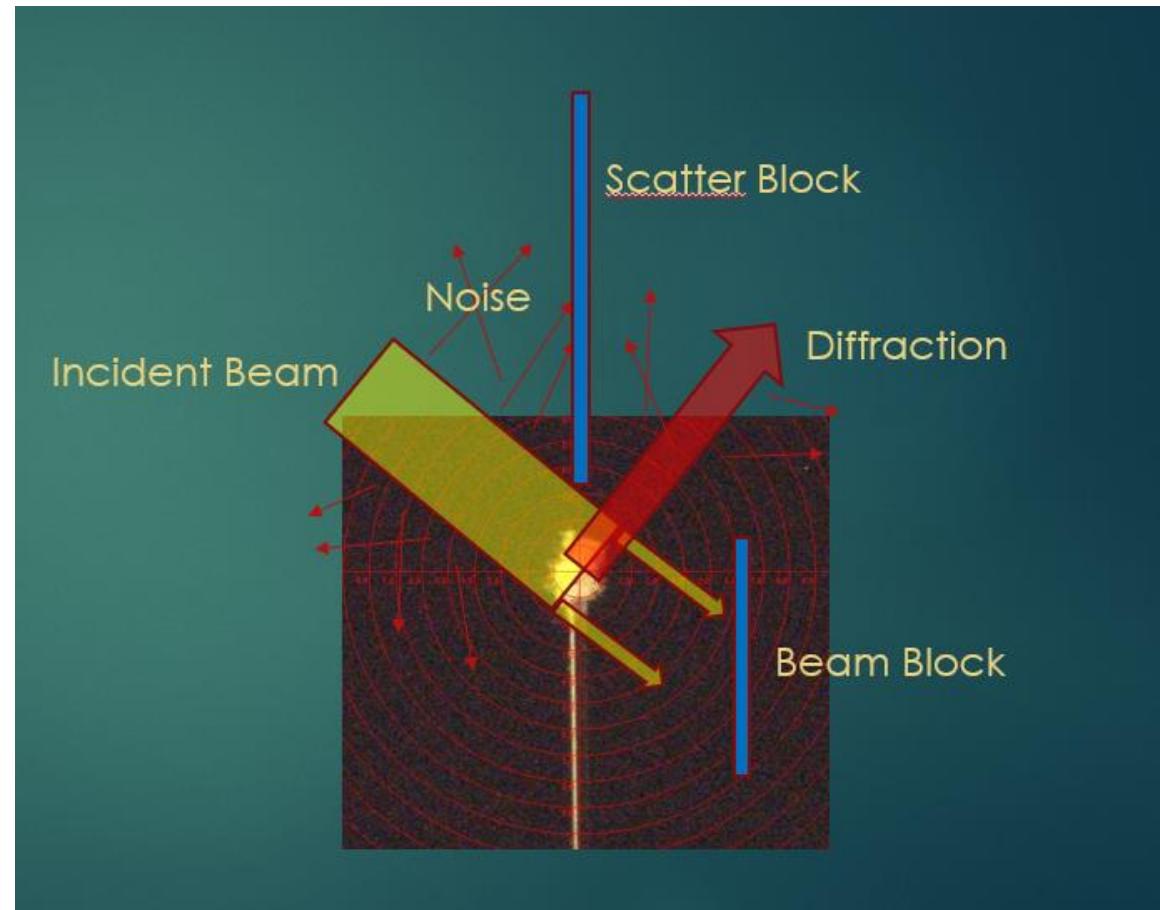
Adapting to your experiment (Noise suppressing)



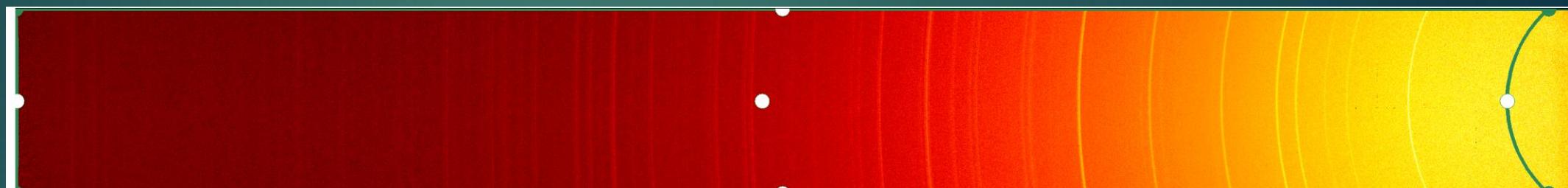
- ▶ Background noise
- ▶ The construction of a scatter/beam block
 - ▶ Lowers the background noise by **95%**
 - ▶ Low intensity peaks better resolved
 - ▶ Experiment time greatly reduced

WHY we need the beam and scatter block?

- ▶ Air Scattering
 - ▶ X-rays plow through air molecules
 - ▶ creating a lot of noise
- ▶ Beam is bigger than the mount
 - ▶ Not all the beam is consumed



Without Noise reduction (Beam-Scatter block)

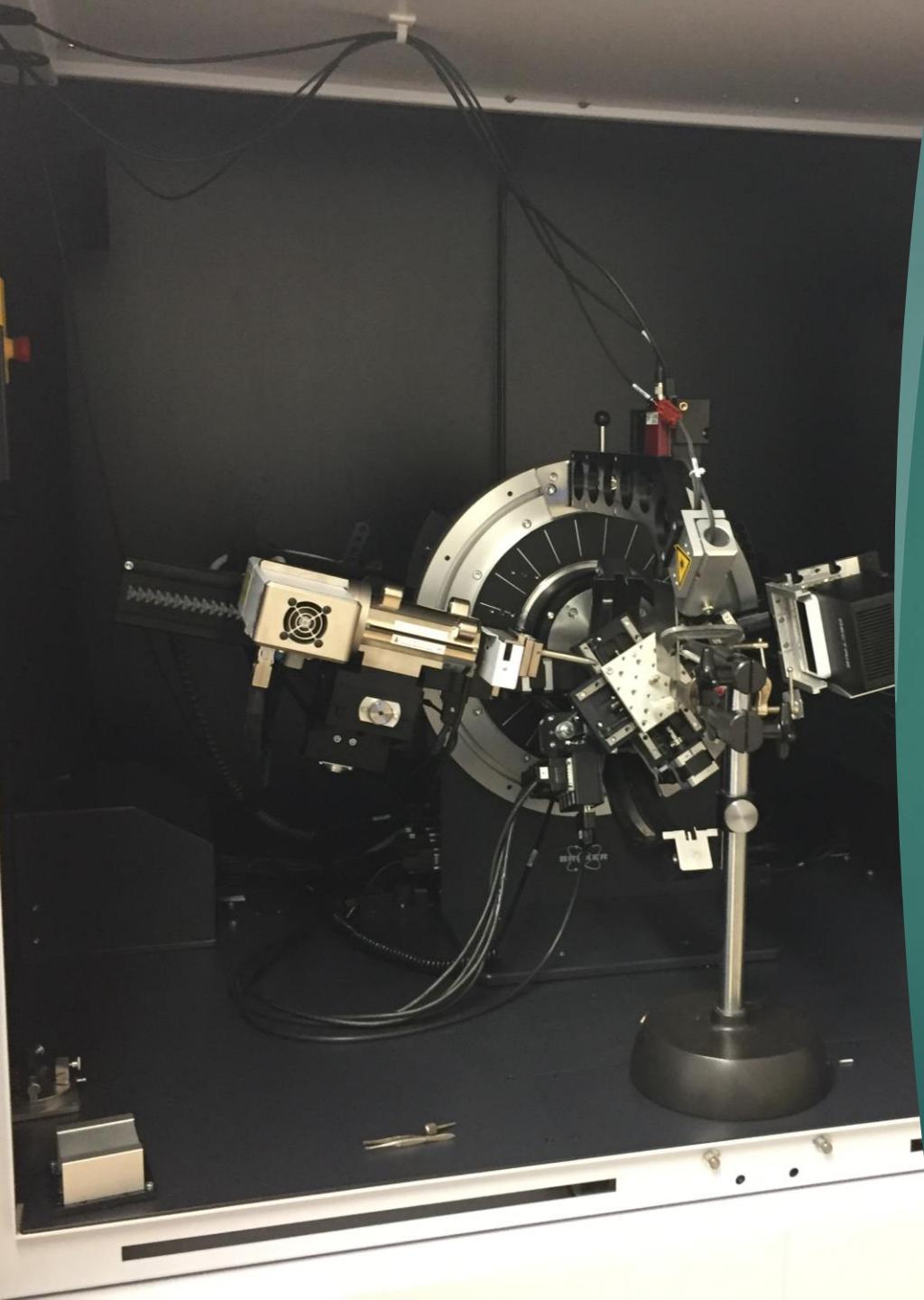


With Noise reduction (Beam-Scatter block)



Calibration method: Did it work?

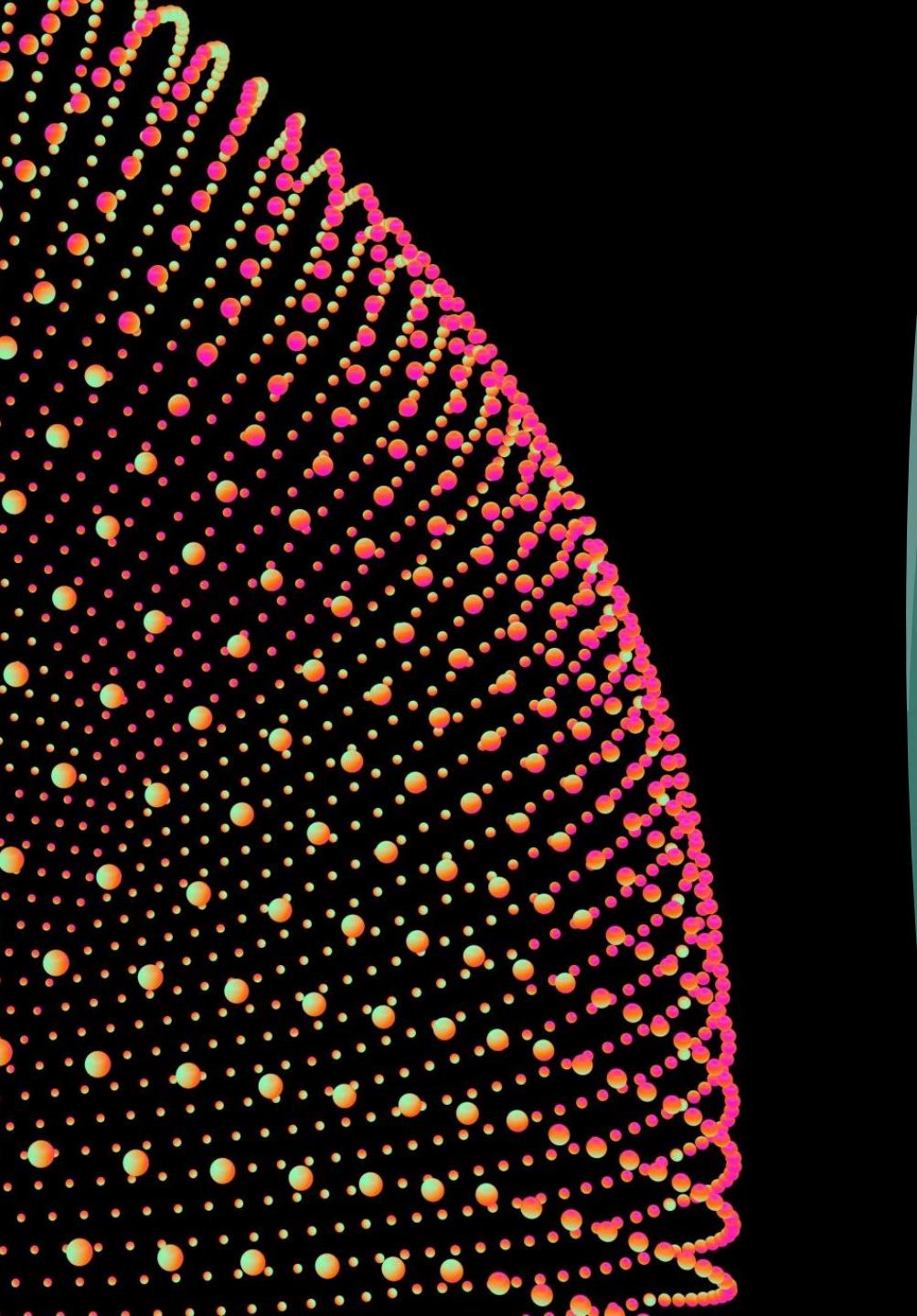
- ▶ Yes
 - ▶ Same experiment
 - ▶ Same geometry
 - ▶ Same calibration parameters
- ▶ Better detector resolution
 - ▶ Resolves completely the experiment
 - ▶ Experiment is now the limiting factor
 - ▶ Exposed previously unnoticeable error (engineering limitation)



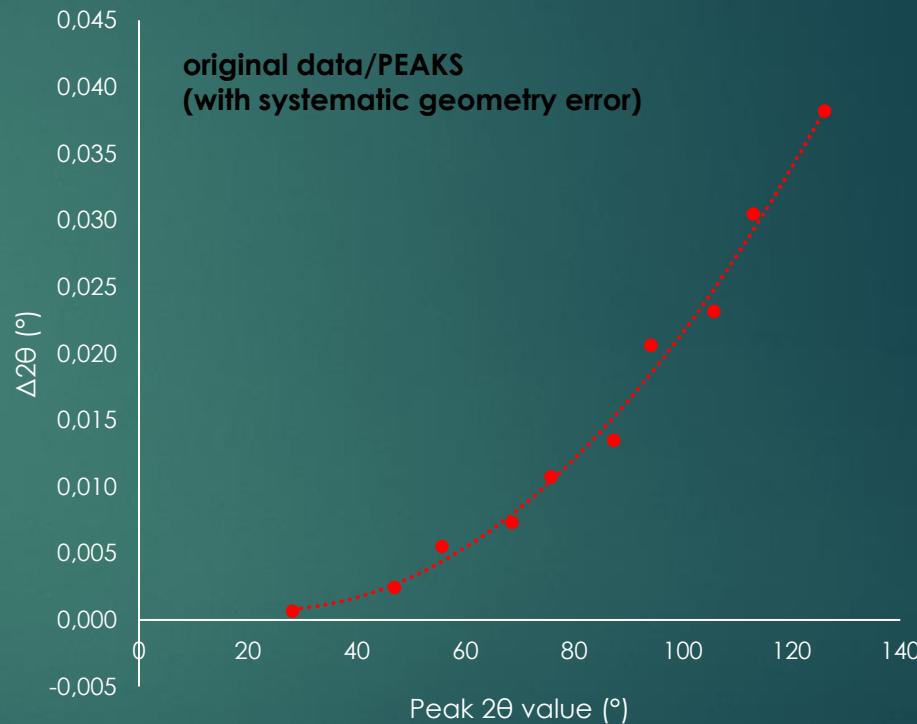
Engineering marvel

- ▶ Data projector
 - ▶ measurement in ångström (\AA)
(0.0000001 mm)
 - ▶ at measurable scale (2- θ ($^{\circ}$))
- ▶ Centered powder mount
 - ▶ 200 μm
 - ▶ in a 34cm sphere
- ▶ Moving parts
 - ▶ Rotation and translation without too much sample movement

Imperfect diffraction sphere?



2-θ error (Measurements vs annealed
CaF₂ standard)



- Possible causes
 - distortion of the sphere
 - sample not precisely in the center

SOLUTION



Correction parameters in TOPAS (BRUKER)

- ▶ FIX target cell
 - ▶ CaF_2 cell dimension (5.46379)
- ▶ Refine Correction Parameters
 - ▶ « Zero Error »
 - ▶ « Sample displacement »
 - ▶ to correct the sphere
- ▶ = Ultimate experiment calibration

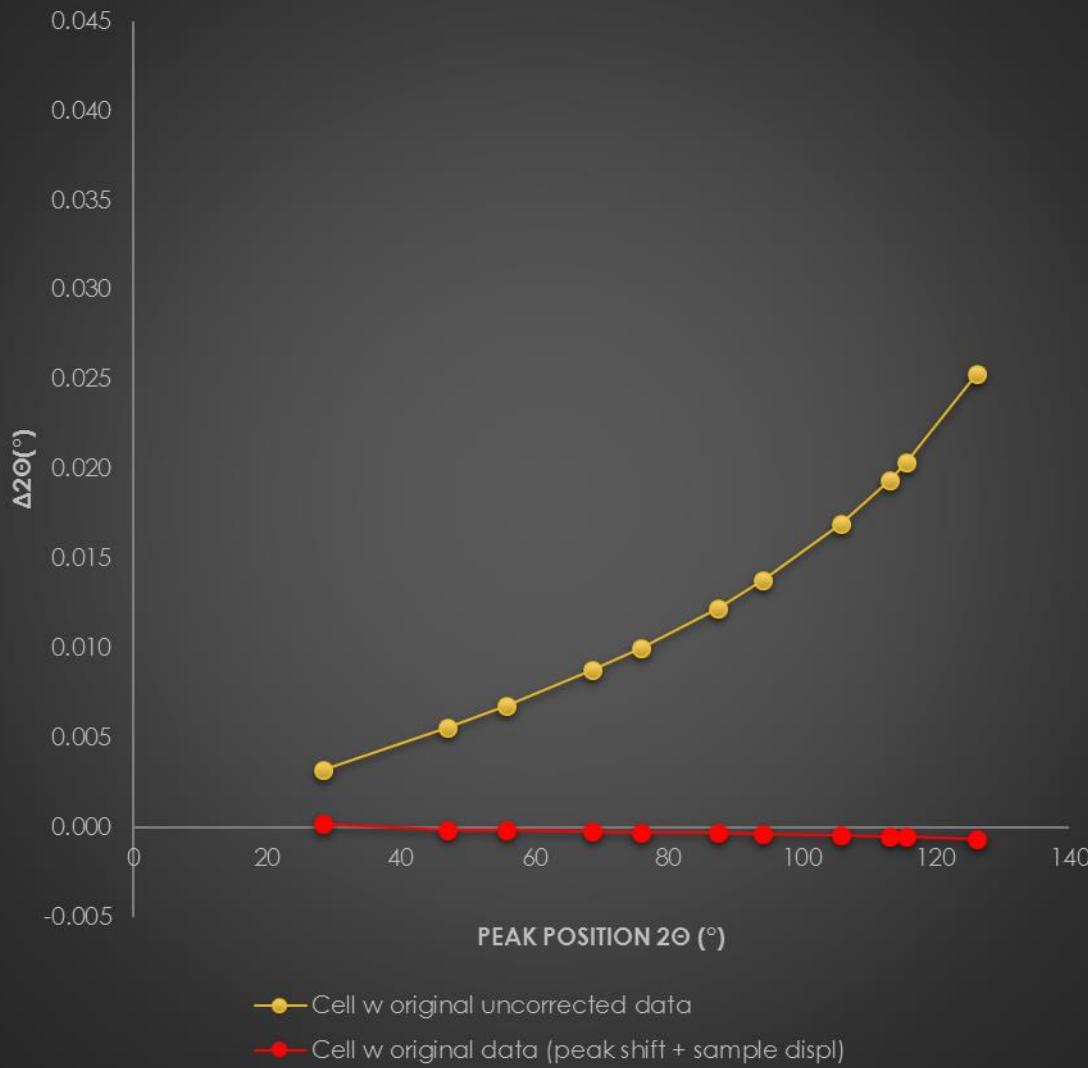
The screenshot shows the TOPAS software interface with the Microstructure tab selected. On the left is a tree view of project files. The main area displays various parameters with their values and status (e.g., Use, Value, Code, Error). A red circle highlights the 'Scale' row, which contains the value '8.46576e-00' under the 'Value' column.

Parameter	Value	Code	Error
Use Phase	✓		
Spacegroup	Fm-3m		
a (Å)	5.4637900	Fix	0.0000000
Scale	8.46576e-00	@	0.00000e+00
Wt% Rietveld	100.000		0.000
Wt% of Spiked	0.000		
Cell Mass	312.299		0.000
Cell Volume (Å³)	163.11054		0.00000
Cry LAC (1/cm)	301.141		0.000
Cry Density (g/cm³)	3.179		0.000
R Bragg	1.955		

The screenshot shows the TOPAS software interface with the Corrections tab selected. On the left is a tree view of project files. The main area displays various correction parameters with their values and status. A red circle highlights the 'Sample displacement (mm)' row, which contains the value '0.1022514' under the 'Value' column.

Parameter	Value	Code	Error
Peak shift			
Zero error	-0.064335	Refine	0
Sample displacement (mm)	0.1022514	Refine	0
Intensity Corrections			
LP factor	0	Fix	0
Surface Rghnss Pitschke et			
Surface Rghnss Suortti			
Sample Convolution			
Absorption (1/cm)	100	Refine	0
Sample Tilt (mm)	0	Refine	0

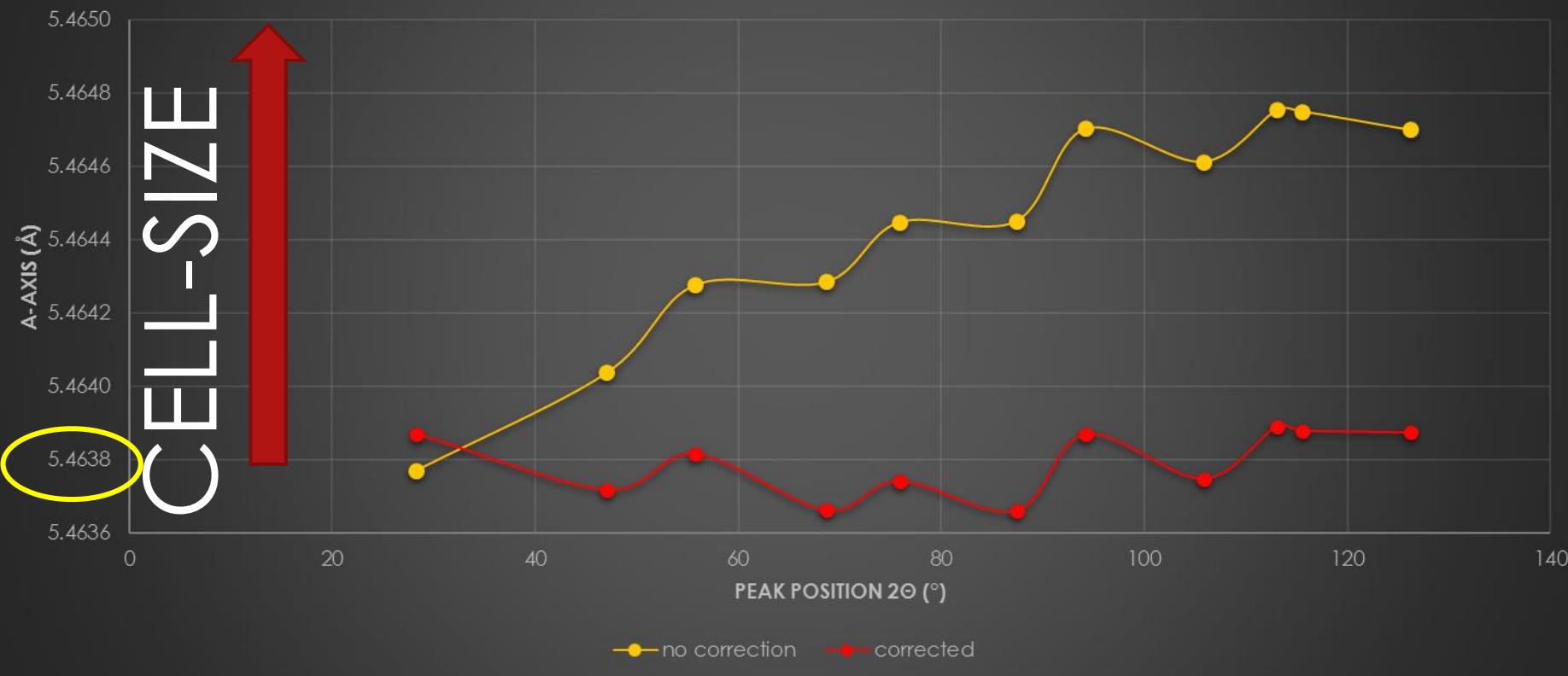
2-theta error (annealed fluorite)



Correction parameters in TOPAS (RESULTS)

- ▶ ALL PEAKS -> SIMILAR ERROR
- Validity tested with NBS Si Standard

CELL SIZE BY SINGLE PEAK



Each peak now belongs to a cell of similar size (CaF_2)

D8 DISCOVER A25 with 2d detector test report

(factory test results)



Date:	Serial # detector: _____							
Customer:	Radius [mm]: 305mm (must be 150 mm or larger)							
SAP# System:	SN# System: _____							
X-ray source	Orientation: 0deg - MaxGammaPos							
Single pinhole	Serial # Optics: _____							
collimator:	X-ray source: Cu							
1mm	Single pinhole: 1mm							
min 2Theta[°]	max 2Theta[°]	Mean value [°]	Gravity Center [°]	Allowed difference +/- 0.045°	Gravity Center + 2Theta Offset	difference after correction	Check	

Manufacturer vs CMN Calibration

+/- 0.045° vs +/ - 0.001

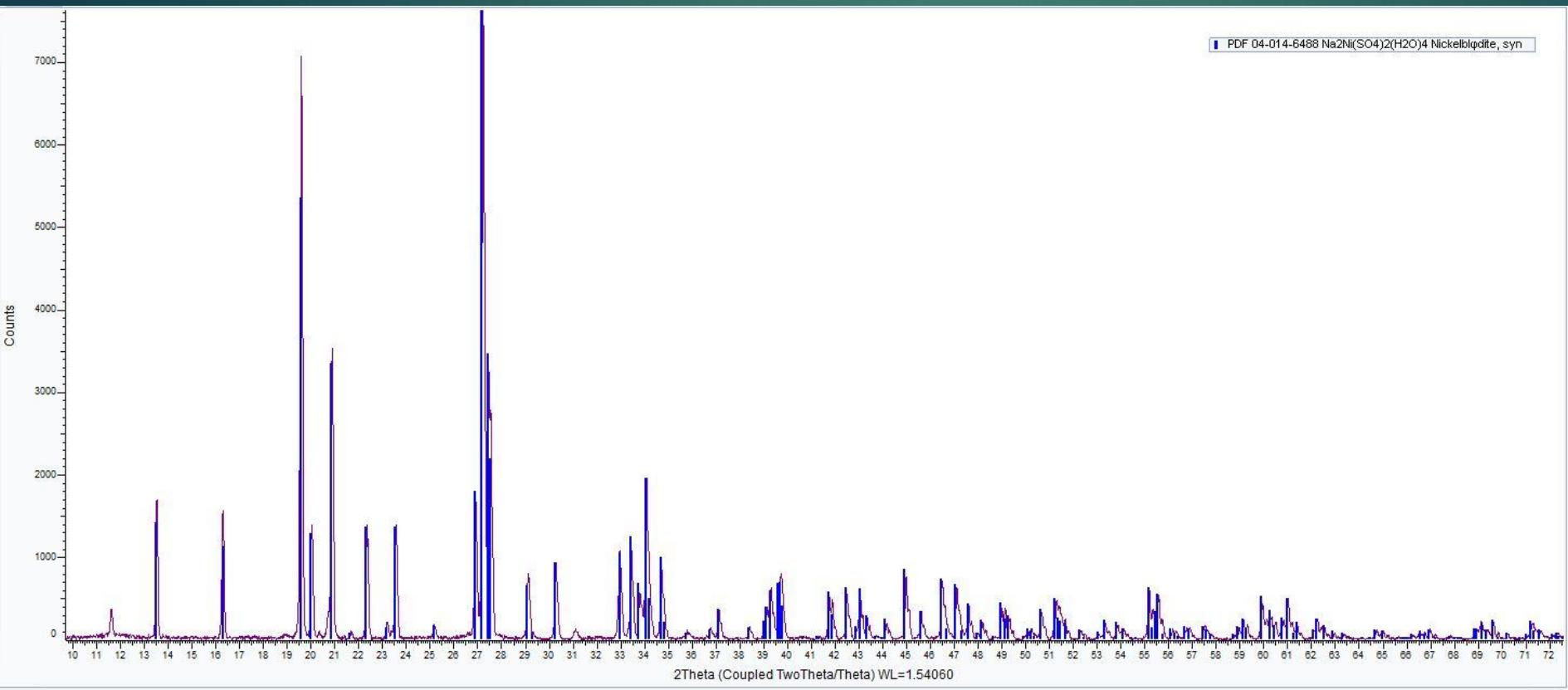
OUR RESEARCH

WHY DATA QUALITY MATTERS



Rietveld structure
solution/refinement

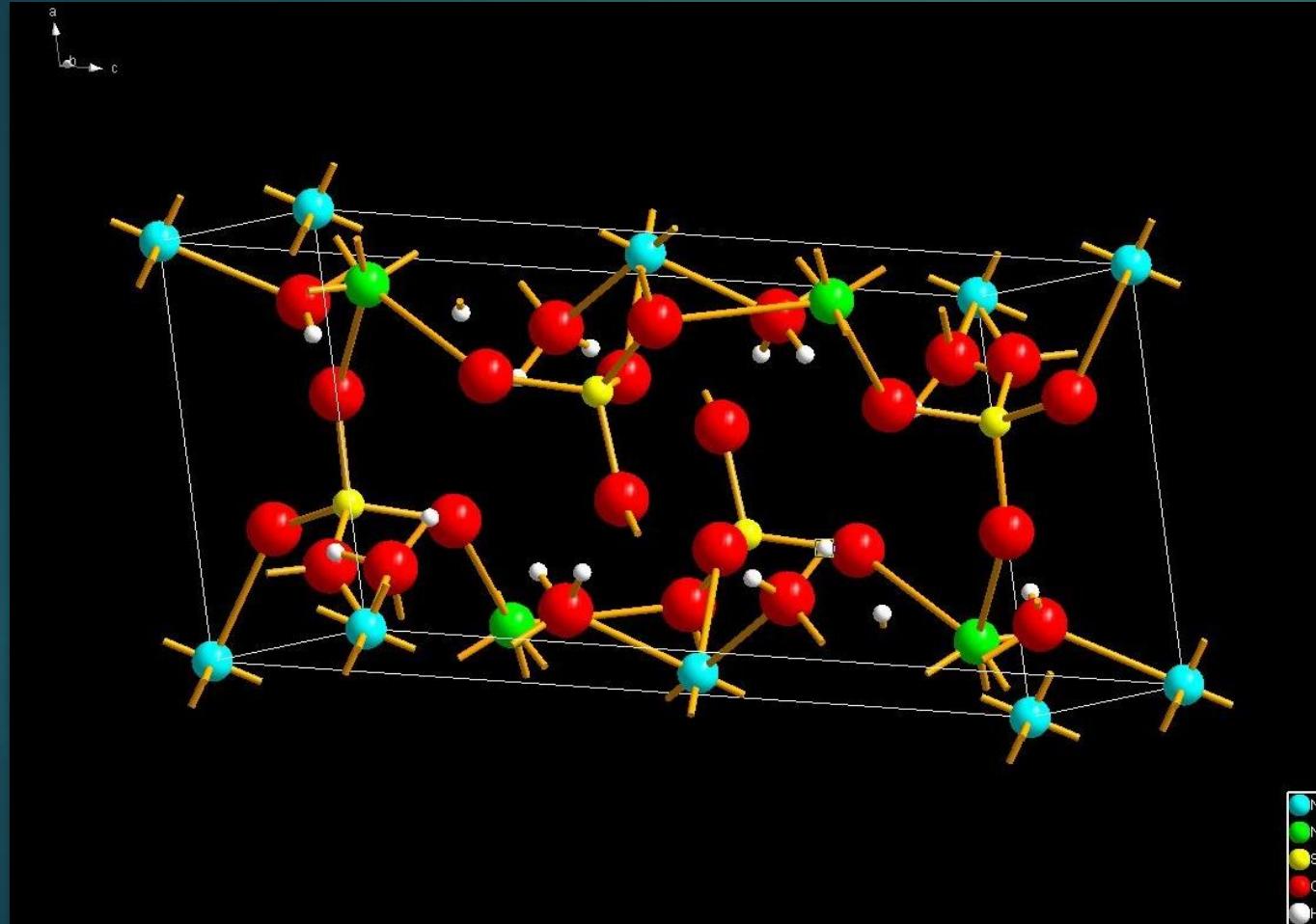
1



Nickelblödite
✓

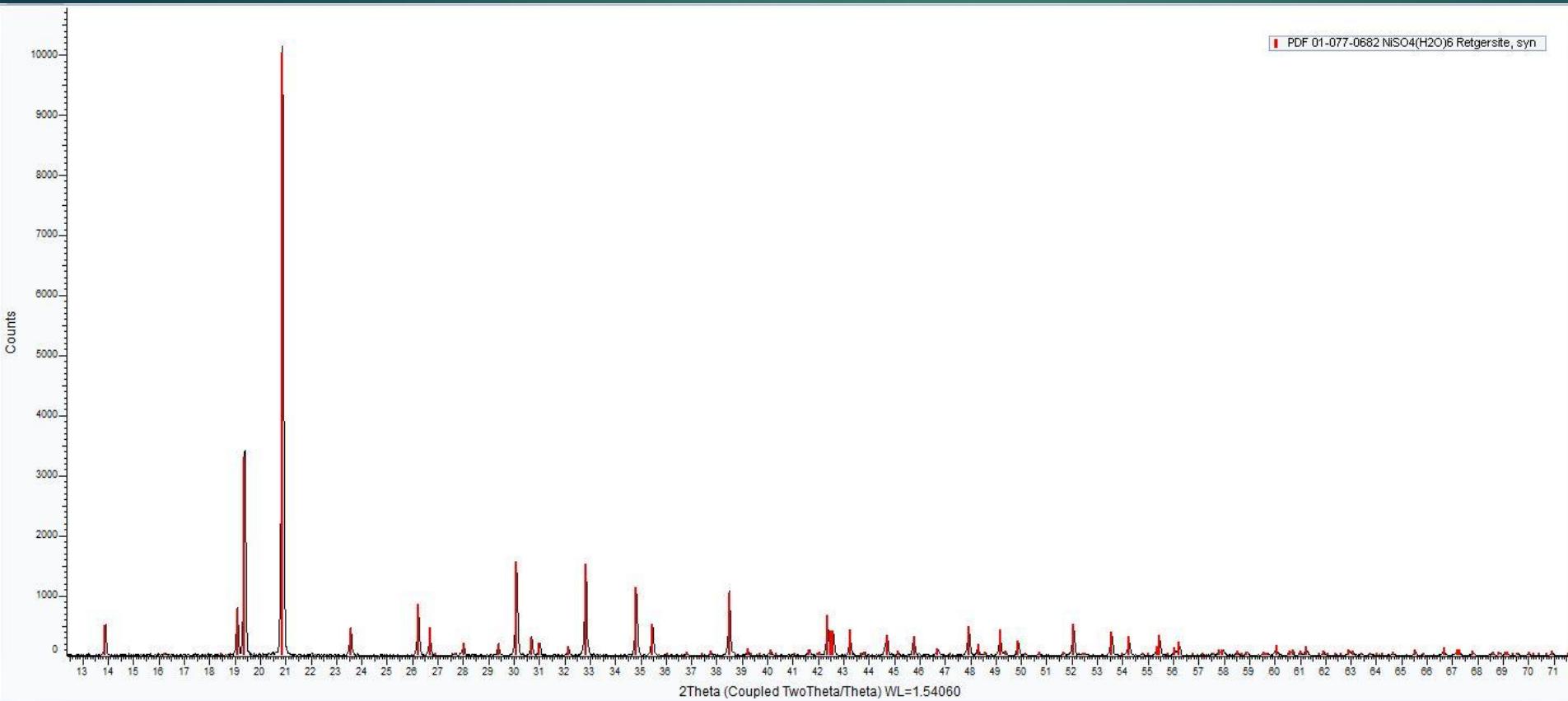
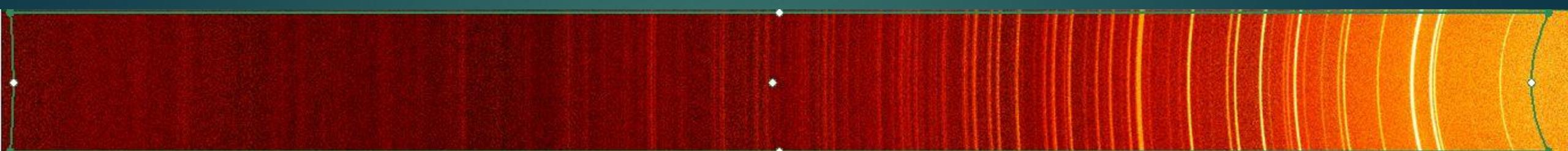


TOPAS SOLUTION

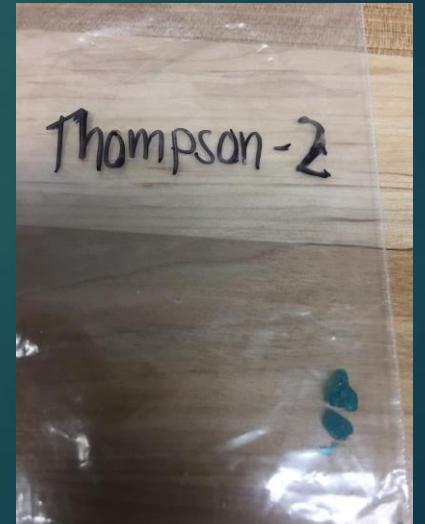


Nickelblödite
 $\text{Na}_2(\text{Ni,Mg})(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$

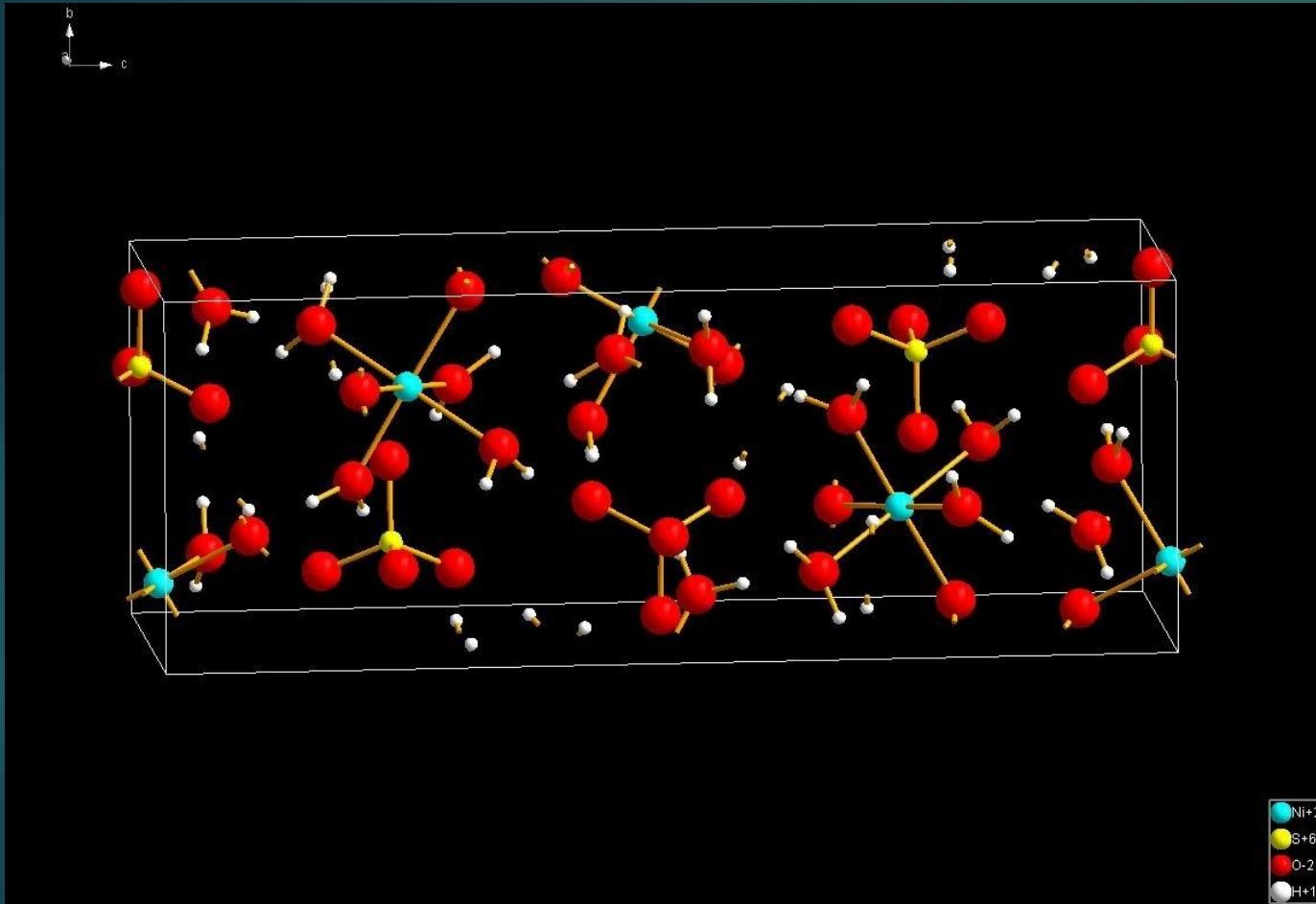
#2



Retgersite
✓



TOPAS SOLUTION

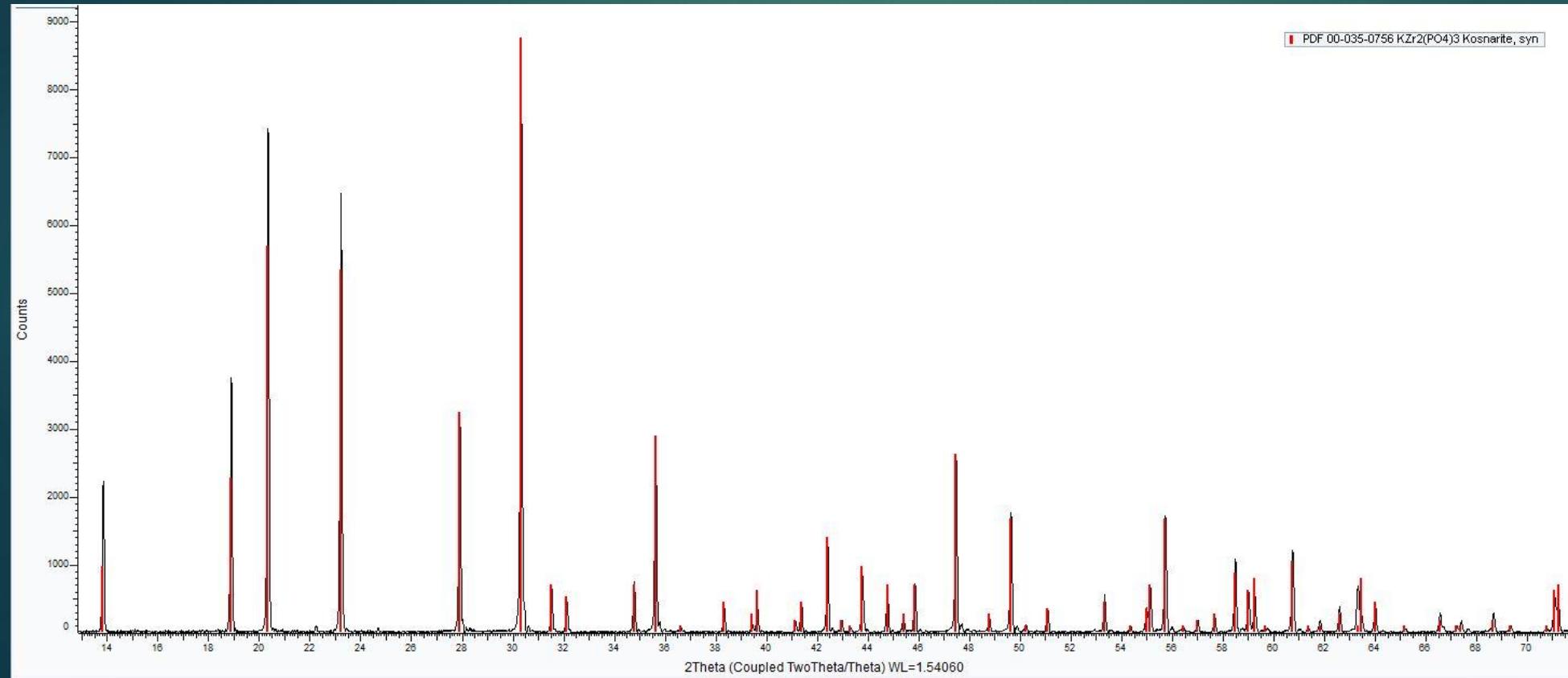
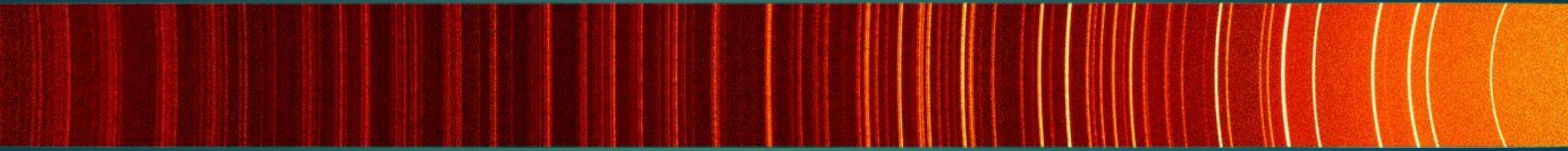


Retgersite

$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$

#3

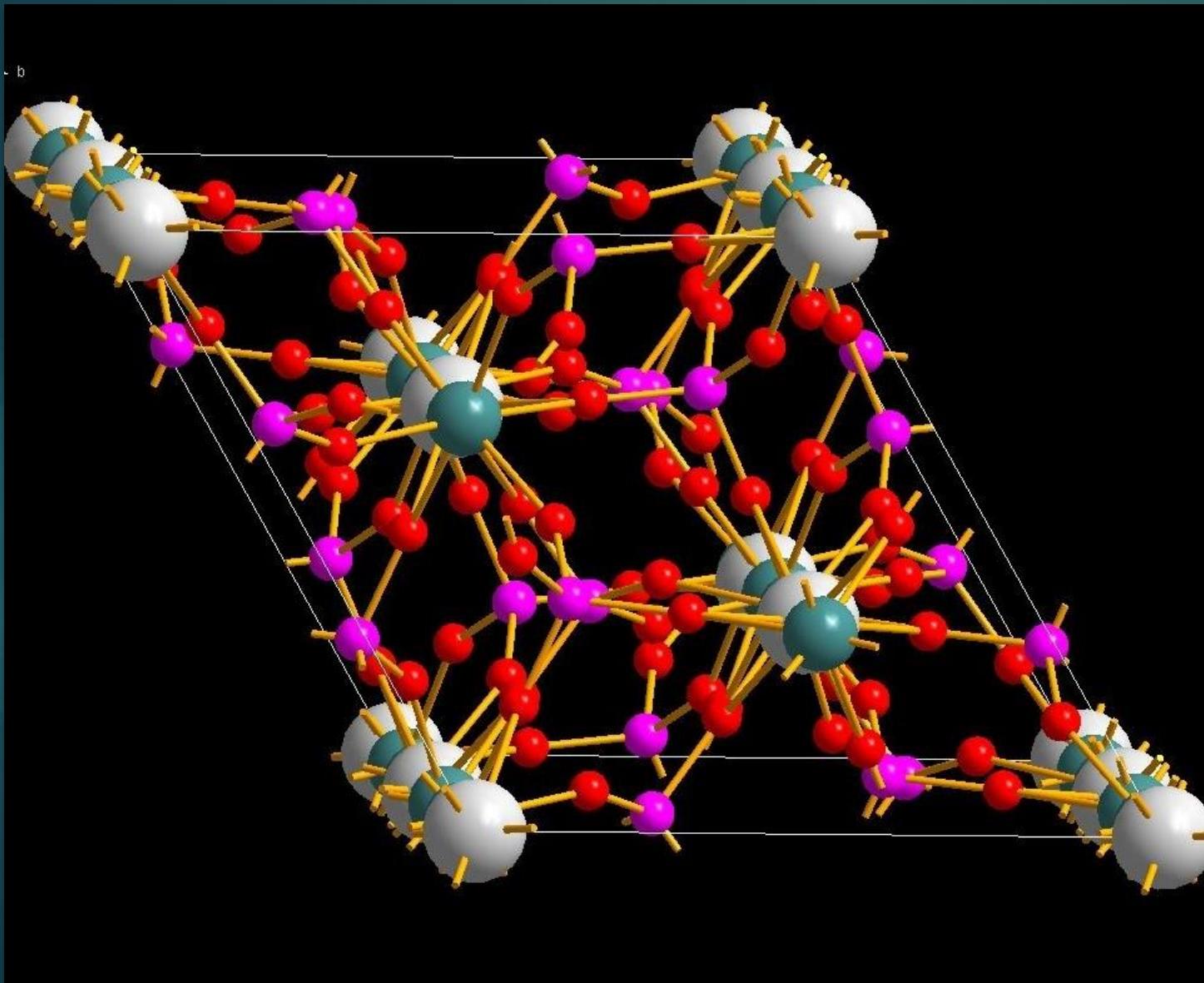
Best diffraction



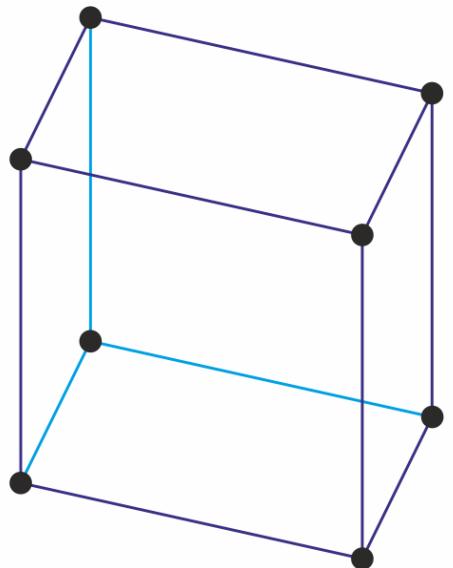
Kosnarite
✓



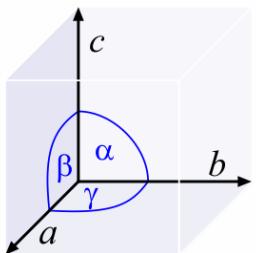
TOPAS SOLUTION



Kosnarite
 $\text{KZr}_2(\text{PO}_4)_3$



CRYSTAL LATTICE
triclinic



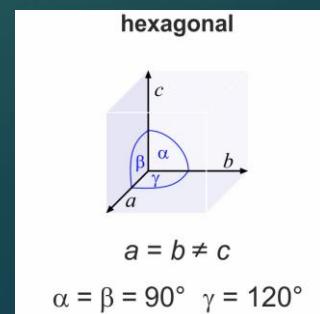
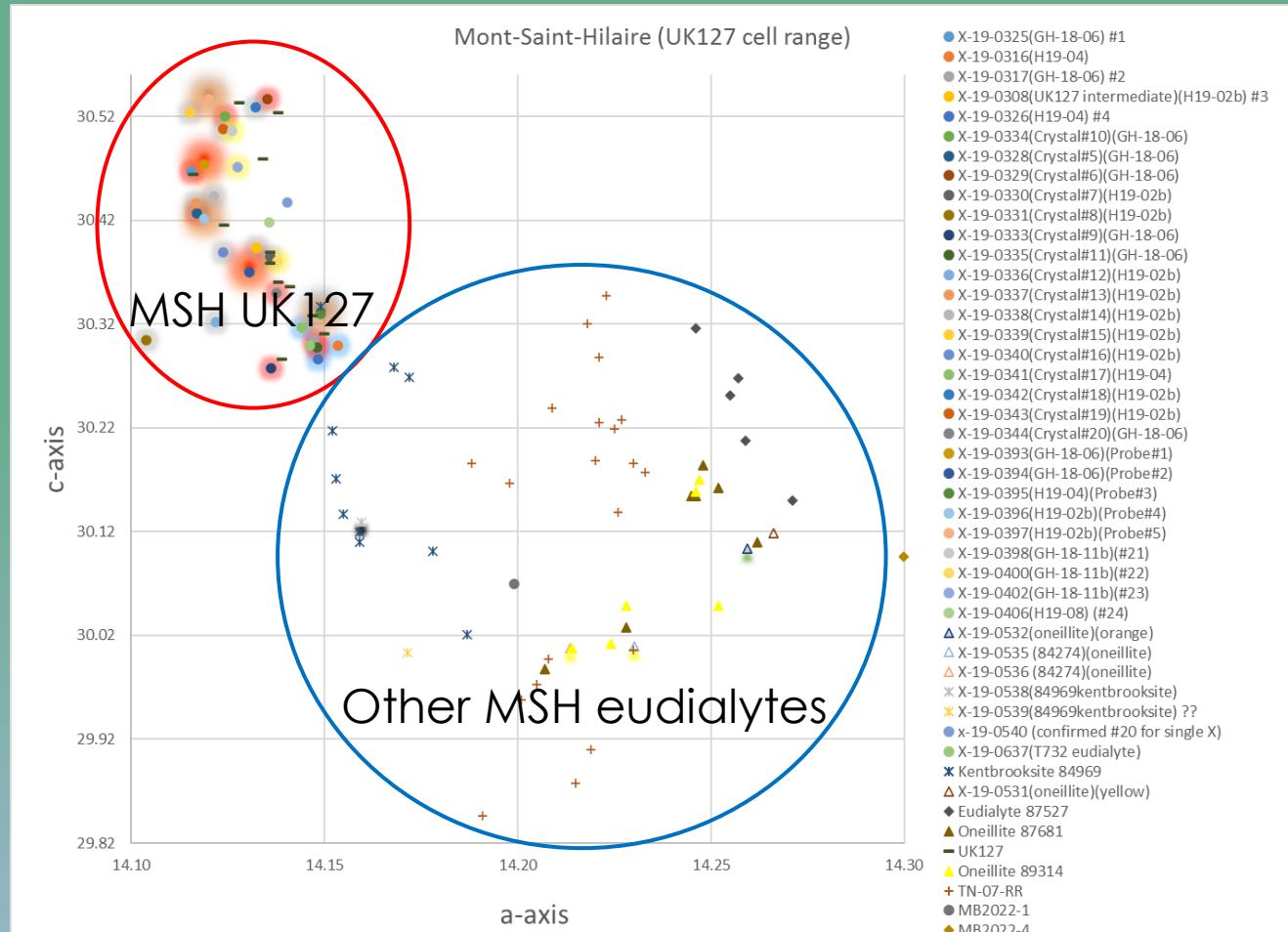
$$a \neq b \neq c$$
$$\alpha \neq \beta \neq \gamma \neq 90^\circ$$

Cell
dimension
refinement

Cell-dimension plot

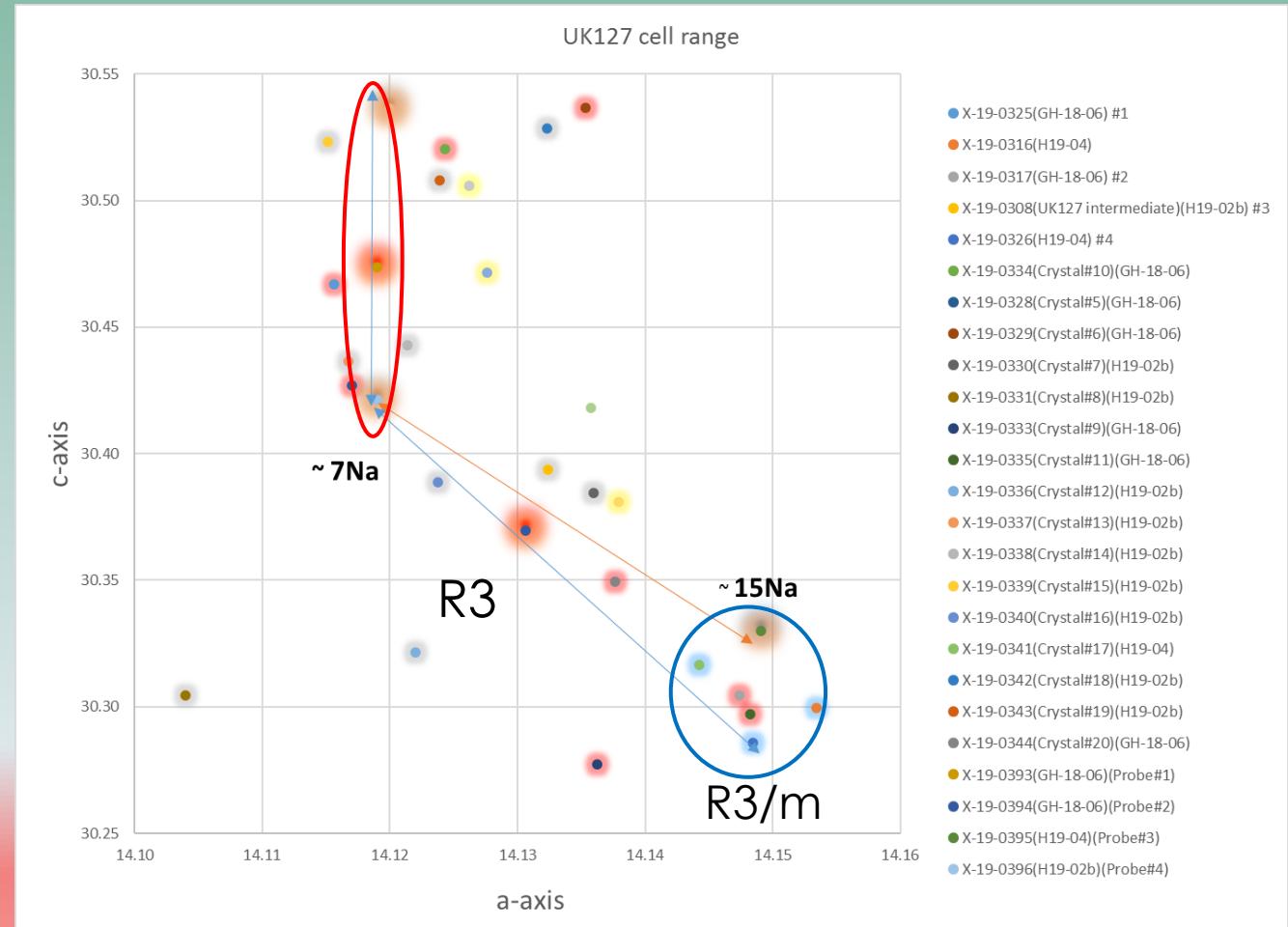
EUDIALYTE GROUP Mont-Saint-Hilaire

- ▶ Trigonal system
- ▶ Symmetry (hexagonal description)
- ▶ only a - and c -axes vary



Eudialyte group (UK127)

- ▶ Identification
 - ▶ Target crystals of interest
- ▶ SAVES TIME
 - ▶ full investigation of complex solid solutions
- ▶ Flexible geometry (rotation+)
 - ▶ Cell dimension of “Single crystal”



NEW MINERALS

(at the CMN)

INDEXING

CALCULATED PATTERN

CELL DIMENSIONS

New mineral description

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NISNITE, Ni_3Sn , A NEW NICKEL MINERAL SPECIES FROM THE JEFFREY MINE, ASBESTOS, QUEBEC

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ABSTRACT

Nisnite, ideally Ni_3Sn , was found during a re-examination of heazlewoodite crystals in rodingite samples from the Jeffrey mine, Asbestos, Quebec. It occurs as bronze-colored metallic, striated, blocky and square to rectangular tabular crystals of up to 100 μm in length, with groupings of ≤ 1 mm growing on heazlewoodite. Crystal groupings exhibit a boxwork-like habit. Reflectance measurements in air gave 43.2 (470 nm), 49.1 (546 nm), 53.2 (589 nm), and 59.0% (650 nm). Minerals closely associated with nisnite are chromite, diopside, grossular, heazlewoodite and shandite. The mineral is cubic, $P\bar{4}/m\bar{3}2m$, with unit-cell parameter refined using powder-diffraction data $a = 3.7349(6)$ Å, $V = 52.10(3)$ Å 3 , $Z = 1$, $D_{\text{calc}} = 9.41$ g/cm 3 . The average results of five and three electron-microprobe analyses on separate crystals gave Ni 57.88, Sn 40.17, sum 98.05 wt.% and Ni 59.24, Sn 41.00, sum 100.24 wt.%, corresponding to $\text{Ni}_{0.98}\text{Sn}_{1.02}$ on the basis of 4 apfu . The structure has been refined to an R index of 0.008% on the basis of 30 unique reflections. The structure of nisnite contains 12-coordinated Sn atoms (12 Ni) and 12-coordinated Ni atoms (8 Ni and 4 Sn). Among the three synthetic Ni_3Sn phases known, nisnite corresponds to the **cep** structure that has been synthesized at high pressure and high temperature.

Keywords: new mineral species, Ni_3Sn , nisnite, rodingite, ophiolite, polymorph, synthetic compound, Jeffrey mine, Asbestos, Québec.

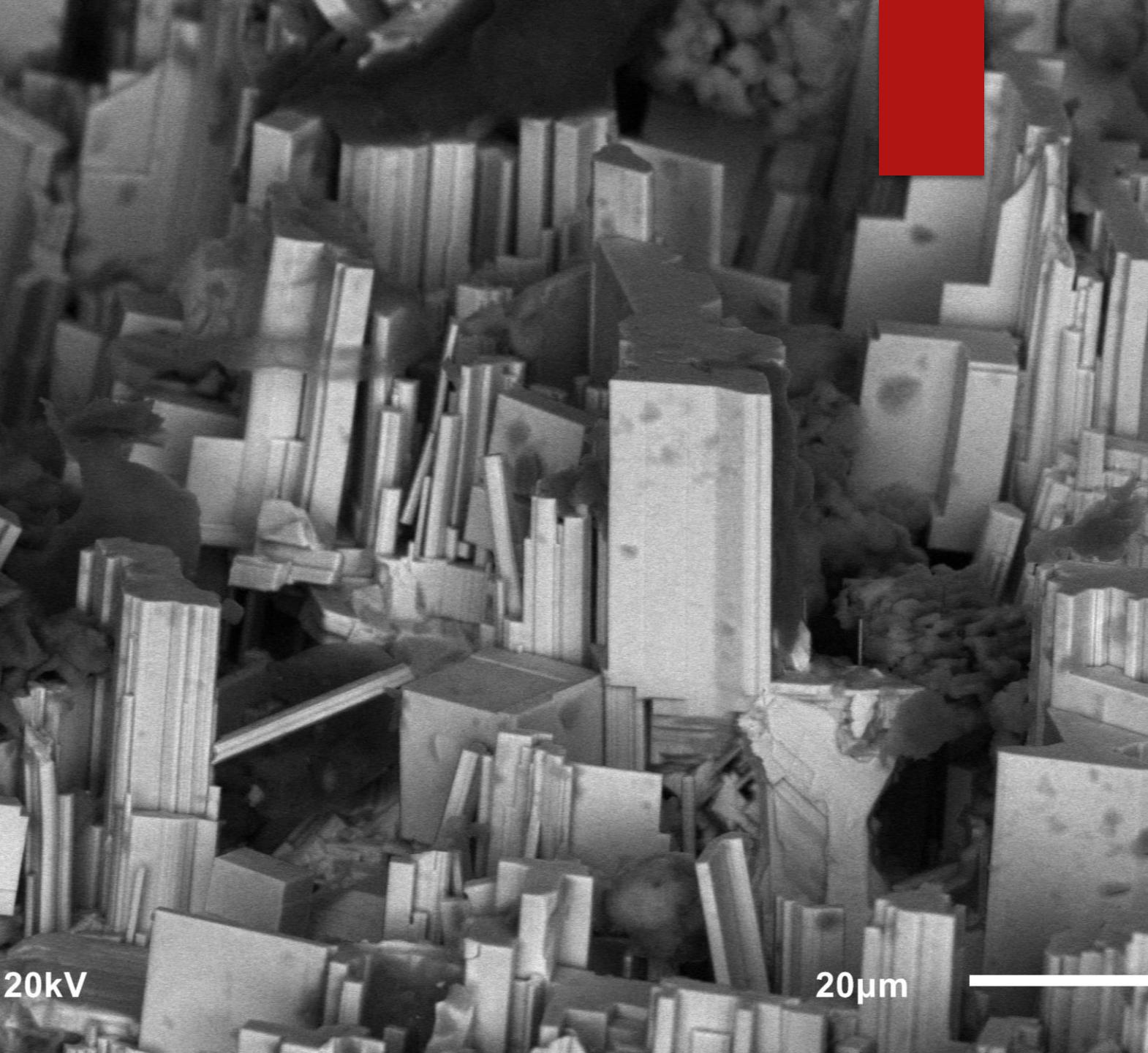
SOMMAIRE

Nous avons découvert la nisnite, de formule idéale Ni_3Sn , au cours d'un ré-examen de cristaux de heazlewoodite dans des échantillons de rodingite provenant de la mine Jeffrey, à Asbestos, Québec. Elle se présente en cristaux carrés ou rectangulaires striés, métalliques, de couleur bronze, atteignant 100 μm de longueur, en groupes de ≤ 1 mm développés sur la heazlewoodite. Ces groupes ont une morphologie réticulaire en boîte. La réflectance mesurée dans l'air donne 43.2 (470 nm), 49.1 (546 nm), 53.2 (589 nm), et 59.0% (650 nm). Lui sont étroitement associés chromite, diopside, grossulaire, heazlewoodite et shandite. Il s'agit d'un minéral cubique, $P\bar{4}/m\bar{3}2m$, avec un paramètre réticulaire a affiné à partir du spectre de diffraction X sur poudre égal à 3.7349(6) Å ($V = 52.10(3)$ Å 3 , $Z = 1$, $D_{\text{calc}} = 9.41$ g/cm 3). Les résultats de cinq et trois analyses avec une microsonde électronique sur deux cristaux sont: Ni 57.88, Sn 40.17, pour un total de 98.05%, et Ni 59.24, Sn 41.00, pour un total de 100.24% (poids), ce qui correspond à $\text{Ni}_{0.98}\text{Sn}_{1.02}$ sur une base de quatre atomes par formule unitaire. Nous en avons affiné la structure, jusqu'à un résidu R de 0.008% en utilisant 30 réflexions uniques. La structure de la nisnite contient des atomes de Sn à coordination 12 (avec 12 Ni) et des atomes de Ni à coordination 12 (avec 8 Ni et 4 Sn). Parmi les trois polymorphes de Ni_3Sn connus, la nisnite correspond à la structure **cep**, synthétisée à pression et température élevées.

(Traduit par la Rédaction)

Mots-clés: nouvelle espèce minérale, Ni_3Sn , nisnite, rodingite, ophiolite, polymorphe, composé synthétique, mine Jeffrey, Asbestos, Québec.

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IMA Voting (Nisnite Ni_2Sn)

- ▶ Mandate
 - ▶ validate that the species is “new” based on existing classifications
 - ▶ Verify data quality
 - ▶ Named in line with provided guidelines
- ▶ 2 years to publish
- ▶ Type specimen
 - ▶ deposited in a museum collection



IMA – Commission on New Minerals, Nomenclature and Classification (CNMNC)
4 January 2010

IMA 2009-083 NISNITE

	Yes	No	Abstain
Mineral	18		
Name	17		1

Consequently both the name and the mineral have been approved.

COMMENTS ON THE MINERAL:

Those who voted YES made the following comments:

1. Does the mineral contain only Ni and Sn or were some other elements measured?
2. The complete results of the crystal structure study (especially atom coordinates) are missing in the proposal.
3. Although the analytical data are good, it would be worthwhile, for the sake of rigour, to show microprobe data for other elements that might substitute into this mineral.
4. The description of the mineral is rather short and formalistic.
5. Good and quite complete description.
6. Very complete mineralogical description.
7. There are some curious errors in Table 1 regarding summations and the range for Ni analytical data. It seems strange that Ni and Sn are the only elements detectable by EMPA.

COMMENTS ON THE NAME:

Those who voted YES made the following comments:

1. In line with nisbite.

Those who ABSTAINED made the following comments:

1. The name is too similar to nisbite (NiSb_2).

XRD lab : New mineral discoveries (40+)



Manganoarrojasite-(KNa) (2020)



Bussyite-(Ce) (2009)



Qaqarssukite-(Ce) (2006)



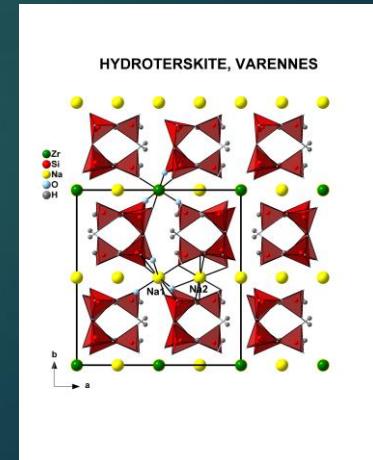
Bussyite-(Y) (2015)



Alicewilsonite-(YCe) (2021)



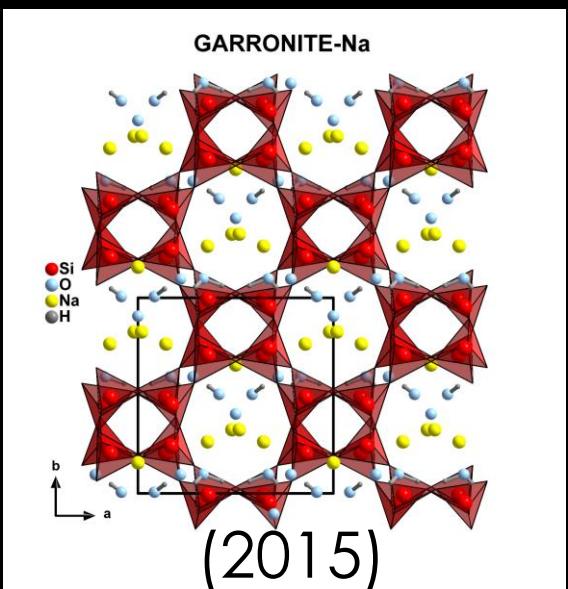
Eldragonite (2011)



Hydroterskite (2015)



Brumadoite (2008)

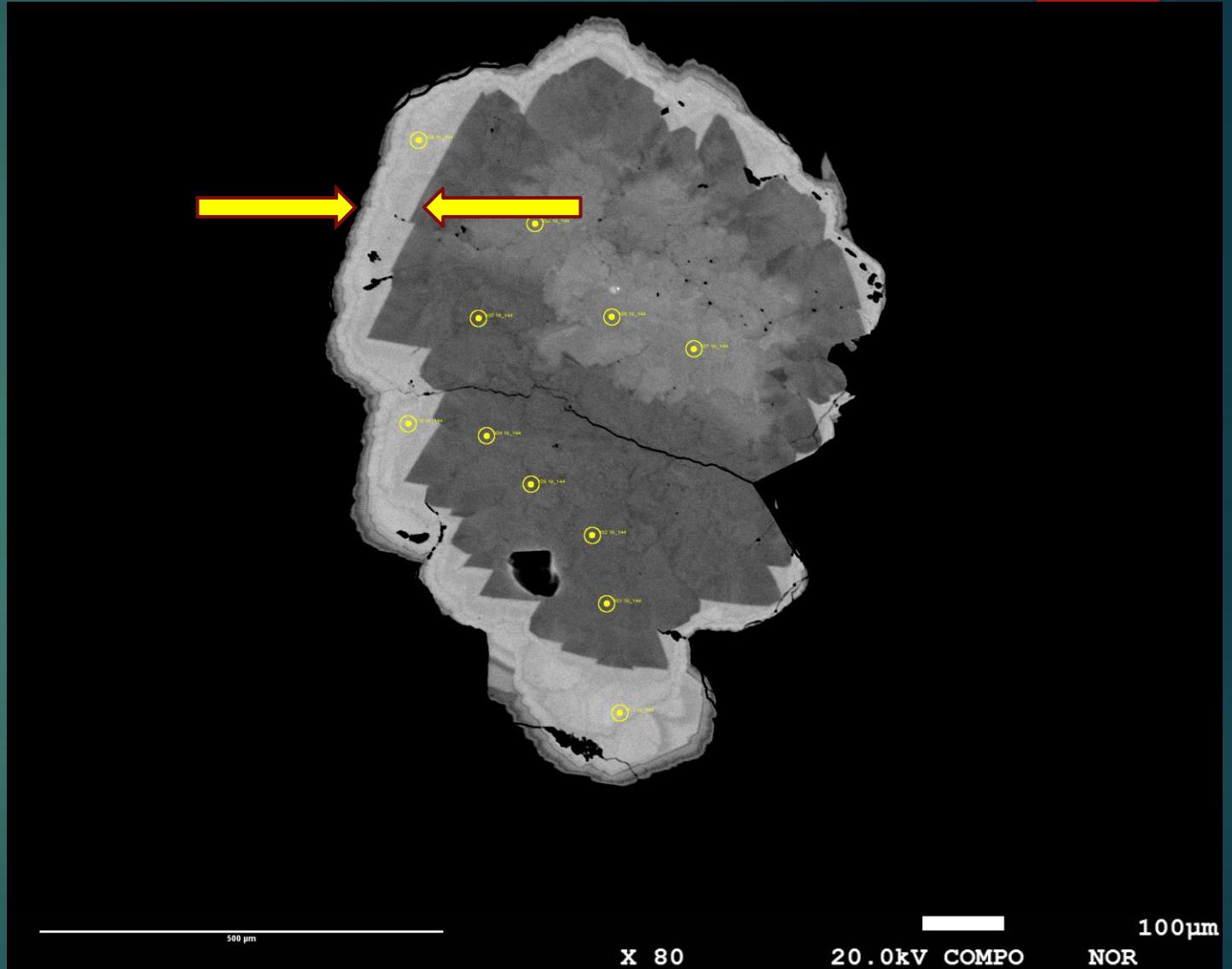


Arisite-(La) and -(Ce) (2010)



Type
Collection

More to
come



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THANK
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