New Rigaku solutions for X-ray and electron diffraction

Laurent LOOS

Responsable commercial

Rigaku Europe SE

Forum Instrumentation de Toulouse

17 Juin 2024

Rigaku Corporate Profile

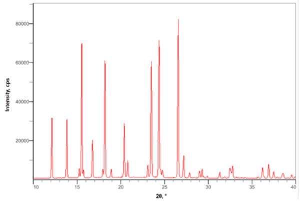
- Business Description: Manufacturing & Sales of Scientific Instruments
- Address: Headquarters・Tokyo Factory・X-ray research Lab
  Matsubara-cho, 3-9-12 Akishima, Tokyo 196-8666

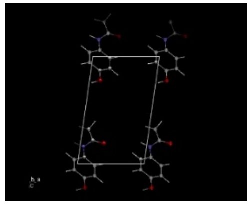
  Osaka Office & Factory
  Akaoji-cho 14-8, Takatsuki, Osaka 569-1146

  Yamanashi Factory
  Wakamiko 4495-8 Sutama-cho, Hokuto, Yamanashi 408-0112
- Representative: President & CEO Hikaru Jun KAWAKAMI
- Founded: 6th December 1951
- Capital: 100 Million Japanese Yen
- Employees: Approx. 1,800 Group Employees (2022)
- Annual Sales: 63 Billion Japanese Yen ($420 million) (FY2022)

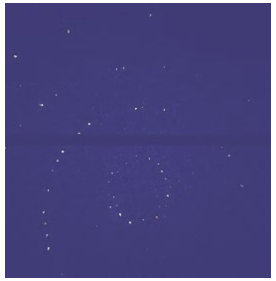
Rigaku's high-performance solutions

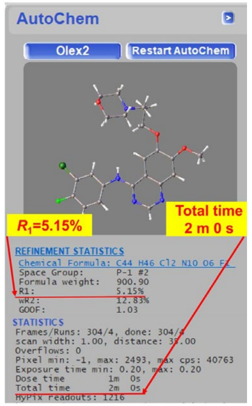
SmartLab
AUTOMATED MULTIPURPOSE X-RAY DIFFRACTOMETER



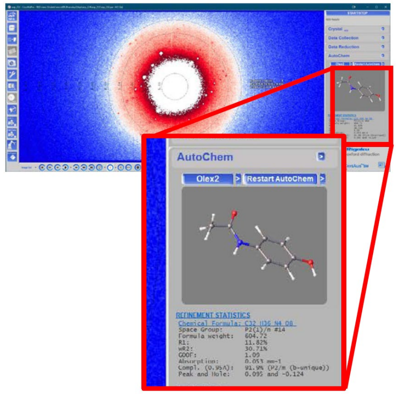


XtaLAB Synergy-R/-DW VHF
SINGLE CRYSTAL X-RAY DIFFRACTOMETER



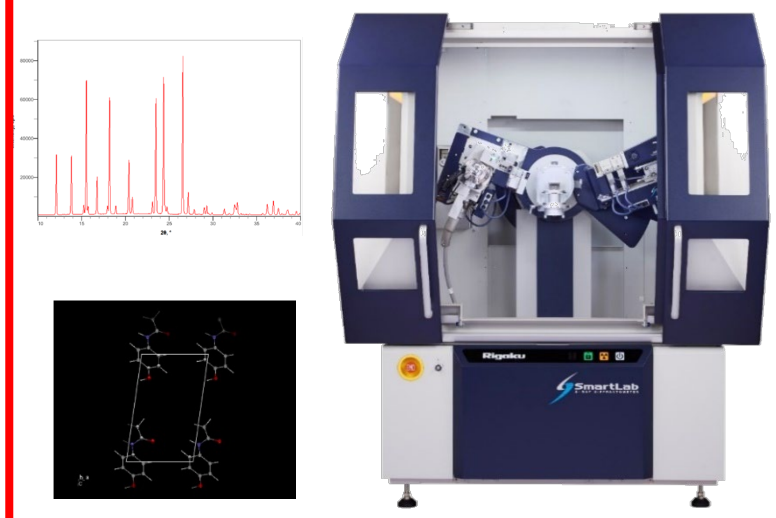


XtaLAB Synergy-ED
SINGLE CRYSTAL ELECTRON DIFFRACTION DIFFRACTOMETER



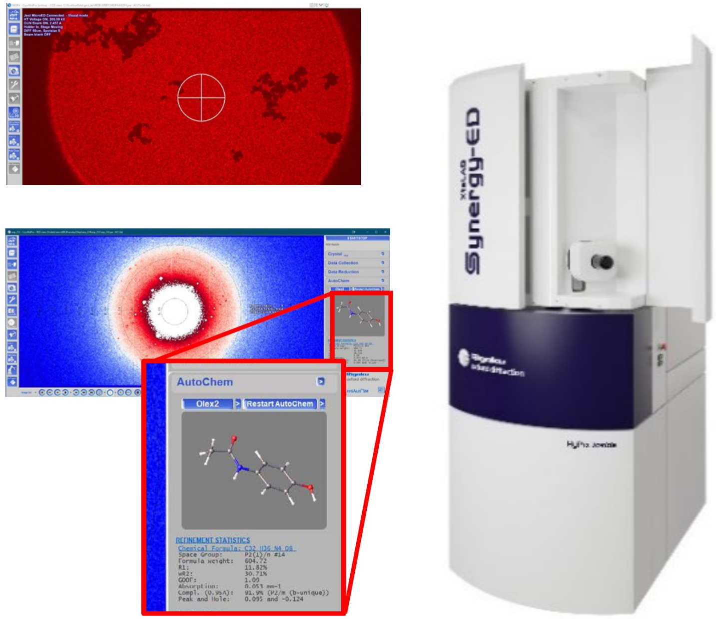
Rigaku's high-performance solutions

SmartLab
AUTOMATED MULTIPURPOSE X-RAY DIFFRACTOMETER



XtaLAB Synergy-R/-DW VHF
SINGLE CRYSTAL X-RAY DIFFRACTOMETER

XtaLAB Synergy-ED
SINGLE CRYSTAL ELECTRON DIFFRACTION DIFFRACTOMETER

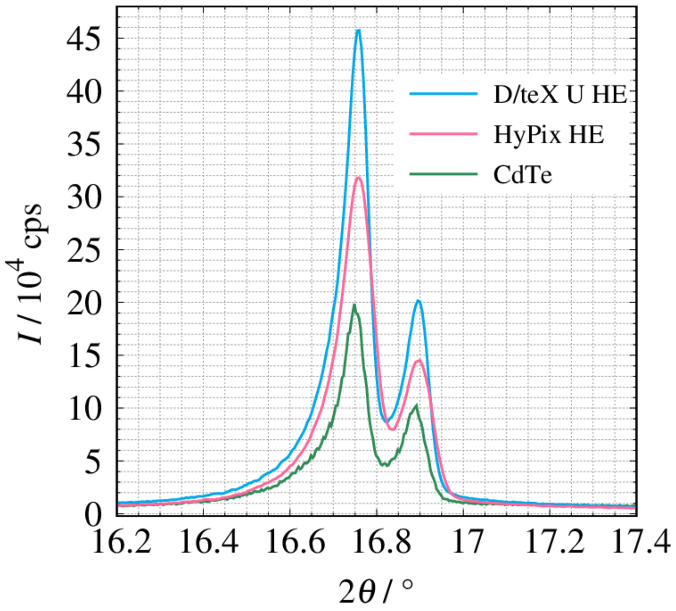


RIGAKU SMARTLAB

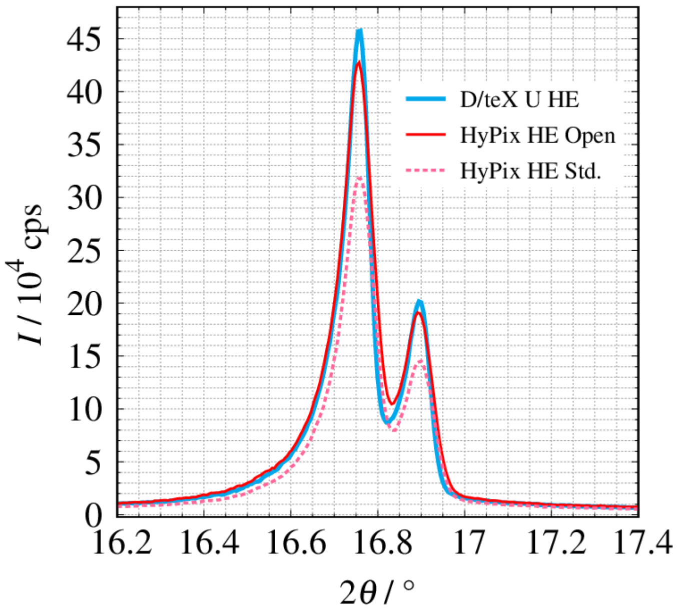
PDF Analysis: Rigaku Recommended Configuration

1. Source: Photon MAX: 6.0kW(Ag) 9.0kW(Mo)
2. Beam: Convergent beam CBO-E
3. Detector: HyPix 3000 HE or D/teX Ultra 250HE
4. Slit: New wide slit for HyPix3000
5. Software: SmartLab Studio II PDF plugin

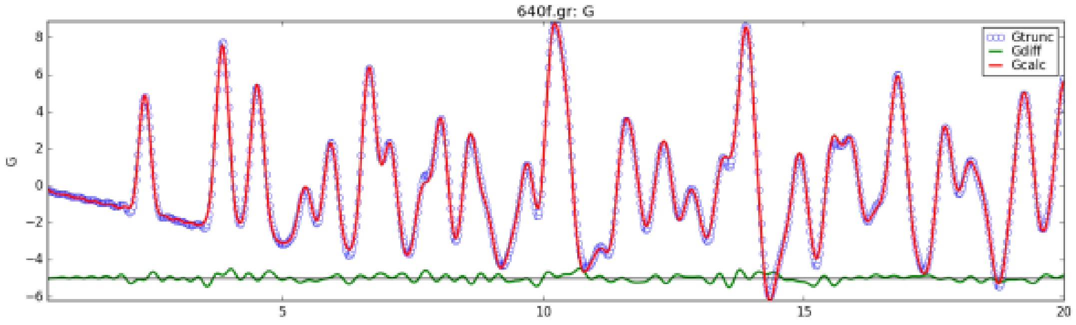
SmartLab Studio II
Guidance™ expert system software



Photon MAX and Detector

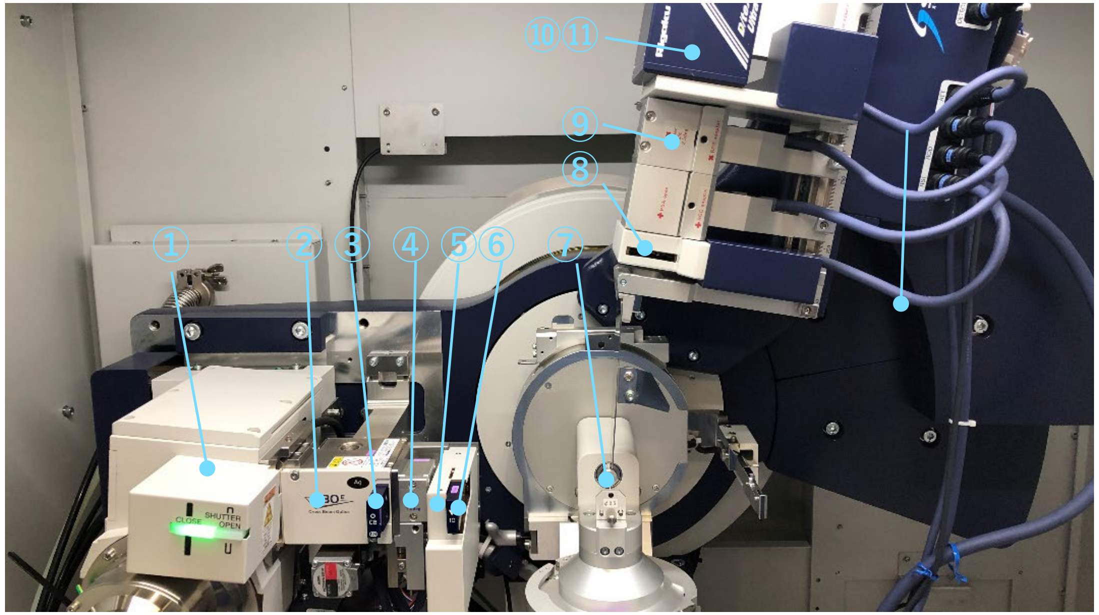


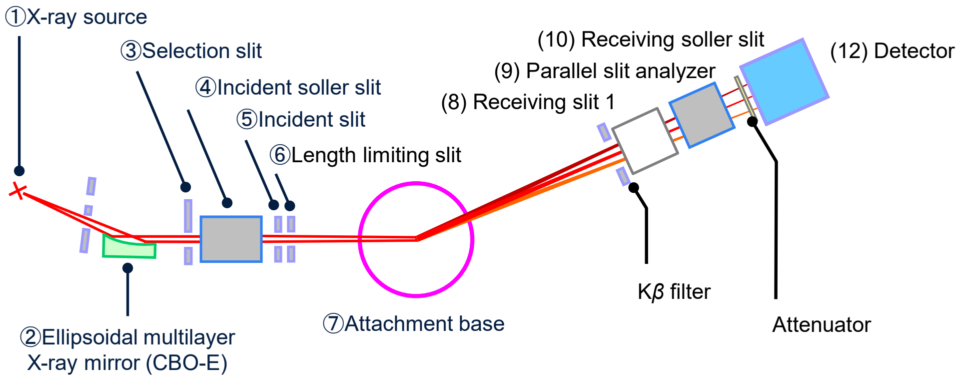
New wide slit



Software

SmartLab Configuration (CB) for PDF application





Apparatus configuration	
X-ray source Filament type Voltage - Current	Ag Kα （λ=0.560880 Å）, Mo Kα （λ=0.710741 Å） Fine focus Ag: 45 kV - 133 mA, Mo: 45 kV - 200 mA
Goniometer radius	300 mm
Optics	CB
Selection Slit Soller slit Length limit slit Divergence scatter cut parts	CBO-E + CB Incident soller slit 5.0° 10 mm Disuse
Attachment base Attachment head Capillary material Capillary diameter	Standard ATT. base Capillary rotation attachment Borosilicate glass 0.5 mm
Receiving scatter cut parts Receiving optical unit 1 Receiving optical unit 2	Scattering protector PSA OPEN Receiving soller slit 5.0°
Direct beam stopper Detector	Use D/teX Ultra 250 HE

Measurement condition	
Measurement mode Energy mode	1D (scan） Standard
Scan axis Scan range Step Speed Number of times	2θ 3-157.5° 0.02° 1 °/min 3
Incident slit Receiving slit 1 Receiving slit 2	0.9mm 15.6 mm 19.1 mm (Open)

RIGAKU SMARTLAB

New 2D high resolution energy detector (e< 340eV)

- Rigaku XSPA-400ER with and energy resolution of 340eV
- Allows to suppress the Kβ lines of Cu
- Better fluorescence suppression

◆Features

1 High energy resolution

✓XRF suppression
✓Kβ peak suppression

2 Pixel detector

✓ 2 dimensional detector

3 Wide dynamic range

✓Linear response over wide dynamic range

4 Filter less

✓ Kβ peak suppression

5 Selectable high intensity/ high angular resolution modes

✓ High intensity mode
✓ High angular resolution mode

RIGAKU SMARTLAB

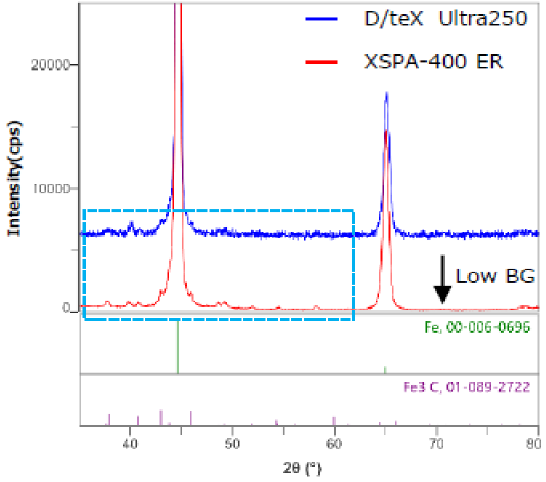
New 2D high resolution energy detector (e< 340eV)

Examples of application

- Improuve the trace phase detection in steel samples

◆**High energy resolution**

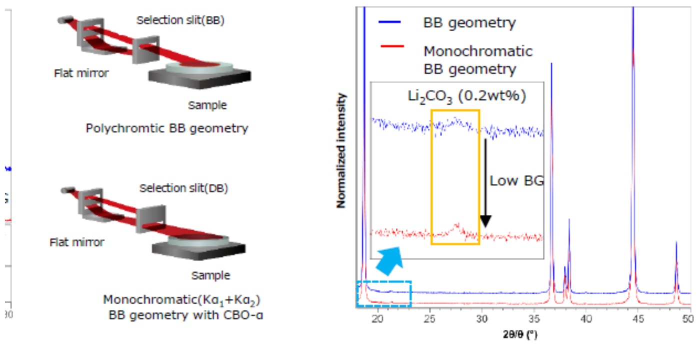
Detection of Fe_3C in CrMo steel.
Low BG improves S/N ratio and
trace phases are observed.



- In combination with the CBO-α we can improve the backgroud and have a better detection of Li2CO3 phase in the sample

◆**XSPA-400 ER with CBO-α for optimal BB data**

Measurement of cathode NMC, $Li(Ni,Mn,Co)O_2$
Low background from XSPA-400 further reduced using CBO-α.

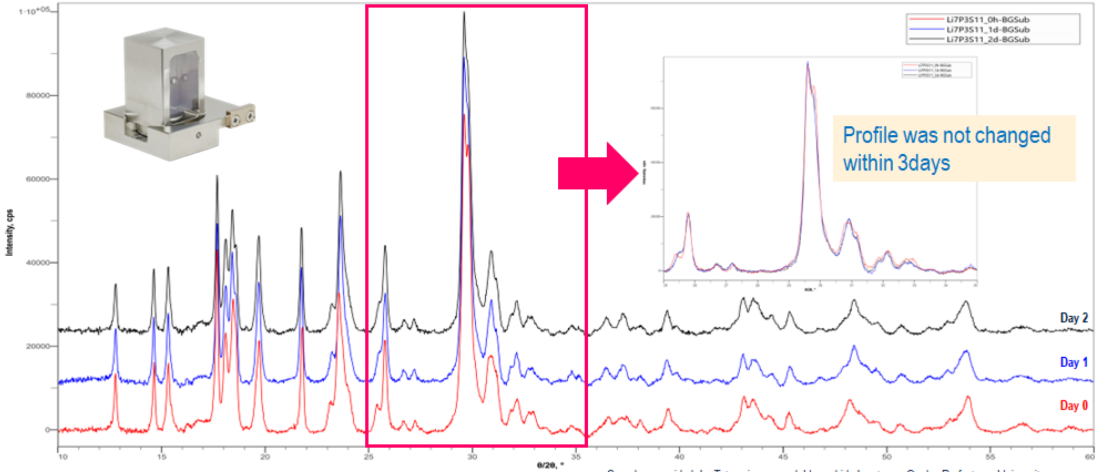


RIGAKU XRD PRODUCT LINE

New Air-Tight Sample holders

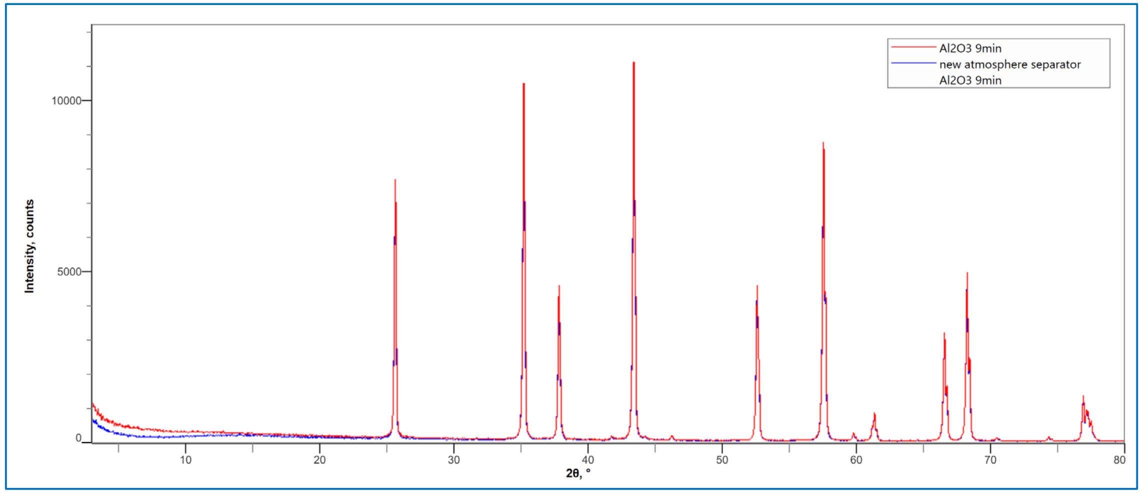
- Long time stability : more than 3 days

Airtightness test: $Li_7P_3S_{11}$ (Sulfide-based solid electrolyte)



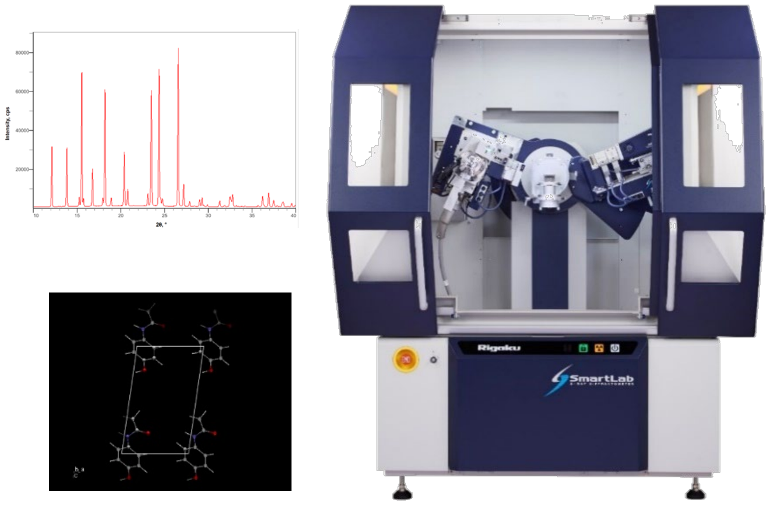
Samples provided by Tatsumisago and Hayashi Laboratory, Osaka Prefecture University

- Low signal attenuation : transmssion better than 97%



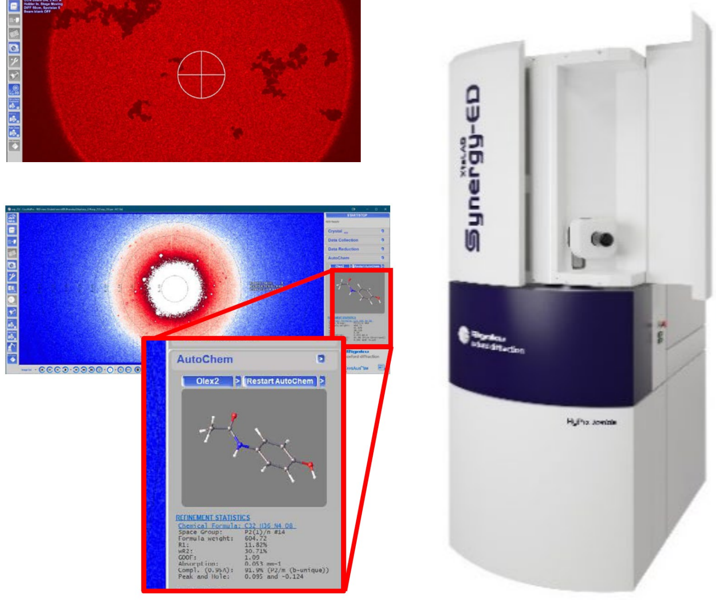
Rigaku's high-performance solutions

SmartLab
AUTOMATED MULTIPURPOSE X-RAY DIFFRACTOMETER



XtaLAB Synergy-R/-DW VHF
SINGLE CRYSTAL X-RAY DIFFRACTOMETER

XtaLAB Synergy-ED
SINGLE CRYSTAL ELECTRON DIFFRACTION DIFFRACTOMETER



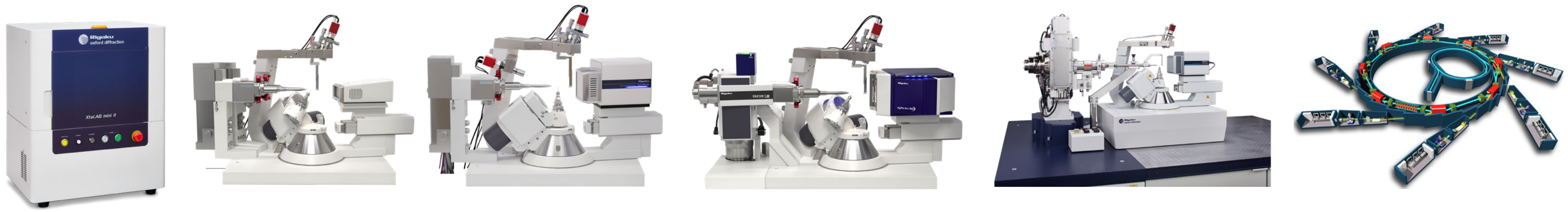
XtaLAB Synergy-ED

- A dedicated electron diffractometer, **made for crystallographers**
- Highly optimized hardware design for 3D-ED
- Seamlessly integrated hard- and software, from instrument control to structure solution

ELECTRON DIFFRACTION: BREAKING THE 1-MICRON BARRIER

- X-rays reach down to micron size crystals
- Electrons reach *up* to micron sized samples, due to orders of magnitude stronger interaction and weaker radiation damage
- X-ray and electron diffraction are **complementary** techniques, to study samples from hundreds of microns down to the nanoscale

1 micron



~300 µm

Decreasing sample size

~1 µm

~0.3 µm

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…many more in preparation

METAL-ORGANIC FRAMEWORKS

As a Prime Target of Electron Diffraction

Angewandte International Edition Chemie
GDCh
A Journal of the German Chemical Society

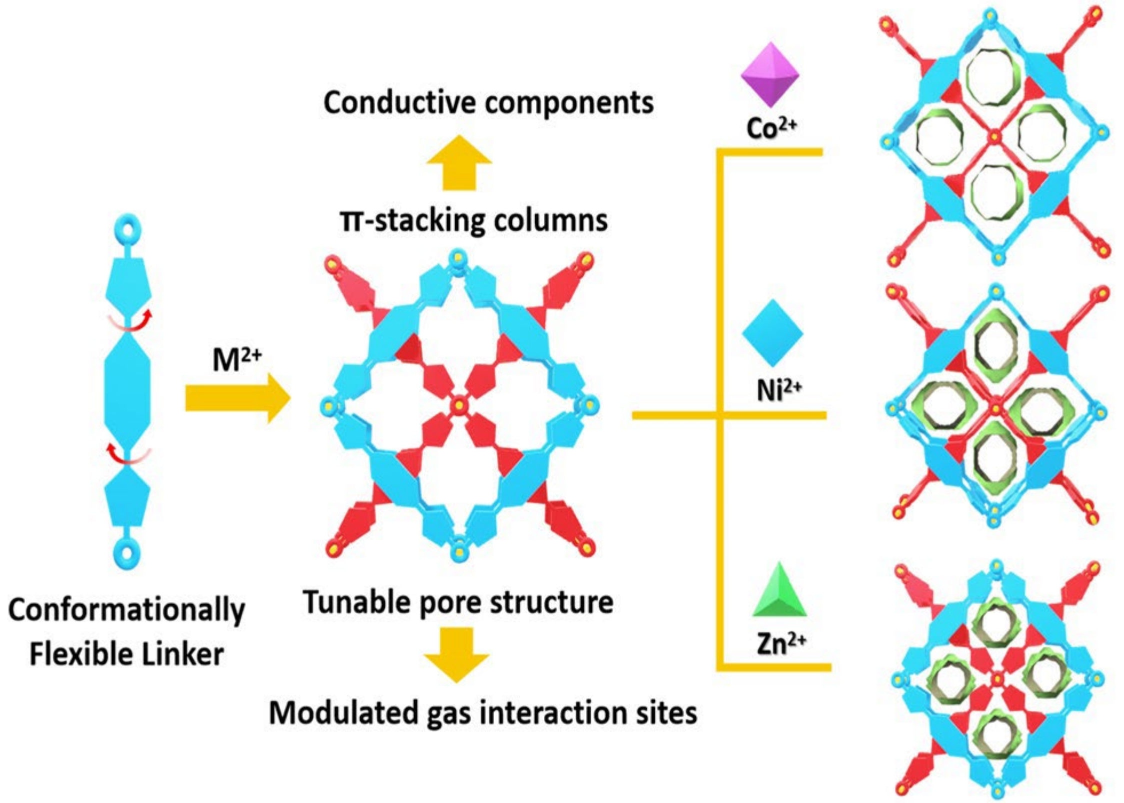
Communication

Fine Pore-Structure Engineering by Ligand Conformational Control of Naphthalene Diimide-Based Semiconducting Porous Coordination Polymers for Efficient Chemiresistive Gas Sensing

Ziqian Xue, Jia-Jia Zheng, Yusuke Nishiyama, Ming-Shui Yao, Yoshitaka Aoyama, Zeyu Fan, Ping Wang, Takashi Kajiwara, Yoshiki Kubota, Satoshi Horike, Ken-ichi Otake, Susumu Kitagawa

First published: 15 November 2022 | https://doi.org/10.1002/anie.202215234

Unfortunately, large single-crystals suitable for single crystal X-ray diffraction (SCXRD) were probably unavailable due to the poor solubility of pyNDI ligands. The three-dimensional electron diffraction (3DED) technique is a powerful method for the structure determination of sub-microcrystals.[12] Therefore, the crystal structures of Co-pyNDI, Zn-pyNDI, and Ni-pyNDI were characterized by the 3DED method (Figures 2a-c). Sets of ED patterns were observed under continuous rotation from 30° to +30° using SynergyED equipment (RIGAKU/JEOL, Tokyo). Each ED pattern was collected every 0.25° and 0.5° with an exposure time of 1.25s and 2.5 s, for NI-pyNDI and Co/Zn-pyNDI, respectively; thus, a data collection time for each set is 5 min. Ab initio structure determination was performed by utilizing SHELXT.



METAL-ORGANIC FRAMEWORKS

As a Prime Target of Electron Diffraction

nature communications

Article

https://doi.org/10.1038/s41467-024-45363-3

A zero-valent palladium cluster-organic framework

Received: 13 September 2023

Accepted: 19 January 2024

Published online: 08 February 2024

Check for updates

Xiyue Liu[1], James N. McPherson[1], Carl Emil Andersen[1], Mike S. B. Jørgensen[1], René Wugt Larsen[1], Nathan J. Yutronkie[2], Fabrice Wilhelm[2], Andrei Rogalev[2], Mónica Giménez-Marqués[3], Guillermo Mínguez Espallargas[3], Christian R. Göb[4] & Kasper S. Pedersen[1]

Journal of Materials Chemistry C

ROYAL SOCIETY OF CHEMISTRY

PAPER

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Cite this: J. Mater. Chem. C, 2023, 11, 11460

Vapor-phase synthesis of low-valent metal–organic frameworks from metal carbonyl synthons†

Carl Emil Andersen,[a] James Neill McPherson,[a] Mónica Giménez-Marqués,[b] Jingguo Li,[c] Mariusz Kubus,[a] Sho Ito,[d] Christian R. Göb,[e] Sascha Ott,[c] René Wugt Larsen,[a] Guillermo Mínguez Espallargas[b] and Kasper S. Pedersen*[a]

Inorganic Chemistry

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Article

Systematic Study on Zirconium Chelidamates: From a Molecular Complex to a M-HOF and a MOF

Mirjam P. M. Poschmann, Özge Alan, Sho Ito, Christian Näther, Gernot Friedrichs, and Norbert Stock*

Cite This: Inorg. Chem. 2023, 62, 12252–12259

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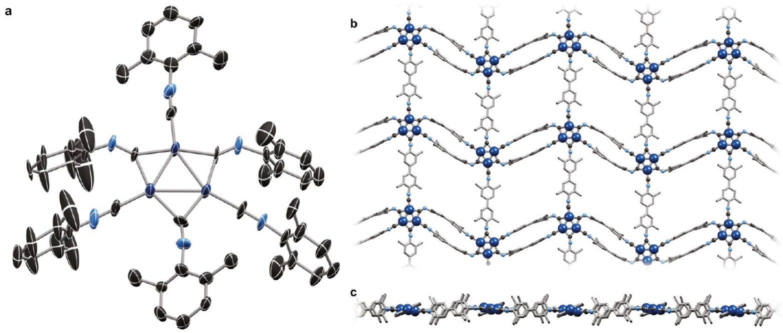

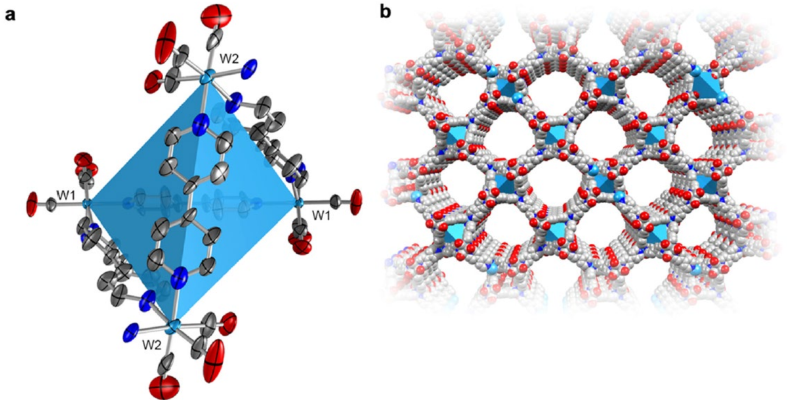

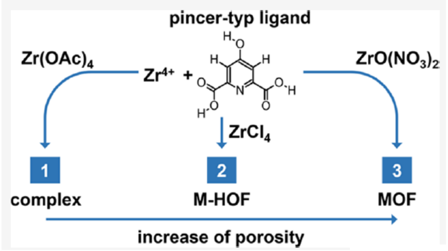

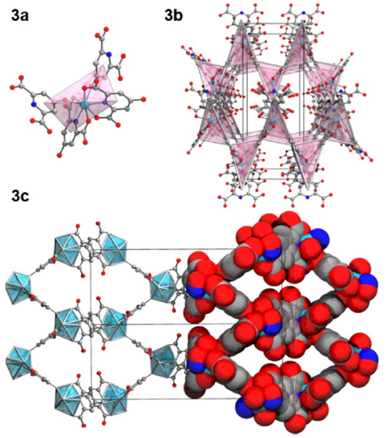

COFS, ZIFS, 1D/2D COORDINATION NETWORKS

As a Prime Target of Electron Diffraction

Rigaku oxford diffraction

communications chemistry

ARTICLE

https://doi.org/10.1038/s42004-023-00853-1 OPEN

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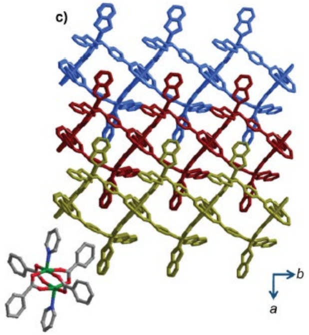
Selective sorption of oxygen and nitrous oxide by an electron donor-incorporated flexible coordination network

Mohana Shivanna[1], Jia-Jia Zheng[1,2], Keith G. Ray[3], Sho Ito[4], Hirotaka Ashitani[5], Yoshiki Kubota[5,6], Shogo Kawaguchi[7], Vitalie Stavila[8], Ming-Shui Yao[1], Takao Fujikawa[1], Ken-ichi Otake[1✉] & Susumu Kitagawa[1✉]



RESEARCH ARTICLE

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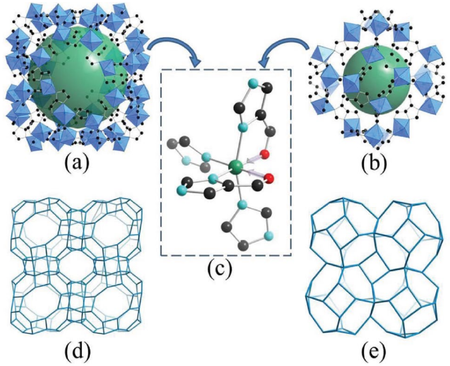
Carbonyl-Supported Coordination in Imidazolates: A Platform for Designing Porous Nickel-Based ZIFs as Heterogeneous Catalysts

*Aljaž Škrjanc, Dominik Jankovič, Anton Meden, Matjaž Mazaj, Erik Svensson Grape, Martin Gazvoda, and Nataša Zabukovec Logar**



research papers

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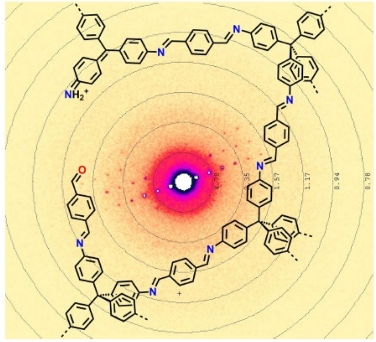
ELECTRON CRYSTALLOGRAPHY

Analysis of COF-300 synthesis: probing degradation processes and 3D electron diffraction structure

Laurens Bourda,[a,b] Subhrajyoti Bhandary,[a] Sho Ito,[c] Christian R. Göb,[d] Pascal Van Der Voort[b] and Kristof Van Hecke[a]*



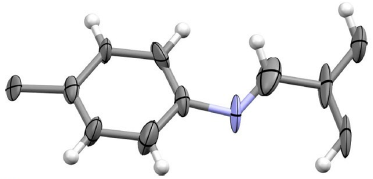


Figure 5
Asymmetric unit of COF-300 as determined by 3DED. Thermal displacement ellipsoids are shown at the 50% probability level.

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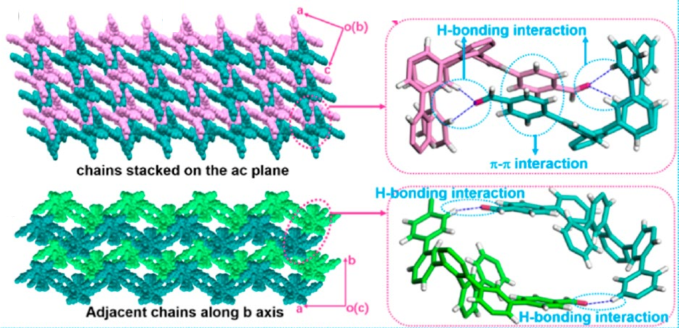
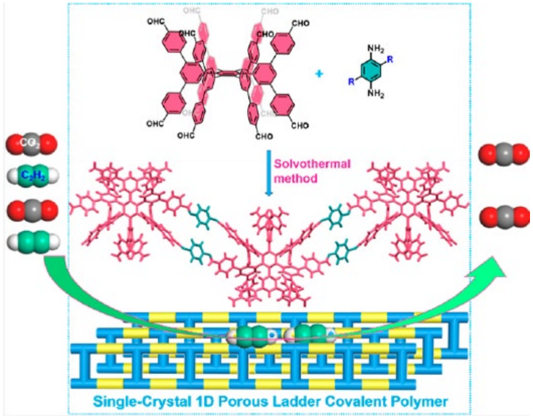
Single-Crystal One-Dimensional Porous Ladder Covalent Polymers

Yi Yang, En Lin, Sa Wang, Ting Wang, Zhifang Wang, and Zhenjie Zhang*

Cite This: *J. Am. Chem. Soc.* 2024, 146, 782–790 Read Online





METAL-ORGANIC FRAMEWORKS

As a Prime Target of Electron Diffraction

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Tailoring Hydrophobicity and Pore Environment in Physisorbents for Improved Carbon Dioxide Capture under High Humidity

Xiaoliang Wang, Maytham Alzayer, Arthur J. Shih, Saptasree Bose, Haomiao Xie, Simon M. Vornholt, Christos D. Malliakas, Hussain Alhashem, Faramarz Joodaki, Sammer Marzouk, Grace Xiong, Mark Del Campo, Pierre Le Magueres, Filip Formalik, Debabrata Sengupta, Karam B. Idrees, Kaikai Ma, Yongwei Chen, Kent O. Kirlikovali, Timur Islamoglu, Karena W. Chapman, Randall Q. Snurr,* and Omar K. Farha*

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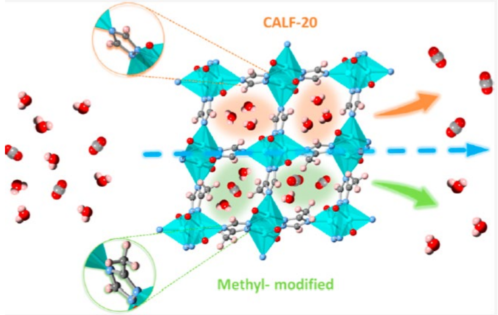
Modulator-Dependent Dynamics Synergistically Enabled Record SO_2 Uptake in Zr(IV) Metal–Organic Frameworks Based on Pyrene-Cored Molecular Quadripod Ligand

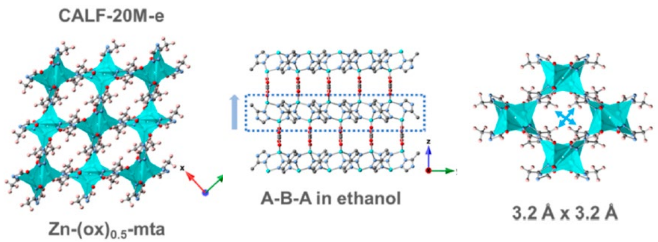
Wei Gong,* Yi Xie, Akihito Yamano, Sho Ito, Xianhui Tang, Eric W. Reinheimer, Christos D. Malliakas, Jinqiao Dong, Yong Cui,* and Omar K. Farha*

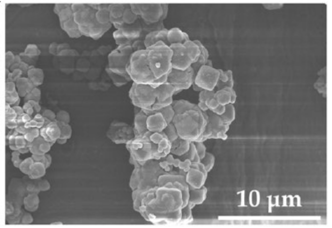
GDCh Research Articles Angewandte Chemie International Edition www.angewandte.org
How to cite: Angew. Chem. Int. Ed. 2024, 63, e202318475
doi.org/10.1002/anie.202318475
Gas Separation Hot Paper

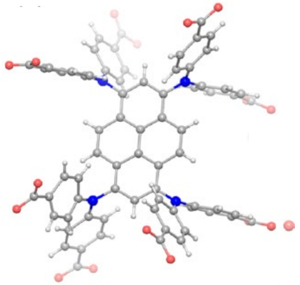
Rational Design and Reticulation of Infinite qbe Rod Secondary Building Units into Metal–Organic Frameworks through a Global Desymmetrization Approach for Inverse C_3H_8/C_3H_6 Separation

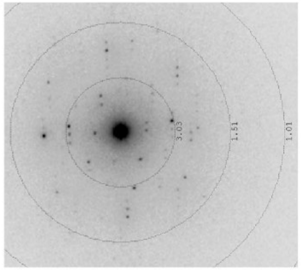
Wei Gong, Yi Xie, Akihito Yamano, Sho Ito, Eric W. Reinheimer, Jinqiao Dong, Christos D. Malliakas, Davide M. Proserpio, Yong Cui, and Omar K. Farha*

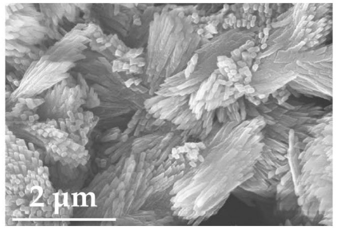


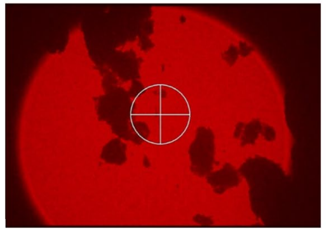


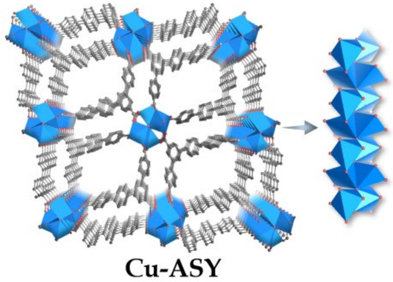


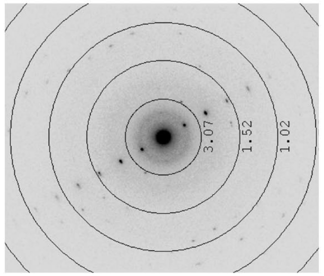




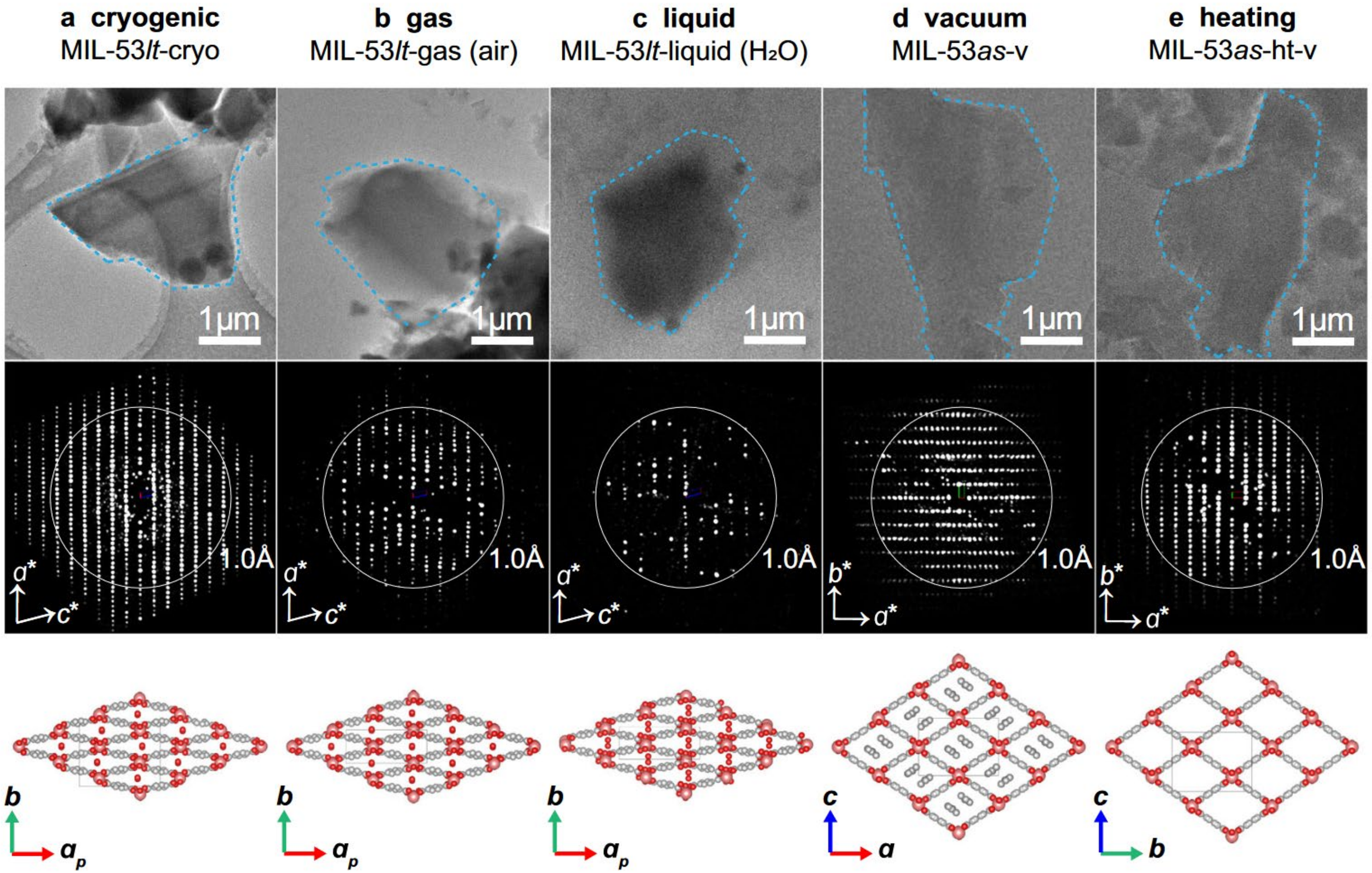








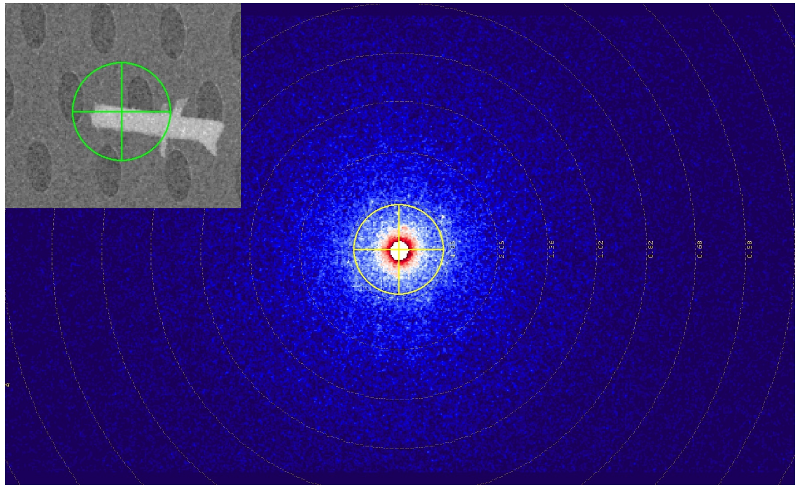
3DED/MICROED IN-SITU



from: Ling et al., Nature Communications 13, 6625 (2022)

CRYO-TRANSFER: STUDY OF HYDRATED SAMPLES

Metal-organic framework is prevented from lattice collapse

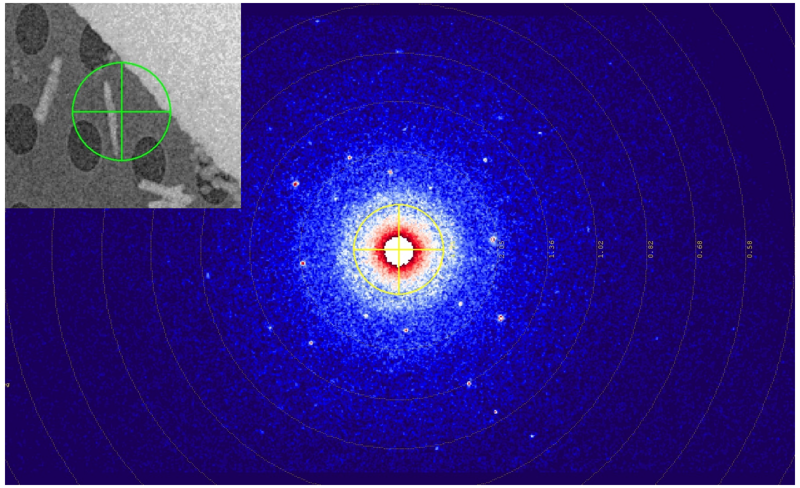


Vacuum

RT
(297 K)

Vacuum

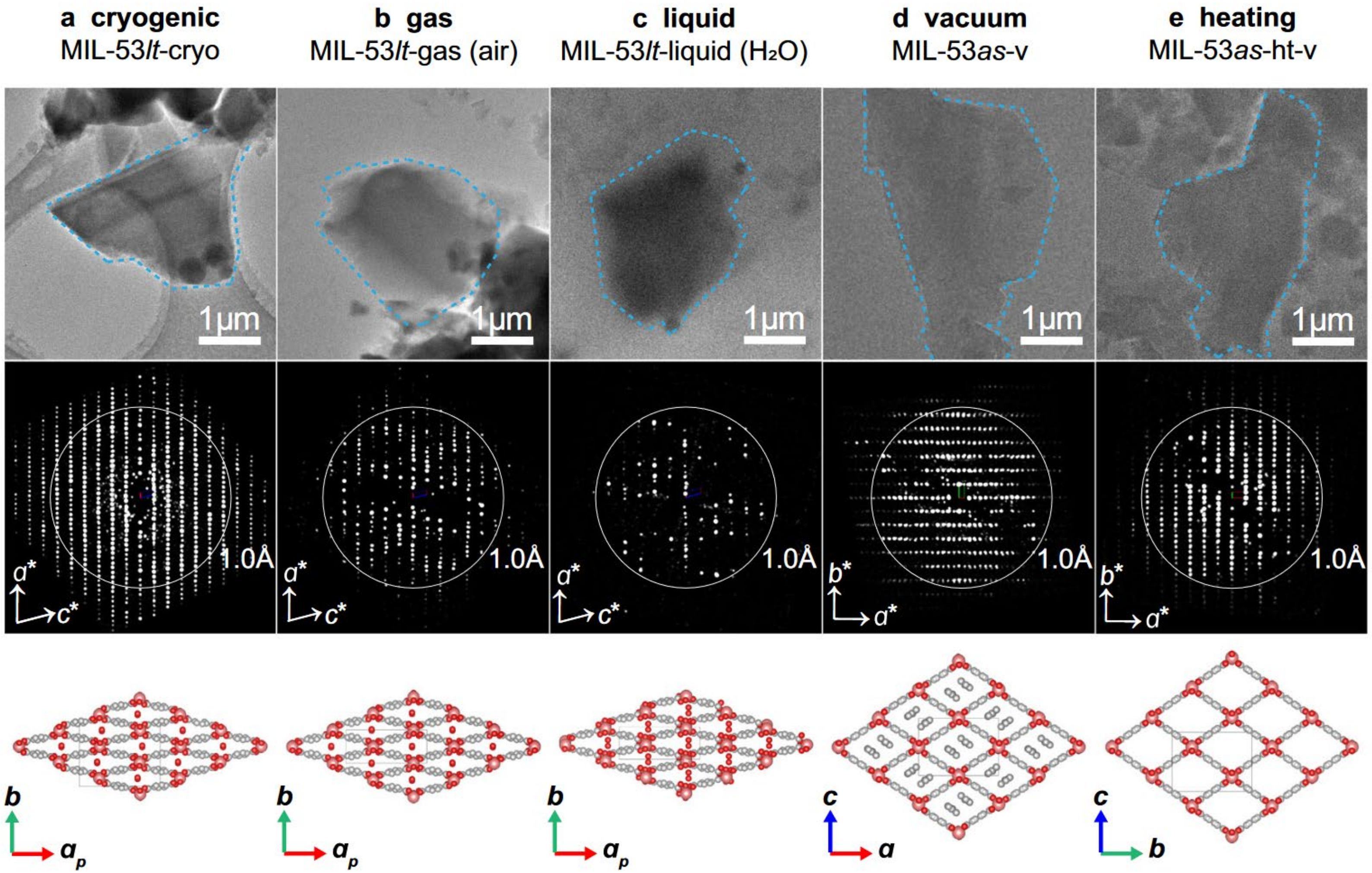
Cryo-Transfer
(100 K)



No useful diffraction data

0.60 Å data, immediate structure

3DED/MICROED IN-SITU



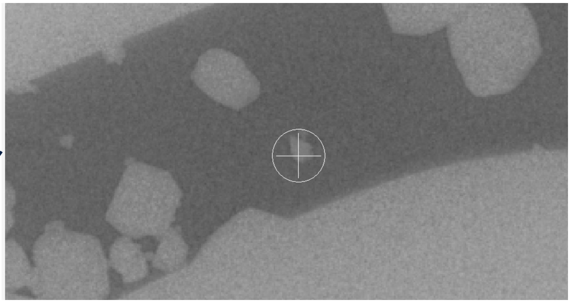
from: Ling et al., Nature Communications 13, 6625 (2022)

MOF PHASE TRANSITION IN-SITU

$Cu(ta)_2$ α/β polymorph transition

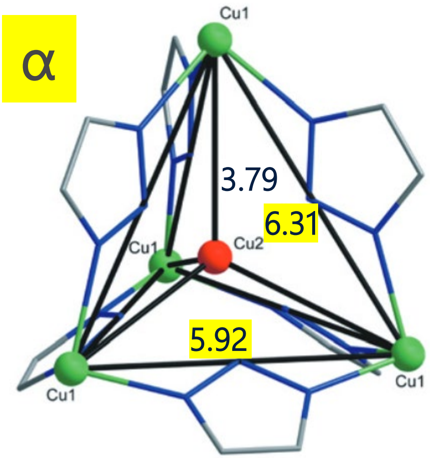
- α-phase at low temperature (RT) shows tetragonal distortion of CuN_6 coordination octahedra
- Reversible phase transition to undistorted β-phase: Grzywa, M. *et al.*, Dalton Trans. 41, 4239 (2012).
- Characterized by SCXRD (α)/PXRD(β), spectroscopic, and thermogravimetric analysis
- ED collection at 20°C / 200°C on identical crystal proves reversible transitions
- Cells, space group, and bond lengths consistent with XRD

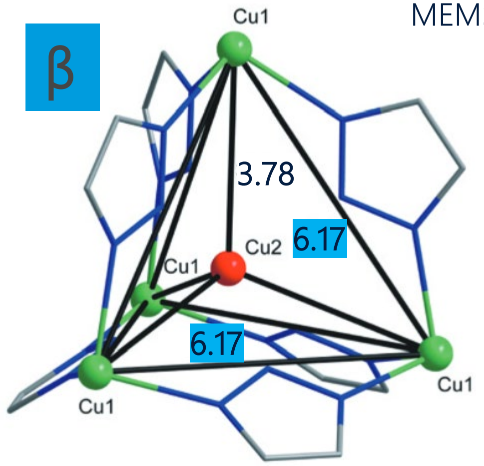
20°

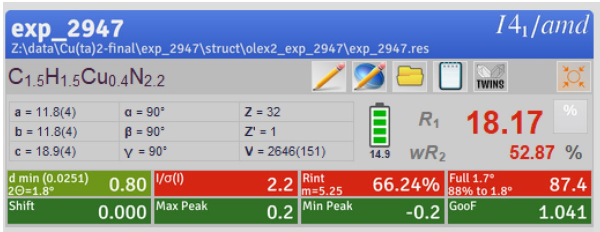


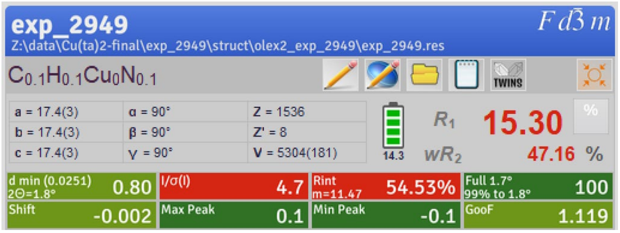
200°

Hummingbird Scientific MEMS heating chip





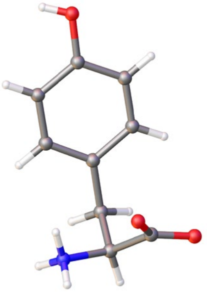




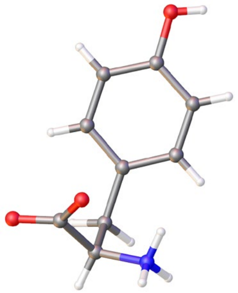
ONGOING PROJECTS

Key workflow R&D topics right now:

- Automated dynamical structure solution

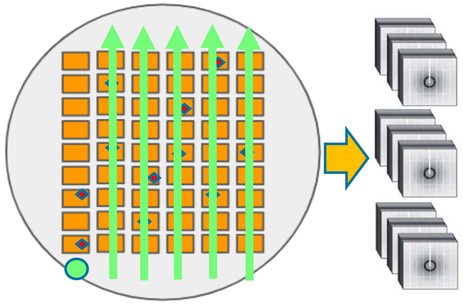


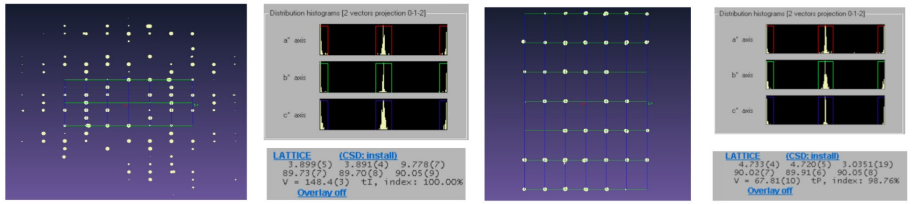
L-tyrosine



D-tyrosine

- Automated polymorph screening (SerialRED)





SYNERGY-ED

- Possible to offer 1 day beam access on Rigaku Europe SE Synergy-ED

- Contact me :
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  - +33 (0)7 88 75 83 89

THANK YOU
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