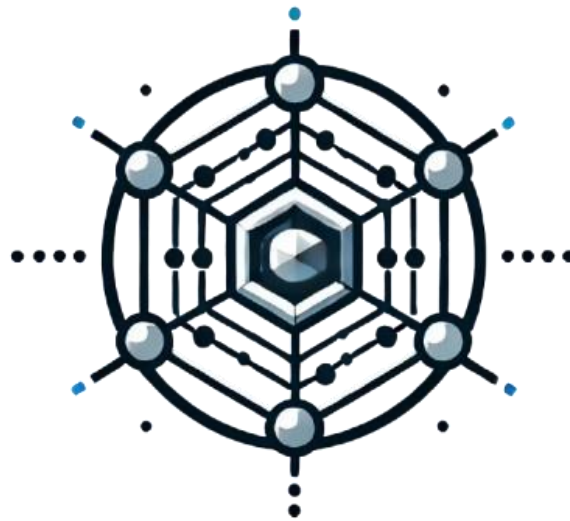




Ben-Gurion University  
of the Negev



## **Single crystal X-ray diffraction service**

**Ilse-Katz Institute of Nanoscale Science & Technology**

# Specifications

---

- HyPix-Arc 100° curved hybrid photon detector.
- Kappa multi-axis goniometer.
- Two radiation sources: Cu, Mo.
- Cryo-system for low temperature measurements.
- Can diffract very small crystals (10  $\mu\text{m}$  and above).
- Can be used for diffracting powder samples (sub-mg amounts of material).



## Workflow for customers

---

- 1) Collecting suitable crystals for diffraction. **May use our crystallization service.**
- 2) Collecting dataset and reduction of the data.
- 3) Solving the crystal structure. **Obtain .cif file.**
- 4) Add .cif file to a personalized database for a research group or company.
- 5) Rietveld refinement of future *powder* samples.

# 1. Crystallization services

**Formulator (Formulatrix)** - A liquid handler that dispenses crystallization solutions for 24- as well as for 96-well grids rapidly and with high accuracy.

- Low volumes, down to 200 nL.
- Dispense a 100  $\mu$ L, 3-ingredient, 96-well crystallization grid in 2.7 mins.
- Supports all microplate types.



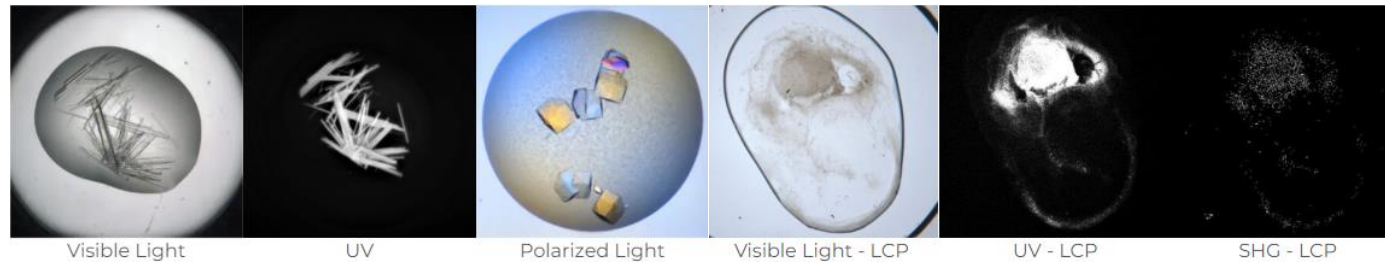
**NT8 Drop Setter (Formulatrix)** - A fast and precise automated nanoliter dispenser for setting up crystallization experiments including sitting drops, hanging drops and seeding.

- Proportionally-Controlled Active Humidification prevents sample evaporation and increases experiment reproducibility.
- Integrated with the Rock Maker crystallization software.

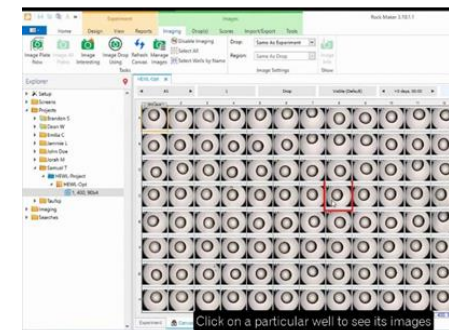


# 1. Crystallization services

**Rock Imager (Formulatrix)** - An automated imaging system for protein crystallization. This apparatus incubates and captures quality images of up to 250 crystallization plates on a user-defined schedule.



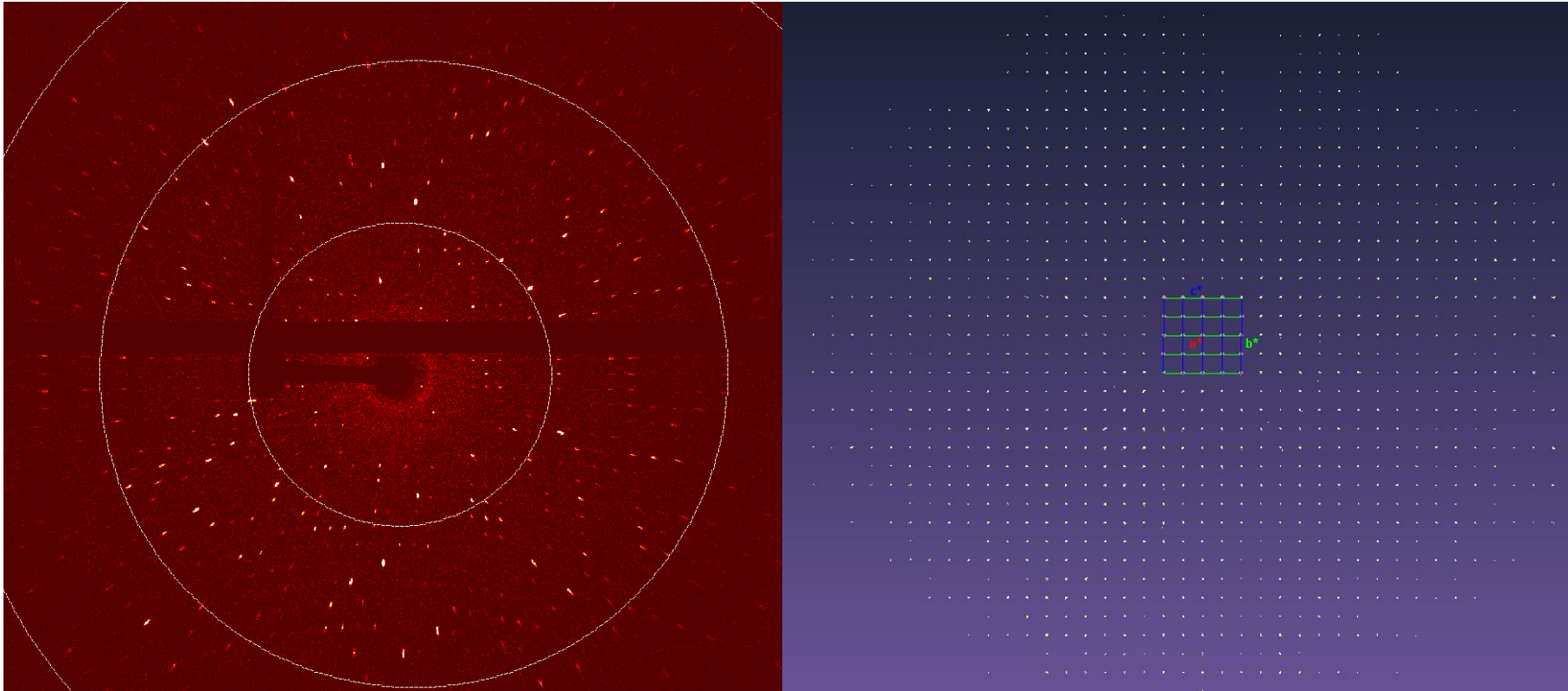
**Rock Maker (Formulatrix)** - Powerful software which provides experimental design, and which automates and tracks the crystallization process. It allows for the rapid creation of complex experiments and crystallization screens in a user-friendly environment.



## 2. Collection of dataset and data reduction

---

- Establishing the presence of a single crystal (screening).
- Pre-experiment to determine initial data collection strategy.
- Collection of dataset according to crystal system and symmetry.



## 2. Collection of dataset and data reduction

- Determination of possible twinning.

### LATTICE

Current cell (CSD#1: 200 +1L)

26.1847(19) 26.1829(14) 6.7223(2) 89.996(4) 89.999(4) 119.987(7) 3991.8(4)

Constrained current cell

26.1845(13) 26.1845(13) 6.7227(3) 90.0 90.0 120.0 3991.8(3)

Lattice reduction

selected cell

26.1900 26.1801 6.7223 89.9978 89.9945 119.9815 hR 24

reduced cell

6.7223 15.2816 15.2828 117.8634 98.4254 98.4299 1330.8

Twin information

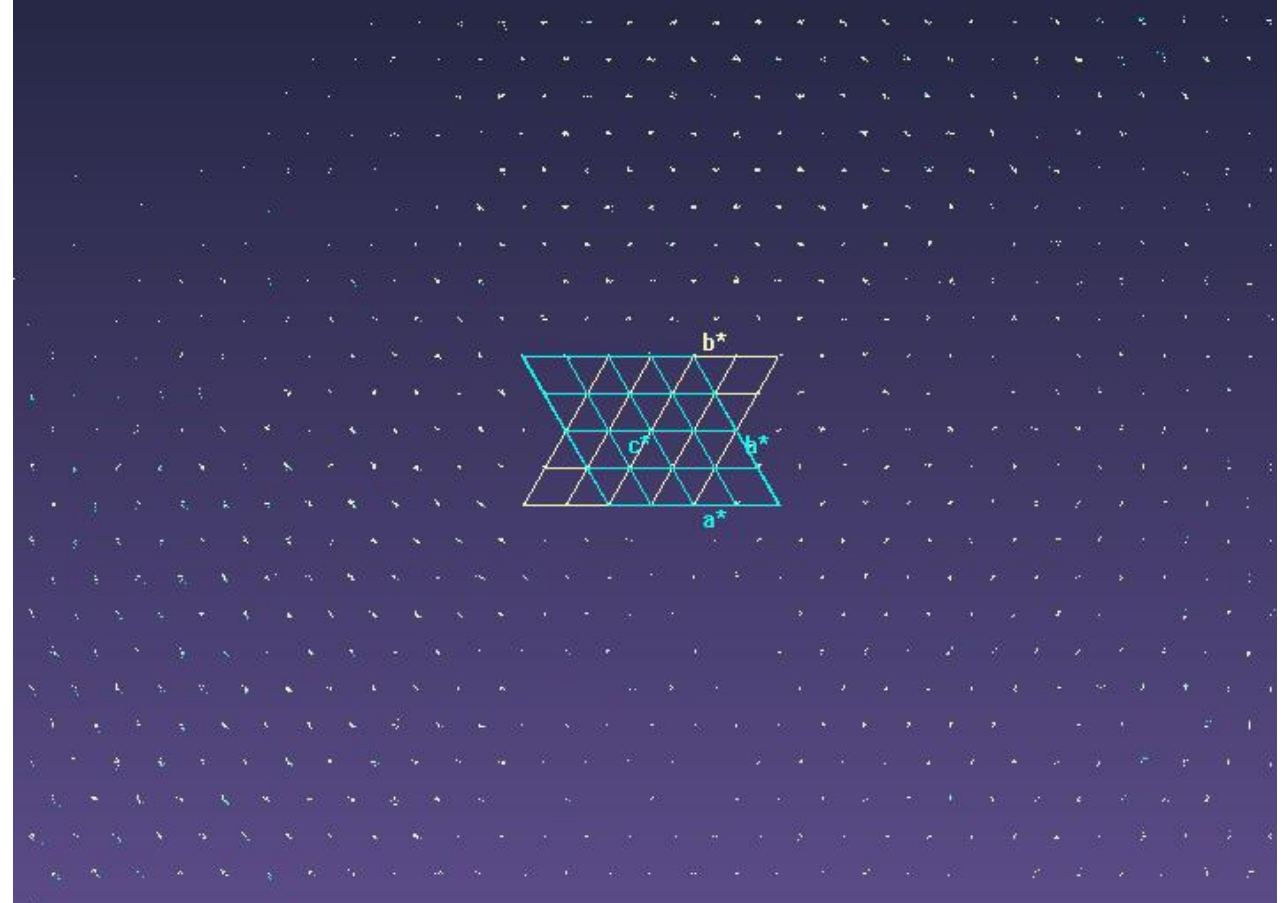
1: 26.1838 26.1835 6.7222 89.995 90.001 119.989 3991.7

2: 26.1858 26.1892 6.7223 89.987 90.005 120.005 3992.2

1: Total: 4901( 90.6%) Separate: 317( 5.9%) Overlapped: 4584( 84.7%)

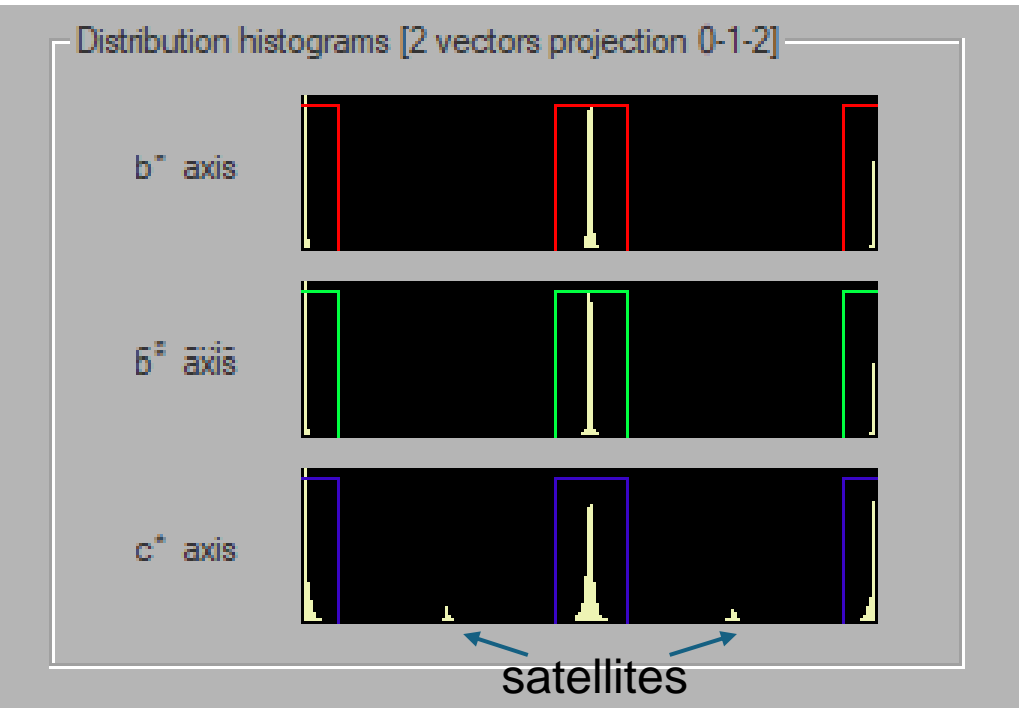
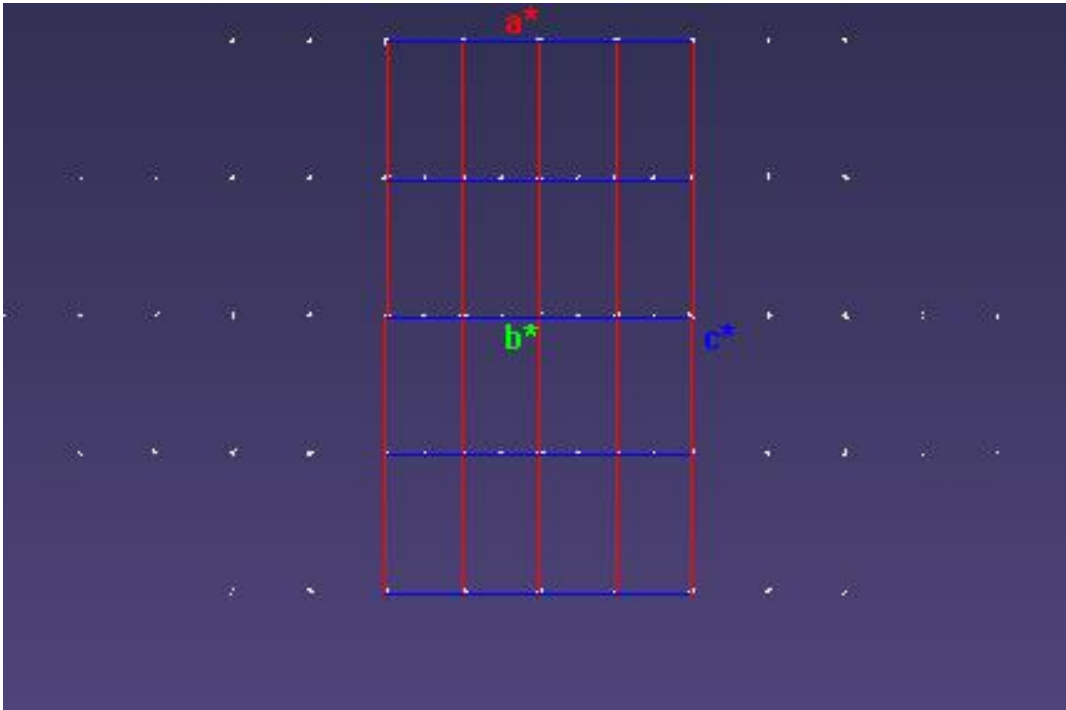
2: Total: 4939( 91.3%) Separate: 355( 6.6%) Overlapped: 4584( 84.7%)

Unindexed: 156( 2.9%)



# 2. Collection of dataset and data reduction

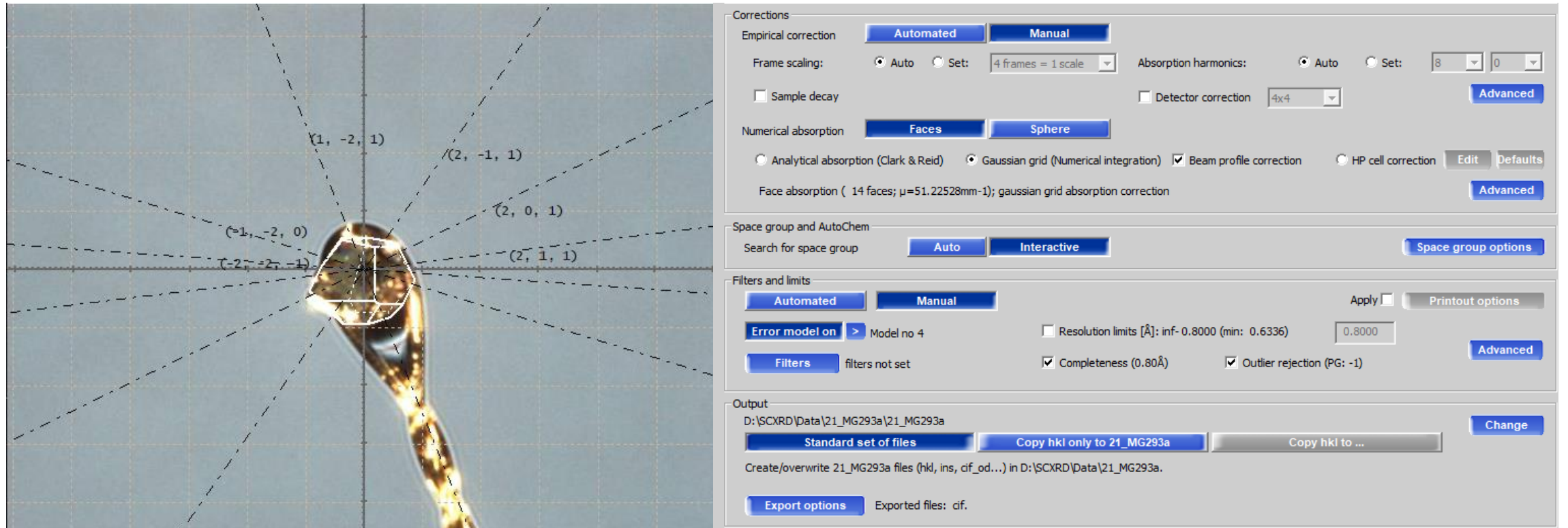
- Determination of modulated structures.





## 2. Collection of dataset and data reduction

- Modeling the crystal for absorption correction.



The screenshot displays the software interface for X-ray diffraction data reduction, showing a 3D model of a crystal and various settings for corrections, space group, filters, and output.

**Corrections**

- Empirical correction: **Automated** / **Manual**
- Frame scaling:  Auto  Set: 4 frames = 1 scale
- Absorption harmonics:  Auto  Set: 8 0
- Sample decay  Detector correction 4x4 **Advanced**
- Numerical absorption: **Faces** / **Sphere**
- Analytical absorption (Clark & Reid)  Gaussian grid (Numerical integration)  Beam profile correction  HP cell correction **Edit Defaults**
- Face absorption ( 14 faces;  $\mu=51.22528\text{mm}^{-1}$ ); gaussian grid absorption correction **Advanced**

**Space group and AutoChem**

- Search for space group: **Auto** / **Interactive** **Space group options**

**Filters and limits**

- Automated** / **Manual** **Apply** **Printout options**
- Error model on** > Model no 4  Resolution limits [ $\text{\AA}$ ]: inf-0.8000 (min: 0.6336) 0.8000 **Advanced**
- Filters** filters not set  Completeness (0.80 $\text{\AA}$ )  Outlier rejection (PG: -1)

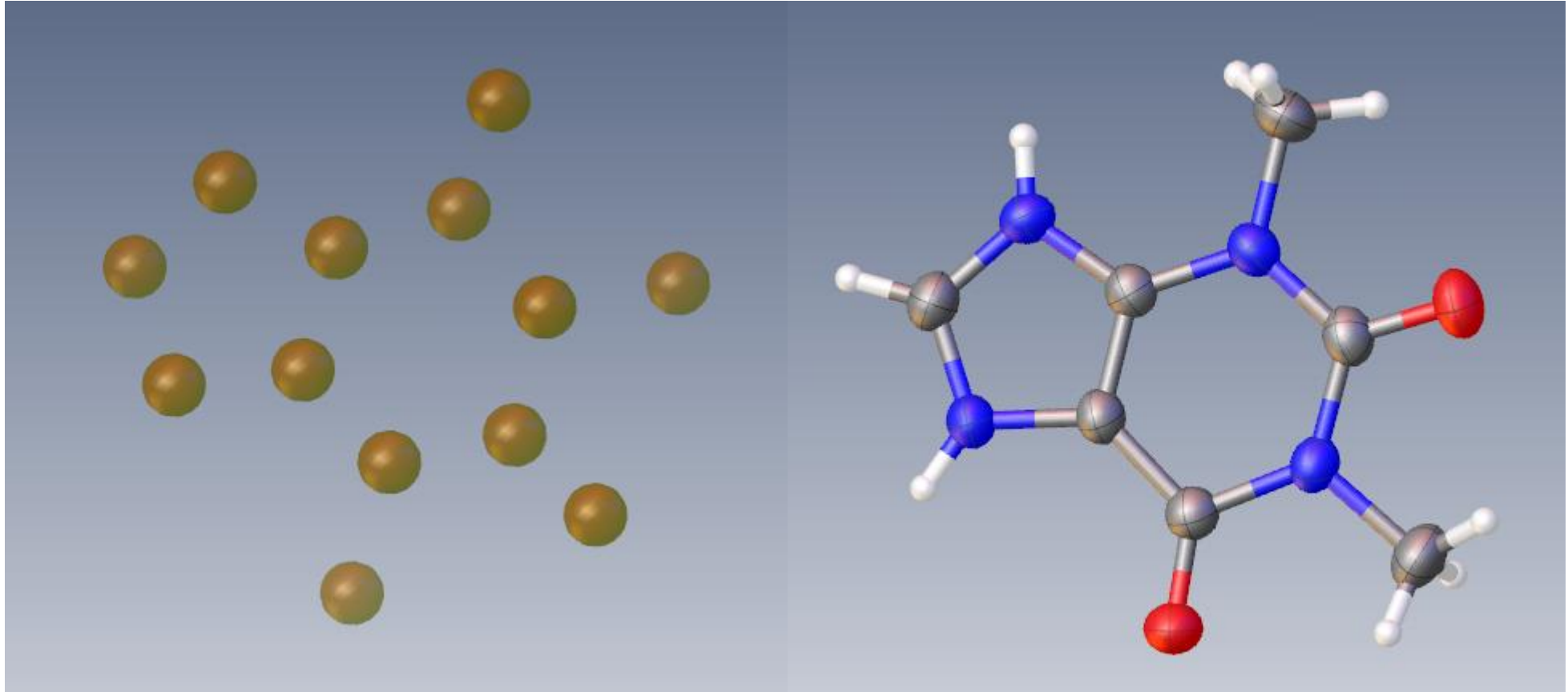
**Output**

- D:\SCXRD\Data\21\_MG293a\21\_MG293a **Change**
- Standard set of files** **Copy hkl only to 21\_MG293a** **Copy hkl to ...**
- Create/overwrite 21\_MG293a files (hkl, ins, cf\_od...) in D:\SCXRD\Data\21\_MG293a.
- Export options** Exported files: cf.

### 3. Solving the crystal structure

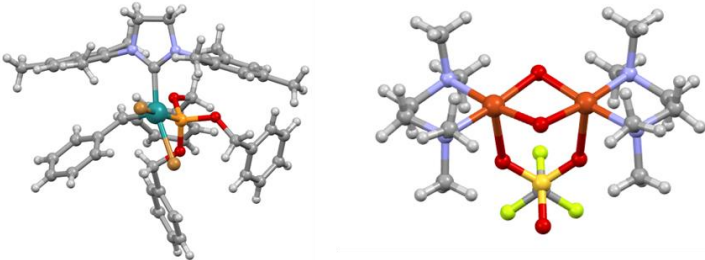
---

- Obtaining an electron density peak map and solution of the structure.

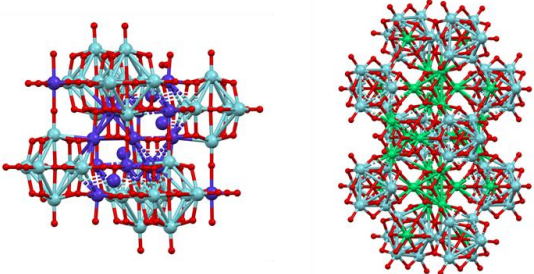


- Preparation for publication.

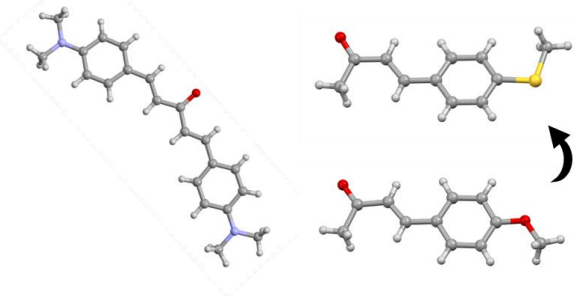
# 3. Solving the crystal structure - examples of solved structures



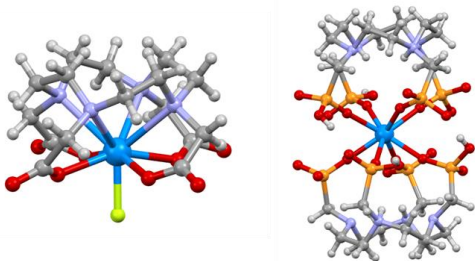
Organometallic catalysts



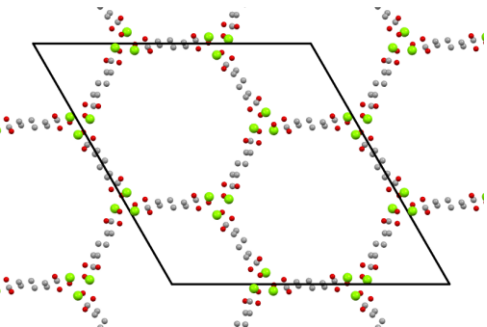
Polyoxometalate catalysts



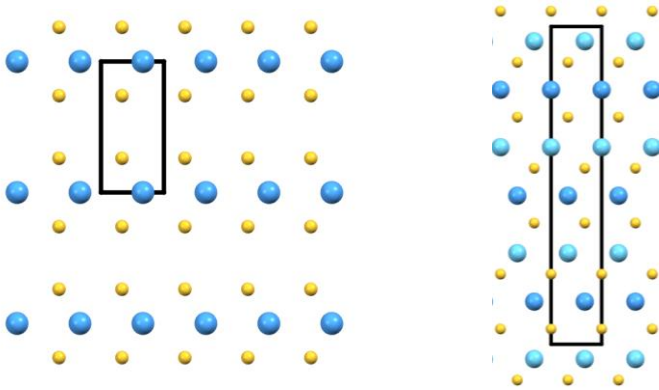
Organic molecules



Chelation complexes



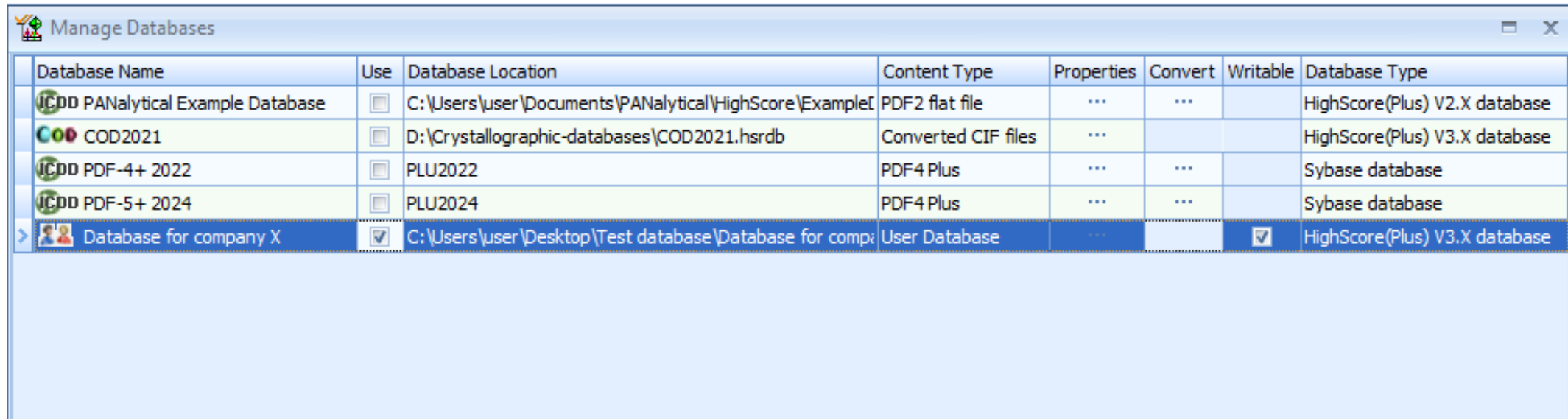
Metal-organic frameworks (MOFs)








Transition metal dichalcogenides (TMDs)

## 4. Custom made database

- Preparation of a custom database for customers.



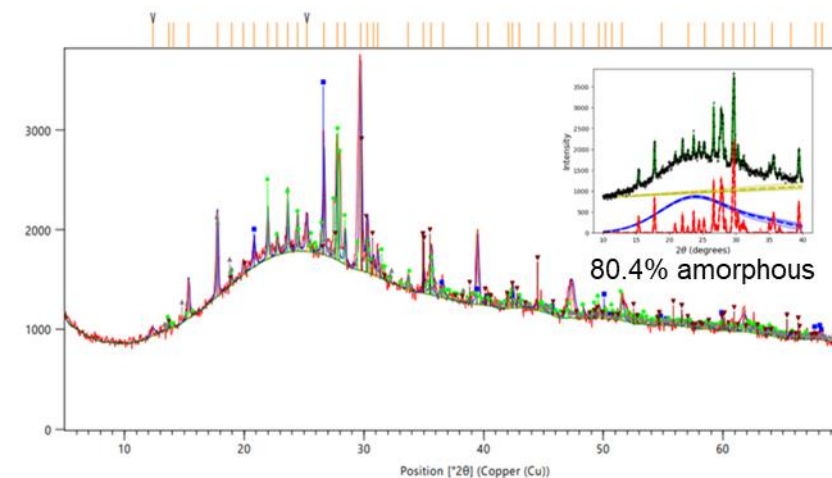
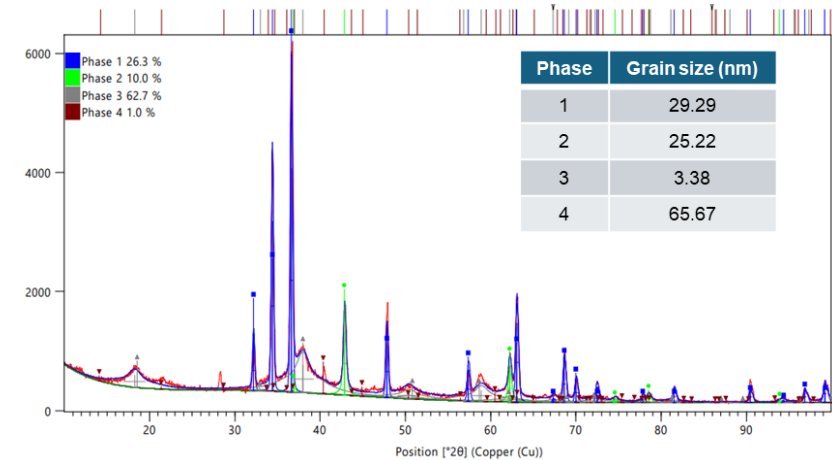
Database Name	Use	Database Location	Content Type	Properties	Convert	Writable	Database Type
 ICDD PANalytical Example Database	<input type="checkbox"/>	C:\Users\user\Documents\PANalytical\HighScore\Example	PDF2 flat file	...	...		HighScore(Plus) V2.X database
 COD2021	<input type="checkbox"/>	D:\Crystallographic-databases\COD2021.hsrdb	Converted CIF files	...			HighScore(Plus) V3.X database
 ICDD PDF-4+ 2022	<input type="checkbox"/>	PLU2022	PDF4 Plus	...	...		Sybase database
 ICDD PDF-5+ 2024	<input type="checkbox"/>	PLU2024	PDF4 Plus	...	...		Sybase database
 Database for company X	<input checked="" type="checkbox"/>	C:\Users\user\Desktop\Test database\Database for comp:	User Database	...		<input checked="" type="checkbox"/>	HighScore(Plus) V3.X database

- Can now be used as reference for future powder samples.

## 5. Rietveld refinement of powder samples

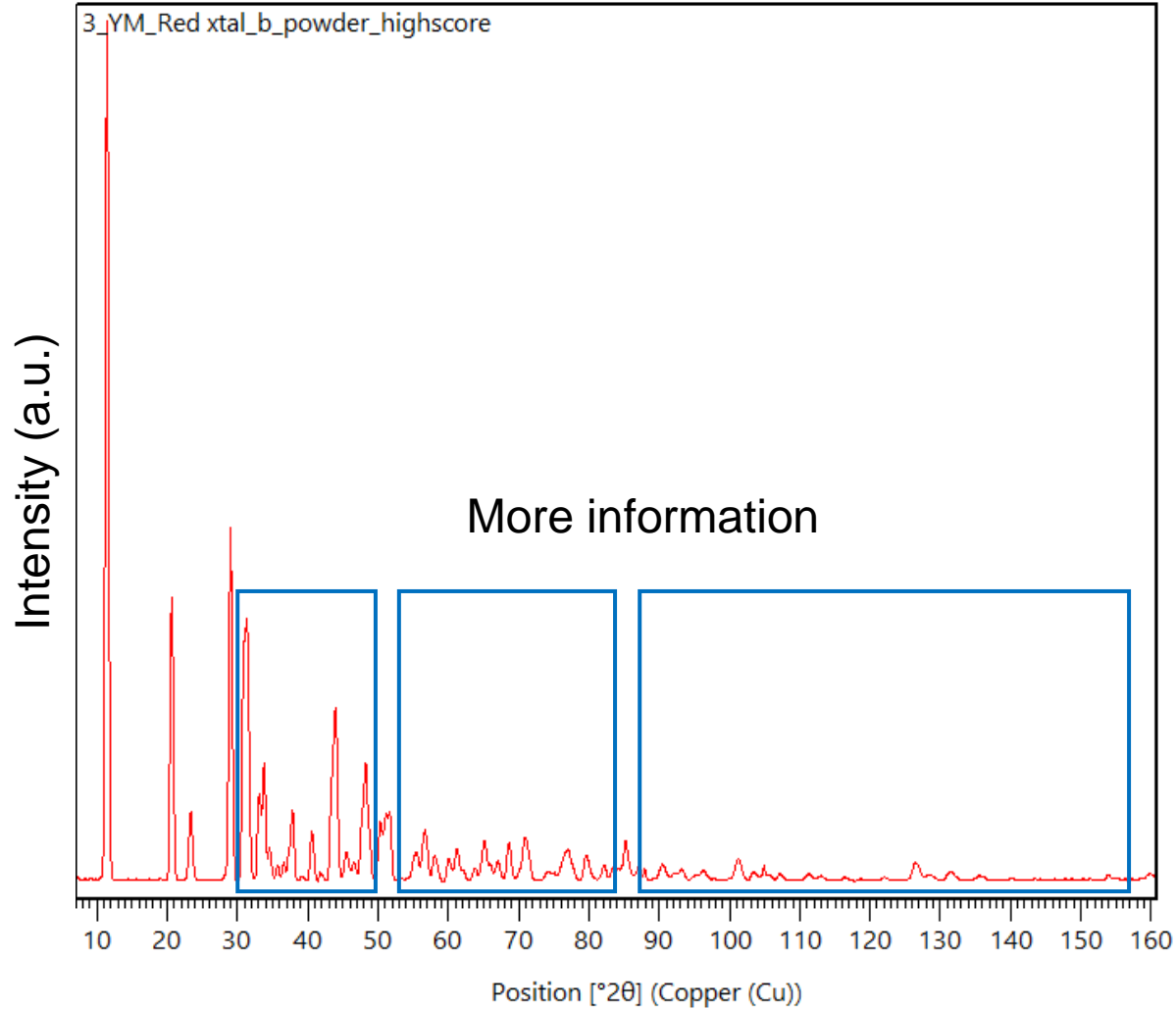
For formulations and/or single/multi-phase materials, it is now possible to use the personalized database to obtain:

- Percent of each phase in the sample.
- Crystallite size of each phase.
- Amorphous percent in the sample.
- Preferred orientation in the sample (relates to morphology of crystals).
- Variations in lattice parameters.
- Substitution of atoms in solids, and more.

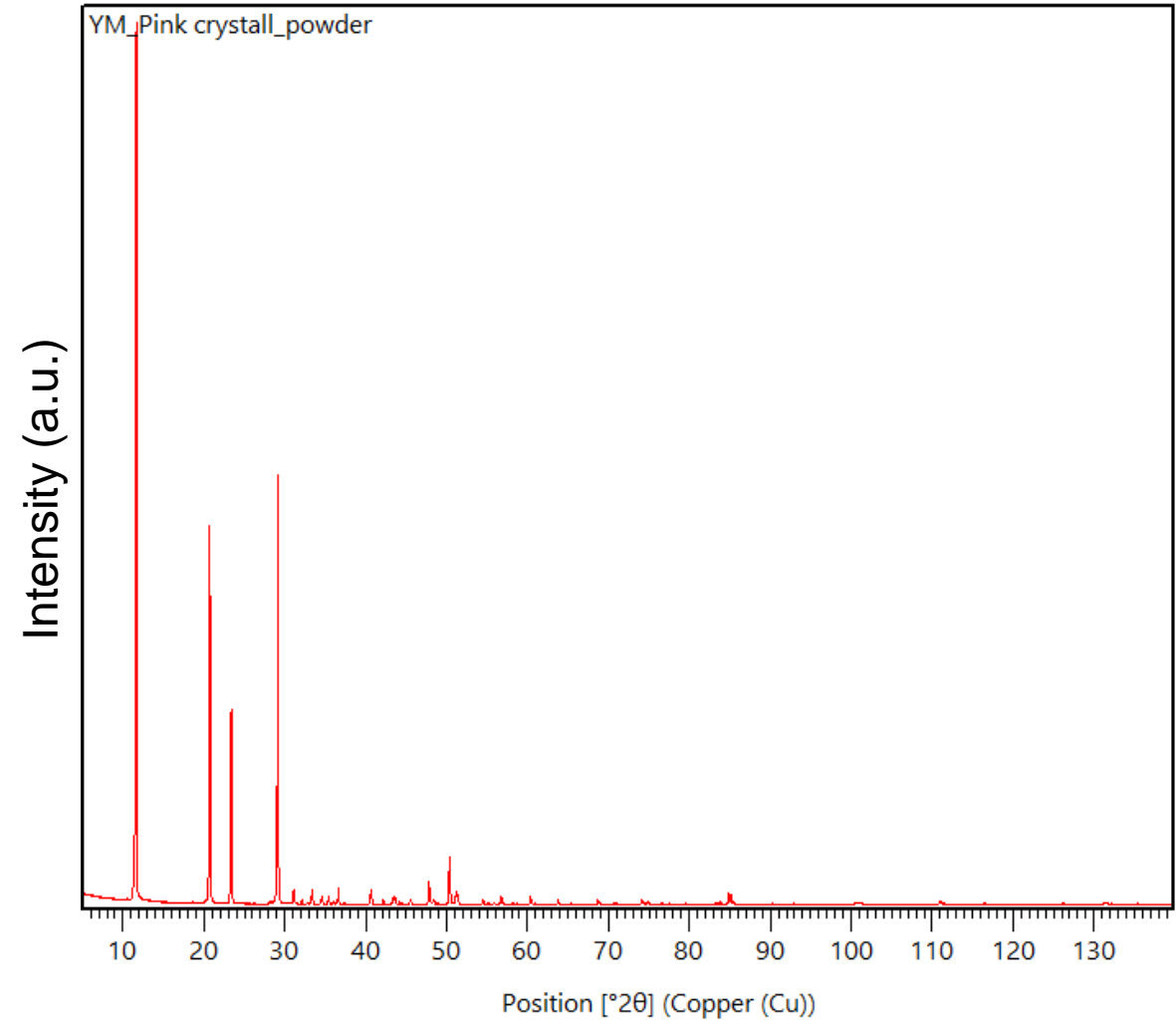


# Powder spectrum

SCXRD (Rigaku)



PXRD (Empyrean)



# Select list of publications from last few years

---

- 1) Libman, A.; Ben-Lulu, M.; Gaster, E.; Bera, R.; Shames, A. I.; Shaashua, O.; Vershinin, V.; Torubaev, Y.; Pappo, D., *J. Am. Chem. Soc.*, **2023**, 21002-21011.
- 2) Alassad, N.; Phatake, R. S.; Baranov, M.; Reany, O.; Lemcoff, N. G., *Catalysts*, **2023**, 1411.
- 3) Vaisman, A.; Vidavsky, Y.; Baranov, M.; Lehrer, A.; Baraban, J. H.; Lemcoff, N. G., *J. Am. Chem. Soc.*, **2023**, 73-78.
- 4) Segalovich-Gerendash, G.; Baranov, M.; Lemcoff, N. G.; Phatake, R., *Organometallics*, **2023**, 825-831.
- 5) Dovrat, G.; Pevzner, S.; Maimon, E.; Vainer, R.; Illiashevsky, O.; Ben-Eliyahu, Y.; Moisy, P.; Bettelheim, A.; Zilbermann, I., *Chem. Eur. J.*, **2022**, e2022018.
- 6) Zhang, G.; Fei, W.; Tubul, T.; Baranov, M.; Leffler, N.; Neyman, A.; Poblet, J. M.; Weinstock, I. A., *Angew. Chem. Int. Ed.*, **2022**, e2022131.
- 7) Manikandan, R.; Shauloff, N.; Nandi, A.; Baranov, M.; Pevzner, A.; Marx, S.; Jelinek, R., *J. Mater. Chem. C*, **2022**, 5458-5465 (see 9152-9152).
- 8) Baranov, M.; Polin, L.; Leffler, N.; Leitus, G.; Shames, A. I.; Weinstock, I. A., *Dalton Trans.*, **2022**, 8600-8604.
- 9) Dyadyuk, A.; Vershinin, V.; Shalit, H.; Shalev, H.; More, N. Y.; Pappo, D., *J. Am. Chem. Soc.*, **2022**, 3676-3684.
- 10) Barrio, J.; Barzilai, S.; Karjule, N.; Amo-Ochoa, P.; Zamora, F.; Shalom, M., *Adv. Opt. Mat.*, **2022**, 2100683.
- 11) Dovrat, G.; Pevzner, S.; Berthon, C.; Lerner, A.; Maimon, E.; Vainer, R.; Karpasas, M.; Ben-Eliyahu, Y.; Moisy, P.; Bettelheim, A.; Zilbermann, I., *Chem. Eur. J.*, **2021**, 8264-8267.
- 12) Zhang, G.; Baranov, M.; Wang, F.; Poblet, J. M.; Kozuch, S.; Leffler, N.; Shames, A. I.; Clemente-Juan, J. M.; Neyman, A.; Weinstock, I. A., *J. Am. Chem. Soc.*, **2021**, 20769-20778.
- 13) Vershinin, V.; Forkosh, H.; Ben-Lulu, M.; Libman, A.; Pappo, D., *J. Org. Chem.*, **2021**, 79-90.
- 14) Azoulay, A.; Barrio, J.; Tzadikov, J.; Volokh, M.; Albero, J.; Gervais, C.; Amo-Ochoa, P.; Garcia, H.; Zamora, F.; Shalom, M., *J. Mat. Chem. A*, **2020**, 8752-8760.
- 15) Tiwari, C. K.; Baranov, M.; Neyman, A.; Neumann, R.; Weinstock, I. A., *Inorg. Chem.*, **2020**, 11945-11952.
- 16) Dovrat, G.; Illy, M.-C.; Berthon, C.; Lerner, A.; Mintz, M. H.; Maimon, E.; Vainer, R.; Ben-Eliyahu, Y.; Moiseev, Y.; Moisy, P.; Bettelheim, A.; Zilbermann, I., *Chem. Eur. J.*, **2020**, 3390-3403.
- 17) Zhang, G.; Gadot, E.; Gan-Or, G.; Baranov, M.; Tubul, T.; Neyman, A.; Li, M.; Clotet, A.; Poblet, J. M.; Yin, P.; Weinstock, I. A., *J. Am. Chem. Soc.*, **2020**, 7295-7300.