Ben-Gurion University of the Negev

IKI

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 μm 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.

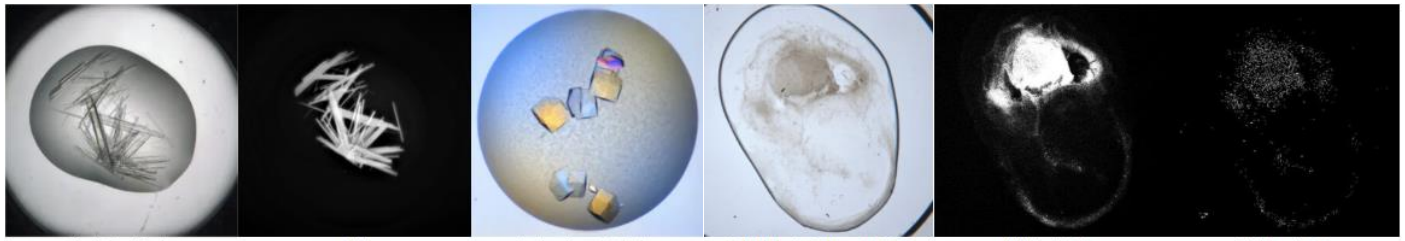
- Low volumes, down to 200 nL.
- Dispense a 100 µL, 3-ingredient, 96-well crystallization grid in 2.7 mins.
- Supports all microplate types.

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

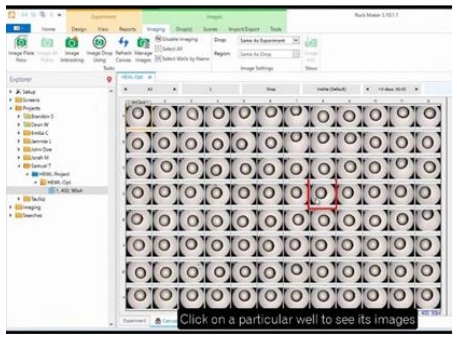
- Proportionally-Controlled Active Humidification prevents sample evaporation and increases experiment reproducibility.
- Integrated with the Rock Maker crystallization software.

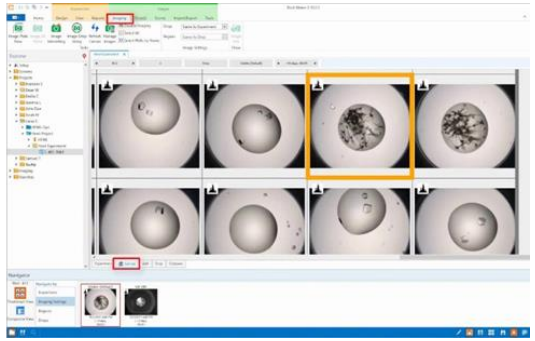
1. Crystallization services

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.



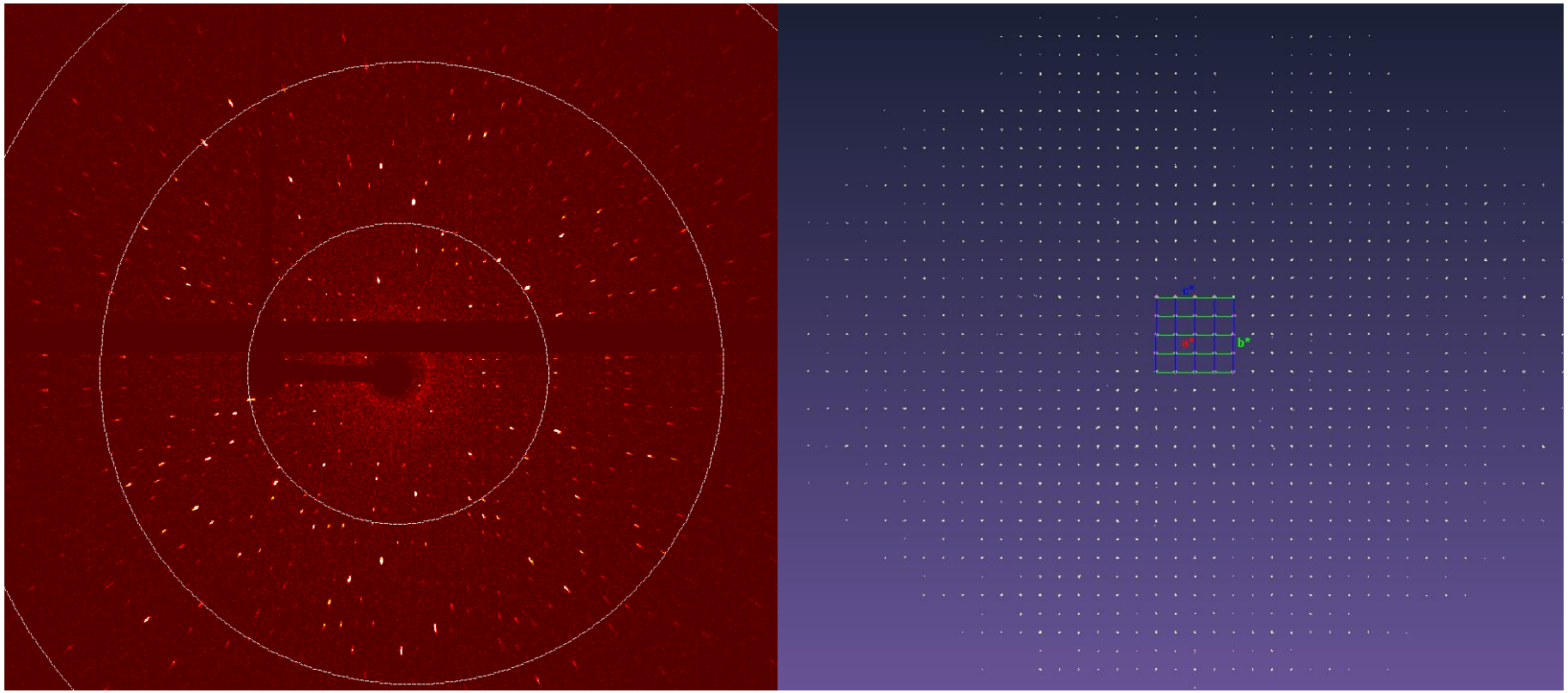


2. Collection of dataset and data reduction

- Establishing the presence of a single crystal (screening).

- Pre-experiment to determine initial data collection strategy.

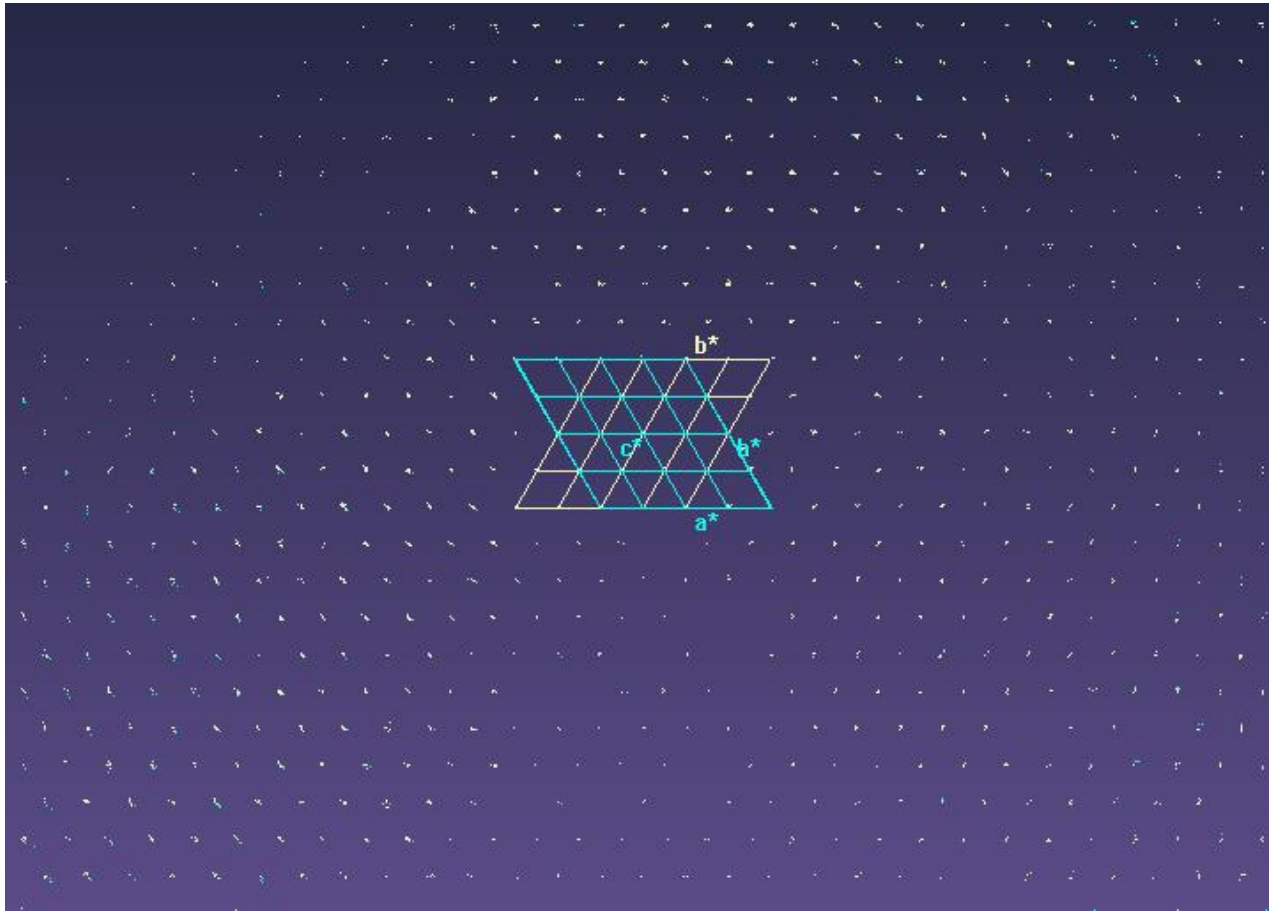
- Collection of dataset according to crystal system and symmetry.



2. Collection of dataset and data reduction

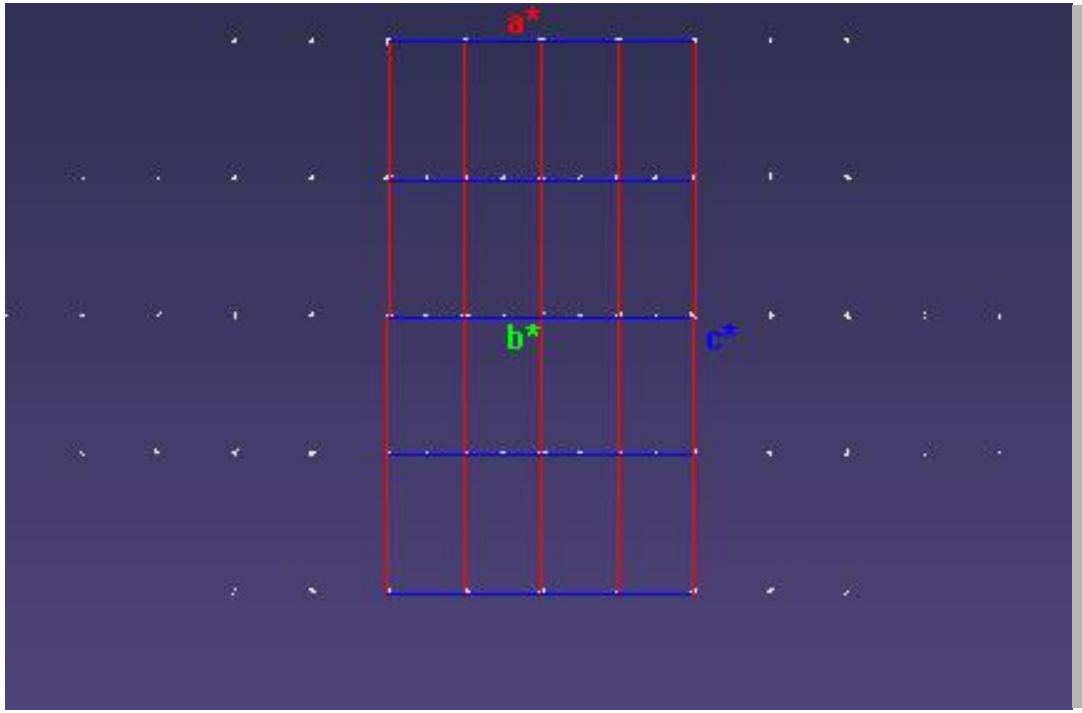
- Determination of possible twinning.

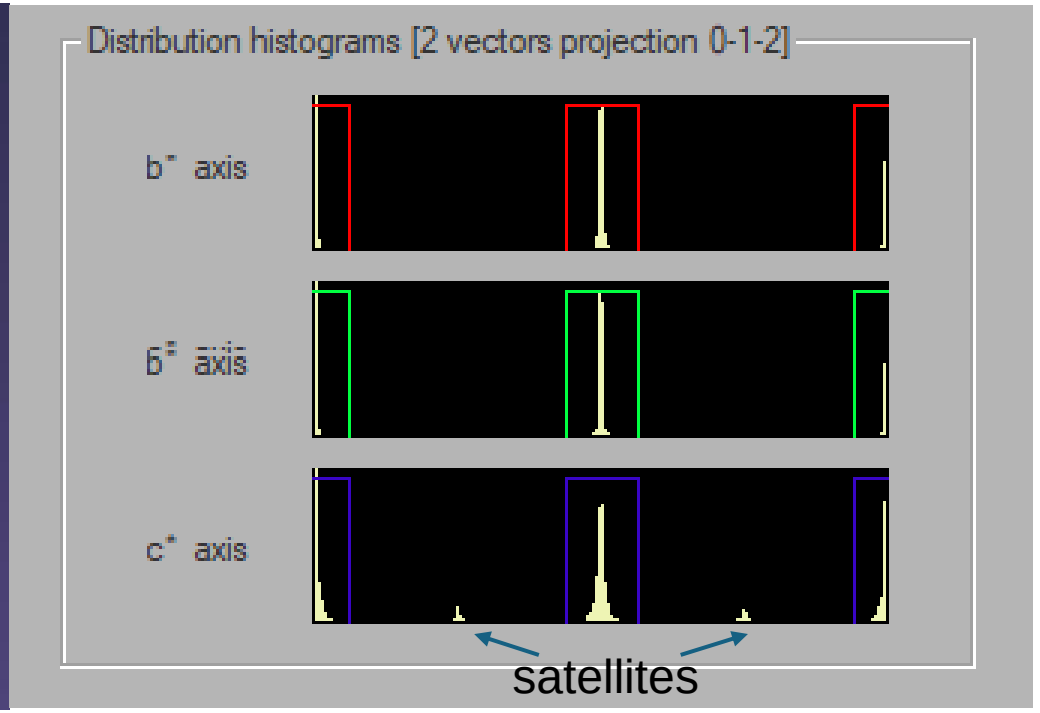
```
LATTICE
  Current cell  (CSD#1: 200 +1L)
    26.1847(19) 26.1829(14) 6.7223(2) 89.996(4) 89.999(4) 119.987(7) 3991.8(4)
  Constrained current cell
    26.1845(13) 26.1845(13) 6.7227(3) 90.0 90.0 120.0 3991.8(3)
  Lattice reduction
     selected cell
       26.1900   26.1801    6.7223   89.9978   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

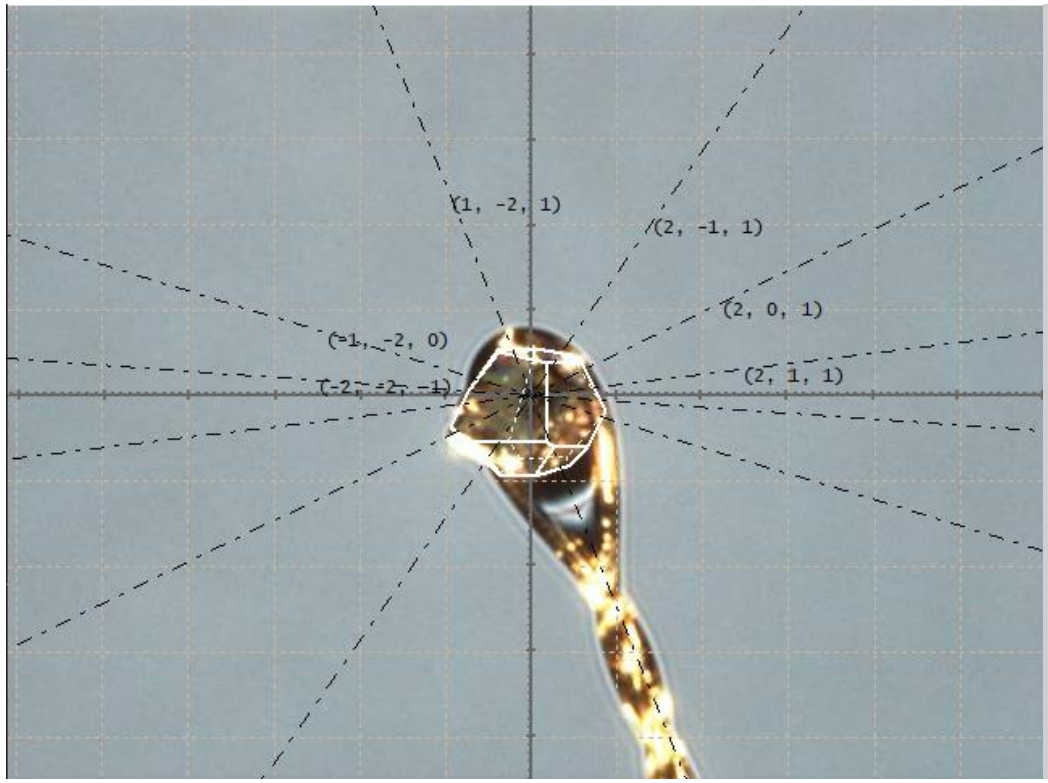
- Determination of modulated structures.

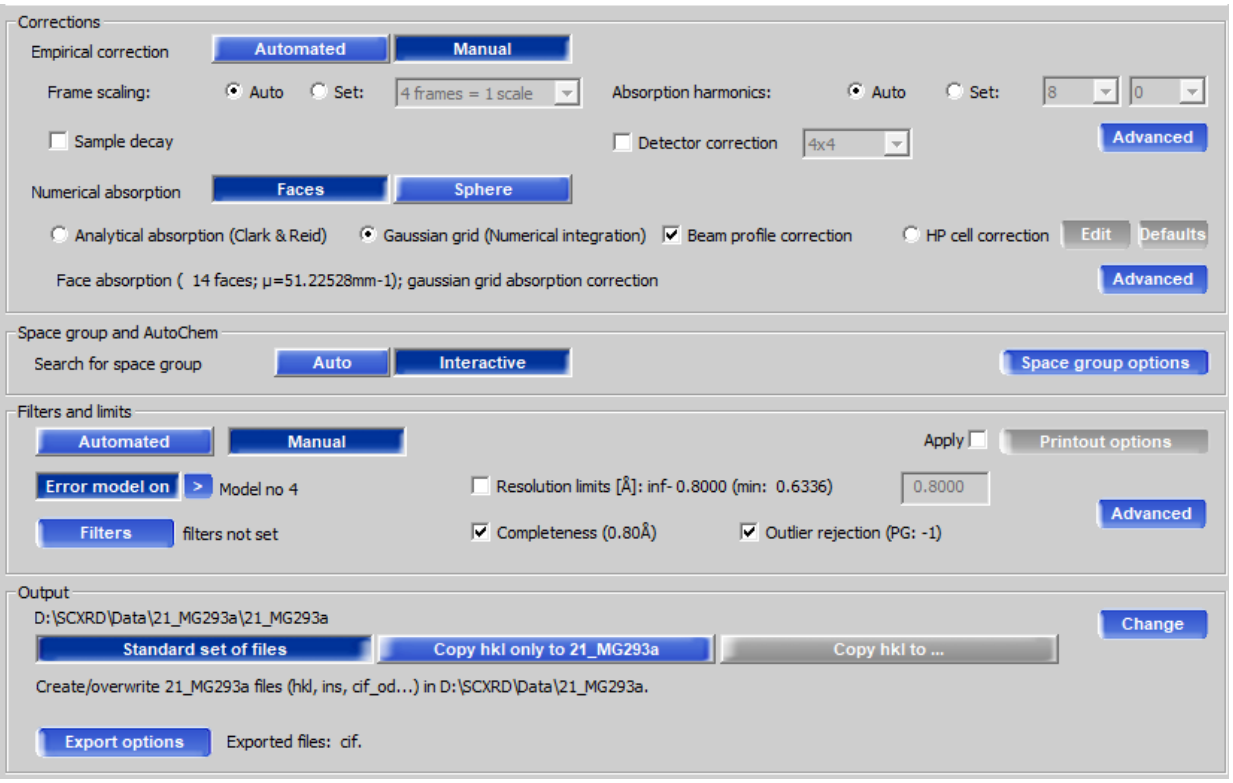




2. Collection of dataset and data reduction

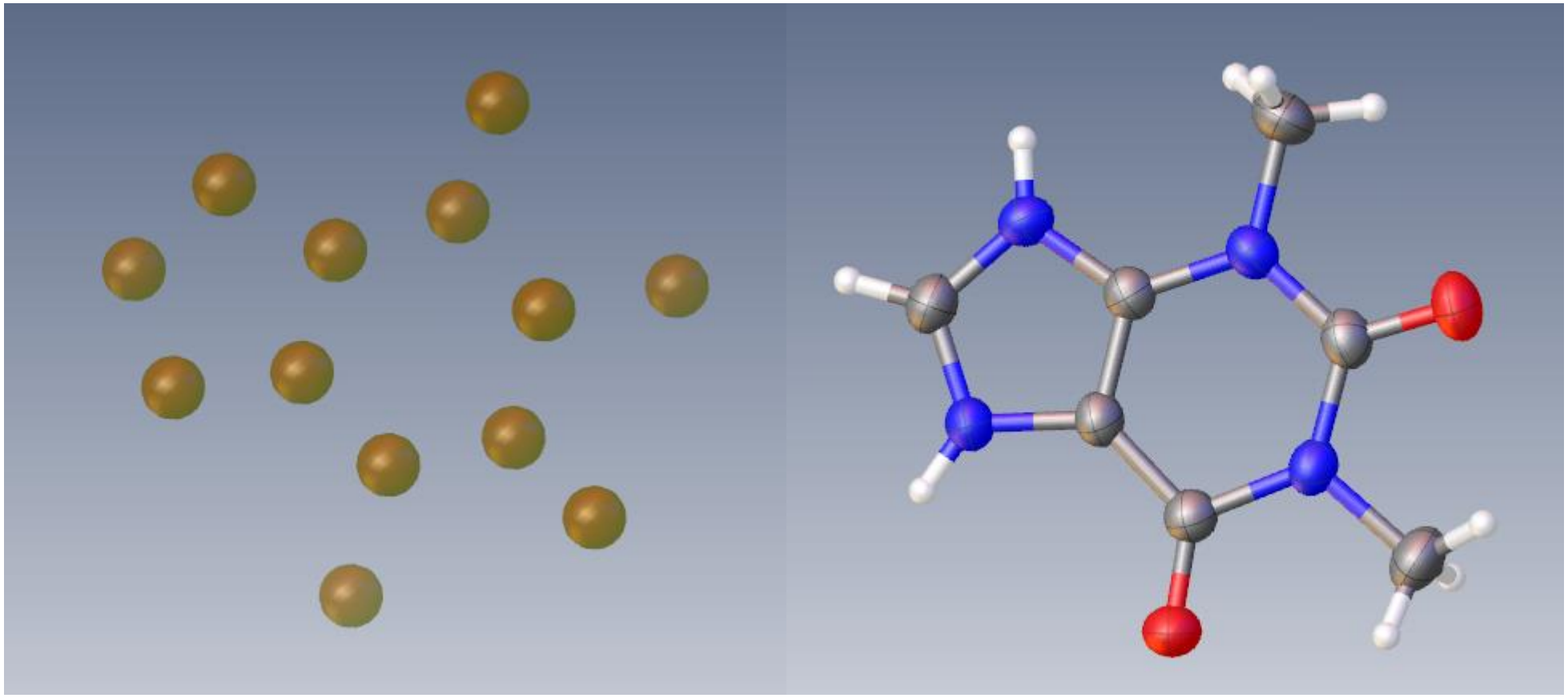
- Modeling the crystal for absorption correction.





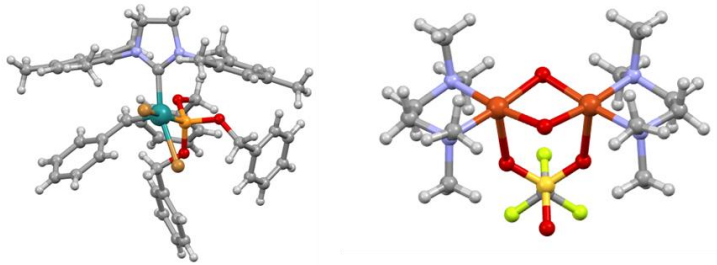
3. Solving the crystal structure

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

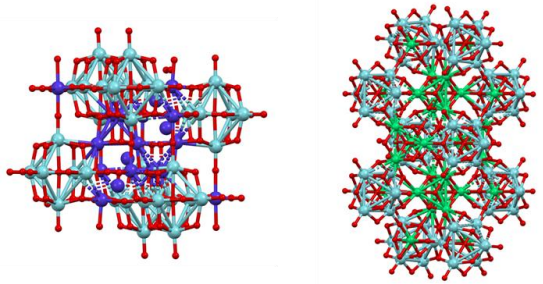


- Preparation for publication.

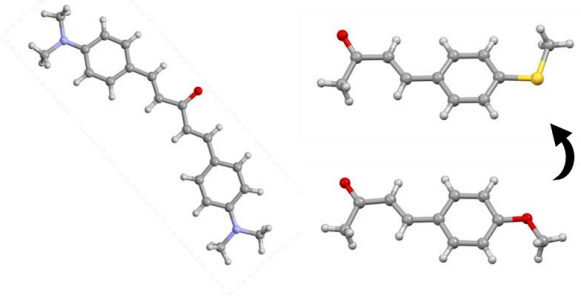
3. Solving the crystal structure - examples of solved structures



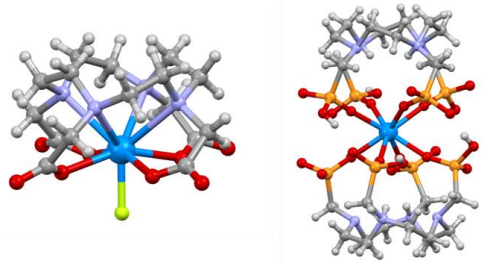
Organometallic catalysts



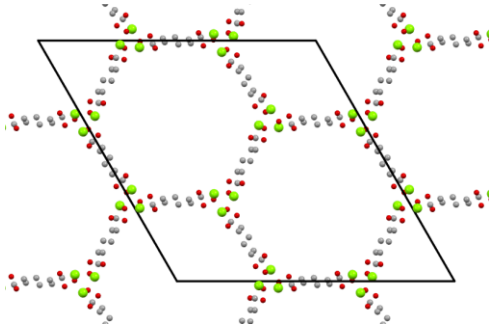
Polyoxometalate catalysts



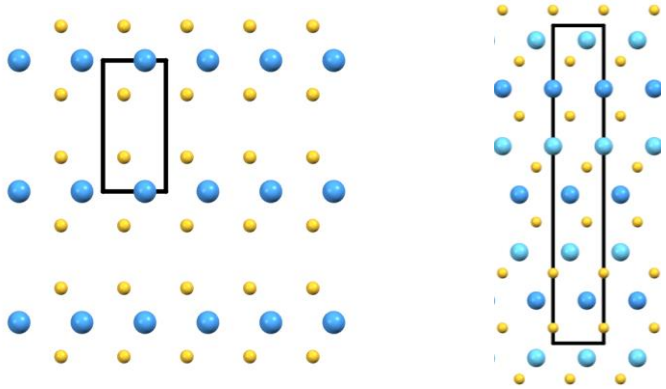
Organic molecules



Chelation complexes



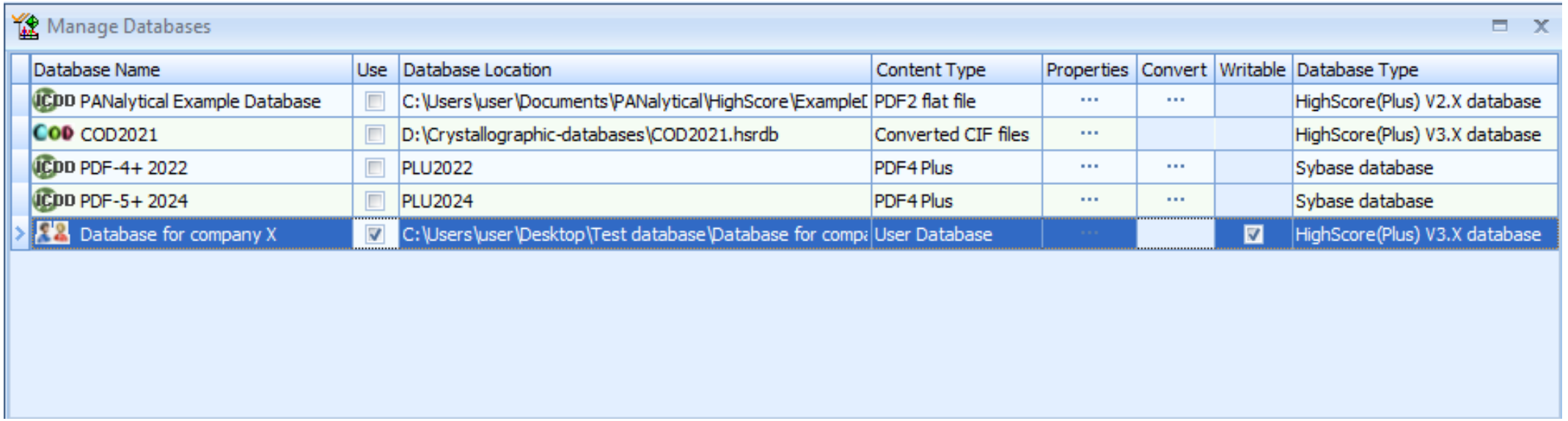
Metal-organic frameworks (MOFs)



Transition metal dichalcogenides (TMDs)

4. Custom made database

- Preparation of a custom database for customers.



- 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.

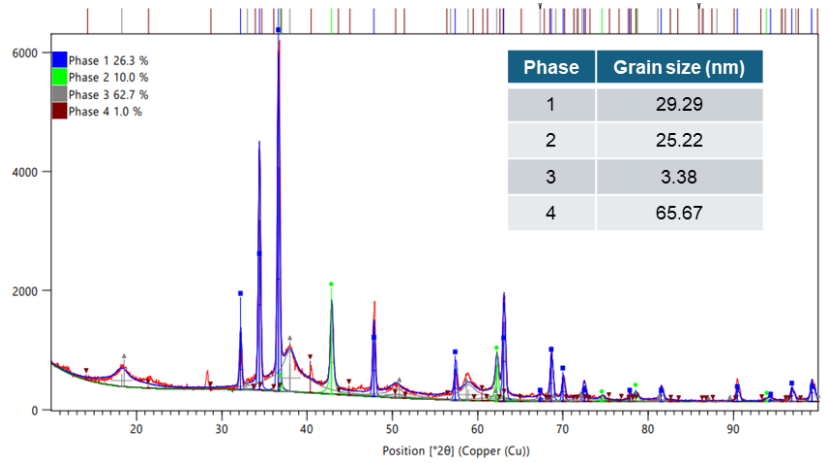
- Crystallite size of each phase.

- Amorphous percent in the sample.

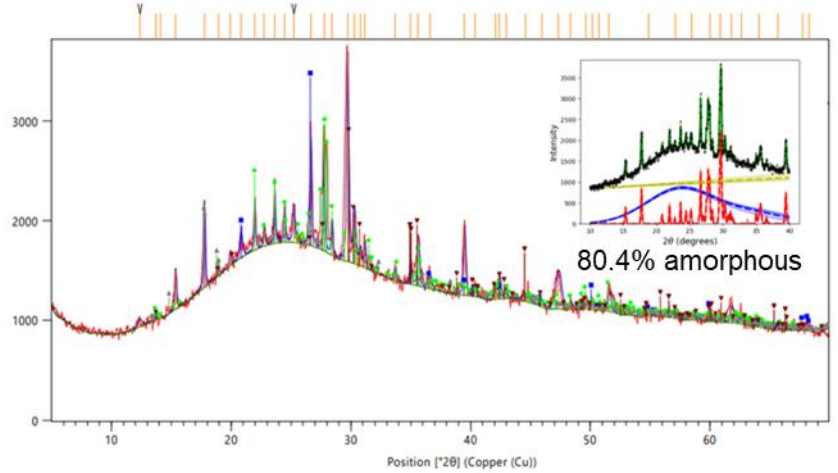
- Preferred orientation in the sample (relates to morphology of crystals).

- Variations in lattice parameters.

- Substitution of atoms in solids, and more.

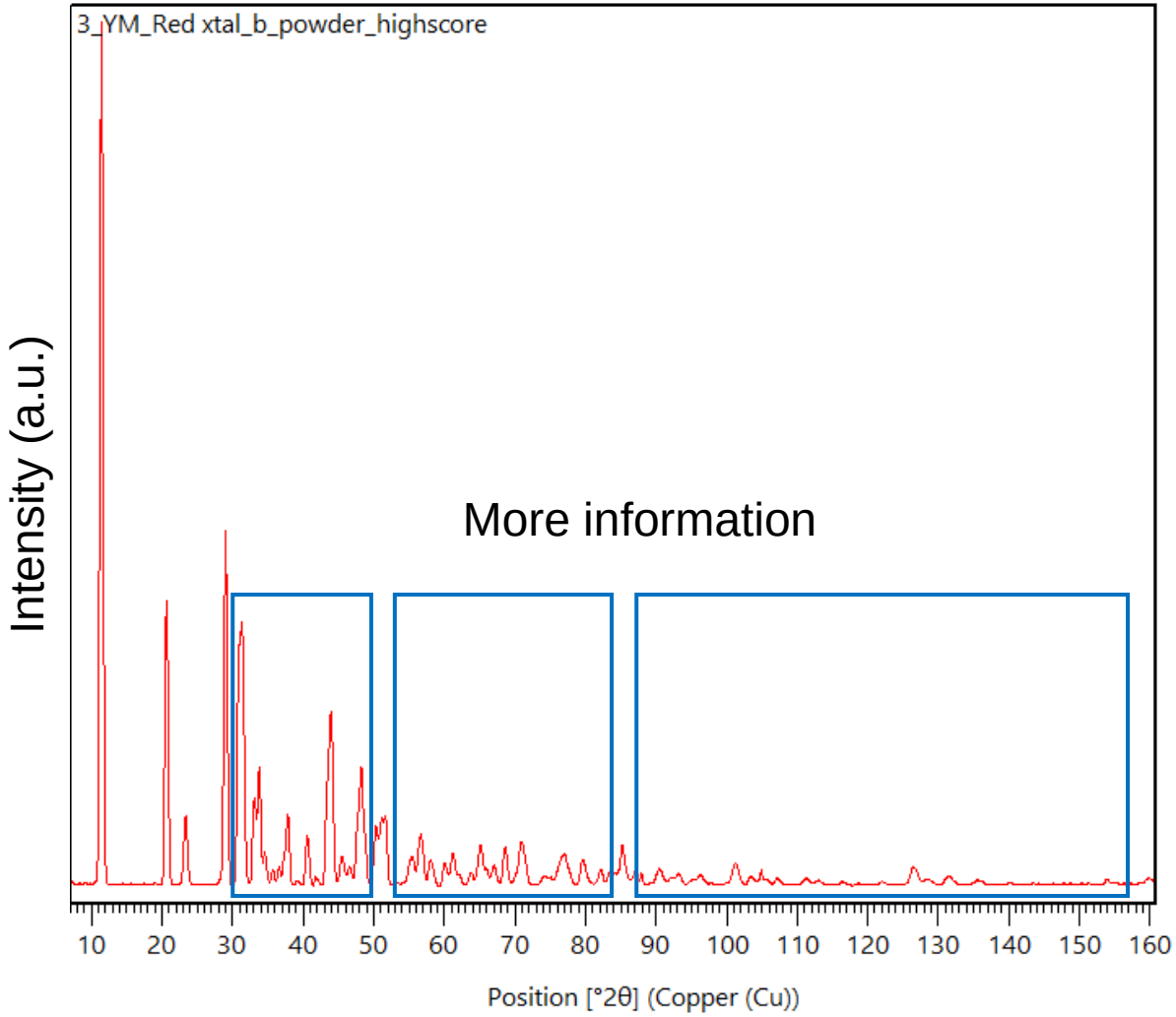


Phase	Grain size (nm)
1	29.29
2	25.22
3	3.38
4	65.67

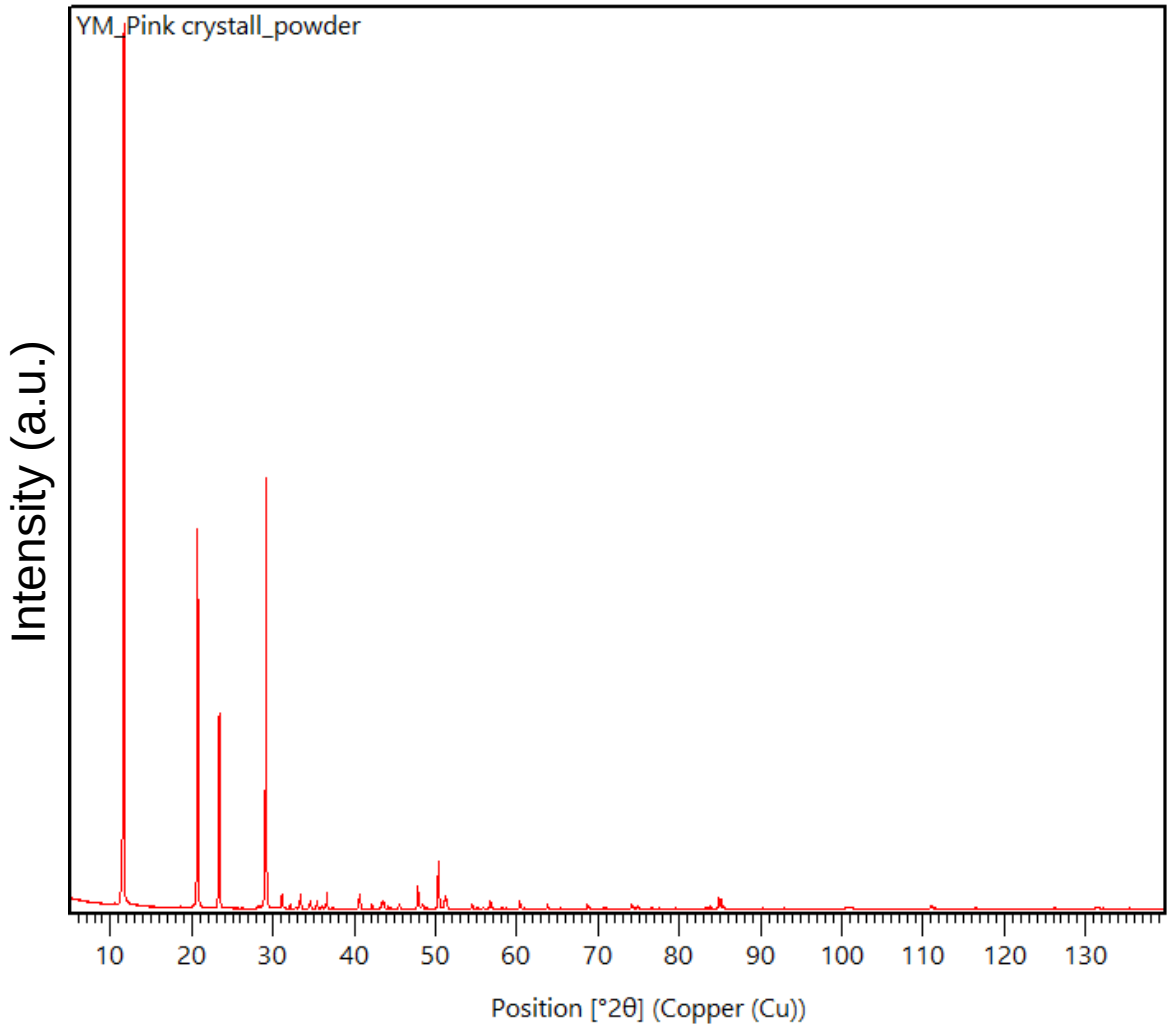


Powder spectrum

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.