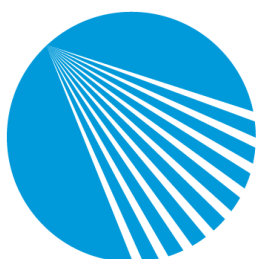


# XtaLAB Synergy-ED Measurement Report

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**Rigaku**  
**oxford diffraction**

# Sample Overview

The following samples were provided and measured on the XtaLAB Synergy-ED:

- **MIL-53-0.05NH<sub>2</sub>-0.95CH<sub>3</sub>** – aluminium metal-organic framework (MOF)
- **MIL-53-0.50NH<sub>2</sub>-0.50CH<sub>3</sub>** – aluminium MOF
- **MIL-53-0.75NH<sub>2</sub>-0.25CH<sub>3</sub>** – aluminium MOF

## Objectives

- Use a tailored electron beam to determine the crystal structure of nanometer-sized crystallites that are too small for home lab X-ray sources or synchrotron radiation.
- Assess data completeness and quality of final structure models.
- Demonstrate the advantages of cryo-transfer and measurement under cryogenic conditions.

A JEOL 200 kV electron source, column and beam optics optimized for electron diffraction purposes.



A Rigaku HyPix-ED detector optimized for operation in the Micro-ED/3DED experimental setup.



A sample stage allowing x, y, z sample alignment and rotation (tilt) about a single axis. A cryo option is available.



[XtaLAB Synergy-ED: Single crystal structures from powders](#)



[3D ED: An update from the Rigaku/JEOL collaboration](#)

*Main Rigaku publications:*

[Structure determination of small molecule compounds by an electron diffractometer for 3D ED/MicroED](#)



[Making the most of 3D ED: Best practices to handle a new tool](#)



## MIL-53-0.05NH<sub>2</sub>-0.95CH<sub>3</sub>

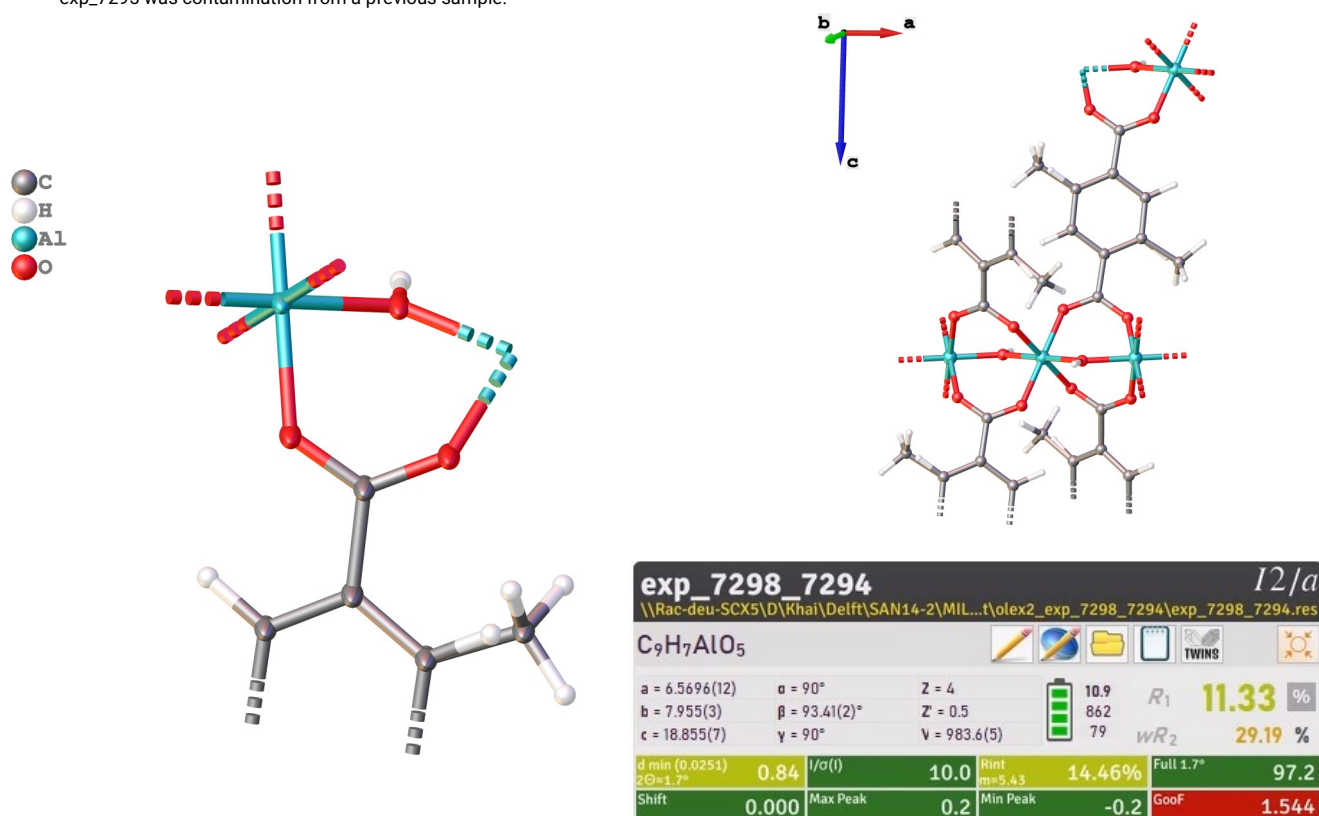
The sample **MIL-53-0.05NH<sub>2</sub>-0.95CH<sub>3</sub>** was provided as colorless solid. The sample was dispersed in ethanol, sonicated, and drop-casted on lacey-carbon supported copper TEM grids. The sample consisted of very small, highly agglomerated plate-like crystallites. The datasets were measured from different crystallites with a wavelength of 0.0251 Å at 175 K. Benefits of collecting data at low temperature include reduction of beam damage, stabilization of the sample *in vacuo* and improvement of resolution. In combination with cryo-transfer (*viz.* freezing samples

prior to introduction to vacuum), solvate and hydrate structures remain unscathed. A total of six datasets were collected. However, only datasets **7294** and **7298** were used for data merging. The individual measurements lasted a bit more than 2 minutes, resulting in a total experiment time of less than 12 minutes. While each individual dataset was suitable to provide a correct structure solution, by merging multiple datasets, more complete, significant and redundant data were obtained and resulted in a good quality structure refinement.

**Table 1.** Data collection parameter overview.

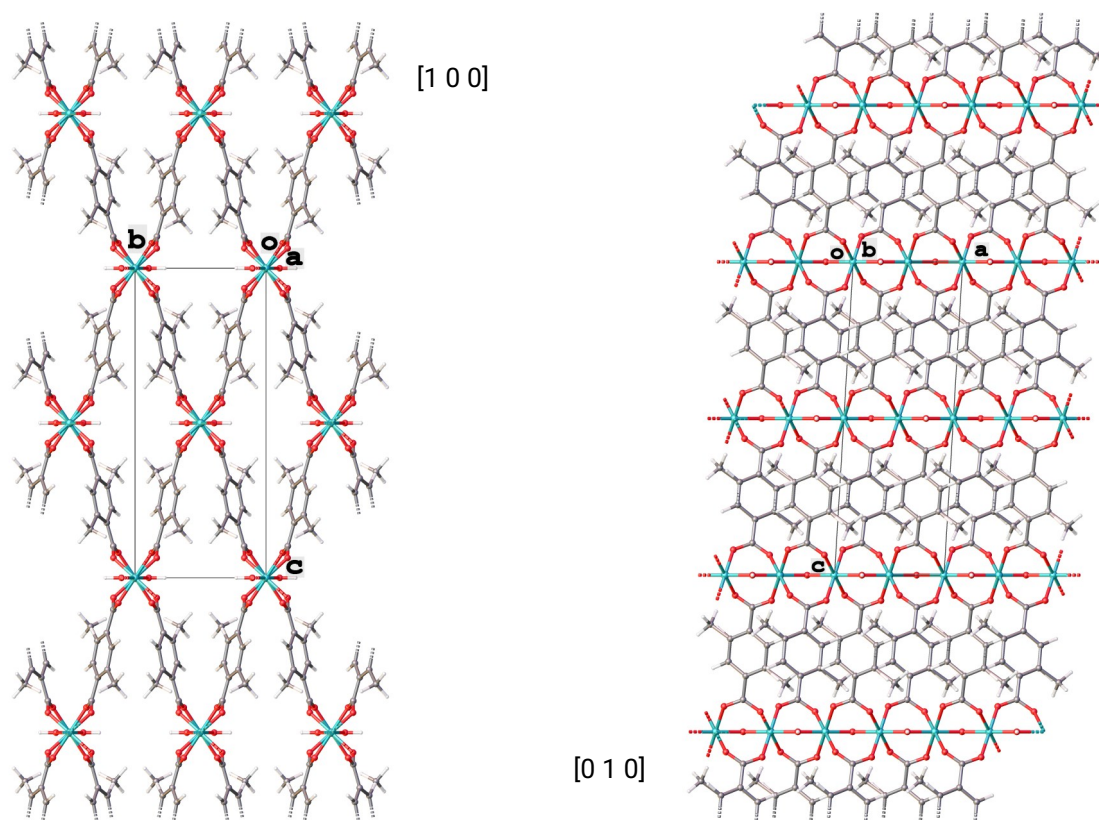
Dataset	Number of frames	Scan range [°]	Scan width [°]	Exposure time [s]	Dose time [min]	Dose [e <sup>-</sup> /(Å <sup>2</sup> )]
<b>7294</b>	480	-60 to +60	0.25	0.25	02:00	2.26
<b>7296</b>	448	-62 to +50	0.25	0.25	01:52	2.11
<b>7297</b>	420	-60 to +45	0.25	0.25	01:45	1.97
<b>7298</b>	496	-62 to +62	0.25	0.25	02:04	1.18
<b>7299</b>	504	-63 to +63	0.25	0.25	02:06	1.19
<b>7300</b>	500	-63 to +62	0.25	0.25	02:05	1.19
					<b>11:52</b>	

\*exp\_7295 was contamination from a previous sample.



**Figure 1.** Asymmetric unit, extended structure, and quality indicators for merged dataset **exp\_7298\_7294** of sample **MIL-53-0.05NH<sub>2</sub>-0.95CH<sub>3</sub>**.

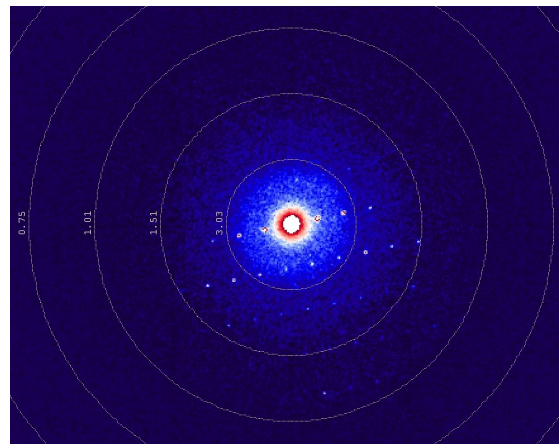
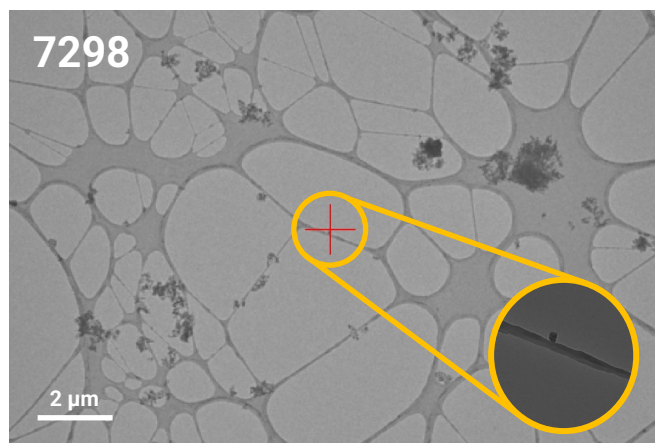
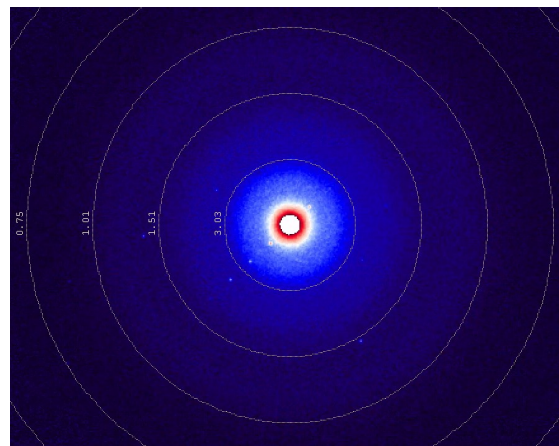
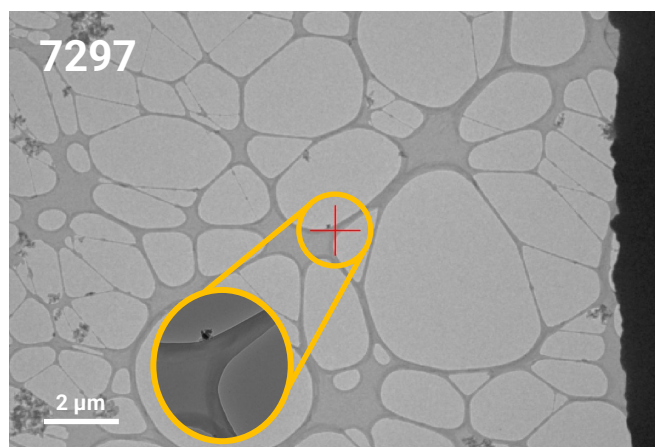
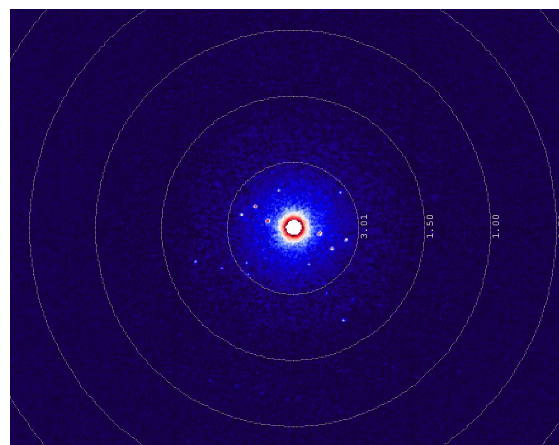
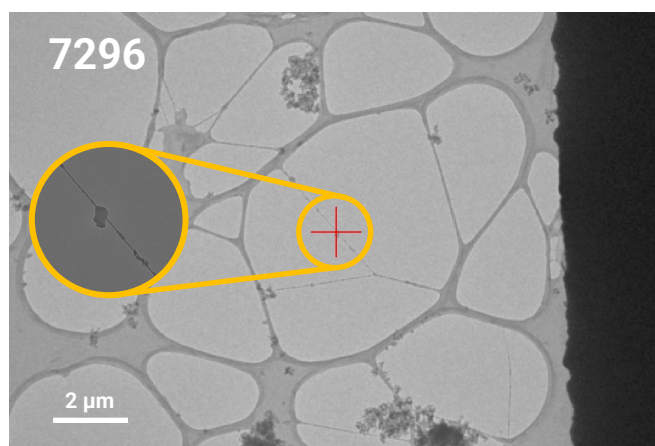
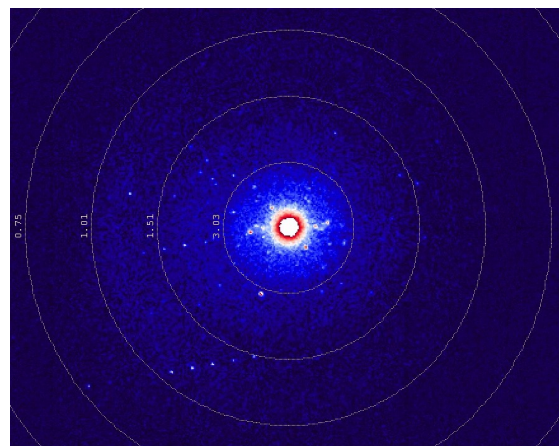
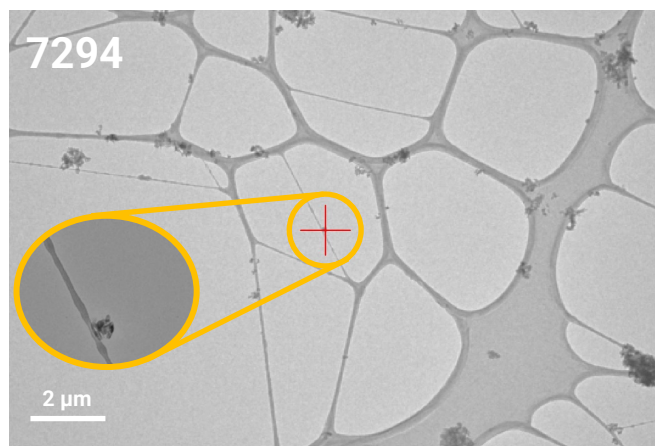




**Figure 2.** Crystal packing of MIL-53-0.05NH<sub>2</sub>-0.95CH<sub>3</sub> (exp\_7298\_7294) viewing along different directions.

**Table 2.** Data quality statistics overview for exp\_7294\_7298 of MIL-53-0.05NH<sub>2</sub>-0.95CH<sub>3</sub>. This dataset was processed up to a resolution of 0.837 Å. Point group symmetry: *I*2/m (b-unique).

Resolution [Å]	Data	Theory	Unique	Compl. [%]	Redund.	$\langle F^2 \rangle$	$\langle F^2 / \sigma(F^2) \rangle$	$R_{\text{int}}$	$R_{\text{pim}}$	$R_{\text{rim}}$	CC1/2
inf- 1.88	433	95	89	93.7	4.9	11895.90	13.40	0.087	0.043	0.096	0.991
1.88- 1.46	448	95	91	95.8	4.9	3020.48	9.01	0.128	0.062	0.144	0.990
1.46- 1.27	442	95	91	95.8	4.9	3012.26	8.18	0.143	0.069	0.153	0.985
1.27- 1.14	476	95	92	96.8	5.2	2006.48	6.23	0.151	0.074	0.152	0.993
1.14- 1.06	471	95	93	97.9	5.1	1728.49	5.88	0.185	0.090	0.208	0.979
1.06- 1.00	492	95	91	95.8	5.4	1089.31	4.80	0.222	0.102	0.259	0.982
1.00- 0.94	460	95	93	97.9	4.9	646.30	3.95	0.312	0.155	0.399	0.932
0.94- 0.90	487	95	92	96.8	5.3	421.77	2.90	0.399	0.186	0.509	0.925
0.90- 0.87	469	95	92	96.8	5.1	488.79	3.45	0.374	0.178	0.480	0.895
0.87- 0.84	499	103	99	96.1	5.0	321.95	2.18	0.470	0.233	0.587	0.795
inf- 0.84	4677	958	923	96.9	5.1	2359.05	5.88	0.144	0.070	0.158	0.992



**Figure 3.** Grain snapshots and exemplary diffraction images of MIL-53-0.05NH<sub>2</sub>-0.95CH<sub>3</sub>.



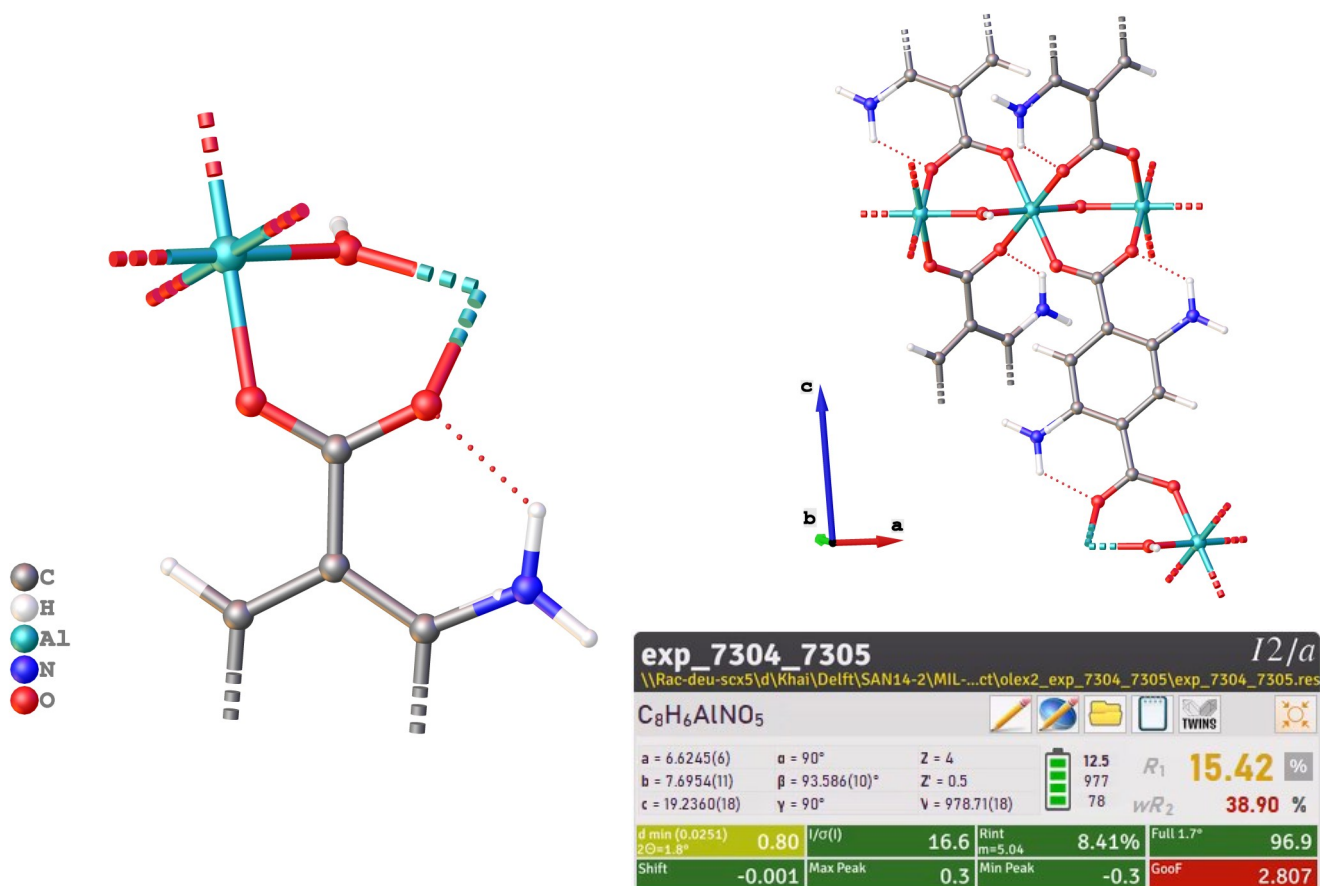
## MIL-53-0.50NH<sub>2</sub>-0.50CH<sub>3</sub>

The sample **MIL-53-0.50NH<sub>2</sub>-0.50CH<sub>3</sub>** was provided as colorless solid. The sample was dispersed in ethanol, sonicated, and drop-casted on lacey-carbon supported copper TEM grids. The sample consisted of decent sized, highly agglomerated plate-like crystallites. The datasets were measured from different crystallites with a wavelength of 0.0251 Å at 175 K. Benefits of collecting data at low temperature includes reduction of beam damage, stabilization of the sample *in vacuo* and improvement of resolution. In combination with cryo-transfer (*viz.* freezing sam-

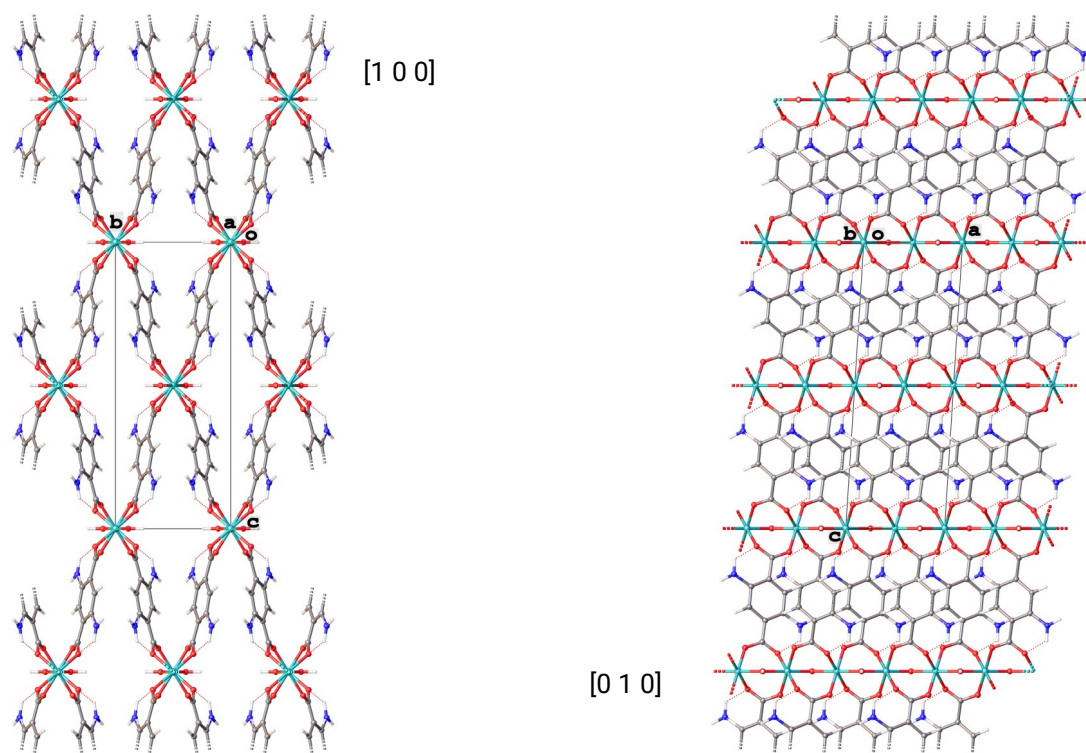
ples prior to introduction to vacuum), solvate and hydrate structures remain unscathed. A total of three datasets were collected. However, only datasets **7304** and **7305** were used for data merging. The individual measurements lasted around 2 minutes, resulting in a total experiment time of less than 6 minutes. While each individual dataset was suitable to provide a correct structure solution, by merging multiple datasets, more complete, significant and redundant data were obtained and resulted in a good quality structure refinement.

**Table 3.** Data collection parameter overview.

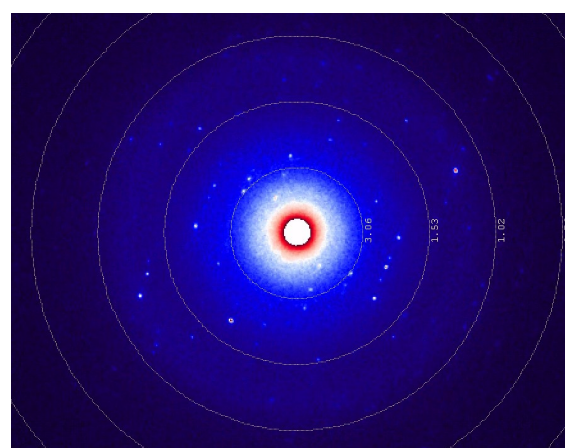
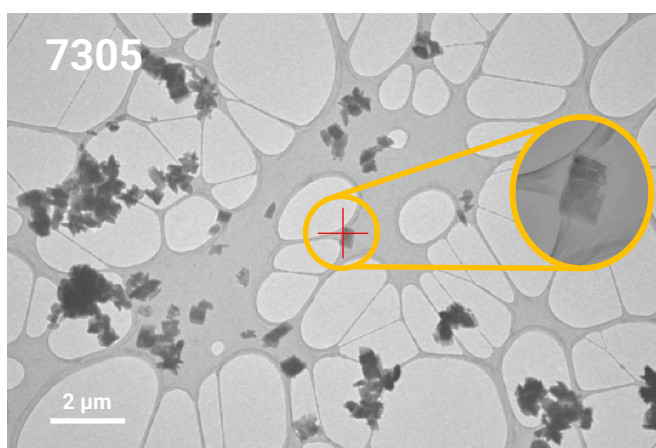
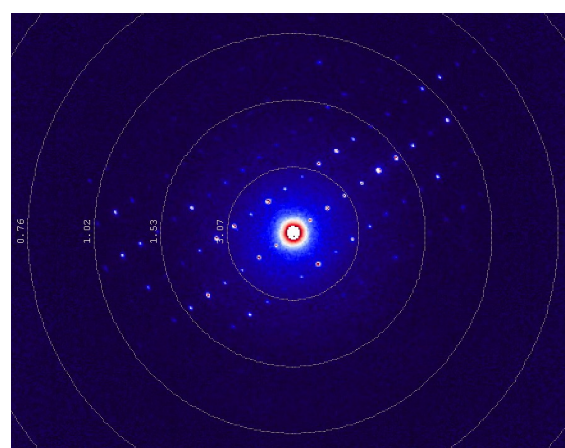
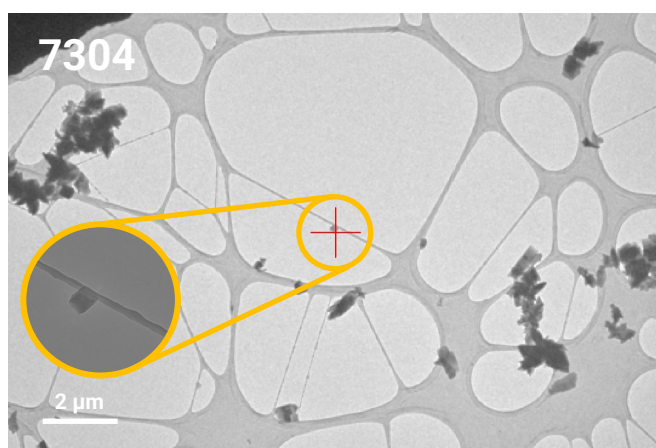
Dataset	Number of frames	Scan range [°]	Scan width [°]	Exposure time [s]	Dose time [min]	Dose [e <sup>-</sup> /(Å <sup>2</sup> )]
<b>7304</b>	504	-62 to +64	0.25	0.25	02:06	1.19
<b>7305</b>	496	-62 to +62	0.25	0.25	02:04	1.18
<b>7306</b>	408	-62 to +40	0.25	0.25	01:42	0.97
					<b>05:52</b>	

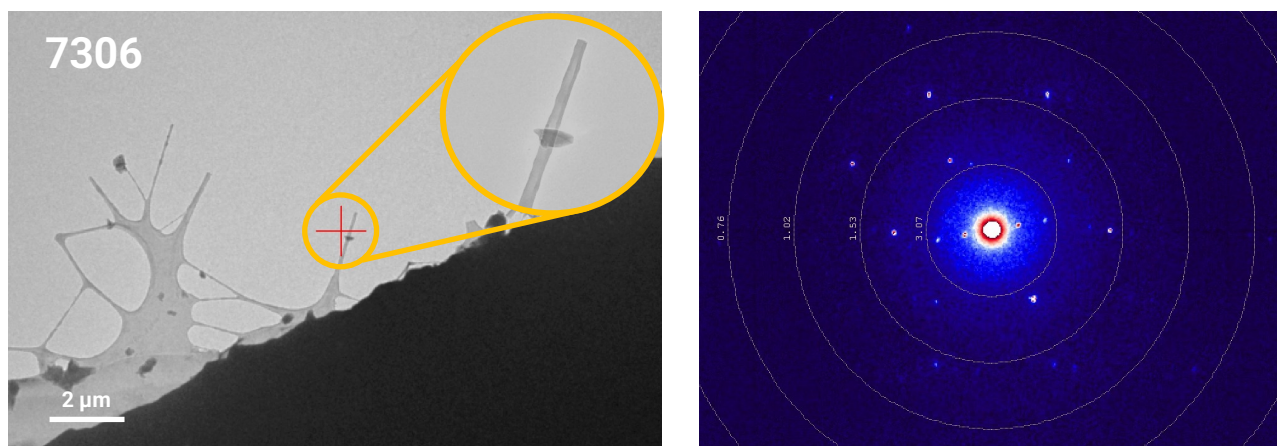


**Figure 4.** Asymmetric unit, extended structure, and quality indicators for merged dataset **exp\_7304\_7305** of sample **MIL-53-0.50NH<sub>2</sub>-0.50CH<sub>3</sub>**.



**Figure 5.** Crystal packing of MIL-53-0.50NH<sub>2</sub>-0.50CH<sub>3</sub> (exp\_7304\_7305) viewing along different directions.





**Figure 6.** Grain snapshots and exemplary diffraction images of **MIL-53-0.50NH<sub>2</sub>-0.50CH<sub>3</sub>**.

**Table 4.** Data quality statistics overview for **exp\_7304\_7305** of **MIL-53-0.50NH<sub>2</sub>-0.50CH<sub>3</sub>**. This dataset was processed up to a resolution of 0.80 Å. Point group symmetry: *I*2/m (b-unique).

Resolution [Å]	Data	Theory	Unique	Compl. [%]	Redund.	$\langle F^2 \rangle$	$\langle F^2 / \sigma(F^2) \rangle$	$R_{\text{int}}$	$R_{\text{pim}}$	$R_{\text{rim}}$	CC1/2
inf- 1.77	443	108	104	96.3	4.3	209604.17	51.59	0.057	0.031	0.071	0.981
1.77- 1.39	452	108	106	98.1	4.3	61053.75	26.91	0.071	0.038	0.089	0.993
1.39- 1.21	474	108	104	96.3	4.6	56010.07	21.69	0.076	0.039	0.074	0.998
1.21- 1.09	488	108	107	99.1	4.6	56291.75	20.53	0.086	0.045	0.106	0.994
1.09- 1.02	493	108	105	97.2	4.7	29513.11	15.89	0.105	0.053	0.118	0.994
1.02- 0.95	527	108	105	97.2	5.0	20981.85	13.33	0.132	0.061	0.152	0.992
0.95- 0.90	519	108	105	97.2	4.9	13227.93	9.92	0.163	0.082	0.202	0.962
0.90- 0.86	536	108	108	100.0	5.0	13350.51	9.56	0.178	0.087	0.218	0.969
0.86- 0.83	500	108	101	93.5	5.0	9855.31	7.42	0.223	0.108	0.252	0.962
0.83- 0.80	490	112	110	98.2	4.5	10136.77	7.86	0.175	0.094	0.227	0.955
inf- 0.80	4922	1084	1055	97.3	4.7	45508.60	17.88	0.085	0.044	0.099	0.990



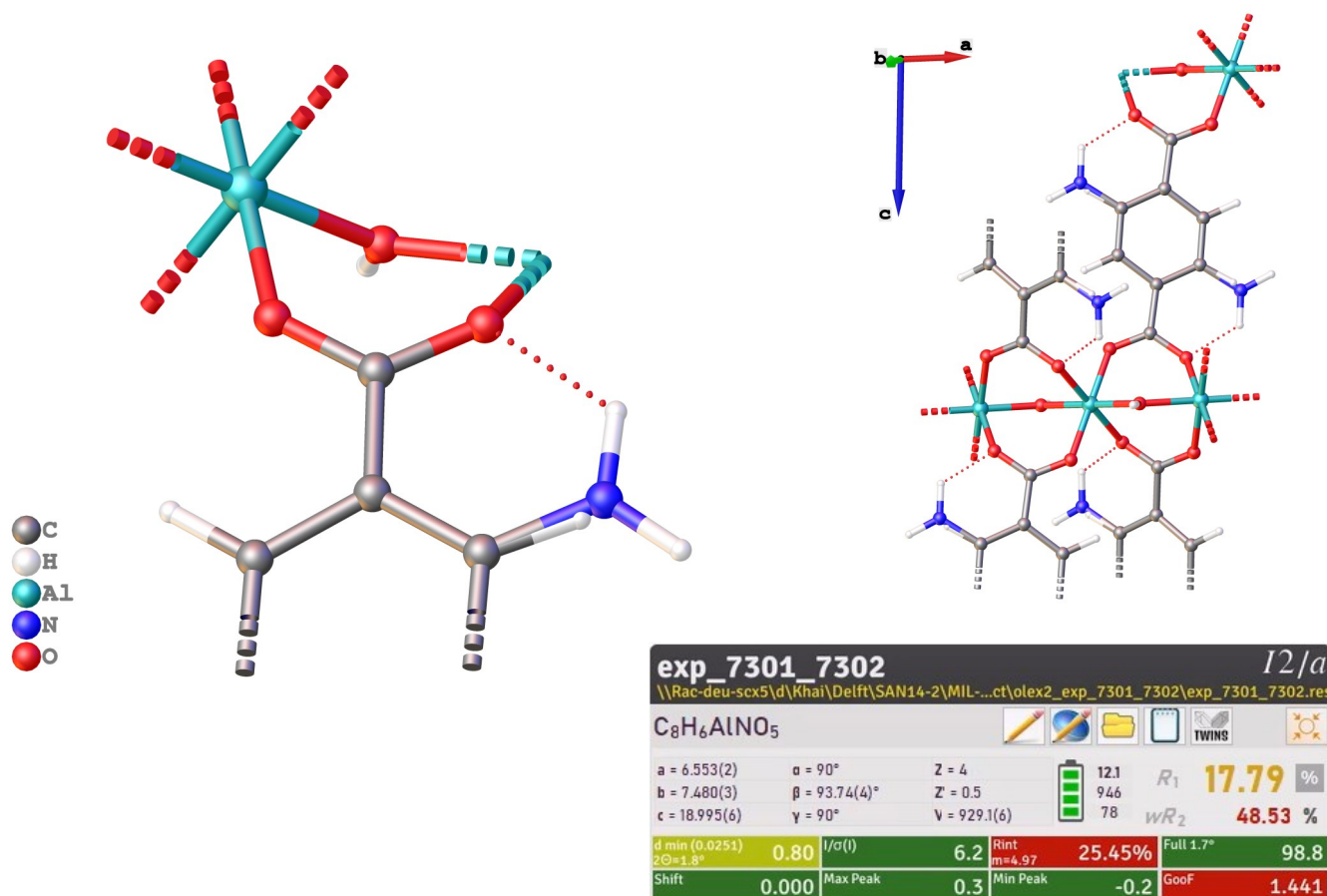
## MIL-53-0.75NH<sub>2</sub>-0.25CH<sub>3</sub>

The sample **MIL-53-0.75NH<sub>2</sub>-0.25CH<sub>3</sub>** was provided as colorless solid. The sample was dispersed in ethanol, sonicated, and drop-casted on lacey-carbon supported copper TEM grids. The sample consisted of very small, highly agglomerated plate-like crystallites. The datasets were measured from different crystallites with a wavelength of 0.0251 Å at 175 K. Benefits of collecting data at low temperature includes reduction of beam damage, stabilization of the sample *in vacuo* and improvement of resolution. In combination with cryo-transfer (*viz.* freezing sam-

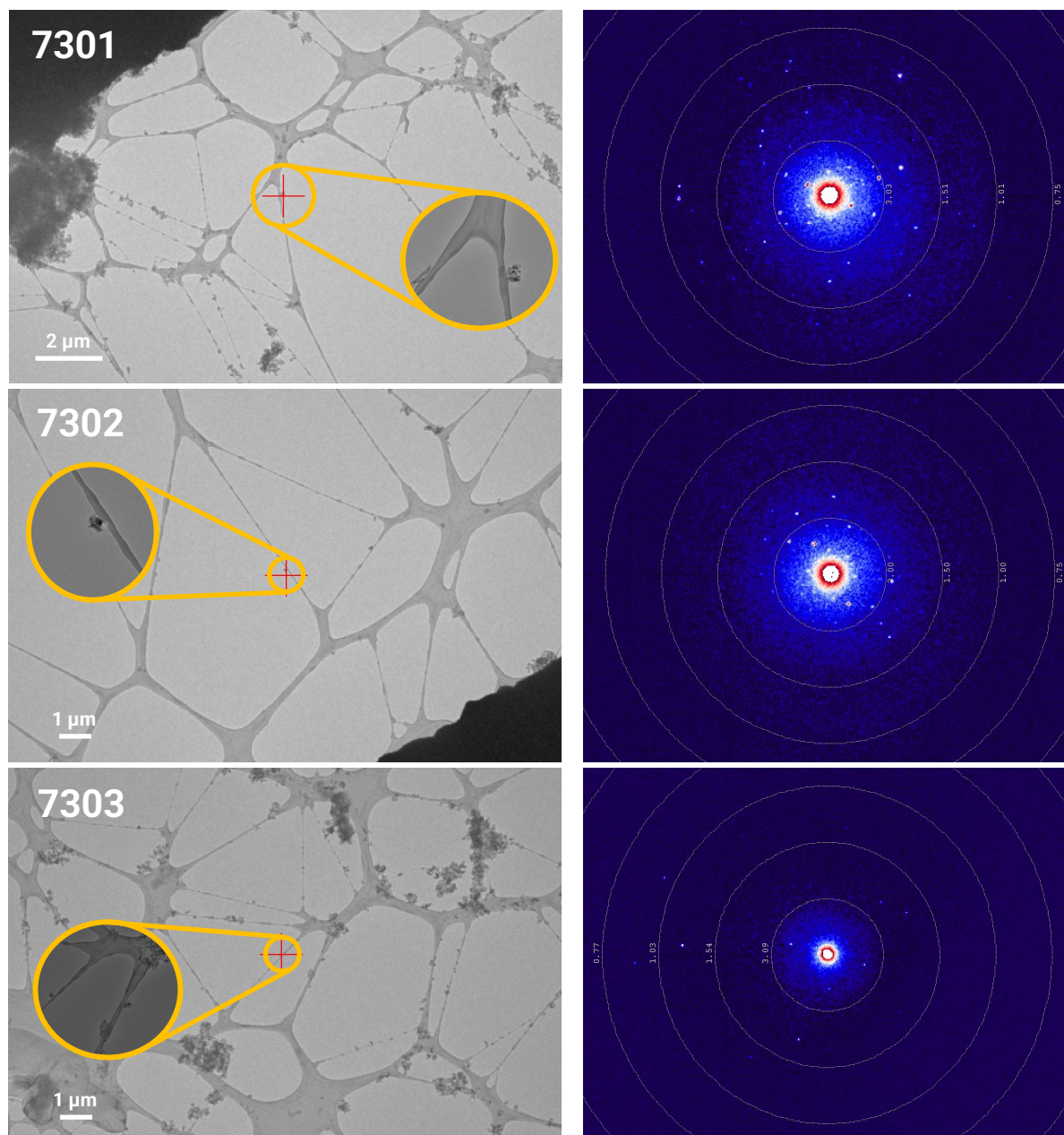
ples prior to introduction to vacuum), solvate and hydrate structures remain unscathed. A total of three datasets were collected. However, only datasets **7301** and **7302** were used for data merging. The individual measurements lasted around 2 minutes, resulting in a total experiment time of ca. 6 minutes. While each individual dataset was suitable to provide a correct structure solution, by merging multiple datasets, more complete, significant and redundant data were obtained and resulted in a good quality structure refinement.

**Table 5.** Data collection parameter overview.

Dataset	Number of frames	Scan range [°]	Scan width [°]	Exposure time [s]	Dose time [min]	Dose [e <sup>-</sup> /(Å <sup>2</sup> )]
<b>7301</b>	480	-60 to +60	0.25	0.25	02:00	1.14
<b>7302</b>	496	-63 to +61	0.25	0.25	02:04	1.18
<b>7303</b>	480	-60 to +60	0.25	0.25	02:00	1.14
					<b>06:04</b>	



**Figure 7.** Asymmetric unit, extended structure, and quality indicators for merged dataset **exp\_7304\_7305** of sample **MIL-53-0.75NH<sub>2</sub>-0.25CH<sub>3</sub>**.



**Figure 7.** Grain snapshots and exemplary diffraction images of **MIL-53-0.75NH<sub>2</sub>-0.25CH<sub>3</sub>**.

**Table 6.** Data quality statistics overview for **exp\_7301\_7302** of **MIL-53-0.75NH<sub>2</sub>-0.25CH<sub>3</sub>**. This dataset was processed up to a resolution of 0.80 Å. Point group symmetry:  $I2/m$  (b-unique).

Resolution [Å]	Data	Theory	Unique	Compl. [%]	Redund.	$\langle F^2 \rangle$	$\langle F^2 / \sigma(F^2) \rangle$	$R_{int}$	$R_{pim}$	$R_{rim}$	CC1/2
inf- 1.78	419	103	99	96.1	4.2	9668.49	14.87	0.116	0.058	0.117	0.980
1.78- 1.39	454	103	100	97.1	4.5	2131.60	7.10	0.205	0.103	0.216	0.977
1.39- 1.21	473	103	101	98.1	4.7	1936.71	5.60	0.211	0.107	0.203	0.991
1.21- 1.09	456	103	101	98.1	4.5	1265.91	3.55	0.287	0.153	0.280	0.975
1.09- 1.02	501	103	100	97.1	5.0	1001.33	3.55	0.354	0.175	0.346	0.959
1.02- 0.95	482	103	102	99.0	4.7	574.23	2.03	0.473	0.243	0.528	0.923
0.95- 0.90	496	103	101	98.1	4.9	433.00	2.03	0.600	0.290	0.744	0.793
0.90- 0.86	494	103	102	99.0	4.8	401.39	1.62	0.580	0.283	0.759	0.827
0.86- 0.83	461	103	101	98.1	4.6	248.82	1.10	0.754	0.397	1.206	0.591
0.83- 0.80	462	107	105	98.1	4.4	247.44	1.08	0.738	0.391	1.349	0.341
inf- 0.80	4698	1034	1012	97.9	4.6	1688.52	4.11	0.237	0.120	0.249	0.983

## Results

- Use a tailored electron beam to determine the crystal structure of nano-sized crystallites that are too small for home lab X-ray sources or synchrotron radiation.

With the XtaLAB Synergy-ED, the worlds first dedicated electron diffractometer, developed in collaboration between Rigaku and JEOL, nano-sized crystallites can be measured at ease. The newest version of *CrysAlis<sup>Pro</sup>* allows the user to operate the ED system in a similar fashion like classic diffractometers. Data reduction, scaling and structure solution are all handled in a single program suite, supported by automatic routines and powerful algorithms implemented in *AutoChem*.

- Assess data completeness and quality of final structure models. Perform dynamical refinement and z-score calculation to determine the absolute structure.

Comprehensive data were obtained for all submitted samples. The structures were solved with SHELXT and refined with olex2.refine in the latest version of *Olex2*. Accurate structure factors for electron beams are automatically added to the .ins file for refinement.

Refinement results of electron diffraction data are affected by dynamical diffraction and the special nature of charged particles. Due to these effects, R-values are systematically higher than for classical X-ray diffraction. However, within the context of electron diffraction data, not taking these effects into account yet, and despite high agglomeration/mosaicity of the tiny crystallites, the results are excellent.

The samples diffracted well. Merging of datasets collected from different grains resulted in full data completeness. Overview of all datasets (blue: **MIL-53-0.05NH<sub>2</sub>-0.95CH<sub>3</sub>**, orange: **MIL-53-0.50NH<sub>2</sub>-0.50CH<sub>3</sub>**, green: **MIL-53-0.75NH<sub>2</sub>-0.25CH<sub>3</sub>**):

Name	Chemical formula	Laue	Volume	Unit cell (as used by data reduction)						Rint	Redundancy
exp_7294	C <sub>9</sub> H <sub>7</sub> Al O <sub>5</sub>	mC	987.06	6.60	7.97	18.80	90.0	93.0	90.0	0.1332	3.1
exp_7296	C <sub>9</sub> H <sub>7</sub> Al O <sub>5</sub>	mC	983.64	6.59	7.96	18.78	90.0	92.9	90.0	0.2144	2.9
exp_7297	C <sub>9</sub> H <sub>7</sub> Al O <sub>5</sub>	mC	984.15	6.55	7.97	18.87	90.0	92.7	90.0	0.2145	2.5
exp_7298	C <sub>9</sub> H <sub>7</sub> Al O <sub>5</sub>	mC	984.07	6.57	7.96	18.86	90.0	93.5	90.0	0.1437	5.1
exp_7299	C <sub>9</sub> H <sub>7</sub> Al O <sub>5</sub>	mC	990.58	6.56	8.01	18.87	90.0	93.2	90.0	0.1684	5.2
exp_7300	C <sub>9</sub> H <sub>7</sub> Al O <sub>5</sub>	mC	994.26	6.57	8.06	18.80	90.0	92.7	90.0	0.1516	3.1
exp_7304	C <sub>8</sub> H <sub>6</sub> Al N O <sub>5</sub>	mC	979.14	6.62	7.70	19.24	90.0	93.6	90.0	0.0847	4.7
exp_7305	C <sub>8</sub> H <sub>6</sub> Al N O <sub>5</sub>	mC	979.65	6.66	7.67	19.23	90.0	94.1	90.0	0.0487	2.8
exp_7306	C <sub>8</sub> H <sub>6</sub> Al N O <sub>5</sub>	mC	958.51	6.58	7.70	18.95	90.0	93.5	90.0	0.0727	2.9
exp_7301	C <sub>8</sub> H <sub>6</sub> Al N O <sub>5</sub>	mC	922.61	7.43	6.56	18.93	90.0	92.6	90.0	0.2365	4.6
exp_7302	C <sub>8</sub> H <sub>6</sub> Al N O <sub>5</sub>	mC	921.58	6.54	7.45	18.97	90.0	94.1	90.0	0.1838	2.8
exp_7303	C <sub>8</sub> H <sub>6</sub> Al N O <sub>5</sub>	mC	927.60	6.57	7.45	19.00	90.0	93.5	90.0	0.1721	2.7

- Advantages of cryo-transfer and measurements under cryogenic conditions.

The obtained results were all collected at 175 K and cryo-transfer technique. No beam damage was observed. Flash-cooling samples prior to introduction to XtaLAB Synergy-ED's high vacuum allows the study of solvated and sensitive compounds, e.g. proteins or MOF crystals. Next to stabilization of samples *in vacuo*, benefits of cryo-transfer plus measuring at low temperature are beam damage reduction, resolution improvement and disorder reduction.

After the post-measurement discussion, we agreed to remeasure the samples again at RT to see if we could observe structural changes.

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