**Typical general procedure writeup for publications when using the Purdue University Panalytical Empyrean X-ray diffractometer (powders and Rietveld analysis)**

**Bragg-Brentano Mode (reflection-transmission stage)**:

Powder diffraction (XRD) data were collected in focusing mode on a Panalytical Empyrean X-ray diffractometer equipped with Bragg-Brentano HD optics, a sealed tube copper X-ray source (λ = 1.54178 Å), soller slits on both the incident and receiving optics sides, and a PixCel3D Medipix detector. Samples were hand ground using an agate mortar and pestle and packed in either metal sample cups with a sample area 16 mm wide and 2 mm deep, or using silicon single crystal zero background sample holders, 16 mm wide and 0.25 mm deep (depending on sample amount available). Anti-scatter slits and divergence slits as well as the mask were chosen based on sample area and starting θ angle. Data were collected between **xx** and **yy**° in 2θ using the Panalytical Data Collector software.1) Search/Match phase identification was performed using the HighScore2) software of Panalytical against the ICCD PDF5 and data base3) and verified against the CCDC4) and ICSD data bases5). Rietveld refinements were performed against the models of the single crystal structure data sets using the HighScore2) software of Malvern-Panalytical. Refinement of preferred orientation was included using a spherical harmonics model. Plots of Rietveld fits for all compounds are given in the SI.

1) Data Collector, XRD Data Collection software, Version 6.1b, PANalytical B.V., Almelo, The Netherlands, 2019.

2) HighScore, Version 5.2, Malvern PANalytical B.V., Almelo, The Netherlands, 2023.

3) ICDD (2024). PDF-5 2024 (Database), edited by Dr. Soorya Kabekkodu, International Centre for Diffraction Data, Newtown Square, PA, USA.

4) C. R. Groom, I. J. Bruno, M. P. Lightfoot and S. C. Ward, Acta Cryst. (2016). B72, 171-179. DOI: 10.1107/S2052520616003954

5) a) Bergerhoff, G. & Brown, I.D. in „Crystallographic Databases“, F.H. Allen et al. (Hrsg.) Chester, International Union of Crystallography, (1987). b) Belsky, A., Hellenbrandt, M., Karen, V. L. & Luksch, P. (2002). Acta Cryst. B58, 364–369.

**Debye-Scherrer Mode (capillary stage)**:

Powder diffraction (XRD) data were collected on a Panalytical Empyrean X-ray diffractometer equipped with a sealed tube copper X-ray source (λ = 1.54178 Å). A parallel and monochromatic beam was created using a hybrid monochromator featuring two Ge 220 single crystals and W/Si (hybrid MPD) mirror focusing optics. Fixed anti scatter slits, a Soller slit and a PixCel3D Medipix detector were used on the receiving optics side. Samples were hand ground using an agate mortar and pestle were packed into capillaries (**specify type and diameter**) and mounted onto a capillary goniometerhead (manufactured by Huber diffraction equipment). Capillaries were aligned in the beam using a digital alignment camera. A mask of 20 mm was used and the divergence slit was chosen based on the diameter of the capillary used. The capillary was rotated at a speed of 5 Hz and data were collected between **xx** and **yy**° in 2θ using the Panalytical Data Collector software.1) ) Search/Match phase identification was performed using the HighScore2) software of Panalytical against the ICCD PDF5 and data base3) and verified against the CCDC4) and ICSD data bases5). Rietveld refinements were performed against the models of the single crystal structure data sets using the HighScore2) software of Malvern-Panalytical. Refinement of preferred orientation was included using a spherical harmonics model. Plots of Rietveld fits for all compounds are given in the SI.

1) Data Collector, XRD Data Collection software, Version 6.1b, PANalytical B.V., Almelo, The Netherlands, 2019.

2) HighScore, Version 5.2, PANalytical B.V., Almelo, The Netherlands, 2020.

3) ICDD (2024). PDF-5 2024 (Database), edited by Dr. Soorya Kabekkodu, International Centre for Diffraction Data, Newtown Square, PA, USA.

4) C. R. Groom, I. J. Bruno, M. P. Lightfoot and S. C. Ward, Acta Cryst. (2016). B72, 171-179. DOI: 10.1107/S2052520616003954

5) a) Bergerhoff, G. & Brown, I.D. in „Crystallographic Databases“, F.H. Allen et al. (Hrsg.) Chester, International Union of Crystallography, (1987). b) Belsky, A., Hellenbrandt, M., Karen, V. L. & Luksch, P. (2002). Acta Cryst. B58, 364–369.