**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 PDF4+ and ICDD PDF4/Organics data base3) and the ICSD data base4). Rietveld refinements were performed against the models of the single crystal structure data sets using the HighScore2) software of 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 4.8, PANalytical B.V., Almelo, The Netherlands, 2018.

3) ICDD (2020). PDF-4+ 2020 and PDF4/Organics 2020 (Database), edited by Dr. Soorya Kabekkodu, International Centre for Diffraction Data, Newtown Square, PA, USA.

4) 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 PDF4+ and ICDD PDF4/Organics data base3) and the ICSD data base4). Rietveld refinements were performed against the models of the single crystal structure data sets using the HighScore2) software of Panalytical. 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 4.8, PANalytical B.V., Almelo, The Netherlands, 2018.

3) ICDD (2020). PDF-4+ 2020 and PDF4/Organics 2020 (Database), edited by Dr. Soorya Kabekkodu, International Centre for Diffraction Data, Newtown Square, PA, USA.

4) 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.

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. The sample ANTAK-RX1 was hand ground using an agate mortar and pestle and packed in a metal sample cup with a sample area 27 mm wide and 1 mm deep. Anti-scatter slit and divergence slit as well as the mask were chosen based on sample area and starting θ angle (DS= 0.25°, ASS=1°, mask=10mm). Data were collected between 10 and 100° in 2θ using the Panalytical Data Collector software.1) ) Search/Match phase identification was performed using the HighScore2) software of Panalytical against the ICCD PDF4+ and ICDD PDF4/Organics data base3) and the ICSD data base4). 3-Amino-5nitro-1,2,4-triazole (ICDD code 02-067-1047 in C2/c) was found as the best match, with no major lines not assigned. No secondary components were identified. Rietveld refinements were performed against the model of the single crystal structure (CCDC code JOWWIB) data set using the HighScore2) software of Panalytical. Refinement of preferred orientation was included using a spherical harmonics model. Plots of Rietveld fits for ANTAK-RX1 are given as pdf files.

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

2) HighScore, Version 4.8, PANalytical B.V., Almelo, The Netherlands, 2018.

3) ICDD (2020). PDF-4+ 2020 and PDF4/Organics 2020 (Database), edited by Dr. Soorya Kabekkodu, International Centre for Diffraction Data, Newtown Square, PA, USA.

4) 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.