Static disorder in a perovskite mixed-valence metal–organic framework

Supporting Information

Ines E. Collings[1,2,*], Paul J. Saines,[3] Mirko Mikolasek,[2]
Tiziana Boffa Ballaran,[4] Michael Hanfland[2]

[1]Center for X-ray Analytics, Swiss Federal Laboratories for Materials Science and Technology, Überlandstrasse 129, 8600 Dübendorf, Switzerland.
[2]European Synchrotron Radiation Facility, 71 avenue des Martyrs, 38000 Grenoble, France.
[3]School of Physical Sciences, University of Kent, Canterbury, CT2 7NH, U.K.
[4]Bayerisches Geoinstitut, University of Bayreuth, 95447 Bayreuth, Germany.
*E-mail: ines.collings@empa.ch

Contents

1 Powder diffraction pattern on DMAFeFe·H_2O-4

Using the same sample for the Mössbauer measurement, the crystallites of DMAFeFe·H_2O-4 packed into the kapton hole sample holder was measured on the ID15B beamline of the European Synchrotron Radiation Facility, Grenoble using monochromatic X-ray radiation ($\lambda = 411219\,\text{Å}$). A $\pm 3°$ ω rotation was performed during a measurement time of 1 s. The 2D diffraction pattern was integrated to a 1D powder profile using Dioptas.[S1] The powder pattern was refined using the Rietveld method in Topas [Figure S1].[S2]

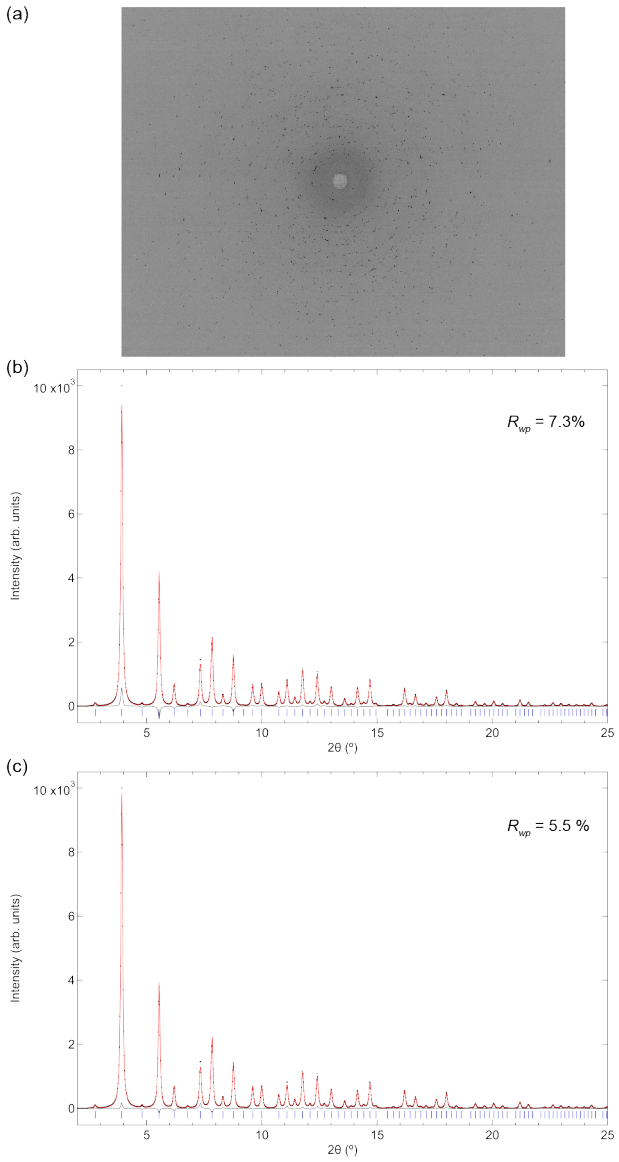

Figure S1: (a) Diffraction measured on the DMAFeFe·H_2O-4 crystallites packed into a kapton window of 200 μm thickness for Mössbauer measurements. (b) Rietveld refinement of DMAFeFe·H_2O-4 from the integrated diffraction image shown in (a) and (c) with preferred orientation taken into consideration. The data are represented in black, the fit in red, the difference curve (data–fit) in grey, and the allowed reflections are indicated by vertical blue bars.

2 Temperature and pressure dependence of DMAFeFe·H_2O-4 volumes

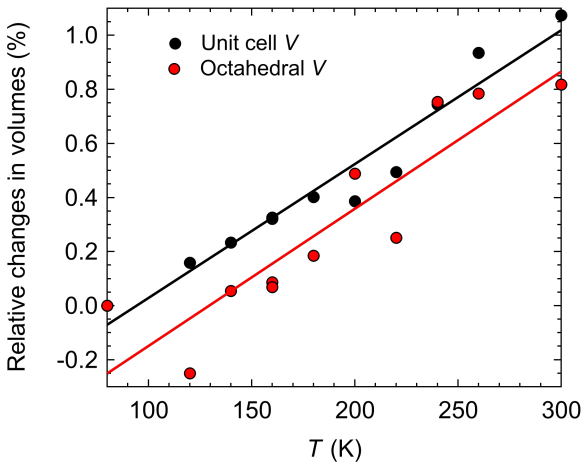

Figure S2: Temperature dependence of the relative changes in unit cell volume (black markers) and octahedral volumes (red markers) with straight line fits shown in solid lines. Octahedral volumes were calculated using VESTA.[S3]

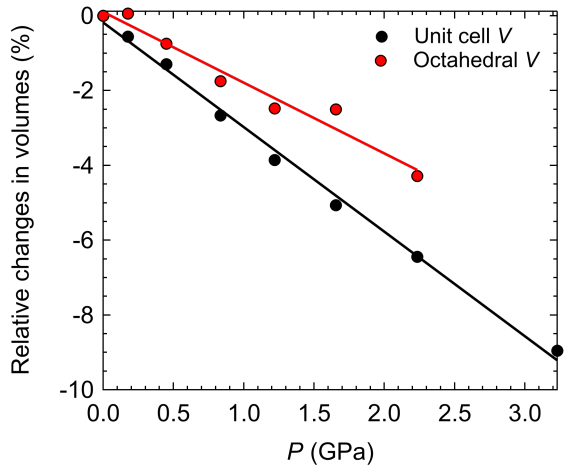

Figure S3: Pressure dependence of the relative changes in unit cell volume (black markers) and octahedral volumes (red markers) with straight line fits shown in solid lines. Octahedral volumes were calculated using VESTA.[S3]

3 Reciprocal space reconstruction

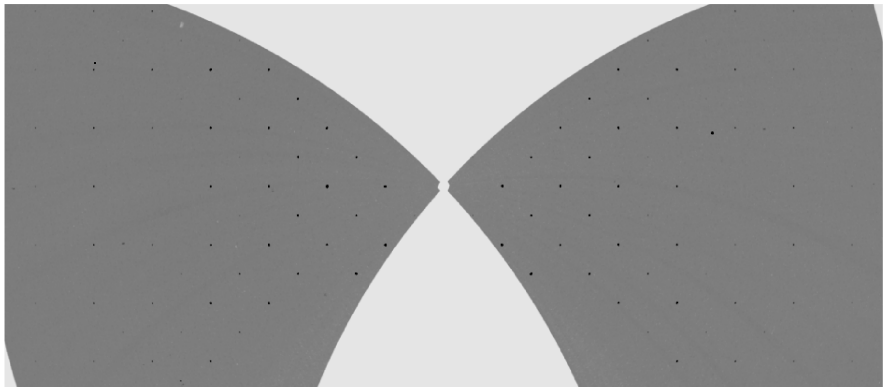

Figure S4: Reciprocal space reconstruction for crystal 2 at ambient conditions, used for the high-pressure experiment.

4 Variable-temperature lattice parameters

Table S1: Unit cell data for DMAFeFe·H_2O-4 as a function of temperature upon heating from 80 K. A greater number of frames were collected for the 80 K and 200 K data sets.

T (K)	a (Å)	V (Å^3)
80	11.9923(3)	1724.67(13)
120	11.9986(7)	1727.4(3)
140	12.0016(7)	1728.7(3)
160	12.0051(6)	1730.2(3)
180	12.0082(7)	1731.6(3)
200	12.0077(3)	1731.33(13)
220	12.0121(9)	1733.2(4)
240	12.0219(8)	1737.5(4)
260	12.0296(8)	1740.8(3)
300	12.0350(6)	1743.2(3)

5 Variable-pressure lattice parameters

Table S2: Unit cell data for DMAFeFe·H_2O-4 as a function of pressure for crystal 1.

P (GPa)	a (Å)	V (Å^3)
0	12.0370(9)	1744.0(4)
0.18(6)	12.0146(2)	1734.31(9)
0.45(5)	11.98482(17)	1721.45(7)
0.84(6)	11.9290(3)	1697.51(13)
1.22(6)	11.8801(5)	1676.71(19)
1.66(7)	11.83018(14)	1655.67(6)
2.24(6)	11.77258(15)	1631.60(6)
3.23(8)	11.6665(3)	1587.91(11)

References

(S1) C. Prescher and V. B. Prakapenka, *High Pressure Res.*, 2015, **35**, 223–230.

(S2) A. A. Coelho, *J. Appl. Crystallogr.*, 2018, **51**, 210–218.

(S3) K. Momma and F. Izumi, *J. Appl. Crystallogr.*, 2011, **44**, 1272–1276.