Electronic Supplementary Information (ESI)

A New High-Pressure Polymorph of $K_2MoO_2F_4$

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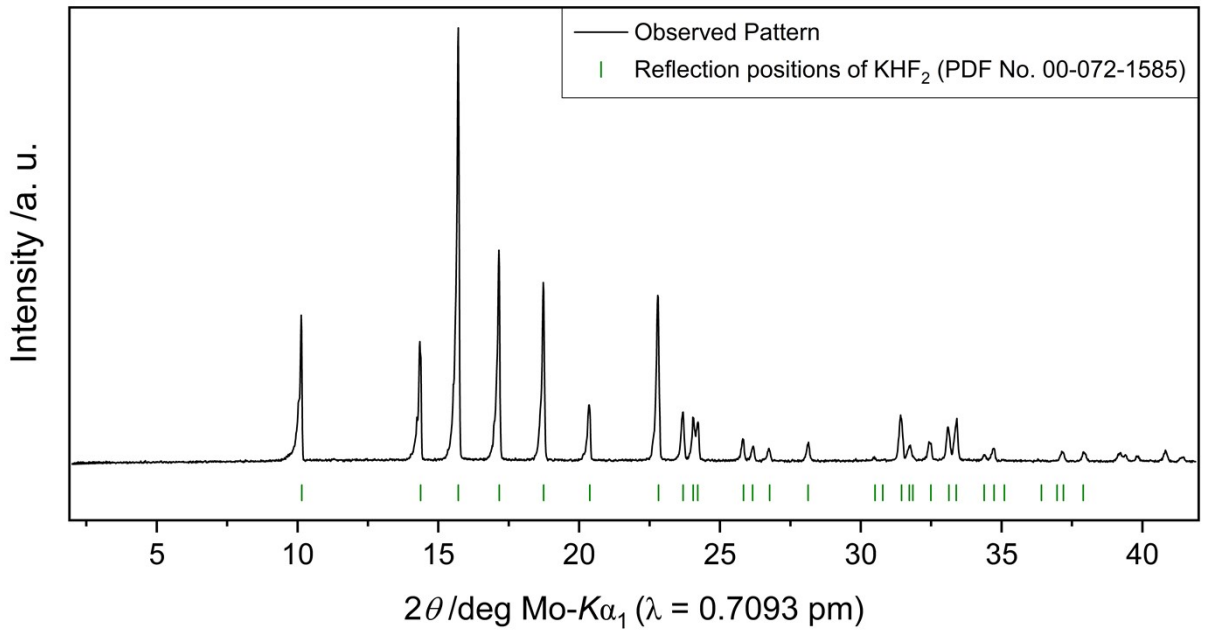

Figure S1: Indexed powder pattern of KHF_2 used as a starting material.

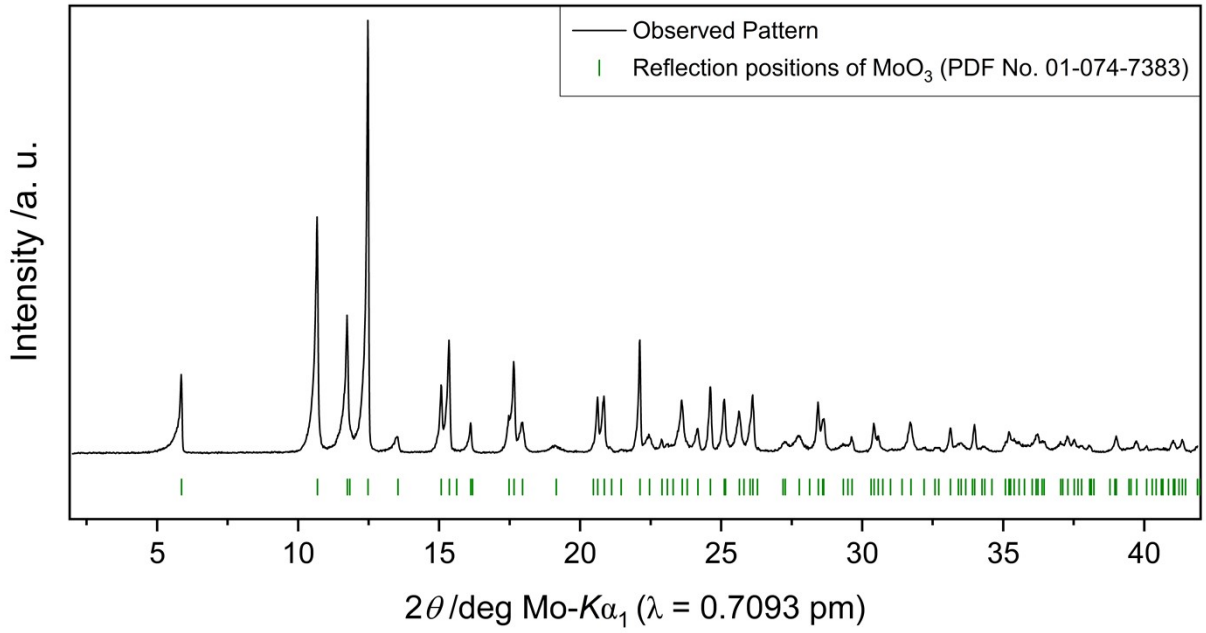

Figure S2: Indexed powder pattern of MoO_3 used as a starting material.

Rietveld analysis was performed using the software package DIFFRAC^{PLUS}-TOPAS 4.2 (Bruker AXS, Karlsruhe, Germany)[1] using a PXRD pattern of the sample and the single-crystal structure solutions of HP-$K_2MoO_2F_4$ and $K_2MoO_2F_4 \cdot H_2O$ (ICSD Collection Code 24913) as a starting point. Peak shape fitting was carried out using pseudo-Voigt profiles and a LaB_6 standard was used for calibration. Additional parameters as well as analysis results are reported in Table S1. Due to high background contribution, overlap of significant reflections and the presence of additional unidentified side phases, the estimated error for the calculated weight percentages is rather large.

Table S1: Rietveld refinement data.

Instrument parameters	
Powder diffractometer	STOE Stadi P
Radiation; wavelength λ /pm	Mo$K\alpha_1$; 70.93
Temperature /K	301(2)
Global R-values	
R_{exp} /%	7.07
R_{wp} /%	9.54
R_p /%	7.22
GooF	1.35
Corrections	
Zero error	-0.0065(14)
LP Factor	12.466
Absorption /cm^{-1}	89.09948
Background	
Chebychev polynomial	Order 50
Structure 1 – HP-$K_2MoO_2F_4$	
Spacegroup	*C*2/*m*
a /Å	13.9027(17)
b /Å	5.81072(55)
c /Å	6.90279(79)
β /deg	90.254(11)
V /Å^3	557.63(11)
Wt% - Rietveld	83(32)
Preferred Orientation Spherical Harmonics	Order 8
Structure 2 – $K_2MoO_2F_4 \cdot H_2O$	
Spacegroup	$P2_1/c$
a /Å	6.3057(35)
b /Å	6.0758(26)
c /Å	18.0912(85)
β /deg	93.653(68)
V /Å^3	691.71(59)
Wt% - Rietveld	17(32)
Preferred Orientation Spherical Harmonics	Order 8

1 A.A. Coelho, *J. Appl. Crystallogr.*, 2018, **51,** 210–218.