Making an order: the concerted alignment of \([\text{MOF}5]^{2-}\) (\(M = \text{Nb} \text{ and } \text{Ta}\)) dipolar anions in one-dimensional coordination chains sustained by tris(3,4,5-trimethyl-1H-pyrazole)copper(II)

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Instrumentation

IR-spectra (400-4000 cm\(^{-1}\)) were measured with a Perkin Elmer FTIR spectrometer (KBr pellets). The room temperature (rt) powder X-ray diffraction (PXRD) patterns were measured using a Stoe STADIP (Cu-K\(_{\alpha1}\), using a 'Mythen’ detector). Regarding peak positions, there is a very good coincidence between the experimental PXRD patterns of the obtained crystalline phases and the simulated patterns based on the single crystal structure (Mercury 3.3.1. software, CCDC). The temperature dependent X-ray measurements were recorded on a Stoe STADIP with a high temperature attachment and an image plate detector system. Simultaneous thermogravimetric/differential thermal analysis/mass spectrometry (TG/DTA-MS) studies were carried out on a Netzsch F1 Jupiter device connected to an Aeolos mass spectrometer. Samples were heated at a rate of 10 K min\(^{-1}\).

Synthesis of the ligand:

**Figure S1.** Comparison of the experimental PXRD pattern of as-synthesized $[\text{Cu(Me$_3$pz)$_3$}[\text{NbOF$_5$}]]_n$ (I) with the simulated pattern calculated from SCXRD data.

**Figure S2.** Comparison of the experimental PXRD pattern of as-synthesized $[\text{Cu(Me$_3$pz)$_3$}[\text{TaOF$_5$}]]_n$ (II) with the simulated pattern calculated from SCXRD data.
Figure S3. IR spectrum of [Cu(Me₃pz)₃{NbOF₅}]ₙ (I): 500 m; 564 vs; 604 s; 710 w; 742 m; 776 w; 918 vs; 1018 m; 1124 w; 1172 m; 1212 w; 1294 m; 1388 w; 1416 m; 1442 m; 1480 w; 1532 m; 1590 m; 1632 w; 2866 w; 2924 m; 3218 s; 3298 vs; 3326 vs.

Figure S4. IR spectrum of [Cu(Me₃pz)₃{TaOF₅}]ₙ (II): 500 w; 524 s; 546 vs; 560 vs; 612 vs; 708 m; 732 s; 768 m; 922 vs; 1018 m; 1124 w; 1172 m; 1214 w; 1292 m; 1388 w; 1414 m; 1442 m; 1482 w; 1532 m; 1590 m; 2866 w; 2924 m; 2986 w; 3114 w; 3242 s; 3320 vs; 3344 vs;
**Figure S5.** Thermo-PXRD pattern for \([\text{Cu(Me}_3\text{pz})_3\text{NbOF}_5]\)\(_n\) (I)

**Figure S6.** Thermo-PXRD pattern for \([\text{Cu(Me}_3\text{pz})_3\text{TaOF}_5]\)\(_n\) (II)
**Figure S7.** Thermoanalytical curves for [Cu(Me₃pz)₃{NbOF₅}]ₙ (I)

![Figure S7](image1)

**Figure S8.** Thermoanalytical curves for [Cu(Me₃pz)₃{TaOF₅}]ₙ (II)

![Figure S8](image2)