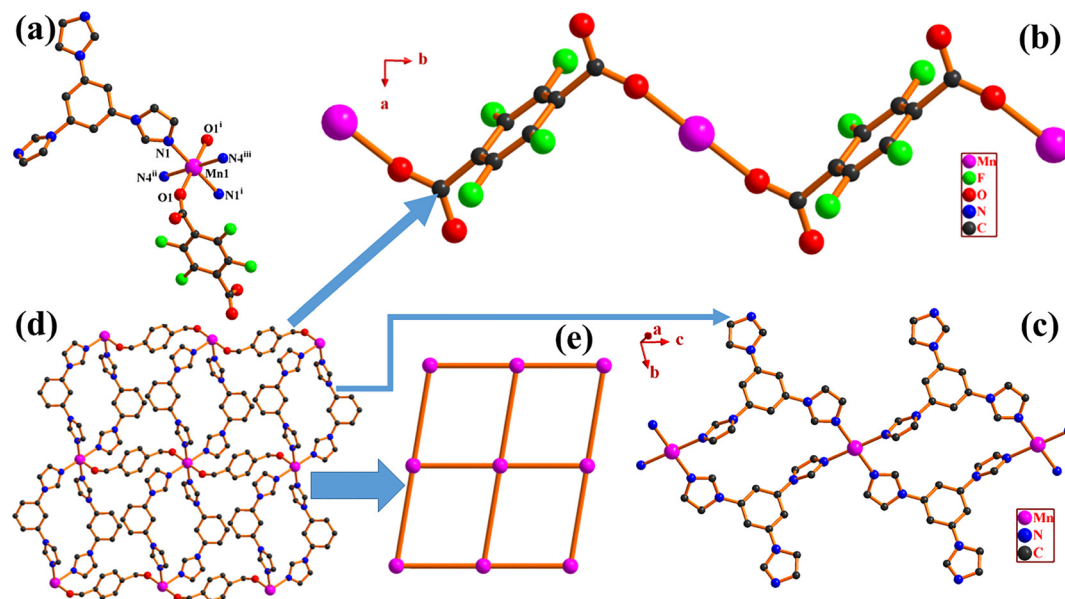


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# The crystal structure of poly(bis( $\mu_2$ -1,3,5-tri(1*H*-imidazol-1-yl)benzene- $\kappa^2N:N'$ )-( $\mu_2$ -2,3,5,6-tetrafluoroterephthalato- $\kappa^2O:O'$ )-manganese(II), $C_{38}H_{24}F_4N_{12}O_4Mn$ )



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## Abstract

$C_{38}H_{24}F_4N_{12}O_4Mn$ , triclinic,  $P\bar{1}$  (no. 2),  $a = 7.5571(5) \text{ \AA}$ ,  $b = 9.9288(5) \text{ \AA}$ ,  $c = 11.5708(8) \text{ \AA}$ ,  $\alpha = 79.816(5)^\circ$ ,  $\beta = 78.050(5)^\circ$ ,  $\gamma = 86.866(4)^\circ$ ,  $V = 835.85(9) \text{ \AA}^3$ ,  $Z = 1$ ,  $R_{gt}(F) = 0.0312$ ,  $wR_{ref}(F^2) = 0.0833$ ,  $T = 298 \text{ K}$ .

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Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

## 1 Source of materials

All chemicals were purchased from commercial sources and used as received. A mixture of  $Mn(NO_3)_2$  (1.0 mL 0.10 mol  $L^{-1}$   $Mn(NO_3)_2$  solution, 0.10 mmol), 2,3,5,6-tetrafluoroterephthalic acid (2,3,5,6-tetrafluoroterephthalic acid is abbreviated as  $H_2TFA$ ) (0.0238 g, 0.10 mmol), 1,3,5-tri(1*H*-imidazol-1-yl)benzene (1,3,5-tri(1*H*-imidazol-1-yl)benzene is abbreviated as TIB) (0.0276 g, 0.1 mmol), NaOH (1.0 mL 0.1 mol  $L^{-1}$  NaOH solution, 0.1 mmol), and  $H_2O$ /anhydrous ethanol (6.0 mL/3.0 mL) was added to a 25 mL Teflon-lined stainless steel reactor and heated at 393 K for

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**Table 1:** Data collection and handling.

|                                                                            |                                                               |
|----------------------------------------------------------------------------|---------------------------------------------------------------|
| Crystal:                                                                   | Colorless block                                               |
| Size:                                                                      | 0.30 × 0.25 × 0.15 mm                                         |
| Wavelength:                                                                | Mo K $\alpha$ radiation (0.71073 Å)                           |
| $\mu$ :                                                                    | 0.48 mm <sup>-1</sup>                                         |
| Diffractometer, scan mode:                                                 | Rigaku XtaLAB Mini (ROW), $\omega$ scans                      |
| $\theta_{\max}$ , completeness:                                            | 25.1°, 100 %                                                  |
| $N(hkl)_{\text{measured}}$ , $N(hkl)_{\text{unique}}$ , $R_{\text{int}}$ : | 4491, 2955, 0.017                                             |
| Criterion for $I_{\text{obs}}$ , $N(hkl)_{\text{gt}}$ :                    | $I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$ , 2,631           |
| $N(\text{param})_{\text{refined}}$ :                                       | 268                                                           |
| Programs:                                                                  | Rigaku, <sup>1</sup> Olex2, <sup>2</sup> SHELX <sup>3,4</sup> |

3 days. After cooling to room temperature at a rate of 283 K h<sup>-1</sup>, colorless block-shaped crystals of I were collected by filtration, washed with anhydrous ethanol and dried in air. Phase pure crystals were obtained by manual separation (yield: 50.62 mg, ca. 60 % based on 4-fluorobenzoic acid). Anal. Calc. for I: C<sub>38</sub>H<sub>24</sub>F<sub>4</sub>N<sub>12</sub>O<sub>4</sub>Mn (%) ( $M_r$  = 843.63): C, 54.05; H, 2.84; N, 19.91. Found: C, 54.06; H, 2.82; N, 19.93.

## 2 Experimental details

CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.<sup>1</sup> Using Olex2,<sup>2</sup> the structure was solved with the ShelXT<sup>3</sup> structure solution program and refined with the ShelXL<sup>4</sup> refinement package. Carbon-bound hydrogen atoms were placed in calculated positions ( $d$  = 0.93 Å for CH and were included in the refinement in the riding model approximation, with  $U_{\text{iso}}(\text{H})$  set to 1.2 $U_{\text{eq}}(\text{C})$  for –CH). The structure was examined using the ADDSYM subroutine of PLATON<sup>5</sup> to ensure that no additional symmetry could be applied to the models.

## 3 Comment

The ceramics are bulk materials formed by sintering randomly oriented inorganic non-metallic crystallites. They are typically opaque due to defects, pores, and intrinsic material birefringence.<sup>6</sup> The optical ceramics, a specialized class of transparent ceramics, eliminate light scattering and combine the advantages of other transparent bulk materials, such as single crystals and glass.<sup>7</sup> They are suitable for high-performance optical windows and laser gain media. However, the optical ceramics impose stringent requirements on raw materials or precursors: (1) high-purity, size-uniform nanocrystallites are essential to eliminate defects and pores; (2) crystallization in the cubic system is mandatory to suppress birefringence.<sup>8</sup>

Additionally, organic and inorganic-organic hybrid materials cannot withstand the high-temperature sintering required for ceramic preparation.<sup>9</sup> Consequently, only a limited number of materials are currently viable for optical ceramics. Coordination polymers, some of them also termed metal-organic frameworks (MOFs), represent a class of crystalline materials with diverse structures and functionalities.<sup>10</sup> While MOF thin films and single crystals show promise for separation, sensing, and optical devices, producing high-quality large-sized samples remains challenging.<sup>11</sup>

The optical ceramics are a type of transparent specialty ceramics that combine the high stability of single crystals with the large-scale advantages of glass, fluids, and other amorphous materials, making them promising laser gain media.<sup>12</sup> Due to the strict requirements for crystal size and symmetry, coupled with the need for high-temperature sintering processes, only a limited number of inorganic non-metallic materials can be used to produce optical ceramics.<sup>13</sup> Considering the structural and functional diversity of metal-organic frameworks (MOFs), metal-organic ceramics hold potential not only for optical devices but also in related fields such as adsorption, separation, and sensing.<sup>14,15</sup> Recently, the Henke research group at TU Dortmund University in Germany achieved the first melting-glassification-ceramic transformation of a cadmium(II)-based MOF glass.<sup>16</sup> Using mechanochemical synthesis, they produced submicron Cd(im)<sub>2</sub> particles with high-density surface defects, successfully lowering the melting point to 455°. This avoided the decomposition window, enabling stable melting and rapid quenching into glass. The study also revealed that under controlled heating conditions, the material could partially recrystallize, transforming into a monolithic glass-ceramic with nanocrystals uniformly embedded in a glass matrix. This structure exhibits enhanced mechanical properties and reversible thermal response characteristics. This research not only uncovers the distinctive thermodynamic phase transition behaviors of  $d$ -block transition metal MOF materials but also opens up new pathways for designing and functionally tailoring MOF glass-ceramics.

On the other hand, both 1,3,5-tris(1-imidazolyl)benzene (TIB)<sup>17</sup> and tetrafluoroterephthalate (H<sub>2</sub>TFA)<sup>18</sup> are rigid organic building blocks with triangular and linear geometries, respectively, which have been proved as versatile linkers to connect metal ions into higher dimensional structures through various coordination modes as well as secondary interactions such as hydrogen bond and halogen bond.<sup>19–21</sup> Although a search in the CSD (Cambridge Structure Database) survey with the help of ConQuest version 1.3 shows 185 and 96 hits based on the TIB and TFA<sup>2-</sup> ligands, respectively, the study of a combination of them in one CP has not been done yet. In order to further study mixed two kinds of ligands based CPs and investigate the influence of

reactant molar ratio on the coordination connectivity and related network, we designed the reactions of them with Mn(II) salt. In this work, we used Hfba and bib as organic ligands for Mn(II) centers. These combinations afforded a two-dimensional CP,  $[Mn(TFA)(TIB)_2]_n$  (I), which has been synthesized by hydrothermal methods and characterized by single-crystal X-ray diffraction (SCXRD).

The single-crystal X-ray diffraction experiment shows that I is a two-dimensional (2D) network coordination polymer and crystallizes in a triclinic crystal system with the  $P\bar{1}$  (no. 2) space group. The asymmetric unit of I consists of a half of the formula as  $\{[Mn(TFA)(TIB)_2]\}$ , i.e., a half of  $Mn^{2+}$  cation, a half of fully deprotonated carboxylate  $H_2TFA$  ligand ( $TFA^{2-}$ ), and a neutral N-containing auxiliary TIB ligand. Each central  $Mn^{2+}$  ion is octahedrally coordinated at the 1d site with a site occupancy factor of 0.50, and it is six-coordinated to two O atoms ( $O1$  and  $O1^i$ ) of two symmetry operation related  $TFA^{2-}$  ligands and four N-donors ( $N1$  and  $N1^i$ ) (symmetry code: i 2-x, 1-y, 2-z) from 4 symmetry operation related TIB ligands, respectively, leading to a perfect octahedral  $\{MnO_2N_4\}$  geometry configuration with the bond lengths of  $Mn1-O1 = Mn1-O1^i = 2.1772(13)$  Å and  $Mn1-N1 = Mn1-N1^i = 2.2926(16)$  Å,  $Mn1-N4^{ii} = Mn1-N4^{iii} = 2.2609(15)$  Å, and with the three axial bond angles of  $O1-Mn1-O1^i = N1-Mn1-N1^i = N4^{ii}-Mn1-N4^{iii} = 180.00^\circ$  (symmetry codes: i 1-x, 2-y, 2-z; ii 1-x, 2-y, 1-z; iii x, y, 1+z), and the bond angles related to the coordination bonds surrounding the central manganese atom are approximately all equal to  $90^\circ$ . The Mn–O bond distance is  $2.1772(13)$  Å ( $Mn1-O1 = Mn1-O1^i$ ) and the Mn–N bond lengths are  $2.2609(15)$  and  $2.2926(16)$  Å ( $Mn1-N1 = Mn1-N1^i$ ,  $Mn1-N4^{ii} = Mn1-N4^{iii}$ ), the N(O)–Mn–O(N) angles fall in the  $87.85(5)^\circ$ – $180.0^\circ$  range, all within the normal range. The detailed coordination modes of the  $TFA^{2-}$  and TIB ligands are shown in Figure. The fully deprotonated carboxylate of the  $TFA^{2-}$  anion coordinates with Mn(II) in a monodentate bridging mode and the TIB ligand features a triangular fork-shaped bridging mode ( $\mu_2$ ) with the dihedral angles among the three coordinated imidazole rings of  $N1/C5/N2/C7/C6$  (Cg1) and  $N3/C14/N4/C16/C15$  (Cg2) (Cg1/Cg2) of  $81.90^\circ$ ,  $N1/C5/N2/C7/C6$  (Cg1) and  $N5/C17/N6/C19/C18$  (Cg3) (Cg1/Cg3) of  $41.51^\circ$ ,  $N3/C14/N4/C16/C15$  (Cg2) and  $N5/C17/N6/C19/C18$  (Cg3) (Cg2/Cg3) of  $47.34^\circ$ , and between the central phenyl ring of  $C8/C9/C10/C11/C12$  (Cg4) and Cg1 (Cg1/Cg4), Cg2 (Cg2/Cg4), Cg3 (Cg3/Cg4) of  $40.33$ ,  $55.17$ ,  $14.32^\circ$ , respectively. While the dihedral angles of 3 imidazole rings of TIB with six-membered benzene ring of  $TFA^{2-}$  ( $C2/C3/C4^{iv}/C2^{iv}/C3^{iv}/C4$ , Cg5, symmetry code: iv 1-x, 3-y, 2-z) are  $13.81^\circ$  (Cg1/Cg5),  $87.04^\circ$  (Cg2/Cg5),  $44.71^\circ$  (Cg3/Cg5), and Cg4/Cg5 of  $47.86^\circ$ , respectively.

It is interesting to note that the Mn(II) cations are connected by  $TFA^{2-}$  dianions to construct a one-dimensional (1D)

$[Mn_2(TFA)]_n$  linear chain along  $b$  axis. The distance between Mn(II) and Mn(II) within the  $[Mn_2(TFA)]_n$  chain is  $9.92(9)$  Å. The  $[Mn_2(TFA)]_n$  chains are further connected by TIB ligands resulting in a two-dimensional (2D) network structure (Figure). The distance between Mn(II) and Mn(II) within the  $[Mn_2(TIB)]_n$  chain is  $11.51(7)$  Å. From a topological perspective, if each  $Mn^{2+}$  core serves as a 4-connected node and is linked by 2  $TFA^{2-}$  and 2 TIB ligands, and each  $TFA^{2-}$  and TIB chelates or  $\mu_2$  bridges two Mn(II) cations acting as a 2-connected linker. The 2D structure of I can be simplified as a 4-c unimodal  $sql$  topology with a Schläfli symbol  $\{4^4.6^2\}$  as analyzed with the TOPOS 4.0 program.<sup>22</sup>

In the crystal of I the analysis of potential hydrogen bonds and schemes with  $d(D\cdots A)$  ( $R(D) + R(A) + 0.50$ ,  $d(H\cdots A)$  ( $R(H) + R(A) - 0.12$  Å,  $D-H\cdots A$ )  $100.0^\circ$  are calculated by PLATON software. The calculation results indicate that there are no classic hydrogen bonds found except for  $C-H\cdots F$  and  $C-H\cdots O$  interactions. All potential hydrogen bond acceptors and donors participate in no classic hydrogen bond interactions. In detail, two weak  $C-H\cdots O$  no classic intermolecular hydrogen bonds are formed between  $C13$  and  $O2^v$  ( $C13-H13\cdots O2^v$ ,  $C13$  as donor,  $O2^v$  as acceptor; bond distances:  $C13-H13 = 0.93$ ,  $H13\cdots O2^v = 2.57$ , and  $C13\cdots O2^v = 3.477(2)$  Å; bond angle:  $C13-H13\cdots O2^v = 164^\circ$ , symmetry code: v x, y-1, z), and between  $C17$  and  $O2$  ( $C17-H17\cdots O2^v$ ,  $C17$  as donor,  $O2$  as acceptor; bond distances:  $C17-H17 = 0.93$ ,  $H17\cdots O2^v = 2.43$ , and  $C17\cdots O2^v = 3.358(3)$  Å; bond angle:  $C17-H17\cdots O2^v = 173^\circ$ ). More significantly, three halogen hydrogen bonds  $C-H\cdots F$  lies in I, i.e.,  $C11-H11\cdots F2^{ii}$  ( $C11$  as donor,  $F2^{ii}$  as acceptor; bond distances:  $C11-H11 = 0.93$ ,  $H11\cdots F2^{ii} = 2.48$ , and  $C11\cdots F2^{ii} = 3.240(2)$  Å; bond angle:  $C11-H11\cdots F2^{ii} = 139^\circ$ ),  $C14-H14\cdots F2^{ii}$  ( $C14$  as donor,  $F2^{ii}$  as acceptor; bond distances:  $C14-H14 = 0.93$ ,  $H14\cdots F2^{ii} = 2.48$ , and  $C14\cdots F2^{ii} = 3.120(2)$  Å; bond angle:  $C14-H14\cdots F2^{ii} = 126^\circ$ ),  $C18-H18\cdots F2^{ii}$  ( $C18$  as donor,  $F2^{ii}$  as acceptor; bond distances:  $C18-H18 = 0.93$ ,  $H18\cdots F2^{ii} = 2.48$ , and  $C18\cdots F2^{ii} = 3.368(2)$  Å; bond angle:  $C18-H18\cdots F2^{ii} = 159^\circ$ ), resulting in a supramolecular three-dimensional (3D)  $C-H\cdots F$  and  $C-H\cdots O$  hydrogen bonding network.

In addition, it obviously exist either offset face-to-face  $\pi\cdots\pi$  or edge-to-face  $C-X\cdots\pi$  ( $X = H, O$ ) stacking interactions calculated by PLATON program. The offset face-to-face  $\pi\cdots\pi$  interactions between imidazole rings (Cg1 and Cg3) and benzene rings (Cg4 and Cg5) may be separated into three groups, i.e. (1)  $Cg1\cdots Cg4^i$ ,  $Cg1\cdots Cg4^v$ ,  $Cg4\cdots Cg1^i$ , and  $Cg4\cdots Cg1^{vi}$ ; (2)  $Cg3\cdots Cg5^{vii}$  and  $Cg5\cdots Cg3^{vii}$ ; (3)  $Cg3\cdots Cg5^{viii}$  and  $Cg5\cdots Cg3^{viii}$  (symmetry codes: vi x, 1+y, z; vii 1-x, 1-y, 1-z; viii 2-x, 1-y, 1-z). Their  $Cg-Cg$ ,  $CgI-Perp$ , and the dihedral angles range from  $3.5815(12)$  to  $3.9663(12)$ ,  $3.1850(7)$  to  $3.8259(10)$  Å, and  $13.16(11)$  to  $14.39(11)^\circ$ , respectively. It is worth mentioning that the adjacent two-dimensional

sheets linked together dependent not only by C–H...O, C–H...F, and  $\pi$ ... $\pi$ , but also by two edge-to-face C–X... $\pi$  (X = H, O) stacking interactions, i.e. between the Cg1<sup>ix</sup> and C15–H15 (C15–H15...Cg1<sup>ix</sup>, symmetry code: ix 2–x, 2–y, 1–z), between the Cg2<sup>ix</sup> and C1–O2 (C1–HO2...Cg2<sup>ix</sup>). The C15–H15...Cg1<sup>ix</sup>, C1–O2...Cg2<sup>ix</sup> (Y–X...Cg) dihedral angle are 130 and 117.59(12)°, and with H15...Cg1<sup>ix</sup>, O2...Cg2<sup>ix</sup> (X...Cg) distances of 2.96 and 3.6020(18) Å, and C15...Cg1<sup>ix</sup>, C1...Cg2<sup>ix</sup> (Y...Cg) distance of 3.638(2) and 4.315(2) Å, respectively. However, their contribution to the overall lattice energy must be very small. Thus a supramolecular 3D network fragment is formed by C–H...X (X = O or F) and  $\pi$ ... $\pi$ , C–X... $\pi$  (X = H, O) interactions stabilizing the coordination polymer.

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