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Two zinc coordination polymers showing five-fold interpenetrated diamondoid network and 2D → 3D inclined polycatenation motif

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ABSTRACT

The reaction of $Zn(NO_3)_2$, 1,4-bis(1,2,4-triazol-4-yl)benzene (btx), 1,4-benzenedicarboxylicate (1,4-bdc) or fumarate (fum) gives two unusual coordination polymers $\{[Zn(btx)(1,4\text{-}bdc)]\ 3H_2O\}_n$ (**1**) and $[Zn(btx)_{0.5}(fum)(H_2O)]_n$ (**2**). **1** shows a 5-fold interpenetrated three-dimensional diamondoid network. **2** displays a 2D → 3D inclined polycatenation motif consisting of two sets of equivalent 2D (6,3) layers. The luminescence and thermal stability were investigated.

In recent years, more and more chemists were involved into rational design and synthesis of coordination frameworks because of their fascinating topology structures and attentional properties such as luminescence, catalysis and gas absorption [1–13]. In order to get such intriguing topologies and functional materials, the crucial step is to employ appropriate organic building blocks as well as metal ions. Auxiliary multicarboxylate ligands can influence the structure of the coordination polymers owing to the fact that they can satisfy charge-balance and even mediate the coordination of the metal centers, and more importantly they can provide diverse ligands and versatile coordination modes [14–18]. Flexible bidentate N-donor ligands such as bis(imidazole) [19,20] and 1-substituted bis(triazole) [21–25] ligands are widely used to construct coordination polymers because flexible ligands can adopt different conformations according to the geometric needs of the different metal ions. Meanwhile many coordination networks are particularly intriguing because of the presence of periodic entanglements, in which independent motifs are entangled together in different modes. Interpenetrating and polycatenation networks are two main subgroups of entanglements which need to break internal connections to separate the individual nets [26–34]. Two entanglements showing interesting 2D → 3D inclined polycatenation based on 2D (4,4) network are synthesized recently [31,32]. We synthesized two interesting entanglements $[2D\text{-}Mn(btb)_2(NCS)_2][1D\text{-}Mn(btb)_2(NCS)_2]$ and $[Cd_3(bbtz)_6(H_2O)_6](BF_4)_6 \cdot 1.75H_2O$ showing 2D (4,4) networks and 1D ribbons of rings polycatenated in a three-dimensional (3D) array [33,34].

In previous work, we synthesized a lot of coordination polymers using flexible 1-substituted bis(triazole) building blocks, such as 1,2-bis(1,2,4-triazol-1-yl)ethane (bte) [35,36], 1,3-bis(1,2,4-triazol-1-yl)propane (btp) [37], 1,4-bis(1,2,4-triazol-1-yl)butane (btb) [38] and 1,4-bis(1,2,4-triazol-1-ylmethyl)benzene (bbtz) [39]. In contrast to 1-substituted-1,2,4-triazole derivatives, the research of coordination polymers based on 4-substituted-1,2,4-triazole derivatives is only just the beginning [40,41]. We also achieved a series of fantastic frameworks based on flexible 4-substituted-1,2,4-triazole ligand 1,2-bis(1,2,4-triazol-4-yl)ethane (btre) (Scheme 1) [42–44], such as $\{[Cd(btre)Cl] \cdot OH\}_n$, $\{[Cd(btre)Cl][CdCl(dca)_2] \cdot 0.5H_2O\}_n$ and $\{[Zn(btre)_{0.5}(OH\text{-}bdc)(H_2O)_2] \cdot 1.5H_2O\}_n$.

The 4-substituted-1,2,4-triazole bridging ligand 1,4-bis(1,2,4-triazol-4-yl)-benzene (btx) (Scheme 1) should be a good rigid building block to construct coordination polymers with large channels and easily result to entanglements. In the present work, two entangled coordination polymers $\{[Zn(btx)(1,4\text{-}bdc)]^{\cdot}3H_2O\}_n$ (**1**) and $[Zn(btx)_{0.5}(fum)(H_2O)]_n$ (**2**) (1,4-bdc = 1,4-benzenedicarboxylicate, fum = fumarate) were synthesized. **1** shows a five-fold interpenetrate 3D diamond network. **2** displays a 2D → 3D inclined polycatenation framework consisting of two sets of equivalent 2D (6,3) layers.

The colorless block crystals of **1** and **2** were prepared by the reaction of $Zn(NO_3)_2$, btx and 1,4-bdc or fum [45]. Single-crystal X-ray analysis [46] revealed that **1** crystallizes in the triclinic system with $P\bar{1}$ space group. The Zn(II) atom is coordinated by two btx nitrogen atoms and three carboxylate oxygen atoms from two 1,4-bdc ligands in a distorted trigonal bipyramidal geometry (Fig. 1). There are two kinds of 1,4-bdc ligands. Two carboxylate groups (O1O2) of one kind of 1,4-bdc ligands act as bis-chelating mode. Two carboxylate groups (O3O4) of the

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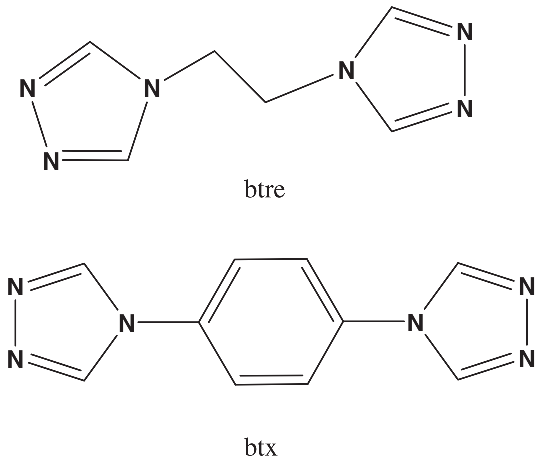

Scheme 1. The ligands btx and btre.

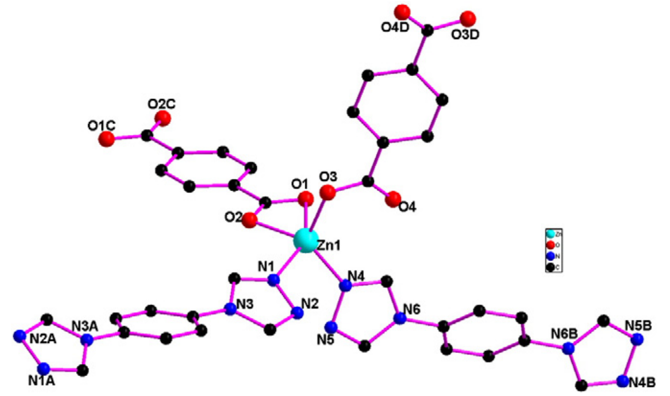

Fig. 1. The coordination environment of the Zn(II) atom in **1** (Symmetry codes: A −x, 2 − y, −z; B −1 − x, −y, 1 − z; C 1 − x, −y, −z; D 1 − x, 1 − y, 1 − z.).

other kind of 1,4-bdc ligands exhibit bis-monodentate mode. The btx ligands adopt bidentate mode. The Zn···Zn distances bridged by 1,4-bdc ligands are 10.916(2) and 11.097(2) Å. The Zn···Zn distances separated by btx ligands are 13.712(3) and 13.533(2) Å.

Each Zn(II) center connects four Zn(II) centers through two btx and two 1,4-bdc bridges in a tetrahedral mode (Fig. 2) and extends to construction of a three-dimensional network (Fig. 3). The topological analysis of **1** has been performed by considering both the metal ion and the ligand as topological nodes. The individual three-dimensional motif can be simplified as a four-connected 6^6 diamondoid topological net where

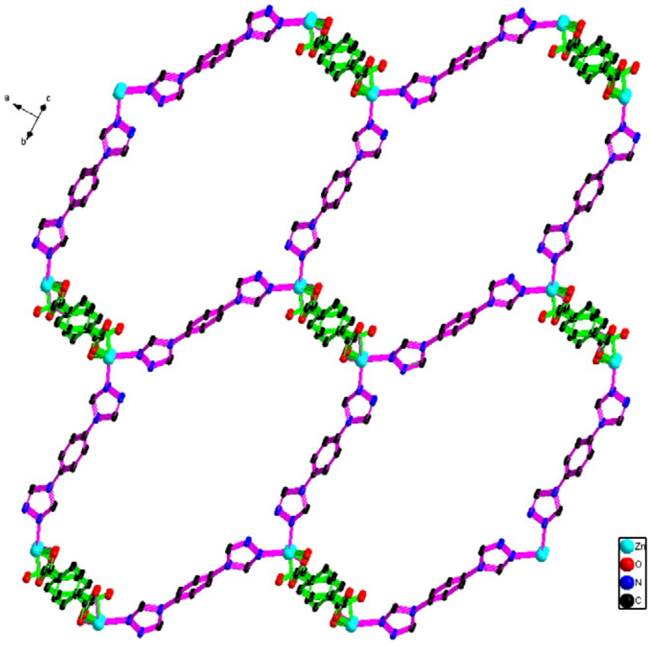

Fig. 3. A single diamondoid network of **1**.

all the shortest circuits are displayed as six-numbered circuits with chair form (Fig. 4) if the Zn(II) atoms are simplified as 4-connected nodes and the btx ligands and 1,4-bdc ligands as 2-connected linkers [47]. Because the single diamondoid network has large spacious voids, it allows adjacent frameworks to interpenetrate to give a 5-fold interpenetrated three-dimensional diamondoid network (Fig. S1). The degrees of the diamondoid net interpenetration ranging from 2- to 11-fold are well-known and have been reported [48], but the 5-fold interpenetrating MOFs in the presence of the mixed organic ligands with different lengths is relatively rare [49,50]. The lattice water molecules exist in the voids of the 5-fold interpenetrating diamondoid network of **1** (Fig. 5).

2 crystallizes in the monoclinic system with $P2_1/c$ space group. The Zn(II) atom is coordinated by one btx nitrogen atom and two fum oxygen atoms and one coordination water oxygen atom in a distorted tetrahedral geometry (Fig. 6). All fum ligands act as bidentate bridging ligands and link two Zn(II) atoms with the Zn···Zn distances of 8.959(2) and 9.334(2) Å. The btx ligands adopt bidentate mode and join two Zn(II) atoms with the Zn···Zn distance of 13.499(2) Å.

Each zinc(II) links three zinc(II) atoms through one btx and two fum ligands and extends to form a 2D (6,3) network (Fig. 7). The edges of the rectangular windows of the layers measure 16.487 × 13.499 Å, as

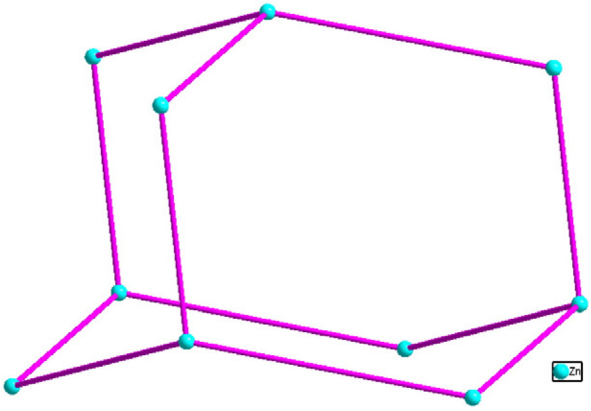

Fig. 2. Schematic plot of a single adamantanoid cage of **1**.

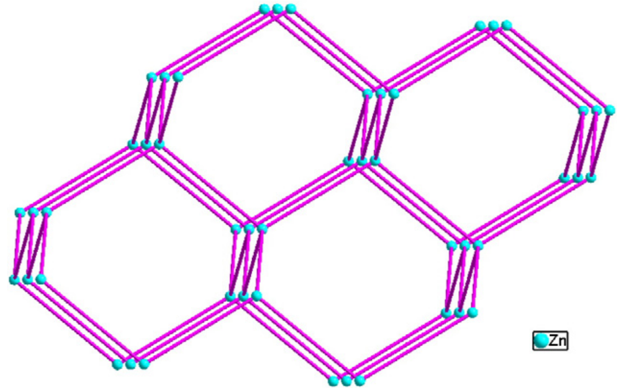

Fig. 4. Schematic depiction of a single diamondoid network of **1**.

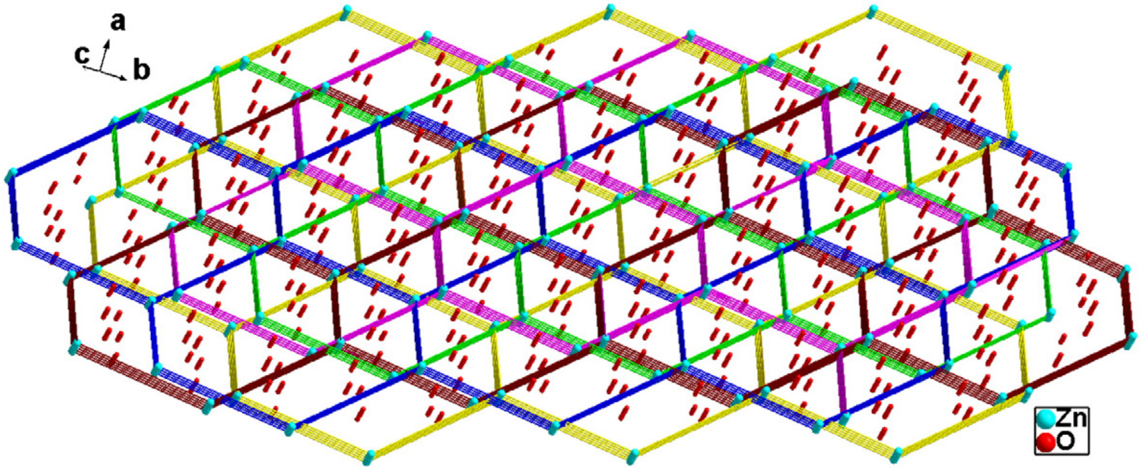

Fig. 5. Schematic depiction of a 5-fold interpenetrated diamondoid network in **1** (showing the lattice water molecules).

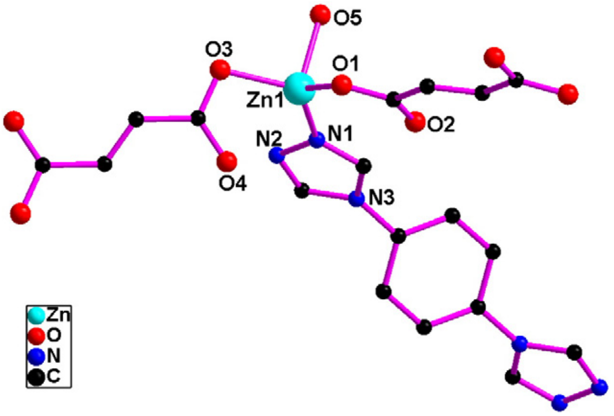

Fig. 6. The coordination environment of the Zn(II) atom in **2**.

defined by Zn···Zn distances; these layers are nearly rectangular in shape, with Zn···Zn···Zn angles of 77.34° and 102.66°.

The interesting feature of the structure is that two sets of layers parallel pack with the angle of 43.1° between two sets of parallel layers. In Fig. 8, a parallel layer set is shown in blue, while one layer of the other different parallel layer set is displayed in pink. Two sets of layers catenate to each other form a 2D + 2D → 3D parallel/parallel inclined polycatenation motif (Fig. 9). Although 2D (6,3) network is rather common, the 2D + 2D → 3D parallel/parallel inclined polycatenation is unusual [31,32]. There are no direct covalent interactions between catenated layers. The catenated motif is stable through the hydrogen bond interactions between the coordination water molecules and carboxylate oxygen atoms (O5···O2 (−x + 1, −y + 1, −z) 2.652(3) Å, O5···O4 (x, −y + 1/2, z + 1/2) 2.657(3) Å).

The measured and simulated PXRDs confirm the purity of **1** and **2** (Figs. S2, S3). Due to the excellent fluorescent properties of d^{10} metal complexes, the solid state photoluminescent properties of **1**, **2** and the free btx ligand were investigated at room temperature. The btx displays the emission band at 432 nm upon excitation at 370 nm, which can probably be assigned to the π–π^* transitions [51]. **1** exhibits the emission band maximum at 394 nm and a shoulder peak at 454 nm, upon excitation at 345 nm (Fig. 10). No emission of **2** was observed at the same experimental condition. Because the Zn(II) ion is difficult to oxidize or to reduce due to the d^{10} configuration, the emissions are neither metal-to-ligand charge transfer (MLCT) nor ligand-to-metal charge transfer (LMCT) [52]. The emissions at 394 and 454 nm can be tentatively attributed to the intraligand charge transition for 1,4-bdc and btx, respectively [51–53], because the emission at 394 nm for **1** is near to that of 1,4-bdc (388 nm) [49]. The emission at 454 nm for **1** shows the obvious red shift (22 nm) compare to free btx ligand.

The thermal stability of the coordination polymers **1** and **2** was examined by thermogravimetric analysis (TG) in dry nitrogen atmosphere from 20 to 800 °C. In the TG curve of **1** (Fig. S4), the lattice water molecules were lost from 45 to 180 °C (Calcd.: 10.90%, found: 10.76%). The anhydrous substance is stable upon heating to 335 °C. Then the weight decrease continuously happened and did not end until 600 °C. The coordination water molecules of **2** were lost from 50 to 170 °C (Calcd.: 5.94%, found: 5.96%). The anhydrous substance is stable upon heating to

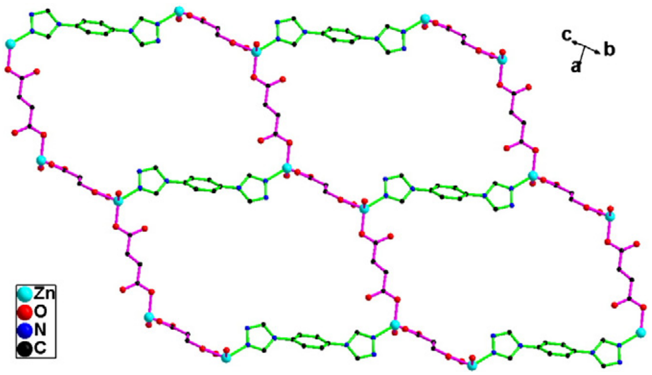

Fig. 7. A two-dimensional (6,3) network in **2**.

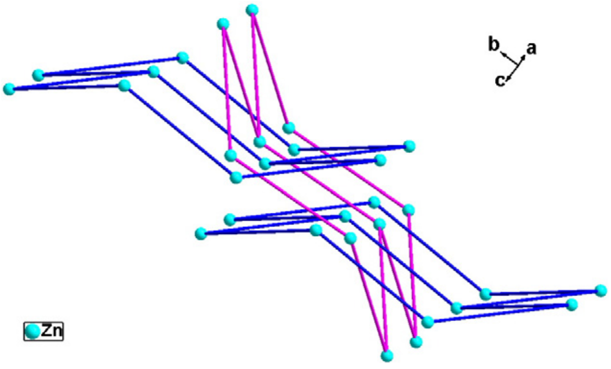

Fig. 8. Schematic perspective of parallel/parallel inclined polycatenation motif in **2**.

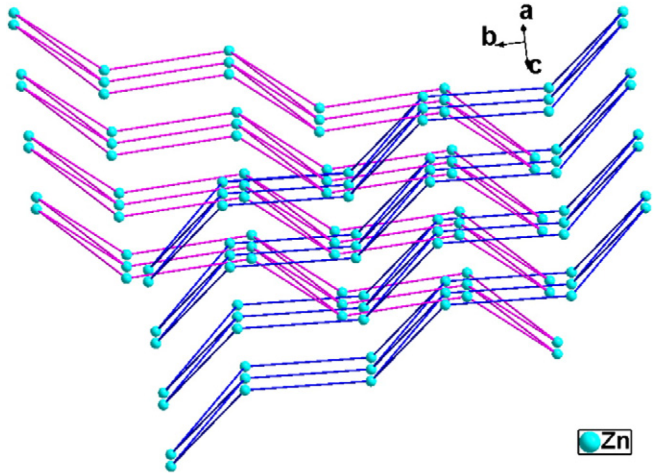

Fig. 9. Schematic depiction of 2D → 3D parallel/parallel inclined polycatenation in **2**.

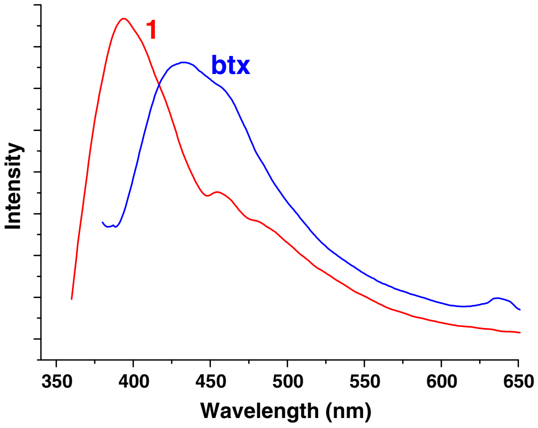

Fig. 10. The emission spectra of **1** and free btx ligand in the solid state at room temperature.

320 °C. Then the weight decrease continuously happened and did not end until 600 °C.

In summary, we successfully synthesized two unusual zinc(II) coordination polymers showing 5-fold interpenetrated three-dimensional diamondoid network and 2D → 3D inclined polycatenation framework consisting of two sets of equivalent 2D (6,3) layers. The result indicates that the combination of rigid 4-substituted bis(1,2,4-triazole) ligand btx and biscarboxylate auxiliary ligand can construct unusual coordination polymers and the structures can be adjusted by the different auxiliary biscarboxylate ligands. Further syntheses and structural studies of novel coordination polymer with rigid 4-substituted 1,2,4-triazole ligands and rigid multicarboxylate ligands are also under way in our laboratory.

Acknowledgments

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Appendix A. Supplementary material

Crystallographic data have been deposited at the Cambridge Crystallographic Data Center as supplementary publications (CCDC-974458, 979321) containing the supplementary crystallographic data. The data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.inoche.2014.02.051.

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[45] Synthesis of $[Zn(btx)(1,4\text{-bdc})]\cdot 3H_2O$ (**1**) and $[Zn(btx)_{0.5}(fum)(H_2O)]_n$ (**2**): A solution of 1,4-H_2bdc or H_2fum (0.2 mmol) in 10 mL of H_2O was adjusted to pH 6 with dilute NaOH solution and $Zn(NO_3)_2\cdot 6H_2O$ (0.2 mmol) was added with stirring. Then 5 mL DMF solution of btx (0.2 mmol) was added and continuously stirred for ten minutes. The mixture solution was filtered and stood for one month to give colorless single crystals of **1** and **2**. Anal. Calcd. for $C_{18}H_{18}N_6O_7Zn$ (**1**) C, 43.61; H, 3.66; N, 16.96. Found: C, 43.55%; H, 3.60%; N, 16.89%. Anal. Calcd. for $C_9H_8N_3O_5Zn$ (**2**) C, 35.61; H, 2.66; N, 13.85. Found: C, 35.54%; H, 2.60%; N, 13.78%.

[46] X-ray single crystal diffraction data collection for **1** and **2** were performed on a Rgaku Mercury CCD. The structure was solved by direct methods and refined ith full-matrix least-squares technique (SHELXTL-97). Crystal data for $C_{18}H_{18}N_6O_7Zn$ (**1**): Mr = 495.75, triclinic, $P\bar{1}$, $a = 7.6033(16)$ Å, $b = 9.352(2)$ Å, $c = 15.193(3)$ Å, $\alpha = 89.330(11)°$, $\beta = 80.570(9)°$, $\gamma = 85.897(11)°$, $V = 1063.0(4)$ Å^3, $Z = 2$, $Dc = 2.042$ g/cm^3, $\mu = 1.209$ mm^{-1}, $F(000) = 508$, S = 1.035, $R_1 = 0.0624$, wR_2 = 0.1565. Crystal data for $C_9H_8N_3O_5Zn$ (**2**) Mr = 303.55, monoclinic, $P2_1/c$, $a = 7.2580(16)$ Å, $b = 14.804(3)$ Å, $c = 10.706(2)$ Å, $\beta = 100.989(5)°$, $V = 1129.2(4)$ Å^3, $Z = 4$, $Dc = 1.786$ g/cm^3, $\mu = 2.193$ mm^{-1}, $F(000) = 612$, S = 1.016, $R_1 = 0.0312$, wR_2 = 0.

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