Lan Zhang*, Lu Wang, Wu-Xiu Ding, Qiang Wang and Meng-Juan Chen

Crystal structure of (OC-6-13)-aqua-tris(3-bromopyridine- κ^1N)-bis(trifluoroacetato- κ^1O) cadmium(II) $C_{19}H_{14}Br_3CdF_6N_3O_5$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size	0.15 × 0.11 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	5.41 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.4°, >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	28241, 5452, 0.042
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4422
$N(\text{param})_{\text{refined}}$:	372
Programs:	Bruker [1], SHELX [2], Diamond [3], Olex2 [4]

Source of material

All reagents were of analytical grade and used as purchased. A mixture of cadmium hydroxide (0.002 mol, 0.294 g), trifluoroacetic acid (HTFA) (0.004 mol, 0.459 g) and ethanol (30 mL) were added to a conical flask in turn. After refluxing for 1 h, 3-bromopyridine (3-BP) (0.006 mol, 0.950 g) was added and mixed with stirring. Continuous heating under reflux was performed for 4 h. The solution was cooled, filtered and recrystallized. Colorless block crystals of the title compound were obtained after 1 month.

Experimental details

The structure was solved by SHELXT with direct methods. Absorption corrections were applied by using multi-scan program [1]. One of the CF_3 groups was refined with a disorder model, and ISOR and RIGU restraints were applied to get a proper model (see the Figure). All the hydrogen on C were placed at calculated positions (0.95 Å) and refined with the riding model, with $U_{\text{iso}}(H)$ set to $1.2U_{\text{eq}}(C)$. The H atoms of the water molecule were located from the difference Fourier map.

Comment

With the increasing interest in the CF_3 group for the development of pharmaceuticals, agricultural chemicals

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Abstract

$C_{19}H_{14}Br_3CdF_6N_3O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 9.2591(4)$ Å, $b = 9.6573(5)$ Å, $c = 16.9256(9)$ Å, $\alpha = 102.707(2)^\circ$, $\beta = 96.854(2)^\circ$, $\gamma = 112.550(2)^\circ$, $V = 1328.20(12)$ Å³, $Z = 2$, $R_{\text{gt}}(F^2) = 0.0291$, $wR_{\text{ref}}(F^2) = 0.0625$, $T = 150.15$ K.

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Table 1 contains crystallographic data, and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

*Corresponding author: Lan Zhang, School of Environmental Engineering and Chemistry, Luoyang Institute of Science and Technology, Luoyang Henan 471023, People's Republic of China, E-mail: lanzhang1110@163.com. <https://orcid.org/0000-0003-4921-2680>

Lu Wang, Qiang Wang and Meng-Juan Chen, School of Environmental Engineering and Chemistry, Luoyang Institute of Science and Technology, Luoyang Henan 471023, People's Republic of China
Wu-Xiu Ding, School of Civil Engineering, Luoyang Institute of Science and Technology, Luoyang Henan 471023, People's Republic of China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
Cd1	0.30460 (3)	0.68572 (3)	0.69225 (2)	0.03136 (7)
Br1	0.68636 (5)	0.30490 (5)	0.64930 (3)	0.04763 (11)
Br2	−0.23747 (5)	0.60816 (5)	0.86902 (3)	0.05333 (12)
Br3	0.73100 (5)	0.97817 (5)	1.03919 (2)	0.05318 (12)
F1	0.3589 (3)	1.0221 (3)	0.49948 (16)	0.0584 (6)
F2	0.1073 (3)	0.9036 (3)	0.45142 (16)	0.0641 (7)
F3	0.2015 (4)	1.0589 (3)	0.57501 (16)	0.0669 (7)
F4 ^a	0.1415 (19)	0.3070 (16)	0.9060 (11)	0.098 (4)
F4A ^b	0.3769 (19)	0.500 (2)	0.9347 (11)	0.087 (5)
F5 ^a	0.3309 (13)	0.5375 (10)	0.9474 (6)	0.108 (4)
F5A ^b	0.170 (3)	0.290 (2)	0.9007 (17)	0.073 (6)
F6 ^a	0.3659 (8)	0.3350 (9)	0.8789 (5)	0.128 (3)
F6A ^b	0.1227 (15)	0.4828 (13)	0.9267 (6)	0.103 (4)
O1	0.3370 (3)	0.8566 (3)	0.61373 (15)	0.0365 (5)
O2	0.1032 (3)	0.6880 (3)	0.52694 (15)	0.0412 (6)
O3	0.3079 (3)	0.5588 (3)	0.79412 (16)	0.0433 (6)
O4	0.0998 (4)	0.3243 (4)	0.75271 (19)	0.0766 (11)
O5	0.1291 (3)	0.4374 (3)	0.61826 (16)	0.0445 (6)
H5A	0.042576	0.436784	0.588048	0.067*
H5B	0.088133	0.381815	0.652429	0.067*
N1	0.5051 (3)	0.6284 (3)	0.64113 (17)	0.0336 (6)
N2	0.1125 (3)	0.7632 (3)	0.74139 (18)	0.0349 (6)
N3	0.5056 (3)	0.9057 (3)	0.80080 (18)	0.0349 (6)
C1	0.2184 (4)	0.8155 (4)	0.5550 (2)	0.0328 (7)
C2	0.2200 (4)	0.9501 (4)	0.5195 (2)	0.0391 (8)
C3	0.2166 (5)	0.4317 (4)	0.8017 (2)	0.0423 (9)
C4 ^a	0.2698 (13)	0.4017 (13)	0.8843 (6)	0.060 (2)
C4A ^b	0.216 (3)	0.428 (2)	0.8936 (12)	0.050 (4)
C5	0.5326 (4)	0.5089 (4)	0.6548 (2)	0.0355 (8)
H5	0.470083	0.447623	0.685653	0.043*
C6	0.6497 (4)	0.4726 (4)	0.6253 (2)	0.0351 (8)
C7	0.7413 (4)	0.5573 (4)	0.5788 (2)	0.0392 (8)
H7	0.822145	0.532772	0.558012	0.047*
C8	0.7096 (4)	0.6787 (4)	0.5642 (2)	0.0420 (9)
H8	0.768148	0.739453	0.532009	0.050*
C9	0.5928 (4)	0.7117 (4)	0.5963 (2)	0.0404 (8)
H9	0.573665	0.796870	0.586278	0.048*
C10	0.0088 (4)	0.6794 (4)	0.7796 (2)	0.0370 (8)
H10	0.009941	0.584030	0.785110	0.044*
C11	−0.1001 (4)	0.7275 (4)	0.8114 (2)	0.0366 (8)
C12	−0.1071 (4)	0.8636 (4)	0.8021 (2)	0.0407 (8)
H12	−0.183900	0.896236	0.822082	0.049*
C13	−0.0001 (5)	0.9500 (5)	0.7631 (2)	0.0441 (9)
H13	−0.000674	1.044653	0.756010	0.053*
C14	0.1086 (4)	0.8977 (4)	0.7343 (2)	0.0388 (8)
H14	0.183750	0.959353	0.708305	0.047*
C15	0.5502 (4)	0.8920 (4)	0.8760 (2)	0.0346 (7)
H15	0.495706	0.795353	0.887326	0.041*
C16	0.6722 (4)	1.0132 (4)	0.9374 (2)	0.0390 (8)
C17	0.7517 (5)	1.1547 (5)	0.9230 (3)	0.0552 (11)
H17	0.835496	1.239830	0.964828	0.066*
C18	0.7058 (6)	1.1688 (5)	0.8462 (3)	0.0683 (14)
H18	0.758856	1.264411	0.833617	0.082*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C19	0.5821 (5)	1.0433 (5)	0.7870 (3)	0.0543 (11)
H19	0.550341	1.055610	0.734467	0.065*

^aOccupancy: 0.655(8). ^bOccupancy: 0.345(8).

and functional materials, fluorine-containing compounds have received significant attention [5–8]. Especially the incorporation of CF_3 -containing groups has been an efficient strategy for the design of new drugs and agrochemical [9]. 3-Bromopyridine (C_5H_4BrN , 3-BP) is an important pyridine derivative which exhibits biological and pharmacological performance. In some studies, it has been shown that derivatives and metal complexes of 3-BP show antidepressant, antibacterial and catalytic properties [10, 11].

The asymmetric unit of the title compound $[Cd(C_2F_3O_2)_2(C_5H_4Br)_3(H_2O)]$ consists of a mononuclear Cd(II) complex (see the Figure). The geometry about central Cd(II) is a slightly distorted octahedron, the ligating atoms being two O atoms from two *trans*-coordinated TFA anions (O1 and O3), one O atom from a H_2O molecule (O5), and two N atoms from two 3-BP ligands (N1 and N2). The axial positions are occupied by O5 and N3 atoms. The four equatorial O1, O3, N1 and N2 atoms are not coplanar, and the coordination plane comprising O1, N1 and N2 makes a dihedral angle of $9.47(9)^\circ$ with the plane formed by atoms O3, N1 and N2. The *trans* angles of O1–Cd1–O3 and N1–Cd1–N2 are $168.04(9)^\circ$ and $175.30(10)^\circ$, respectively, which are slightly deviating from linearity. The *cis* angles of N–Cd–N are $87.61(10)^\circ$ and $90.13(10)^\circ$, while the O–Cd–O bond angles are $80.14(9)^\circ$ and $111.81(9)^\circ$. The Cd–N bond lengths are in the range of 2.337(3) to 2.406(3) Å that are in agreement with that found in $[Cd(CH_3COO)_2(C_6H_6N_2O)_2]$ [12]. The bond lengths of Cd–O fall in the range of 2.272(2) to 2.329(2) Å, which are comparable with the values for the similar complex $[Cd(CH_3COO)_2(C_6H_7N_3O)_2] \cdot 2C_2H_5OH$ [13]. The oxygen of the water molecule in the structure of the title compound participates in the formation of intermolecular hydrogen bond (O5–H5A...O2¹: 2.750(3) Å and 135.6° : $-x$, $-y+1$, $-z+1$) and intramolecular hydrogen bond (O5–H5A...O4: 2.731(4) Å and 153.4°) with TFA anions.

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