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Crystal structure of bis(4-bromo-2-(((3-bromopropyl)imino)methyl)phenolato- $\kappa^2\text{N,O}$)-oxido-vanadium(IV), $\text{C}_{20}\text{H}_{20}\text{Br}_4\text{N}_2\text{O}_3\text{V}$

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.21 × 0.20 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	7.21 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ϕ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13114, 4152, 0.062
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2792
$N(\text{param})_{\text{refined}}$:	281
Programs:	CrysAlis ^{PRO} [1], SHELX [2], WinGX/ORTEP [3], PLATON [4]

Abstract

$\text{C}_{20}\text{H}_{20}\text{Br}_4\text{N}_2\text{O}_3\text{V}$, monoclinic, $P2_1/c$ (no. 14), $a = 12.6011(10)$ Å, $b = 16.5997(8)$ Å, $c = 12.9206(12)$ Å, $\beta = 119.006(11)^\circ$, $V = 2363.7(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0518$, $wR_{\text{ref}}(F^2) = 0.1062$, $T = 153(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

In a 250 ml round bottom flask containing 50 ml of absolute methanol were added 5 mmol of 3-bromopropyl-ammonium hydrochloride and 5 mmol of 5-bromo-2-hydroxybenzaldehyde. Then 5 mmol of NaOH was dissolved in 2 ml H₂O, were added to the above solution and the contents were refluxed for 3 h at 343 K. After cooling to room temperature, 12 ml methanol containing 2.5 mmol of vanadyl acetylacetonate were added. To this solution was added 30 ml trichloromethane and filtered off. The contents of the flask were refluxed with stirring for 3 h at 343 K.

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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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.83724 (8)	1.03500 (4)	0.19060 (8)	0.0663 (3)
Br2	0.09345 (6)	0.31912 (4)	−0.02620 (7)	0.0533 (2)
Br3A ^a	0.1145 (5)	0.8078 (6)	0.3151 (7)	0.0748 (18)
Br3B ^b	0.1334 (10)	0.7774 (9)	0.3256 (6)	0.091 (2)
Br4	1.01107 (6)	0.62854 (4)	0.46765 (6)	0.0455 (2)
C1	0.7401 (5)	0.7283 (3)	0.2097 (5)	0.0310 (14)
H1	0.817775	0.721207	0.219818	0.037*
C2	0.6922 (5)	0.8086 (3)	0.1860 (5)	0.0276 (14)
C3	0.7709 (6)	0.8716 (4)	0.1938 (5)	0.0392 (16)
H3	0.850941	0.860512	0.214053	0.047*
C4	0.7290 (6)	0.9491 (4)	0.1717 (5)	0.0407 (17)
C5	0.6100 (7)	0.9674 (4)	0.1380 (5)	0.0432 (18)
H5	0.582808	1.020436	0.122137	0.052*
C6	0.5327 (6)	0.9069 (4)	0.1283 (5)	0.0407 (16)
H6	0.451814	0.918907	0.103447	0.049*
C7	0.5724 (6)	0.8271 (3)	0.1550 (5)	0.0354 (15)
C8	0.7507 (5)	0.5875 (3)	0.2356 (5)	0.0302 (14)
H8A	0.825148	0.596940	0.232740	0.036*
H8B	0.700683	0.551187	0.171465	0.036*
C9	0.7807 (5)	0.5483 (3)	0.3516 (5)	0.0357 (16)
H9A	0.705398	0.535403	0.351216	0.043*
H9B	0.822318	0.497891	0.357560	0.043*
C10	0.8575 (5)	0.5977 (4)	0.4590 (5)	0.0383 (16)
H10A	0.873834	0.567082	0.529135	0.046*
H10B	0.813373	0.645852	0.457699	0.046*
C11	0.2554 (5)	0.6137 (3)	0.1052 (5)	0.0307 (14)
H11	0.174267	0.624504	0.081002	0.037*
C12	0.2836 (5)	0.5337 (3)	0.0807 (5)	0.0281 (14)
C13	0.1914 (5)	0.4760 (3)	0.0390 (5)	0.0316 (14)
H13	0.112924	0.490402	0.020702	0.038*
C14	0.2179 (5)	0.3982 (4)	0.0254 (5)	0.0341 (15)
C15	0.3314 (5)	0.3756 (3)	0.0489 (5)	0.0310 (14)
H15	0.347331	0.321934	0.040746	0.037*
C16	0.4230 (5)	0.4323 (3)	0.0848 (5)	0.0338 (15)
H16	0.499274	0.417173	0.097088	0.041*
C17	0.4013 (5)	0.5122 (3)	0.1028 (5)	0.0301 (14)
C18	0.2788 (6)	0.7455 (4)	0.1776 (7)	0.055 (2)
H18A	0.191134	0.741046	0.139079	0.066*
H18B	0.299095	0.791159	0.143735	0.066*
C19	0.3315 (6)	0.7597 (5)	0.3171 (8)	0.084 (3)
H19A	0.319009	0.711308	0.351806	0.101*
H19B	0.418160	0.769198	0.353641	0.101*
C20	0.2766 (7)	0.8253 (5)	0.3423 (8)	0.086 (3)
H20A	0.275403	0.870800	0.294665	0.104*
H20B	0.327181	0.840010	0.424635	0.104*
N1	0.3306 (4)	0.6709 (3)	0.1568 (4)	0.0323 (12)
N2	0.6856 (4)	0.6650 (3)	0.2184 (4)	0.0260 (11)
O1	0.4910 (3)	0.5647 (2)	0.1400 (3)	0.0335 (10)
O2	0.4935 (3)	0.7703 (2)	0.1468 (3)	0.0372 (11)
O3	0.5665 (4)	0.6648 (2)	0.3545 (3)	0.0445 (11)
V1	0.51793 (9)	0.66669 (6)	0.21604 (9)	0.0291 (3)

^aOccupancy: 0.48 (2), ^bOccupancy: 0.52 (2).

The green crystals that formed were filtered off after five days at room temperature.

Experimental details

Hydrogen atoms are positioned using the riding models of the SHELX system. Their U_{iso} values were set to $1.2U_{\text{eq}}$ of the parent atoms. One of Br atom is disordered over two positions in a 0.52:0.48 ratio.

Comment

Schiff base ligands play a central role in the coordination chemistry [5]. They have a variety of applications due to its structural features, including catalytic applications, biological roles, glycogen metabolism, as well as insulin mimicking and organic synthesis [6–10]. In our previous publications, we studied the Schiff base complexes, which exhibit insulin mimicking, and catalytic activity in the epoxidation reaction [11, 12]. Halogenated organic compounds gives suitable properties for different uses such as synthetic intermediates and starting compounds, designer molecules for material science, industrial chemicals, and bioactive compounds [13]. In order to expand its application to Schiff base, we present in this paper the synthesis of a halogenated schiff base and the crystal structures of the title compound.

The molecular structure of the title compound with the atom-numbering scheme is illustrated in the figure. The crystallographic data showed that the vanadium (IV) ion was surrounded by two oxygen and two nitrogen atoms from two Schiff base ligands and one oxygen atom from an oxido ligand in the five-coordinated geometry. The geometry around the vanadium(IV) ion is a distorted square pyramidal. The V–O and V–N bond distances of the Schiff base ligand in the equatorial plane (defined by the two organic ligands), V1–O1 = 1.910(5) Å, V1–O2 = 1.898(6) Å, V1–N1 = 2.105(7) Å, V1–N2 = 2.101(7) Å, compare well with the V–O and V–N distances found in similar complexes [14, 15]. The axial V1–O3 = 1.584(6) Å is in good agreement with the V–O distances in related complexes [14, 15]. The O(3)–V(1)–N(2) angle is 85.7(2)°, while O(3)–V(1)–O(2), O(2)–V(1)–N(2), N(2)–V(1)–N(1) angles are 115.8(3), 87.1(2) and 162.2(3)°, respectively.

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