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Crystal structure of *catena*-poly[(5*H*-pyrrolo[3,2-*b*:4,5-*b'*]dipyridine- κ_2N,N')-(μ^4 -hexaoxidodivanadato)dizinc(II)], $C_{10}H_9N_3O_6V_2Zn$

Table 1: Data collection and handling.

Crystal:	White block
Size:	0.23 × 0.21 × 0.19 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.95 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	28.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8830, 3424, 0.052
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2282
$N(\text{param})_{\text{refined}}$:	200
Programs:	Bruker [1], SHELX [2]

Source of material

To a solution of vanadiumoxy acetylacetonate (1.5 mmol) in H₂O (10 mL) was successively added 2,2'-dipyridine amine (1 mmol) and zinc chloride (0.5 mmol), and then maintained for 4 h at room temperature with consistent stirring, and then heated for four days at 160 °C. After the reaction was completed, the filtrate was left to slowly evaporate at room temperature. The orange-green crystals were obtained. Yield: 23.2%. Anal. Calcd. for $C_{10}H_9N_3O_6V_2Zn$: C, 27.65; H, 2.09; N, 9.67; found: C, 27.56; H 2.14; N 9.78.

Experimental details

All hydrogen atomic positions were taken from a difference Fourier map. Hydrogen atoms were assigned with isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C and N})$. All the H atoms were refined as riding on their parent atom.

Comment

Polyoxovanadates (POVs) belong to the large class of vanadium-oxide clusters with reversible multi-electron redox transformations properties under mild conditions, which allow them to act as multi-functional catalysts in homogeneous or heterogeneous catalytic systems [3–6]. The organic-inorganic hybrid POVs cluster can endow them

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Abstract

$C_{10}H_9N_3O_6V_2Zn$, monoclinic, $C2/c$ (no. 15), $a = 8.0106(17)$ Å, $b = 29.957(6)$ Å, $c = 12.050(3)$ Å, $\beta = 95.025(4)^\circ$, $V = 2880.6(10)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0512$, $wR_{\text{ref}}(F^2) = 0.1975$, $T = 296(2)$ K.

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A part of the coordination polymer 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.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	0.1988 (9)	0.4502 (2)	0.3684 (6)	0.0461 (16)
H1	0.166675	0.452583	0.440473	0.055*
C2	0.2184 (10)	0.4881 (2)	0.3096 (7)	0.0539 (19)
H2	0.199601	0.515954	0.340344	0.065*
C3	0.2672 (10)	0.4844 (2)	0.2026 (7)	0.0514 (18)
H3	0.283273	0.509899	0.160769	0.062*
C4	0.2919 (10)	0.4428 (2)	0.1584 (6)	0.0484 (17)
H4	0.322859	0.439859	0.086116	0.058*
C5	0.2700 (8)	0.40536 (19)	0.2227 (5)	0.0325 (13)
C6	0.2759 (9)	0.3203 (2)	0.2089 (5)	0.0380 (15)
C7	0.3209 (9)	0.2870 (2)	0.1342 (5)	0.0414 (15)
H7	0.361140	0.294659	0.066710	0.050*
C8	0.3040 (10)	0.2428 (2)	0.1632 (6)	0.0505 (18)
H8	0.335352	0.220193	0.116422	0.061*
C9	0.2387 (10)	0.2325 (2)	0.2644 (6)	0.0474 (17)
H9	0.220103	0.203135	0.285055	0.057*
C10	0.2044 (9)	0.2669 (2)	0.3300 (5)	0.0409 (15)
H10	0.165389	0.260178	0.398522	0.049*
N1	0.2245 (6)	0.40826 (16)	0.3260 (4)	0.0327 (11)
N2	0.2216 (6)	0.31022 (17)	0.3049 (4)	0.0316 (11)
N3	0.2993 (8)	0.36405 (17)	0.1734 (4)	0.0462 (15)
H3A	0.339045	0.366013	0.109544	0.055*
O1	0.4147 (7)	0.34645 (18)	0.5235 (4)	0.0611 (16)
O2	0.7334 (7)	0.3766 (3)	0.5800 (5)	0.085 (2)
O3	0.4566 (8)	0.43049 (18)	0.5876 (5)	0.0686 (16)
O4	0.500000	0.3646 (3)	0.750000	0.0564 (19)
O5	0.0329 (6)	0.34579 (15)	0.5204 (4)	0.0446 (11)
O6	0.0230 (8)	0.42726 (18)	0.6023 (4)	0.0651 (16)
O7	0.000000	0.3567 (2)	0.750000	0.0457 (17)
V1	0.52194 (12)	0.38141 (4)	0.61144 (7)	0.0310 (3)
V2	−0.04763 (12)	0.37835 (3)	0.61285 (8)	0.0291 (3)
Zn1	0.21188 (9)	0.35568 (2)	0.42672 (5)	0.0328 (2)

more properties and thus exploration of new functionalized organic-inorganic hybrid POVs is an interesting aspect in chemistry [7, 8]. Therefore, lots of studies on transition-metal functionalized POVs structures have been widely reported [9]. In addition, as a class of versatile tridentate N-ligands 2,2'-dipyridine amine (dpa) can be used not only as a base but also as an interesting ligand, and numerous of dpa-based complexes have been reported [10]. However, the examples of dpa-based zinc-polyoxovanadates are still rare.

Single crystal diffraction analysis of the title complex showed that the asymmetric structural unit consists of one neutral bidentate dpa, a zinc cation and a formal [V₂O₆]_∞ unit. The zinc center is coordinated by two nitrogen atoms of dpa and two oxygen atoms from the POV and formed the distorted T_d coordination, which was similar with those of reported zinc compounds [11]. The V–O bond distances of VO₄ unit are in the range of 1.579(5) and 1.785(3) Å, which

is similar with those of reported vanadates complexes [12–14].

Additionally, Zn–O and V–O bonds between the basic units form the infinite chain organic-inorganic hybrid polymer illustrated in the Figure.

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