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The crystal structure of tris(1-ethylimidazole- κ^1N)-(sulfato- κ^2O,O')vanadium(IV), $C_{15}H_{24}N_6O_5SV$

Table 1: Data collection and handling.

Crystal:	Blue plate
Size:	$0.19 \times 0.16 \times 0.07$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.63 mm^{-1}
Diffractometer, scan mode:	CCD, φ and ω
θ_{max} , completeness:	25.3° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11434, 3692, 0.063
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2699
$N(\text{param})_{\text{refined}}$:	256
Programs:	Bruker [1], SHELX [2], Olex2 [3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.64695(19)	0.3047(3)	0.3291(2)	0.0198(7)
H1	0.688429	0.275393	0.292903	0.024*
C2	0.5264(2)	0.3790(3)	0.3655(2)	0.0257(7)
H2	0.469309	0.410915	0.361046	0.031*
C3	0.58492(19)	0.3600(3)	0.4466(2)	0.0235(7)
H3	0.575105	0.376268	0.509541	0.028*
C4	0.5290(2)	0.3381(4)	0.1892(2)	0.0293(8)
H4A	0.484774	0.260872	0.178396	0.035*
H4B	0.573489	0.311682	0.152089	0.035*
C5	0.4900(2)	0.4836(4)	0.1540(2)	0.0452(10)
H5A	0.444471	0.508290	0.188907	0.068*
H5B	0.466638	0.476425	0.085822	0.068*
H5C	0.533645	0.560311	0.164514	0.068*
C6	0.74230(19)	−0.0441(3)	0.4228(2)	0.0243(7)
H6	0.759494	−0.012938	0.365597	0.029*
C7	0.6967(2)	−0.1823(4)	0.5275(2)	0.0333(8)
H7	0.676364	−0.264023	0.558284	0.040*
C8	0.7129(2)	−0.0469(4)	0.5641(2)	0.0283(8)
H8	0.705379	−0.016325	0.625675	0.034*
C9	0.7073(2)	−0.3020(4)	0.3686(3)	0.0440(10)
H9A	0.678455	−0.385569	0.393776	0.053*
H9B	0.671101	−0.269782	0.308367	0.053*
C10	0.7896(3)	−0.3522(4)	0.3482(3)	0.0508(11)
H10A	0.817870	−0.270703	0.321685	0.076*
H10B	0.780307	−0.433001	0.302055	0.076*
H10C	0.825276	−0.386443	0.407219	0.076*
C11	0.8997(2)	0.1735(4)	0.7014(2)	0.0262(7)
H11	0.859245	0.198646	0.740012	0.031*
C12	1.0167(2)	0.0960(4)	0.6582(2)	0.0309(8)
H12	1.072075	0.057659	0.659553	0.037*
C13	0.9616(2)	0.1415(4)	0.5806(2)	0.0286(8)
H13	0.972064	0.140214	0.517154	0.034*
C14	1.0140(2)	0.0929(5)	0.8359(2)	0.0440(10)
H14A	1.046147	−0.000599	0.843043	0.053*

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Abstract

$C_{15}H_{24}N_6O_5SV$, monoclinic, $P2_1/c$ (no. 14), $a = 16.0059 \text{ \AA}$, $b = 9.1008 \text{ \AA}$, $c = 14.2686 \text{ \AA}$, $\beta = 100.352^\circ$, $V = 2044.6(2) \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.0400$, $wR_{\text{ref}}(F^2) = 0.0852$, $T = 173(2) \text{ K}$.

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The complex title 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

$\text{VOSO}_4 \cdot 2 \text{ H}_2\text{O}$ (0.199 g, 1 mmol) in 1-ethylimidazole (6 mL) was sealed in a 20 mL vial, which was heated to 90° under autogenously pressure for 72 h. Yield: 60% based on V. Anal.

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H14B	0.967887	0.085482	0.873534	0.053*
C15	1.0721(3)	0.2170(5)	0.8733(3)	0.0596(13)
H15A	1.118376	0.223056	0.836861	0.089*
H15B	1.095709	0.199754	0.940610	0.089*
H15C	1.040119	0.309428	0.866771	0.089*
N1	0.66062(15)	0.3136(3)	0.42325(16)	0.0175(5)
N2	0.56687(16)	0.3426(3)	0.29145(17)	0.0216(6)
N3	0.74200(15)	0.0407(3)	0.49793(17)	0.0206(6)
N4	0.71516(18)	−0.1797(3)	0.43794(19)	0.0285(7)
N5	0.88789(16)	0.1901(3)	0.60755(17)	0.0211(6)
N6	0.97685(17)	0.1161(3)	0.73432(18)	0.0293(7)
O1	0.83011(13)	0.2731(2)	0.42317(14)	0.0237(5)
O2	0.78286(13)	0.4676(2)	0.56484(13)	0.0204(5)
O3	0.71898(12)	0.2765(2)	0.63494(13)	0.0191(5)
O4	0.80374(13)	0.4561(2)	0.73786(14)	0.0274(5)
O5	0.66665(13)	0.5260(2)	0.64964(15)	0.0271(5)
S1	0.74209(5)	0.43673(8)	0.65095(5)	0.01873(18)
V1	0.77923(3)	0.26022(6)	0.50944(3)	0.01657(13)

Calcd. for C₁₅H₂₄N₆O₅SV (%): C, 39.91; H, 5.36; N, 18.62. Found: C, 39.78; H, 5.51; N, 18.49.

Experimental details

The structure was solved by direct methods with the SHELXS program. All H-atoms were positioned with idealized geometry and refined isotropic ($U_{iso}(H) = 1.2U_{eq}(C)$), using a riding model with C(imodazole)—H = 0.950 Å, C(methylene)—H = 0.99 Å and C(methyl)—H = 0.98 Å, respectively.

Comment

The range of potential applications of vanadium complexes range from catalysis to bioactivity [4–10]. The design and synthesis of vanadium complexes will be very meaningful. In this text, we describe the synthesis of an interesting vanadium complex by a simple solvothermal reaction.

The central vanadium atom adopts a '4 + 1 + 1' geometry formed by three N atoms from 1-eIM, two bridging oxygen O_b from sulfate and one terminal oxygen O_t with the V—O and V—N bonds within the range of 1.597–2.186 Å and 2.082–2.125 Å, respectively [11]. The sulfate ligand is coordinated in a chelating bidentate mode with the S—O bonds within 1.448–1.518 Å. Although the central vanadium possesses distorted octahedral coordination geometry which is same to the previous reports in the similar system [12]. But in the previous reports, the four coordinated equatorial atoms are N atoms from 1-eIM. It is noteworthy that the 1-eIM plays a dual function as solvent and ligands.

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References

1. Bruker. SAINTv8.37A. Bruker AXS Inc, Madison, WI, USA (2015).
2. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
4. Barthelet, K.; Riou, D.; Ferey, G.: [V^{III}(H₂O)]₃O(O₂CC₆H₄CO₂)₃·(Cl, 9H₂O) (MIL-59): a rare example of vanadocarboxylate with a magnetically frustrated three-dimensional hybrid framework. *Chem. Commun.* **14** (2002) 1492–1493.
5. Laye, R. H.; Wei, Q.; Mason, P. V.; Shanmugam, M.; Teat, S. J.; Brechin, E. K.; Collison, D.; McInnes, E. J. L.: A highly reduced vanadium(III/IV) polyoxovanadate comprising an octavanadyl square-prism surrounding a dimetallic vanadium(III) fragment. *J. Am. Chem. Soc.* **128** (2006) 9020–9021.
6. Hu, X. K.; Wang, H.; Huang, B.; Li, N.; Hu, K. H.; Wu, B. L.; Xiao, Z. C.; Wei, Y. H.; Wu, P. F.: A new scheme for rational design and synthesis of polyoxovanadate hybrids with high antitumor activities. *J. Inorg. Biochem.* **193** (2019) 130–132.
7. Steens, N.; Ramadan, A. M.; Absillis, G.; Parac-Vogt, T. N.: Hydrolytic cleavage of DNA-model substrates promoted by polyoxovanadates. *Dalton Trans.* **39** (2010) 585–592.
8. Boulmier, A.; Feng, X. X.; Oms, O.; Mialane, P.; Riviere, E.; Shin, C. J.; Yao, J. Q.; Kubo, T.; Furuta, T.; Oldfield, E.; Dolbecq, A.: Anticancer activity of polyoxometalate-bisphosphonate complexes: synthesis, characterization, *in vitro* and *in vivo* results. *Inorg. Chem.* **56** (2017) 7558–7565.
9. Dankhoff, K.; Ahmad, A.; Weber, B.; Biersack, B.; Schobert, R.: Anticancer properties of a new non-oxido vanadium(IV) complex with a catechol-modified 3,3'-diindolylmethane ligand. *J. Inorg. Biochem.* **194** (2019) 1–6.
10. Wang, L. J.; Fan, H. T.; Du, C. J.; Xing, X. J.; Zhao, Y. Y.; Chen, B. K.; Wang, L. Y.: Synthesis, structure and properties of a co-crystallized complex based on polyoxovanadate [V^{IV}₁₂V^V₆O₄₂]^{6−} and a mononuclear vanadium complex [VON(CH₂CH₂O)₃]. *Inorg. Chem. Commun.* **63** (2016) 39–41.
11. Biswal, D.; Pramanik, N. R.; Chakrabarti, S.; Drew, M. G. B.; Acharya, K.; Chandra, S.: Syntheses, crystal structures, DFT calculations, protein interaction and anticancer activities of water soluble dipicolinic acid-imidazole based oxido vanadium(IV) complexes. *Dalton Trans.* **46** (2017) 16682–16702.
12. Chen, B. K.; Huang, X. Q.; Wang, B.; Lin, Z. G.; Hu, J. F.; Chi, Y. N.; Hu, C. W.: Three new imidazole-functionalized hexanuclear oxido vanadium clusters with exceptional catalytic oxidation properties for alcohols. *Chem. Eur. J.* **19** (2013) 4408–4413.