Wei Wei, Qiong Wu, Heng-Qiang Zhang*, Rui Liu and Hong-Li Chen

Crystal structure of bis(4-bromo-2-(((3-chloropropyl)imino)methyl)phenolato- $\kappa^2 N,O$)-oxidovanadium(IV), $C_{20}H_{20}Br_2Cl_2N_2O_3V$

Table 1: Data collection and handling.

Crystal:	Pink block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	4.15 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ϕ and ω
$\theta_{\max}$, completeness:	26.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	15913, 4522, 0.068
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3426
$N(\text{param})_{\text{refined}}$:	271
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

<https://doi.org/10.1515/ncrs-2018-0456>

Received October 28, 2018; accepted January 11, 2019; available online February 4, 2019

Abstract

$C_{20}H_{20}Br_2Cl_2N_2O_3V$, monoclinic, $P2_1/c$ (no. 14), $a = 11.9615(8)$ Å, $b = 16.5865(7)$ Å, $c = 12.9110(10)$ Å, $\beta = 115.879(9)^\circ$, $V = 2304.7(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0459$, $wR_{\text{ref}}(F^2) = 0.1132$, $T = 153(2)$ K.

CCDC no.: 1890454

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

*Corresponding author: Heng-Qiang Zhang, Department of Chemistry, Hebei Normal University for Nationalities, Chengde 067000, People's Republic of China, e-mail: zhanghengqiang80@163.com

Wei Wei: Department of Architecture Engineering, Chengde Petroleum College, Chengde 067000, People's Republic of China

Qiong Wu, Rui Liu and Hong-Li Chen: Department of Chemistry, Hebei Normal University for Nationalities, Chengde 067000, People's Republic of China

Source of material

In a 250 mL flask containing 50 mL of absolute methanol were added 5 mmol of 3-chloropropyl-ammonium hydrochloride and 5 mmol of 5-bromo-2-hydroxybenzaldehyde. Then 5 mmol of NaOH was dissolved in 2 mL H₂O, added to the above solution and the contents were refluxed for 3 h at 343 K. After cooling to room temperature, we added 10 mL methanol containing 2.5 mmol of vanadyl acetylacetonate. To this solution was added 30 mL trichloromethane and filtered off. The filtrate was refluxed with stirring for 3 h at 343 K. The pink crystals that formed were filtered off after seven days at room temperature.

Experimental details

All hydrogen atoms were added using a riding model. Their U_{iso} values were set to 1.2 U_{eq} of the parent atoms.

Comment

Vanadium compounds having a special physiological function of analog insulin, which can improve diabetes health. Their actions have been reported several times [5]. Schiff bases are universal ligands that can form metal compounds with many properties [6–8]. Studying some structure of such Schiff base vanadium complexes may help to understand the vanadium biochemistry.

The structure of the complex consists of a monomeric oxo-vanadium(IV) species bonded by two chelate rings of the Schiff base, namely, V1/O3/C11/C12/C17/N2 and

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
Br1	1.39699(4)	0.68672(2)	0.51587(4)	0.03736(16)
Br2	0.62594(5)	−0.03933(3)	0.27680(5)	0.05447(19)
C1	1.0908(3)	0.4870(2)	0.4017(3)	0.0235(9)
C2	1.0631(3)	0.5681(2)	0.4115(3)	0.0267(9)
H2	0.982001	0.582228	0.395633	0.032*
C3	1.1516(3)	0.6268(2)	0.4435(3)	0.0279(9)
H3	1.130510	0.680452	0.446462	0.033*
C4	1.2737(4)	0.6054(2)	0.4715(3)	0.0265(9)
C5	1.3053(4)	0.5264(2)	0.4660(3)	0.0275(9)
H5	1.387745	0.512871	0.486506	0.033*
C6	1.2140(3)	0.4661(2)	0.4298(3)	0.0249(9)
C7	1.2514(3)	0.3848(2)	0.4194(3)	0.0259(9)
H7	1.336360	0.375389	0.448188	0.031*
C8	1.2413(4)	0.2458(2)	0.3784(4)	0.0339(11)
H8A	1.231414	0.210911	0.434153	0.041*
H8B	1.329543	0.254703	0.403898	0.041*
C9	1.1889(4)	0.2042(2)	0.2632(4)	0.0376(11)
H9A	1.224808	0.150593	0.273671	0.045*
H9B	1.100124	0.197757	0.237044	0.045*
C10	1.2098(4)	0.2451(3)	0.1712(4)	0.0428(12)
H10A	1.188972	0.301755	0.169671	0.051*
H10B	1.155138	0.221886	0.097333	0.051*
C11	0.9126(4)	0.1696(2)	0.3513(3)	0.0271(9)
C12	0.7853(4)	0.1879(2)	0.3062(3)	0.0260(9)
C13	0.7004(4)	0.1245(2)	0.2873(4)	0.0326(10)
H13	0.616031	0.135502	0.260167	0.039*
C14	0.7420(4)	0.0467(2)	0.3090(4)	0.0340(11)
C15	0.8676(4)	0.0292(2)	0.3539(4)	0.0375(11)
H15	0.894496	−0.024015	0.369339	0.045*
C16	0.9509(4)	0.0891(2)	0.3754(4)	0.0346(11)
H16	1.035122	0.076798	0.406696	0.041*
C17	0.7385(3)	0.2684(2)	0.2824(3)	0.0254(9)
H17	0.654921	0.275279	0.264695	0.030*
C18	0.7331(4)	0.4102(2)	0.2637(4)	0.0291(10)
H18A	0.776003	0.444317	0.330551	0.035*
H18B	0.650132	0.400386	0.256621	0.035*
C19	0.7233(4)	0.4545(2)	0.1578(4)	0.0295(10)
H19A	0.678258	0.504413	0.150875	0.035*
H19B	0.806276	0.468500	0.168277	0.035*
C20	0.6599(4)	0.4083(3)	0.0479(4)	0.0364(11)
H20A	0.707436	0.360014	0.052037	0.044*
H20B	0.657705	0.440930	−0.015323	0.044*
Cl1	1.36945(11)	0.23572(8)	0.19286(12)	0.0541(4)
Cl2	0.50465(10)	0.38037(6)	0.01971(11)	0.0456(3)
N1	1.1797(3)	0.32450(17)	0.3747(3)	0.0261(8)
N2	0.7996(3)	0.33249(17)	0.2828(3)	0.0225(7)
O1	0.9994(2)	0.43342(14)	0.3672(2)	0.0289(7)
O2	0.9545(3)	0.32848(16)	0.1685(2)	0.0345(7)
O3	0.9979(2)	0.22705(15)	0.3714(2)	0.0318(7)
V1	0.98367(6)	0.32950(4)	0.30079(6)	0.02260(18)

V1/N1/C7/C6/C1/O1. The vanadium (IV) centre is five-coordinated in a distorted tetragonal pyramidal geometry containing one oxido ligand, two oxygen atoms (O1, O3), two nitrogen atoms (N1, N2) of the two Schiff base ligands, denoted as VO₃N₂. Bond angle sum of complex (N1–V1–O1, O1–V1–N2, N2–V1–O3, O3–V1–N1) is 344.2°, which is not closed to 360°. The V=O distance of 1.587(3) Å, typical for a V=O double bond in similar compounds: 1.586(6) Å [9] and 1.589(3) Å [10]. There also exists non classical intramolecular C–H···O and C–H···Cl hydrogen bonds stabilizing the structure.

Acknowledgements: The authors thank the projects of the program for the Department of Science and Technology of Hebei province (no. 17272610) for financial support.

References

1. Rigaku, O. D.: CrysAlis PRO. Rigaku Oxford Diffraction Ltd, Yarnton, Oxfordshire, England (2015).
2. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
3. Farrugia, L. J.: WinGX and ORTEP for Windows: an update. *J. Appl. Crystallogr.* **45** (2012) 849–854.
4. Spek, A. L.: Structure validation in chemical crystallography. *Acta Crystallogr.* **D65** (2009) 148–155.
5. You, Z. L.; Cui, Y. M.; Ma, Y. P.; Wang, C.; Zhou, X. S.; Li, K.: Synthesis, characterization and urease inhibitory activity of oxovanadium(V) complexes with similar Schiff bases. *Inorg. Chem. Commun.* **14** (2011) 636–640.
6. Ren, D. X.; Xing, N.; Shan, H.; Chen, C.; Cao, Y. Z.; Xing, Y. H.: Novel vanadium(III) complexes with rigid phenylpolycarboxylate ligands: synthesis, structures and application in C–H bond activation. *Dalton Trans.* **14** (2013) 5355–5365.
7. Lemons, B. G.; Richens, D. T.; Anderson, A.; Sedgwick, M.; Crans, D. C.; Johnson, M. D.: Stabilization of a vanadium(V)-catechol complex by compartmentalization and reduced solvation inside reverse micelles. *New J. Chem.* **24** (2013) 75–81.
8. Leon, I. E.; Di Virgilio, A. L.; Barrio, D. A.; Arrambide, G.; Gambino, D.; Etcheverry, S. B.: Hydroxylamido-amino acid complexes of oxovanadium(V). Toxicological study in cell culture and in a zebrafish model. *Metallomics* **4** (2012) 1287–1296.
9. Grivani, G.; Tahmasebi, V.; Khalaji, A. D.: A new oxidovanadium(IV) complex containing an *O,N*-bidentate Schiff base ligand: synthesis, characterization, crystal structure determination, thermal study and catalytic activity for an oxidative bromination reaction. *Polyhedron* **68** (2014) 144–150.
10. Grivani, G.; Ghavami, A.; Kučeráková, M.; Dušek, M.; Khalaji, A. D.: Synthesis, characterization, crystal structure determination, thermal study and catalytic activity of a new oxidovanadium Schiff base complex. *J. Mol. Struct.* **1076** (2014) 326–332.