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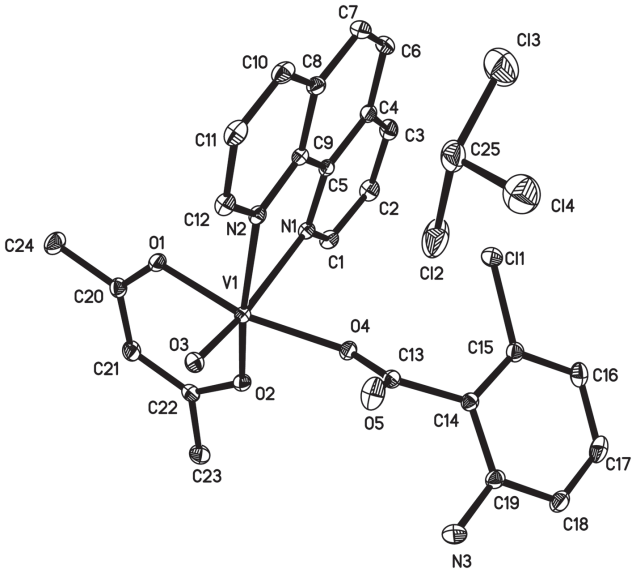
Crystal structure of (acetylacetonato-κ²*O*,*O*′)-(2-amino-6-chlorobenzoato-κ*O*)-oxido(1,10-phenanthroline-κ²*N*,*N*′)vanadium(IV) – trichloromethane (1/1)



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Abstract

$C_{25}H_{21}Cl_4N_3O_5V$, triclinic, $P\bar{1}$ (no. 2), $a = 10.8055(5)$ Å, $b = 11.5657(6)$ Å, $c = 12.0494(6)$ Å, $\alpha = 78.058(1)°$, $\beta = 80.405(2)°$, $\gamma = 69.388(1)°$, $V = 1371.69(12)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0549$, $wR_{ref}(F^2) = 0.1673$, $T = 295(2)$ K.

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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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.79 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	25.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	16340, 4936, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4183
$N(param)_{refined}$:	360
Programs:	Bruker programs [1], SHELX [2, 3]

Source of material

In a 250 mL round bottom flask containing 20 mL of absolute ethyl alcohol were added 1 mmol of 2-amino-6-chlorobenzoic acid with stirring. Then 1 mmol of vanadyl acetylacetonate, dissolved in 15 mL absolute methanol and 15 mL trichloromethane, were added to the above solution and the contents were refluxed for 1 h at 80°C. An amount of 20 mL absolute ethyl alcohol containing 1 mmol of 1,10-phenanthroline were added. The contents of the flask were refluxed with stirring for 1 h at 80°C. The reddish brown crystals that formed were filtered off and after 3 days 4°C the crystals were washed with absolute ethyl alcohol, n-hexane and dried in an oven.

Experimental details

The H atoms bonded to N3 was located in a difference map and refined without restraints. Other atoms were placed in calculated positions, with C—H = 0.93 for phenyl, 0.96 for methyl and 0.97 Å for methylene H atoms, and refined as riding, with $U_{iso}(H) = 1.2_{eq}(C)$ for phenyl and methylene H, and $1.5_{eq}U(C)$ for methyl H.

Discussion

Vanadium was considered to be an important pharmacologically and nutritionally important trace element [4]. Their properties have been reported in many articles [5–10]. Vanadyl acetylacetonate had a good hypoglycemic activity [11], in addition, 1,10-phenanthroline were selected as ancillary ligands for the VO(acac) scaffold also exhibited significantly potency [12]. We have synthesized the title complex as we want to study the structure of vanadium compounds and its relationship with hypoglycemic activity and enrich vanadium bioinorganic chemistry.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
V1	0.69247(5)	0.63377(5)	0.23284(4)	0.03762(19)
N1	0.9206(2)	0.5443(2)	0.2162(2)	0.0379(6)
N2	0.7442(2)	0.5268(2)	0.3954(2)	0.0389(6)
N3	0.4964(4)	0.3600(4)	0.0352(4)	0.0728(10)
O1	0.7399(2)	0.7714(2)	0.2759(2)	0.0497(6)
O2	0.6993(2)	0.7202(2)	0.07284(19)	0.0485(5)
O3	0.5368(2)	0.6817(2)	0.2737(2)	0.0530(6)
O4	0.7163(2)	0.4846(2)	0.16426(19)	0.0443(5)
O5	0.5602(3)	0.3992(3)	0.2416(3)	0.0796(9)
Cl1	0.95472(10)	0.23989(10)	0.17492(11)	0.0722(3)
Cl2	0.6711(3)	0.20639(16)	0.45548(14)	0.1443(9)
Cl3	0.8471(2)	0.0386(2)	0.6193(2)	0.1546(9)
Cl4	0.5975(2)	0.0148(2)	0.61263(17)	0.1281(6)
C1	1.0065(3)	0.5535(3)	0.1245(3)	0.0476(8)
H1	0.9753	0.6076	0.0589	0.057*
C2	1.1417(4)	0.4847(4)	0.1235(3)	0.0576(9)
H2	1.1987	0.4929	0.0577	0.069*
C3	1.1897(3)	0.4065(4)	0.2176(3)	0.0562(9)
H3	1.2800	0.3612	0.2170	0.067*
C4	1.1029(3)	0.3933(3)	0.3171(3)	0.0454(7)
C5	0.9683(3)	0.4647(3)	0.3103(2)	0.0367(6)
C6	1.1431(4)	0.3161(4)	0.4222(3)	0.0571(9)
H6	1.2321	0.2685	0.4272	0.069*
C7	1.0540(4)	0.3109(4)	0.5145(3)	0.0583(9)
H7	1.0832	0.2607	0.5823	0.070*
C8	0.9160(3)	0.3810(3)	0.5102(3)	0.0455(7)
C9	0.8731(3)	0.4564(3)	0.4079(2)	0.0369(6)
C10	0.8188(4)	0.3813(4)	0.6049(3)	0.0565(9)
H10	0.8424	0.3321	0.6746	0.068*
C11	0.6903(4)	0.4543(4)	0.5928(3)	0.0571(9)
H11	0.6257	0.4568	0.6547	0.069*
C12	0.6564(3)	0.5249(3)	0.4878(3)	0.0485(8)
H12	0.5679	0.5734	0.4810	0.058*
C13	0.6584(3)	0.4031(3)	0.1758(3)	0.0412(7)
C14	0.7156(3)	0.3116(3)	0.0925(3)	0.0403(7)
C15	0.8492(3)	0.2418(3)	0.0794(3)	0.0479(8)
C16	0.9044(4)	0.1682(4)	−0.0056(4)	0.0634(10)
H16	0.9943	0.1213	−0.0122	0.076*
C17	0.8205(5)	0.1673(4)	−0.0801(4)	0.0718(12)
H17	0.8557	0.1219	−0.1400	0.086*
C18	0.6876(5)	0.2311(4)	−0.0683(3)	0.0633(10)
H18	0.6337	0.2282	−0.1196	0.076*
C19	0.6318(4)	0.3006(3)	0.0202(3)	0.0492(8)
C20	0.7296(4)	0.8777(3)	0.2182(3)	0.0509(8)
C21	0.7005(4)	0.9135(4)	0.1058(3)	0.0571(9)
C22	0.6897(3)	0.8353(3)	0.0384(3)	0.0480(8)
C23	0.6659(4)	0.8828(4)	−0.0843(3)	0.0648(10)
H23A	0.7413	0.8393	−0.1320	0.097*
H23B	0.6532	0.9708	−0.1009	0.097*
H23C	0.5881	0.8686	−0.0987	0.097*
C24	0.7529(5)	0.9703(4)	0.2770(4)	0.0770(13)
H24A	0.7079	0.9675	0.3527	0.116*
H24B	0.7192	1.0530	0.2348	0.116*
H24C	0.8465	0.9492	0.2809	0.116*
C25	0.6867(7)	0.1208(5)	0.5903(5)	0.0953(17)
H25	0.647(6)	0.179(6)	0.645(5)	0.114*
H3B	0.462(4)	0.417(4)	0.094(3)	0.050(10)*
H3A	0.441(4)	0.356(4)	−0.002(3)	0.050(10)*
H21	0.683(4)	0.991(5)	0.075(4)	0.075(14)*

The structure of the compound title compound is shown in the Figure. It is a neutral oxovanadium complex with mixed acac, 1,10-phenanthroline and 2-amino-6-chlorobenzoato ligands. The central vanadium atom has a octahedral environment being coordinated by two oxygen atoms of acac, two nitrogen atoms of 1,10-phenanthroline, one oxygen atom of 2-amino-6-chlorobenzoate and one oxido ligand. The ligands of 1,10-phenanthroline and acac form a five-membered and six-memberedchelate ring, separately. The plane defined by the 1,10-phenanthroline ligand is almost perpendicular to that of the ring defined by the acac ligand. The V—O(1), V—O(2) and V—O(4) bond lengths (1.982 Å, 2.018 Å, 1.983 Å) are obviously longer than that of V—O(3) distance (1.60 Å), and suggest that V—O(1) , V—O(2) and V—O(4) are single bonds, while V—O(3) is a typical double bond [13].

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