Crystal structure of tris(isopropoxy)bis(μ -isopropoxy)divanadium, $C_{12}H_{28}O_4V$

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Source of material: Vanadium(V)triisopropoxy oxid (242 mg, 1 mmol) was added to samariumdiodide (404 mg, 1 mmol) in 20 ml THF. The reaction mixture was stirred for 48 h, filtered and concentrated in vacuo. At 195 K green-blue crystals (14 mg, 5 %) of the title compound among other products were obtained.

To the best of our knowledge, the title compound is the first fully structural characterized homoleptic bis(μ -oxo) vanadium alkoxy complex according to a CSD search (see ref. 1).

Table 1. Parameters used for the X-ray data collection

Crystal:	light blue prism, size 0.4 x 0.5 x 0.6 mm
Wavelength:	Mo $K\alpha$ radiation (0.71069 Å)
μ :	6.05 cm ⁻¹
Diffractometer:	STOE-IPDS
Scan mode:	100 exposures, $\Delta\phi = 2^\circ$
$T_{\text{measurement}}$:	200 K
$2\theta_{\text{max}}$:	48.5°
$N(hkl)_{\text{unique}}$:	2454
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	266
Programs:	SHELXS-86, SHELXL-93

$C_{12}H_{28}O_4V$, triclinic, $P\bar{1}$ (No. 2), $a = 9.595(2)$ Å, $b = 9.662(2)$ Å, $c = 9.959(2)$ Å, $\alpha = 103.72(1)^\circ$, $\beta = 109.20(1)^\circ$, $\gamma = 98.47(1)^\circ$, $V = 821.1$ Å³, $Z = 2$, $R(F) = 0.032$, $R_w(F^2) = 0.083$.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(1)	2i	0.388(3)	1.756(3)	1.446(3)	0.033(6)
H(2A)	2i	0.177(3)	1.598(3)	1.435(3)	0.048(8)
H(2B)	2i	0.145(3)	1.526(3)	1.276(3)	0.047(8)
H(2C)	2i	0.133(4)	1.680(3)	1.324(3)	0.066(9)
H(3)	2i	0.233(3)	1.147(3)	1.059(3)	0.055(8)
H(4A)	2i	0.312(4)	1.228(4)	0.893(4)	0.08(1)
H(4B)	2i	0.176(4)	1.313(4)	0.874(3)	0.08(1)
H(4C)	2i	0.138(4)	1.135(4)	0.816(4)	0.069(9)
H(5)	2i	0.516(4)	1.256(3)	1.092(4)	0.08(1)
H(6A)	2i	0.551(8)	1.054(7)	1.133(6)	0.19(3)
H(6B)	2i	0.748(6)	1.155(5)	1.263(6)	0.13(2)
H(6C)	2i	0.677(5)	1.111(5)	1.088(5)	0.10(2)
H(7)	2i	0.309(3)	1.260(3)	1.583(3)	0.034(6)
H(8A)	2i	0.365(4)	1.045(3)	1.454(3)	0.058(8)
H(8B)	2i	0.191(4)	0.982(4)	1.362(4)	0.08(1)
H(8C)	2i	0.257(4)	1.004(4)	1.545(5)	0.10(1)
H(9A)	2i	0.469(4)	1.734(3)	1.238(3)	0.063(9)
H(9B)	2i	0.302(3)	1.765(3)	1.198(3)	0.058(8)
H(9C)	2i	0.327(3)	1.606(3)	1.149(3)	0.043(7)
H(10A)	2i	0.071(4)	1.304(4)	1.494(4)	0.07(1)
H(10B)	2i	0.070(4)	1.171(4)	1.560(4)	0.08(1)
H(10C)	2i	0.012(3)	1.139(3)	1.383(4)	0.056(8)
H(11A)	2i	0.031(3)	1.211(3)	1.130(4)	0.064(9)
H(11B)	2i	-0.032(5)	1.128(4)	0.960(4)	0.10(1)
H(11C)	2i	0.013(4)	1.307(4)	1.013(4)	0.09(1)
H(12A)	2i	0.653(5)	1.472(5)	1.186(5)	0.11(2)
H(12B)	2i	0.761(5)	1.392(5)	1.135(6)	0.13(2)
H(12C)	2i	0.774(7)	1.455(6)	1.292(7)	0.14(2)

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2i	0.3498(3)	1.6707(3)	1.3672(3)	0.039(1)	0.031(1)	0.034(1)	0.017(1)	0.005(1)	0.012(1)
C(2)	2i	0.1875(3)	1.6128(3)	1.3484(4)	0.036(2)	0.055(2)	0.046(2)	0.024(1)	0.010(1)	0.013(2)
C(3)	2i	0.1985(3)	1.2239(3)	1.0436(3)	0.047(2)	0.032(1)	0.032(1)	0.005(1)	0.002(1)	0.005(1)
C(4)	2i	0.2074(4)	1.2272(4)	0.8949(3)	0.068(2)	0.060(2)	0.031(1)	0.010(2)	0.006(1)	0.004(1)
C(5)	2i	0.6046(3)	1.2692(3)	1.1847(3)	0.054(2)	0.074(2)	0.032(1)	0.032(2)	0.018(1)	0.008(1)
C(6)	2i	0.6622(6)	1.1324(5)	1.1725(6)	0.093(3)	0.087(3)	0.085(3)	0.049(2)	0.048(3)	0.004(2)
C(7)	2i	0.2430(3)	1.1984(3)	1.4858(3)	0.036(1)	0.039(1)	0.034(1)	0.002(1)	0.008(1)	0.013(1)

Table 3. (Continued)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(8)	2i	0.2685(4)	1.0466(4)	1.4587(5)	0.057(2)	0.047(2)	0.101(3)	0.017(2)	0.026(2)	0.033(2)
C(9)	2i	0.3634(3)	1.6927(3)	1.2268(3)	0.051(2)	0.045(2)	0.040(2)	0.014(1)	0.008(1)	0.022(1)
C(10)	2i	0.0829(3)	1.1984(4)	1.4781(4)	0.045(2)	0.074(2)	0.054(2)	0.008(2)	0.021(2)	0.018(2)
C(11)	2i	0.0386(3)	1.2139(4)	1.0386(4)	0.043(2)	0.075(2)	0.049(2)	-0.001(2)	0.003(2)	0.013(2)
C(12)	2i	0.728(1)	1.3988(6)	1.2141(8)	0.191(6)	0.086(3)	0.104(4)	0.011(4)	0.108(4)	0.026(3)
O(1)	2i	0.5541(2)	1.2899(2)	1.3063(2)	0.048(1)	0.057(1)	0.0323(9)	0.0298(8)	0.0167(8)	0.0135(8)
O(2)	2i	0.2975(2)	1.3548(2)	1.1572(2)	0.0362(9)	0.0307(8)	0.0272(8)	0.0068(6)	0.0045(7)	0.0066(6)
O(3)	2i	0.4436(2)	1.5748(2)	1.4229(2)	0.0287(8)	0.0282(8)	0.0284(8)	0.0113(6)	0.0073(6)	0.0114(6)
O(4)	2i	0.2667(2)	1.2594(2)	1.3764(2)	0.037(1)	0.0426(9)	0.0371(9)	-0.0006(7)	0.0073(8)	0.0167(7)
V(1)	2i	0.41239(4)	1.36902(4)	1.34801(4)	0.0287(2)	0.0265(2)	0.0257(2)	0.0086(2)	0.0086(2)	0.0080(2)

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References

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