

crystallization yields exclusively the dication with the bridging dppe as described here.

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*Acta Cryst.* (1991). **C47**, 650–651

## A Vanadyl Complex of a Tetradentate Ligand Featuring a *cis*-N<sub>2</sub>S<sub>2</sub> Donor Ligand Set

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(Received 16 July 1990; accepted 20 August 1990)

**Abstract.** [N,N'-Dimethyl-2,2'-(ethylenediimino-κ<sup>2</sup>-N,N')diethanethiolato-κ<sup>2</sup>S,S']oxovanadium(IV), [VO(C<sub>8</sub>H<sub>18</sub>N<sub>2</sub>S<sub>2</sub>)], *M<sub>r</sub>* = 273.3, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 7.620 (1), *b* = 12.702 (2), *c* = 12.782 (1) Å, β = 103.75 (1)°, *V* = 1202 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.510 Mg m<sup>-3</sup>, Mo *Kα*, λ = 0.7107 Å, μ = 1.074 mm<sup>-1</sup>, *F*(000) = 572, *T* = 293 (1) K, *R* = 0.038 for 1759 observed reflections. The V atom in [VO(C<sub>8</sub>H<sub>18</sub>N<sub>2</sub>S<sub>2</sub>)] exists in a distorted square-pyramidal geometry with the square plane defined by a *cis*-N<sub>2</sub>S<sub>2</sub> donor set provided by the dianion L<sup>2-</sup> [where LH<sub>2</sub> is N,N'-dimethyl-2,2'-(ethylene-diimino)diethanethiol]; V—N 2.151 (3), 2.164 (3); V—S 2.361 (1), 2.335 (1) Å. The V atom lies 0.6986 (5) Å out of the N<sub>2</sub>S<sub>2</sub> plane in the direction of the apical O atom [V—O 1.596 (2) Å].

**Experimental.** [VO(acac)<sub>2</sub>] (acac = acetylacetonate) and the free ligand, LH<sub>2</sub> (Wilson, Kony, Tiekink, Pilbrow, Spence & Wedd, 1988), were refluxed in methanol to produce a mixture of [V(acac)<sub>3</sub>] and [VO(C<sub>8</sub>H<sub>18</sub>N<sub>2</sub>S<sub>2</sub>)]. Recrystallization from hot methanol produced a low yield (6%) of well formed crystals of [VO(C<sub>8</sub>H<sub>18</sub>N<sub>2</sub>S<sub>2</sub>)]. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo *Kα* radiation; ω:2θ scan technique. Cell parameters by least squares on 25 reflections (2 ≤ θ ≤ 11°) (de Boer & Duisenberg, 1984) on a spherical crystal of 0.33 mm diameter; no absorption correction applied. 3447 reflections (1.5 ≤ θ ≤ 27.5°) measured in the range -9 ≤ *h* ≤ 9, 0 ≤ *k*

≤ 16, -16 ≤ *l* ≤ 1. No significant variation in the net intensities of three reflections (2̄10, 020, 241) measured every 7200 s. 2762 unique reflections (*R*<sub>int</sub> 0.024) and 1759 satisfied *I* ≥ 2.5σ(*I*). Structure solved by Patterson method, full-matrix least-squares refinement on 128 parameters based on *F* (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms included at their calculated positions. Evidence of disorder in the structure is seen in the high thermal motion associated with the C(4) and C(5) atoms and the short C(4)—C(5) bond distance of 1.262 (7) Å. At convergence *R* = 0.038, *wR* = 0.046, *w* = 0.57/[σ<sup>2</sup>(*F*) + 0.0015|*F*|<sup>2</sup>], *S* = 0.96, (Δ/σ)<sub>max</sub> ≤ 0.002, Δρ<sub>max</sub> = 0.40, Δρ<sub>min</sub> = -0.59 e Å<sup>-3</sup>; no extinction correction applied. Scat-

Table 1. Atomic coordinates and *B*<sub>eq</sub> values (Å<sup>2</sup>)

	<i>B</i> <sub>eq</sub> = (8π <sup>2</sup> /3) trace U.			
	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub>
V	0.23878 (7)	0.16363 (4)	0.18823 (4)	2.46
S(1)	0.3692 (2)	0.3332 (1)	0.2013 (1)	4.48
S(2)	-0.0256 (1)	0.2247 (1)	0.0717 (1)	4.83
O(1)	0.3542 (4)	0.0900 (2)	0.1286 (2)	4.39
N(1)	0.3575 (4)	0.1602 (2)	0.3587 (2)	3.03
N(2)	0.0589 (4)	0.0526 (2)	0.2379 (2)	3.79
C(1)	0.4460 (6)	0.3401 (3)	0.3453 (3)	4.57
C(2)	0.3692 (5)	0.2681 (3)	0.4053 (3)	3.62
C(3)	0.5427 (7)	0.1166 (4)	0.3807 (4)	5.84
C(4)	0.2406 (10)	0.0958 (5)	0.4110 (3)	9.29
C(5)	0.1194 (9)	0.0383 (8)	0.3544 (4)	12.36
C(6)	0.0639 (8)	-0.0507 (4)	0.1835 (6)	8.31
C(7)	-0.1287 (6)	0.0910 (4)	0.2114 (4)	5.63
C(8)	-0.1864 (6)	0.1296 (5)	0.0992 (4)	6.40

Table 2. Selected interatomic distances (Å) and bond angles (°)

V—S(1)	2.361 (1)	V—S(2)	2.335 (1)
V—N(1)	2.151 (3)	V—N(2)	2.164 (3)
V—O(1)	1.596 (2)	S(1)—C(1)	1.816 (4)
S(2)—C(8)	1.813 (5)	N(1)—C(2)	1.489 (4)
N(1)—C(3)	1.479 (5)	N(1)—C(4)	1.482 (5)
N(2)—C(5)	1.461 (5)	N(2)—C(6)	1.491 (6)
N(2)—C(7)	1.471 (5)	C(1)—C(2)	1.496 (5)
C(4)—C(5)	1.262 (7)	C(7)—C(8)	1.481 (6)
S(1)—V—S(2)	91.6 (1)	S(1)—V—N(1)	83.0 (1)
S(1)—V—N(2)	149.2 (1)	S(1)—V—O(1)	107.3 (1)
S(2)—V—N(1)	138.1 (1)	S(2)—V—N(2)	83.9 (1)
S(2)—V—O(1)	112.3 (1)	N(1)—V—N(2)	80.4 (1)
N(1)—V—O(1)	109.0 (1)	N(2)—V—O(1)	102.6 (1)
V—S(1)—C(1)	100.3 (1)	V—S(2)—C(8)	100.1 (2)

tering factors for neutral V atom corrected for  $f'$  and  $f''$  from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149) and for the remaining atoms as incorporated in *SHELX76* (Sheldrick, 1976). All calculations on a Sun4/280 computer system. Atomic parameters are given in Table 1, selected interatomic parameters in Table 2\* and numbering scheme used is shown in Fig. 1 which was drawn with *ORTEP* (Johnson, 1971) at 25% probability levels.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, mean-plane data and all interatomic parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53501 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

*Acta Cryst.* (1991). C47, 651–654

## Structure of Di[(carbonyl)(chloro)(1,4,7-triazacyclononane)(triphenylphosphine)-osmium(II)] Tetrachlorozincate(II)

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(Received 3 July 1990; accepted 23 August 1990)

**Abstract.**  $[\text{OsCl}(\text{CO})\{\text{P}(\text{C}_6\text{H}_5)_3\}(\text{C}_6\text{H}_{15}\text{N}_3)]_2[\text{ZnCl}_4]$ ,  $M_r = 1497.51$ , triclinic,  $P\bar{1}$ ,  $a = 16.000$  (4),  $b = 17.262$  (5),  $c = 10.432$  (3) Å,  $\alpha = 94.52$  (2),  $\beta = 92.22$  (2),  $\gamma = 88.78$  (2)°,  $V = 2870$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.73$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu =$

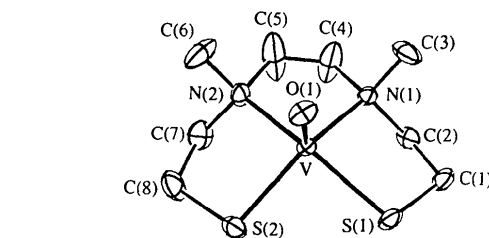


Fig. 1. Molecular structure and crystallographic numbering scheme employed for  $[\text{VO}(\text{C}_8\text{H}_{18}\text{N}_2\text{S}_2)]$  (Johnson, 1971).

**Related literature.** The structure determination of the title compound forms part of a wider study of complexes of this type (Wilson *et al.*, 1988; Wedd & Spence, 1990).

The Australian Research Council is thanked for support.

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$52.25$  cm<sup>-1</sup>,  $F(000) = 1464$ ,  $T = 296$  K,  $R = 0.045$ , 6364 unique observed reflections. The reaction of the dimer,  $\{\text{OsBr}_2(\text{CO})[\text{P}(\text{C}_6\text{H}_5)_3]_2\}_2$ , with an equimolar amount of the cyclic amine 1,4,7-triazacyclononane (containing  $\text{ZnCl}_2$  as an impurity) yielded a few clear crystals of the title compound as a minor product of the reaction. The structure is comprised of two