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Chlorobis(η^5-cyclopentadienyl)oxoniobium(V)

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Abstract. $[Nb(Cl)(O)(C_5H_5)_2]$, $M_r = 274{\cdot}54$, orthorhombic, *Fdd*2, $a = 14{\cdot}676\ (4)$, $b = 44{\cdot}39\ (1)$, $c = 6{\cdot}200\ (1)$ Å, $V = 4039\ (2)$ Å^3, $Z = 16$, $D_x = 1{\cdot}806$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 6{\cdot}87$ cm^{-1}, $F(000) = 2176$, $T = 296$ K, $R_F = 3{\cdot}09\%$ for 803 observed $[F_o > 6\sigma(F_o)]$ reflections and 99 parameters. This Nb^V compound has a typical bent metallocene structure with a centroid–metal–centroid angle of 128·2 (1)°. The Cl—Nb—O angle of 98·4 (2)° is perpendicular to, and bisected by, the centroid–metal–centroid plane. When projected down the centroid–centroid vector, the cyclopentadienyl rings are in a staggered conformation.

Experimental. Pale yellow needles (0·08 × 0·08 × 0·50 mm) prepared by the aerobic oxidation of $[(\eta^5\text{-}C_5H_5)_2NbH(tolAs)_2]$ in chloroform-*d*. Nicolet *R*3*m* diffractometer with graphite monochromator; ω scans; lattice parameters from least-squares fit of 25 reflections ($20 \le 2\theta \le 25°$); absorption correction was applied ($\mu = 6{\cdot}87$ cm^{-1}, thin needle, $T_{max}/T_{min} = 1{\cdot}23$); $2\theta_{max} = 48°$ ($h = +17$, $k = \pm 51$, $l = +8$); standard reflections 711, 2,16,2, 1,11,3. 1892 reflections collected, 876 unique ($R_{int} = 2{\cdot}65\%$), 803 observed with $F_o > 6\sigma(F_o)$, 73 unobserved reflections. Direct-methods (*SOLV*) structure solution; least-squares refinement (on *F*) on 99 parameters; all non-H atoms anisotropic, H atoms with idealized contributions, cyclopentadienyl rings fixed as rigid planar pentagons (C—C = 1·420 Å), $R_F = 3{\cdot}09\%$, $wR_F = 3{\cdot}30\%$, $S = 1{\cdot}170$, $w^{-1} = \sigma(F_o) + gF_o^2$, $g = 0{\cdot}001$; $(\Delta/\sigma)_{max} = 0{\cdot}003$; $\Delta\rho_{max} = 0{\cdot}50$, $\Delta\rho_{min} = -0{\cdot}59$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149); *SHELXTL* computer program (Sheldrick, 1984).

Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* (Å$^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
Nb	7250·9 (4)	580·3 (1)	5000	27·1 (2)
Cl	7011 (2)	1047 (1)	6993 (5)	60 (1)
O	7349 (4)	337 (1)	7180 (10)	44 (2)
C(1)	8916 (4)	501 (2)	5090 (14)	61 (4)
C(2)	8810	818	4899	67 (4)
C(3)	8402	879	2869	69 (4)
C(4)	8256	600	1805	88 (6)
C(5)	8574	366	3178	64 (4)
C(6)	5665 (5)	729 (2)	3941 (21)	111 (8)
C(7)	5617	457	5137	103 (7)
C(8)	6045	227	3905	80 (5)
C(9)	6356	358	1948	69 (5)
C(10)	6121	668	1971	85 (6)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Nb—Cnt(1)	2·171 (6)	Nb—O	1·737 (6)
Nb—Cnt(2)	2·182 (6)	Nb—Cl	2·439 (2)
O—Nb—Cl	98·4 (2)	Cnt(1)—Nb—Cnt(2)	128·2 (1)
O—Nb—Cnt(1)	108·1 (3)	Cl—Nb—Cnt(1)	104·3 (2)
O—Nb—Cnt(2)	108·2 (3)	Cl—Nb—Cnt(2)	105·5 (2)

Cnt(1) = centroid of atoms C(1)–C(5).
Cnt(2) = centroid of atoms C(6)–C(10).

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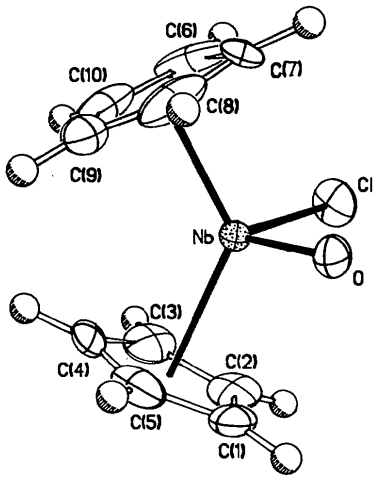

Fig. 1. Molecular structure and labeling scheme for $[Nb(Cl)(O)(C_5H_5)_2]$.

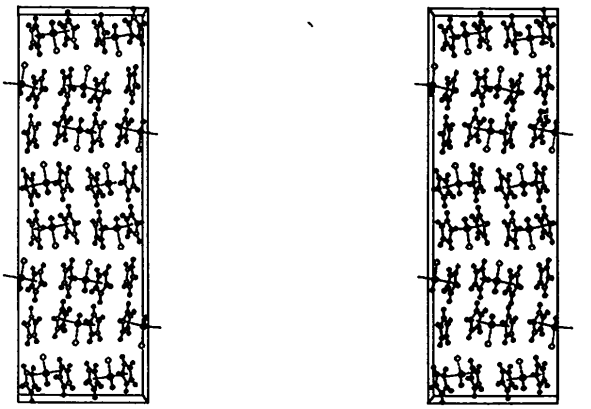

Fig. 2. Unit-cell packing diagram as viewed down the *c* axis.

Atomic and equivalent isotropic thermal parameters are given in Table 1 and selected bond lengths in Table 2. Fig. 1 shows the molecular structure of the compound. A unit-cell packing diagram is shown in Fig. 2.*

Related literature. The title compound was previously prepared by the reaction of niobocene dichloride with various organolithium compounds followed by oxidation with atmospheric oxygen (Baukova, Knizhnikov, Lemenovskii, Nesmeyanov & Perevalova, 1976). A similar compound, μ-oxo-bis[bis(η^5-cyclopentadienyl)]chloroniobium(V) bis-(tetrafluoroborate), has been crystallographically characterized (Cameron, Critchley, Denton, Forder, Prout & Rees, 1974).

* Lists of structure factors, anisotropic thermal parameters and full lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54012 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of uns-*cis*-*mer* (Pyridine-2-carboxylato-*N*,*O*)(ethylenediaminediacetato-*N*,*N*′,*O*,*O*′)cobalt(III)

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Abstract. $C_{12}H_{14}CoN_3O_6$, $M_r = 355{\cdot}19$, orthorhombic, *Pccn*, $a = 8{\cdot}838$ (3), $b = 24{\cdot}221$ (5), $c = 12{\cdot}553$ (2) Å, $V = 2687{\cdot}03$ Å^3, $Z = 8$, $D_x = 1{\cdot}756$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 12{\cdot}4$ cm^{-1}, $F(000) = 1456$, room temperature, final $R = 0{\cdot}052$ for 2044 unique reflections. The geometry about the Co atom is roughly octahedral with the edda (ethylenediaminediacetato) ligand assuming an uns-*cis* configuration such that there is one out-of-plane acidate (*R*) ring and one in-plane amino acidate (*G*) ring.

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