

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and equivalent isotropic vibrational parameters for non-hydrogen atoms ( $\text{\AA}^2 \times 10^3$  except Pd,  $\text{\AA}^2 \times 10^4$ )

	x	y	z	$U_{eq}^*$
Pd(1)	0	5000	5000	381 (1)
N(1)	611 (3)	5753 (3)	7168 (5)	44 (1)
N(2)	-950 (3)	5229 (3)	6233 (6)	43 (1)
O(1)	-1721 (3)	4944 (2)	5643 (6)	63 (1)
C(1)	170 (3)	5982 (3)	8297 (6)	38 (1)
C(2)	-720 (3)	5703 (3)	7810 (6)	39 (1)
C(3)	-1262 (3)	5916 (4)	8933 (6)	43 (1)
C(4)	-964 (3)	6408 (3)	10480 (6)	46 (1)
C(5)	-76 (3)	6683 (3)	10995 (6)	48 (1)
C(6)	481 (3)	6496 (3)	9993 (6)	43 (1)
C(7)	-1520 (5)	6673 (5)	11713 (9)	67 (1)
C(8)	1412 (3)	6773 (5)	10597 (8)	60 (1)
O(1W)	2500	6005 (5)	7500	57 (1)

$$* U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i a_j^*$$

**Related literature.** The title molecule shares some structural features with the following:  $\alpha$ -bis(1,2-benzoquinone dioximato)palladium(II) (Kistenmacher & Destro, 1983),  $\beta$ -bis(1,2-benzoquinone dioximato)palladium(II), (Endres, Mégnamisi-Bélombé, Little & Wolfe, 1979), 1:1 pyridine adduct of (4-methyl-1-quinone 2-oximato)copper(II) (McPartlin, 1973).

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## Structure of [ZnCl(C<sub>4</sub>H<sub>8</sub>O)( $\mu$ -Cl)]<sub>∞</sub>

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**Abstract.** *catena*-Poly[[chloro(tetrahydrofuran)zinc( $\mu$ -chloro)],  $M_r = 208.39$ , orthorhombic,  $Pc2_1b$ ,  $a = 6.928$  (1),  $b = 7.306$  (1),  $c = 15.197$  (3)  $\text{\AA}$ ,  $V = 769.2$  (4)  $\text{\AA}^3$ ,  $Z = 4$ ,  $D_x = 1.80$  g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71073$   $\text{\AA}$ ,  $\mu = 38.8$  cm<sup>-1</sup>,  $F(000) = 416$ ,  $T = 295$  K,  $R = 0.037$ ,  $wR = 0.044$  for 1065 unique observed reflections [ $I_o \geq 2.5\sigma(I)$ ]. Zn is tetrahedrally coordinated by a C<sub>4</sub>H<sub>8</sub>O (THF) ligand [Zn–O = 1.981 (3)  $\text{\AA}$ ], a terminal Cl [Zn–Cl = 2.169 (1)  $\text{\AA}$ ] and two bridging Cl [Zn–Cl = 2.289 (4) and 2.323 (4)  $\text{\AA}$ ]. Infinite chains of [ZnCl(THF)( $\mu$ -Cl)] units are formed along the 2<sub>1</sub> axis of the crystal.

**Experimental.** Colourless needles of [ZnCl(THF)( $\mu$ -Cl)]<sub>∞</sub> were obtained as a coproduct (with [( $\eta$ -C<sub>5</sub>H<sub>5</sub>)MoCl(O)<sub>2</sub>( $\mu$ -O)]) of the oxidation of [( $\eta$ -C<sub>5</sub>H<sub>5</sub>)MoCl(O)<sub>2</sub>]<sub>2</sub>( $\mu$ -Cl)<sub>2</sub>Zn(THF)] by O<sub>2</sub> (Bottomley,

Table 2. Selected bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

Pd(1)–N(1)	1.984 (4)	Pd(1)–N(2)	2.023 (4)
N(1)–C(1)	1.292 (6)	N(2)–O(1)	1.272 (6)
N(2)–C(2)	1.333 (6)	C(1)–C(2)	1.441 (6)
C(1)–C(6)	1.446 (6)	C(2)–C(3)	1.405 (6)
C(3)–C(4)	1.336 (7)	C(4)–C(5)	1.436 (7)
C(4)–C(7)	1.505 (7)	C(5)–C(6)	1.348 (7)
C(6)–C(8)	1.503 (7)		
N(2)–Pd(1)–N(1)	78.7 (2)	C(3)–C(2)–C(1)	121.7 (4)
C(1)–N(1)–Pd(1)	116.1 (3)	C(4)–C(3)–C(2)	120.3 (5)
C(2)–N(2)–Pd(1)	115.4 (3)	C(5)–C(4)–C(3)	118.7 (4)
C(2)–C(1)–N(1)	116.8 (4)	C(6)–C(5)–C(4)	124.1 (4)
C(6)–C(1)–C(2)	116.9 (4)	C(5)–C(6)–C(1)	118.2 (4)
C(1)–C(2)–N(2)	112.8 (4)		

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Ferris & White, 1988). The crystals were extremely hygroscopic. One, of dimensions 0.15  $\times$  0.15  $\times$  0.40 mm, was sealed in a capillary in a dry box and then mounted on an Enraf–Nonius CAD-4 diffractometer. Lattice constants were obtained by accurate centring of 16 reflections in the range  $30 < 2\theta < 40^\circ$ . Intensities were measured using the  $\omega/2\theta$  scan mode to a  $2\theta_{\text{max}}$  of  $50^\circ$  ( $h_{\text{max}}$  7,  $k_{\text{max}}$  8,  $l_{\text{max}}$  17). Three standard reflections were monitored every hour. There was no significant change in their intensity. The intensities of 1883 reflections were measured and averaged to yield 1337 unique reflections (including Friedel pairs), of which 1065 were judged as being significant by the criterion that  $I > 2.5\sigma(I)$ . No absorption correction was made. The structure was solved using direct methods (*MULTAN*80, Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and all other

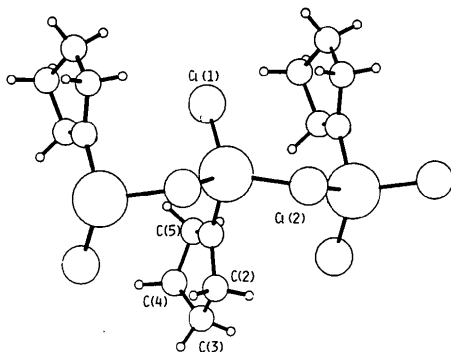
Table 1. Atomic coordinates of the non-H atoms with e.s.d.'s in parentheses

	x	y	z	$B_{\text{iso}}^*(\text{\AA}^2)$
Zn	0.01585 (7)	0.25	0.26797 (3)	2.97 (4)
Cl(1)	0.19891 (21)	0.2519 (8)	0.38372 (8)	4.95 (7)
Cl(2)	-0.18343 (17)	0.0033 (6)	0.2489 (4)	4.01 (11)
O	0.1798 (4)	0.2445 (15)	0.16115 (20)	3.70 (24)
C(2)	0.1105 (7)	0.254 (3)	0.0715 (3)	4.5 (5)
C(3)	0.2814 (10)	0.244 (3)	0.0154 (4)	7.1 (6)
C(4)	0.4474 (10)	0.286 (3)	0.0737 (5)	7.1 (7)
C(5)	0.3870 (8)	0.237 (4)	0.1619 (4)	5.7 (4)

\*  $B_{\text{iso}}$  is the mean of the principal axes of the thermal ellipsoid.

Table 2. Important interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

Zn-Cl(1)	2.169 (1)	Cl(1)-Zn-Cl(2)	117.4 (2)
Zn-Cl(2)	2.289 (4)	Cl(1)-Zn-Cl(2a)	115.6 (2)
Zn-Cl(2a)	2.323 (4)	Cl(1)-Zn-O	109.2 (1)
Zn-O	1.981 (3)	Cl(2)-Zn-Cl(2a)	104.8 (1)
O-C(2)	1.447 (5)	Cl(2)-Zn-O	103.1 (3)
C(2)-C(3)	1.460 (8)	Cl(2a)-Zn-O	105.4 (3)
C(3)-C(4)	1.484 (12)	Zn-Cl(2)-Zn	106.4 (1)
C(4)-C(5)	1.450 (14)		
C(5)-O	1.437 (6)		

Fig. 1. Structure of  $[\text{ZnCl}(\text{C}_4\text{H}_8\text{O})(\mu\text{-Cl})]_\infty$ .

computations were performed using *NRCVAX* (Gabe, Lee & Le Page, 1985). The non-centrosymmetric  $Pc2_1b$  is a polar space group; the origin was chosen as the Zn atom at  $x, 0.25, z$  and the sense of the  $b$  axis was determined by a Rogers' (1981)  $\eta$  refinement of Friedel pairs of reflections. The initial value of  $\eta$  was 0.5 and refinement gave 0.05 (12). Refinement in the centrosymmetric space group  $Pcmb$  with a (chemically unreasonable) planar THF gave a significantly [0.005 confidence level (Hamilton, 1965)] higher  $R$  value (0.039) and a difference synthesis showed no evidence of disorder. Thus  $Pc2_1b$  is a more chemically reasonable choice of space group. The function minimized was  $\sum w(\Delta F)^2$ , where  $w = 1/[\sigma(F)^2 + 0.001F^2]$  and  $\sigma$  was obtained from counting statistics. All atoms except H were refined anisotropically. The H atoms were allowed to ride on the C atom to which they were attached ( $\text{C-H} = 0.96 \text{ \AA}$ ) and were given the fixed isotropic thermal parameters of the C atom obtained in

an isotropic refinement. Full-matrix refinement of 73 parameters gave a final  $R = 0.037$ ,  $wR = 0.044$  and a goodness of fit of 1.106. The largest  $\Delta/\sigma$  was 0.271. A final difference synthesis had a maximum peak of  $0.82 \text{ e \AA}^{-3}$  close to the Zn atom, and a minimum hole of  $-0.73 \text{ e \AA}^{-3}$ . Scattering factors for neutral atoms, corrected for the real and imaginary parts of the anomalous dispersion, were obtained from *International Tables for X-ray Crystallography* (1974). Positional parameters are listed in Table 1, selected bond lengths and angles in Table 2, and a diagram of the structure is shown in Fig. 1.\*

**Related literature.** The structure of  $[\text{ZnCl}_3(\text{THF})]^-$  has been determined; it contains tetrahedrally coordinated Zn in isolated anions (Folting, Huffman, Bansemer & Caulton, 1984; Folting, Huffman, Bansemer, Caulton, Martin & Smith, 1984). In  $\text{ZnCl}_2 \cdot 1.3\text{H}_2\text{O}$  chains of  $[\text{ZnCl}(\mu\text{-Cl})]_\infty$  are present, as in  $[\text{ZnCl}(\text{THF})(\mu\text{-Cl})]_\infty$ , but the  $\text{H}_2\text{O}$  ligands are coordinated to Zn ions on the periphery of the chain (Follner & Brehler, 1970). Anhydrous  $\text{ZnCl}_2$  has close-packed Cl ions with Zn in tetrahedral holes (Yakel & Brynstad, 1978).

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\* Lists of H-atom positions, anisotropic thermal parameters, structure-factor amplitudes and further bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51583 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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