

(Hodgson & Raymond, 1972). This relationship is discussed in the paper reporting the structure of [K(C<sub>4</sub>H<sub>10</sub>O<sub>2</sub>)<sub>2</sub>][Yb(C<sub>8</sub>H<sub>8</sub>)<sub>2</sub>] (Kinsley, Streitwieser & Zalkin, 1985).

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## Structure of Dichlorobis(pyridine *N*-oxide)zinc(II)

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**Abstract.** [ZnCl<sub>2</sub>(C<sub>5</sub>H<sub>5</sub>NO)<sub>2</sub>],  $M_r = 326.5$ , orthorhombic, *Fdd2*,  $a = 12.318(4)$ ,  $b = 28.176(6)$ ,  $c = 7.268(2)$  Å,  $V = 2522.5(7)$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.72$ ,  $D_m = 1.70$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 24.1$  cm<sup>-1</sup>,  $F(000) = 1312$ ,  $T = 297$  K. Final  $R = 0.019$  for 599 observed reflections. The [ZnCl<sub>2</sub>(C<sub>5</sub>H<sub>5</sub>NO)<sub>2</sub>] molecule has a distorted tetrahedral geometry (crystallographically required *C<sub>2</sub>*) with Zn–O and Zn–Cl distances of 1.992(2) and 2.214(1) Å respectively. The pyridine rings are planar within 0.009(3) Å.

**Experimental.** The title complex was prepared from approximately 1:1 molar ratios of anhydrous zinc dichloride and pyridine *N*-oxide in methanol/ethanol mixtures and recrystallized from the same solvents. Diffractometer used: Enraf–Nonius CAD-4 equipped with graphite monochromator. Cell dimensions: from 15 reflections in range  $22 \leq 2\theta \leq 30^\circ$ . Density measured by flotation. Crystal dimensions:  $\sim 0.3 \times 0.25 \times 0.4$  mm. Crystal faces not readily indexed, and therefore no absorption corrections made; estimate of maximum relative error in intensity due to absorption *ca* 12%. Total of 606 independent reflections ( $h$ , 0–14;  $k$ , 0–33;  $l$ , 0–8) measured in range  $\sin \theta/\lambda \leq 0.6$  Å<sup>-1</sup> using  $\omega$ – $2\theta$  scan technique. Four standards monitored, no variation with time. Standard deviations assigned as  $\sigma(I) = [\sigma_{\text{count}}^2 + (0.04I)^2]^{1/2}$ ; 599 reflections with  $F_o \geq 2\sigma(F_o)$  used for refinement. Structure solved by Patterson and Fourier methods: least-squares refinement (based on  $F$ ) carried out using *LINEX*, a modified version of *ORFLS* (Busing, Martin & Levy, 1962); function minimized  $\sum w(|F_o| - |F_c|)^2$  with weights

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$w = [2LpF_o/\sigma(I)]^2$ . Pyridine-ring H atoms included as fixed contributions with C–H set at 0.98 Å. Initial refinement with all *hkl* indices positive gave  $R = 0.035$ , but with an unsatisfactory goodness of fit. Since *Fdd2* is a polar space group, the refinement was continued with all indices reassigned with negative values, and this converged at  $R = 0.019$ ,  $wR = 0.028$  and  $S = 1.13$  for the 77 variables refined. At convergence all  $\Delta p_i < 0.02 \times \sigma(p_i)$  and maximum and minimum values in final difference map 0.40 and  $-0.55$  e Å<sup>-3</sup>. Atomic scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974); anomalous-dispersion corrections for Zn and Cl included (Cromer & Liberman, 1970). Calculations carried out on a CDC-Cyber 175 computer. Atomic coordinates are listed in Table 1\* and bond distances and angles in Table 2. An *ORTEP* view (Johnson, 1965) of the molecule is given in Fig. 1.

**Related literature.** Structures related to the present compound: [CuCl<sub>2</sub>(C<sub>5</sub>H<sub>5</sub>NO)<sub>2</sub>]<sub>2</sub> (Morrow, 1974), [CuCl<sub>2</sub>(C<sub>5</sub>H<sub>5</sub>NO)<sub>2</sub>] (Estes & Hodgson, 1976) and [ZnI<sub>2</sub>(C<sub>5</sub>H<sub>5</sub>NO)<sub>2</sub>] (Sawitzki & von Schnering, 1974).

We thank the University Computation Center for a generous allocation of computer time.

\* Lists of anisotropic thermal parameters, observed and calculated structure-factor amplitudes, H-atom coordinates and least-squares-planes' data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42858 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^4$ ) for  $[\text{ZnCl}_2(\text{C}_5\text{H}_5\text{NO})_2]$

	x	y	z	$U_{eq}^*$
Zn	0	0	0	357 (3)
Cl	1492 (1)	187 (1)	1535 (2)	512 (5)
O	388 (2)	-542 (1)	-1634 (4)	517 (12)
N	-389 (2)	-798 (1)	-2448 (4)	360 (10)
C(1)	-337 (3)	-1275 (1)	-2269 (5)	436 (16)
C(2)	-1059 (3)	-1558 (1)	-3168 (6)	485 (17)
C(3)	-1863 (3)	-1357 (1)	-4249 (5)	531 (18)
C(4)	-1917 (3)	-871 (1)	-4377 (5)	494 (18)
C(5)	-1178 (3)	-596 (1)	-3471 (5)	435 (16)

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

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) in dichlorobis(pyridine *N*-oxide)zinc(II)

Zn—O	1.992 (2)	Zn—Cl	2.214 (1)
	O—N	1.338 (4)	
N—C(1)	1.353 (4)	N—C(5)	1.350 (4)
C(1)—C(2)	1.360 (5)	C(4)—C(5)	1.365 (5)
C(2)—C(3)	1.384 (5)	C(3)—C(4)	1.375 (6)
O—Zn—O'	106.8 (2)	Cl—Zn—Cl'	119.5 (1)
O—Zn—Cl	106.4 (1)	O—Zn—Cl'	108.5 (1)
	Zn—O—N	120.4 (1)	
O—N—C(1)	117.4 (3)	O—N—C(5)	122.1 (2)
N—C(1)—C(2)	120.2 (3)	N—C(5)—C(4)	120.4 (3)
C(1)—C(2)—C(3)	120.1 (3)	C(5)—C(4)—C(3)	120.1 (3)
C(2)—C(3)—C(4)	118.6 (3)	C(1)—N—C(5)	120.5 (3)

(') denotes twofold-related atoms.

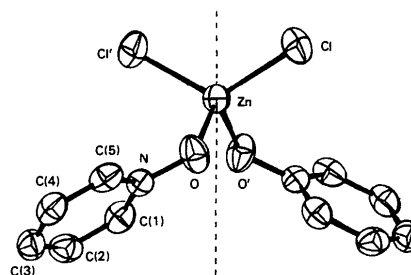


Fig. 1. An ORTEP plot of the  $[\text{ZnCl}_2(\text{C}_5\text{H}_5\text{NO})_2]$  molecule viewed perpendicular to  $[001]$ . Ellipsoids are at the 50% probability level.

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## Structure of Methyl Tetrahydro-3,4-dihydroxy-2,4,5-trimethyl-2-furancarboxylate at 163 K

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**Abstract.**  $\text{C}_9\text{H}_{16}\text{O}_5$ ,  $M_r = 204.22$ , monoclinic,  $P2_1$ ,  $a = 10.5910$  (14),  $b = 18.908$  (3),  $c = 7.9060$  (8)  $\text{\AA}$ ,  $\beta = 100.849$  (13) $^\circ$ ,  $V = 1554.9$  (3)  $\text{\AA}^3$ ,  $Z = 6$ ,  $D_x = 1.308$   $\text{g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069$   $\text{\AA}$ ,  $\mu = 0.997$   $\text{cm}^{-1}$ ,  $F(000) = 660$ ,  $R = 0.0389$  for 3978 reflections ( $F \geq 4\sigma_F$ ). The asymmetric unit is a trimer linked through three hydrogen bonds [O...H distances 1.91 (3), 1.97 (3), 2.09 (4)  $\text{\AA}$ ] and a bifurcated hydro-

gen bond [O...H distances 2.32 (4), 2.37 (4)  $\text{\AA}$ ]. In addition, trimers are linked along **a** and along **b** by hydrogen bonds [O...H distances 1.94 (4), 2.05 (3)  $\text{\AA}$ , respectively].

**Experimental.** Title compound prepared from D-ribonic acid. Absolute configuration correct as shown [m.p. 408 K;  $[\alpha]_D^{25^\circ\text{C}} = -32^\circ$  ( $\text{CHCl}_3$ , 0.92  $\text{g dm}^{-3}$ )]. Transparent, colorless crystals obtained from ethyl acetate/hexane solution. Crystal for data collection cut from a large plate having a corrugated surface. Summary of data collection and structural refinement in Table 1.

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