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Structure of μ-[1,2-Bis(*tert*-butylamino-1:2κN)-1,2-di(2-pyridyl-κN)ethane]-bis(ethylzinc)

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Abstract. $[Zn_2(C_2H_5)_2(C_{20}H_{28}N_4)]$, $M_r = 513{\cdot}35$, orthorhombic, *Pbca*, $a = 16{\cdot}98$ (2), $b = 17{\cdot}76$ (1), $c = 17{\cdot}63$ (1) Å, $U = 5317$ (8) Å^3, $Z = 8$, $D_x = 1{\cdot}283$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 18{\cdot}6$ cm^{-1}, $F(000) = 2160$, $T = 295$ K, $R = 0{\cdot}059$ for 2624 reflections with $I \geq 2{\cdot}5\sigma(I)$. Each of the two amido N atoms bridges between the two ethylzinc moieties, while each of the two pyridyl N atoms coordinates to a Zn. Each Zn is tetrahedrally coordinated by three N and one C atom. The length of the newly formed C—C bond of 1·569 (8) Å is significantly longer than the normal C(sp^3)—C(sp^3) single-bond value.

Experimental. Crystals were obtained by recrystallization from benzene. X-ray data for a transparent colourless crystal (0·20 × 0·45 × 0·80 mm), sampled under nitrogen in a Lindemann-glass capillary, were collected on an Enraf–Nonius CAD-4 diffractometer using Zr-filtered Mo $K\alpha$ radiation. Lattice parameters and their estimated standard deviations were derived from the setting angles of 16 reflections ($10 < \theta < 10{\cdot}5°$). The space group was determined from the observed systematic absences. A total of 5695 reflections [$\theta < 25°$; $\omega/2\theta$ scan; $\Delta\omega = 0{\cdot}40 + 0{\cdot}35\tan(\theta)°$; $0 \leq h \leq 20$, $0 \leq k \leq 21$, $0 \leq l \leq 20$] were scanned. Four reference reflections (221, $2\bar{2}1$, $2\bar{2}\bar{1}$, $22\bar{1}$) showed a small decay of 2% over the 128 h of X-ray exposure time. The data were corrected for Lp and the decay but not for absorption. The 2624 reflections with $I \geq 2{\cdot}5\sigma(I)$ were used in the subsequent analysis. The structure was solved for the heavy atoms with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and completed with the non-hydrogen atoms by standard Fourier methods. Subsequent refinement was carried out by blocked full-matrix least-squares techniques on F with *SHELX*76 (Sheldrick, 1976). All non-hydrogen atoms were refined with anisotropic thermal parameters. H atoms were introduced on calculated positions and refined with fixed geometry with respect to their carrier atoms and one common isotropic temperature factor [0·161 (4) Å^2]. Convergence was reached at $R = 0{\cdot}059$ [$wR = 0{\cdot}076$, $w^{-1} = \sigma^2(F) + 0{\cdot}0012F^2$, 2624 reflections, 296 parameters, $S = 2{\cdot}35$; $(\Delta/\sigma)_{av} = 0{\cdot}3$]. A final difference Fourier map did not show features outside the range of 0·79 and −0·51 e Å^{-3}. Fig. 1 shows the

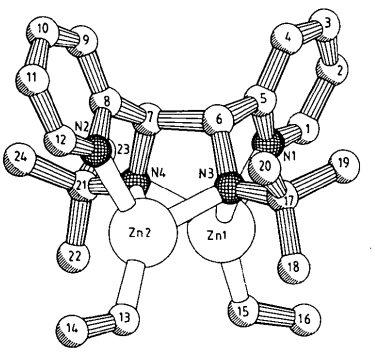

Fig. 1. View of the molecule with adopted numbering. Hydrogen atoms omitted for clarity.

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Table 1. *Final coordinates and equivalent isotropic thermal parameters of the non-*H *atoms with their e.s.d.'s in parentheses*

$$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
Zn(1)	0·19065 (4)	0·09490 (4)	0·33287 (4)	0·0706 (3)
Zn(2)	0·19757 (4)	0·02457 (5)	0·19412 (5)	0·0765 (3)
N(1)	0·0863 (3)	0·1437 (3)	0·3772 (3)	0·073 (2)
N(2)	0·0978 (3)	−0·0240 (3)	0·1396 (3)	0·074 (2)
N(3)	0·1409 (2)	0·1237 (2)	0·2284 (3)	0·059 (1)
N(4)	0·1375 (3)	−0·0030 (2)	0·2924 (3)	0·065 (2)
C(1)	0·0671 (4)	0·1736 (4)	0·4445 (4)	0·093 (2)
C(2)	−0·0064 (5)	0·2061 (4)	0·4574 (5)	0·102 (2)
C(3)	−0·0600 (4)	0·2058 (4)	0·3999 (5)	0·102 (2)
C(4)	−0·0423 (4)	0·1728 (4)	0·3319 (4)	0·079 (2)
C(5)	0·0330 (3)	0·1431 (3)	0·3210 (3)	0·063 (2)
C(6)	0·0594 (3)	0·1043 (3)	0·2497 (4)	0·061 (2)
C(7)	0·0583 (3)	0·0173 (3)	0·2650 (3)	0·062 (2)
C(8)	0·0381 (4)	−0·0214 (3)	0·1900 (4)	0·068 (2)
C(9)	−0·0363 (4)	−0·0480 (4)	0·1725 (4)	0·085 (2)
C(10)	−0·0466 (4)	−0·0816 (4)	0·1025 (5)	0·099 (2)
C(11)	0·0122 (5)	−0·0849 (4)	0·0529 (5)	0·106 (2)
C(12)	0·0855 (5)	−0·0550 (4)	0·0725 (4)	0·097 (2)
C(13)	0·3046 (4)	0·0084 (5)	0·1525 (7)	0·132 (2)
C(14)	0·3195 (6)	−0·0452 (7)	0·1086 (8)	0·209 (3)
C(15)	0·2922 (4)	0·1145 (5)	0·3837 (5)	0·115 (2)
C(16)	0·3031 (5)	0·1812 (5)	0·4241 (6)	0·157 (2)
C(17)	0·1507 (4)	0·1947 (4)	0·1869 (4)	0·083 (2)
C(18)	0·2388 (4)	0·2102 (4)	0·1791 (5)	0·102 (2)
C(19)	0·1134 (5)	0·2617 (4)	0·2258 (6)	0·137 (2)
C(20)	0·1170 (6)	0·1865 (5)	0·1063 (5)	0·154 (2)
C(21)	0·1414 (5)	−0·0735 (4)	0·3367 (5)	0·093 (2)
C(22)	0·2276 (5)	−0·0881 (5)	0·3544 (6)	0·124 (2)
C(23)	0·0983 (6)	−0·0666 (5)	0·4103 (5)	0·147 (2)
C(24)	0·1097 (6)	−0·1414 (4)	0·2928 (6)	0·142 (2)

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

Zn(1)–N(1)	2·122 (6)	C(3)–C(4)	1·37 (1)
Zn(1)–N(3)	2·090 (6)	C(4)–C(5)	1·396 (9)
Zn(1)–N(4)	2·085 (5)	C(5)–C(6)	1·502 (9)
Zn(1)–C(15)	1·974 (8)	C(6)–C(7)	1·569 (8)
Zn(2)–N(3)	2·095 (5)	C(7)–C(8)	1·529 (9)
Zn(2)–N(4)	2·069 (6)	C(8)–C(9)	1·38 (1)
Zn(2)–N(2)	2·130 (6)	C(9)–C(10)	1·38 (1)
Zn(2)–C(13)	1·981 (8)	C(10)–C(11)	1·33 (1)
N(1)–C(1)	1·340 (9)	C(11)–C(12)	1·40 (1)
N(1)–C(5)	1·342 (8)	C(13)–C(14)	1·25 (2)
N(2)–C(8)	1·349 (9)	C(15)–C(16)	1·39 (1)
N(2)–C(12)	1·321 (9)	C(17)–C(18)	1·53 (1)
N(3)–C(6)	1·475 (7)	C(17)–C(19)	1·51 (1)
N(3)–C(17)	1·467 (8)	C(17)–C(20)	1·54 (1)
N(4)–C(7)	1·474 (7)	C(21)–C(22)	1·52 (1)
N(4)–C(21)	1·477 (9)	C(21)–C(23)	1·49 (1)
C(1)–C(2)	1·39 (1)	C(21)–C(24)	1·53 (1)
C(2)–C(3)	1·36 (1)		

N(1)–Zn(1)–N(3)	83·5 (2)	C(2)–C(3)–C(4)	120·5 (7)
N(1)–Zn(1)–N(4)	96·0 (2)	C(3)–C(4)–C(5)	118·9 (6)
N(1)–Zn(1)–C(15)	119·4 (3)	N(1)–C(5)–C(4)	120·9 (5)
N(3)–Zn(1)–N(4)	74·2 (2)	N(1)–C(5)–C(6)	114·9 (5)
N(3)–Zn(1)–C(15)	135·2 (3)	C(4)–C(5)–C(6)	124·2 (5)
N(4)–Zn(1)–C(15)	132·9 (3)	N(3)–C(6)–C(5)	112·7 (5)
N(3)–Zn(2)–N(4)	74·4 (2)	N(3)–C(6)–C(7)	106·6 (4)
N(2)–Zn(2)–N(3)	96·0 (2)	C(5)–C(6)–C(7)	107·7 (5)
N(3)–Zn(2)–C(13)	130·5 (3)	N(4)–C(7)–C(6)	106·6 (4)
N(2)–Zn(2)–N(4)	83·7 (2)	N(4)–C(7)–C(8)	112·2 (5)
N(4)–Zn(2)–C(13)	136·7 (4)	C(6)–C(7)–C(8)	107·3 (5)
N(2)–Zn(2)–C(13)	120·3 (3)	N(2)–C(8)–C(7)	114·6 (5)
Zn(1)–N(1)–C(1)	133·7 (4)	N(2)–C(8)–C(9)	121·8 (6)
Zn(1)–N(1)–C(5)	106·7 (4)	C(7)–C(8)–C(9)	123·4 (6)
C(1)–N(1)–C(5)	119·5 (5)	C(8)–C(9)–C(10)	117·5 (6)
Zn(2)–N(2)–C(8)	106·7 (4)	C(9)–C(10)–C(11)	120·7 (7)
Zn(2)–N(2)–C(12)	134·3 (5)	C(10)–C(11)–C(12)	119·5 (8)
C(8)–N(2)–C(12)	119·0 (6)	N(2)–C(12)–C(11)	121·3 (7)
Zn(1)–N(3)–Zn(2)	82·1 (1)	Zn(2)–C(13)–C(14)	121·6 (7)
Zn(1)–N(3)–C(6)	95·6 (4)	Zn(1)–C(15)–C(16)	119·8 (6)
Zn(1)–N(3)–C(17)	127·2 (4)	N(3)–C(17)–C(18)	108·1 (5)
Zn(2)–N(3)–C(6)	107·9 (3)	N(3)–C(17)–C(19)	113·7 (6)
Zn(2)–N(3)–C(17)	121·7 (4)	N(3)–C(17)–C(20)	109·7 (6)
C(6)–N(3)–C(17)	115·7 (4)	C(18)–C(17)–C(19)	108·0 (6)
Zn(1)–N(4)–Zn(2)	82·9 (2)	C(18)–C(17)–C(20)	107·3 (7)
Zn(1)–N(4)–C(7)	107·6 (3)	C(19)–C(17)–C(20)	109·7 (7)
Zn(1)–N(4)–C(21)	120·4 (4)	N(4)–C(21)–C(22)	107·2 (6)
Zn(2)–N(4)–C(7)	96·7 (3)	N(4)–C(21)–C(23)	111·6 (6)
Zn(2)–N(4)–C(21)	128·4 (4)	N(4)–C(21)–C(24)	112·6 (7)
C(7)–N(4)–C(21)	114·9 (5)	C(22)–C(21)–C(23)	107·9 (8)
N(1)–C(1)–C(2)	121·8 (7)	C(22)–C(21)–C(24)	108·0 (7)
C(1)–C(2)–C(3)	118·4 (7)	C(23)–C(21)–C(24)	109·4 (7)

molecule with adopted numbering. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Data on the geometry are assembled in Table 2. Scattering factors of Cromer & Mann (1968) corrected for anomalous dispersion (Cromer & Liberman, 1970) were used. Geometrical calculations and illustrations were performed with the programs *PLATON* and *PLUTON* of the *EUCLID* package (Spek, 1982).

Related literature. The title compound was obtained from the reaction of *N-tert*-butyl-2-pyridine aldimine with diethylzinc *via* dimerization of the initially formed persistent *N-tert*-butyl-2-pyridine aldimine ethylzinc radical (Jastrzebski, Klerks, van Koten & Vrieze, 1981; van Koten, Jastrzebski & Vrieze, 1983). In solution this radical is in equilibrium with the title compound.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44071 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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