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Acta Cryst. (1984). C**40**, 613–616

Tetrameric Methylzinc Acetoximate,* $[Zn_4(CH_3)_4(C_3H_6NO)_4]$

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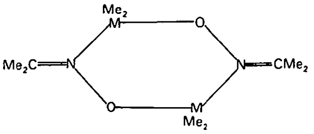
Abstract. $M_r = 610{\cdot}0$, monoclinic, $P2_1/c$, $a = 9{\cdot}258\ (6)$, $b = 13{\cdot}58\ (3)$, $c = 24{\cdot}10\ (5)$ Å, $\beta = 105{\cdot}4\ (2)^\circ$, $U = 2921$ Å^3, $Z = 4$, $D_m = 1{\cdot}40\ (2)$, $D_x = 1{\cdot}39$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 4{\cdot}1$ mm^{-1}, $F(000) = 1248$, $T = 293$ K. Final $R = 0{\cdot}101$ for 3081 observed reflection intensities estimated visually. The molecule exists in the crystal as discrete tetramers in agreement with the solution data. The four Zn atoms lie at the corners of a slightly distorted tetrahedron and above each face is an acetoximate group in which the O atom is attached to two Zn atoms and the N to a third. A cage structure results which has four five-membered rings and two adjacent six-membered rings but no four-membered rings so that the four Zn atoms are in three different environments.

Introduction. The title compound was prepared as described by Coates & Ridley (1966). Though dimeric compounds $(Me_2C{=}NOMMe_2)_2$ (M = Al, Ga, In) have been prepared and are considered to have the cyclic structure (I) (Jennings & Wade, 1967), methylzinc acetoximate is tetrameric in benzene solution (Coates & Ridley, 1966). The present structure analysis was undertaken to establish the degree of association in the solid state and to investigate the manner by which association takes place, whether nitrogen is involved in coordination or whether the cubane structure found for $(MeZnOMe)_4$ (Shearer & Spencer, 1980) results.



(I)

Experimental. Recrystallized from pentane as air-sensitive colourless needles, elongated along **a**, rectangular cross-section, sealed in Pyrex capillaries under dry N_2; unit-cell dimensions from zero-level precession photographs using Mo $K\alpha$ radiation; D_m by flotation in *o*-bromotoluene/bromobenzene; intensity data recorded on layers 0*kl*–8*kl* using Ni-filtered Cu $K\alpha$ radiation by the equi-inclination Weissenberg multiple-film technique. There was a marked deterioration in the shape of the reflections after about five days exposure and subsequently the crystals turned from colourless through yellow to dark brown after an exposure time of about a fortnight. Three crystals of approximate dimensions $0{\cdot}13 \times 0{\cdot}13 \times 0{\cdot}9$ mm (for nets $h = 0$–2), $0{\cdot}15 \times 0{\cdot}24 \times 0{\cdot}8$ mm ($h = 3$–6) and $0{\cdot}21 \times 0{\cdot}21 \times 1{\cdot}0$ mm ($h = 7$–8) used for data collection, intensities estimated visually, Lorentz and polarization corrections applied with spot-length corrections (Phillips, 1956) for upper-level reflections; in view of the uncertainty in recognizing the individual crystal faces, together with

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* Tetrakis[μ-(acetone oximato)-μ-*O*,*N*]-tetrakis(methylzinc).

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Table 1. *Fractional positional parameters* ($\times 10^4$) *and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

B_{eq} values calculated by reference to Willis & Pryor (1975).

	x	y	z	B_{eq}(Å^2)
Zn(1)	1639 (2)	3434 (1)	1347 (1)	5·6 (1)
Zn(2)	4224 (2)	2101 (1)	2335 (1)	5·7 (1)
Zn(3)	3544 (2)	1992 (1)	674 (1)	5·5 (1)
Zn(4)	949 (2)	1005 (1)	1290 (1)	5·9 (1)
O(1)	3875 (9)	3180 (6)	1707 (3)	5·4 (3)
O(2)	1494 (9)	2650 (6)	611 (3)	5·6 (3)
O(3)	1048 (9)	2258 (6)	1775 (3)	5·5 (3)
O(4)	3104 (9)	997 (6)	1249 (3)	5·2 (3)
N(1)	4692 (11)	2937 (8)	1299 (4)	5·7 (4)
N(2)	454 (12)	1878 (7)	553 (4)	5·6 (4)
N(3)	1965 (11)	2135 (7)	2330 (3)	5·1 (3)
N(4)	4238 (11)	917 (7)	1777 (3)	5·4 (4)
C(1)	638 (18)	4697 (11)	1344 (7)	6·9 (6)
C(2)	5876 (19)	2345 (16)	3020 (6)	8·7 (10)
C(3)	3920 (21)	1524 (11)	−40 (6)	7·4 (6)
C(4)	60 (23)	−189 (12)	1493 (8)	8·4 (8)
C(5)	5961 (14)	3377 (10)	1385 (5)	6·0 (5)
C(6)	6935 (20)	3121 (14)	996 (7)	8·1 (7)
C(7)	6501 (19)	4134 (12)	1850 (7)	7·9 (7)
C(8)	−505 (16)	1815 (11)	78 (5)	6·7 (7)
C(9)	−509 (26)	2561 (16)	−427 (7)	10·2 (11)
C(10)	−1613 (21)	976 (16)	−22 (8)	9·1 (8)
C(11)	1305 (15)	2113 (9)	2723 (5)	5·9 (5)
C(12)	−364 (19)	2187 (14)	2607 (7)	8·0 (7)
C(13)	2212 (19)	1997 (13)	3340 (6)	7·7 (7)
C(14)	5204 (15)	259 (9)	1781 (4)	6·0 (5)
C(15)	5238 (20)	−347 (12)	1258 (6)	7·9 (7)
C(16)	6410 (18)	86 (13)	2324 (6)	7·6 (7)

the relatively small size of the crystals and the almost square cross-section in two cases, no absorption corrections applied. Zn atoms determined from Patterson function, C, N, O atoms from successive Fourier syntheses; refinement by least squares on F with anisotropic temperature factors in final cycles, $R = 0{\cdot}101$ for 3081 observed reflections (index range h 0 to 8, k 0 to 13, l −28 to 26), max. $\Delta/\sigma = 0{\cdot}12$ in final LS cycle, weighting scheme $\sqrt{w} = [1-\exp(-20)(\sin\theta/\lambda)^2]/(1 + 0{\cdot}2|F_o| + 0{\cdot}0012|F_o|^2 + 0{\cdot}00003|F_o|^3)$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1962). Computer programs were supplied by Professor D. W. J. Cruickshank and Dr J. G. Sime of Glasgow University.

Discussion. Final atomic parameters are given in Table 1.*

The structure is shown in Fig. 1 with the four independent Zn atoms at the corners of a slightly distorted tetrahedron; above each face of the tetrahedron is an acetoximate group in which the O atom is attached to two Zn atoms and the N atom to a third. The orientation of the >O—N=CMe$_2$ group on each Zn$_3$ tetrahedral face is such that the four Zn atoms in the tetramer are found in three different environments. In addition to the methyl C atoms: (i) Zn(1) is bonded to three O atoms, (ii) Zn(2) is bonded to one O and two N atoms, and (iii) Zn(3) and Zn(4) are each bonded to one N and two O atoms; thus the Zn atoms are four-coordinate and the O atoms three-coordinate. This pattern of bonding results in a 'cage' structure which has four puckered five-membered rings and two adjacent six-membered rings, but no four-membered rings are found, *cf.* (MeZnNPh$_2$)$_2$ (Bell, Shearer & Spencer, 1983). The atoms Zn(2), N(4) and O(4) are common to both five- and six-membered rings. Each five-membered ring is composed of two Zn, two O and one N atom. Although both of the six-membered rings contain two Zn, two O and two N atoms, they are, however, intrinsically different. In the first, Zn(2),-N(3),O(3),Zn(4),O(4),N(4), both O atoms are bonded to a common zinc, Zn(4), and similarly both N atoms are bonded to Zn(2), whereas in the other six-membered ring, Zn(3),N(1),O(1),Zn(2),N(4),O(4), each Zn is bonded to one N and one O atom. The six-membered ring formed by Zn(2), N(3), O(3), Zn(4), O(4) and N(4) is analogous in shape to a cyclohexane ring in the chair conformation. The atoms Zn(2), N(3), Zn(4) and O(4) are approximately coplanar and N(4) is situated 0·7 Å to one side of the plane while O(3) is 0·8 Å to the other side.

Bond lengths and angles with e.s.d.'s are listed in Table 2. The mean Zn—C bond length is 1·95 (2) Å; none of the individual values differ significantly from each other and are similar to the values found in (MeZnOMe)$_4$, (MeZnNPh$_2$)$_2$ and Me$_2$Zn which have been discussed earlier (Bell *et al.*, 1983). The Zn—O lengths vary from 2·024 (8) to 2·069 (8) Å and for these extreme cases the difference is significant. In spite of differences in environment of the Zn atoms, however, the Zn—O lengths agree fairly closely with one another and, with the exception of Zn(4)—O(4), the shortest, the

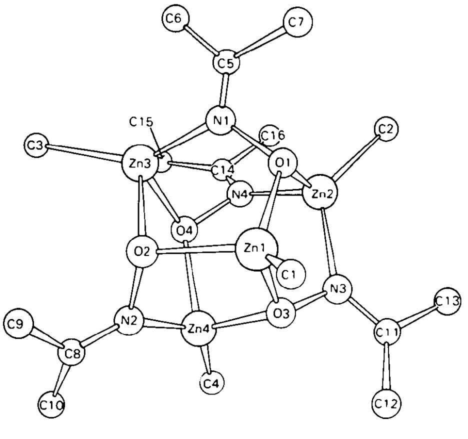

Fig. 1. Structure of (MeZnON=CMe$_2$)$_4$.

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* Lists of structure factors, anisotropic thermal parameters, least-squares planes and intramolecular non-bonded distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39081 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Zn(1)–C(1)	1·948 (15)	O(1)–N(1)	1·430 (13)
Zn(2)–C(2)	1·958 (17)	O(2)–N(2)	1·405 (13)
Zn(3)–C(3)	1·953 (15)	O(3)–N(3)	1·389 (12)
Zn(4)–C(4)	1·940 (18)	O(4)–N(4)	1·422 (12)
Zn(1)–O(1)	2·050 (8)	N(1)–C(5)	1·285 (16)
Zn(1)–O(2)	2·042 (8)	N(2)–C(8)	1·252 (17)
Zn(1)–O(3)	2·053 (8)	N(3)–C(11)	1·257 (16)
Zn(2)–O(1)	2·069 (8)	N(4)–C(14)	1·263 (16)
Zn(3)–O(2)	2·066 (9)	C(5)–C(6)	1·503 (22)
Zn(3)–O(4)	2·052 (8)	C(5)–C(7)	1·507 (21)
Zn(4)–O(3)	2·053 (8)	C(8)–C(9)	1·581 (24)
Zn(4)–O(4)	2·024 (8)	C(8)–C(10)	1·509 (25)
Zn(2)–N(3)	2·089 (10)	C(11)–C(12)	1·498 (21)
Zn(2)–N(4)	2·098 (10)	C(11)–C(13)	1·509 (21)
Zn(3)–N(1)	2·047 (10)	C(14)–C(15)	1·512 (20)
Zn(4)–N(2)	2·082 (10)	C(14)–C(16)	1·497 (21)

N(3)–Zn(2)–N(4)	101·0 (4)	O(3)–N(3)–C(11)	115·6 (10)
O(1)–Zn(1)–O(2)	96·1 (3)	Zn(2)–N(3)–C(11)	133·0 (9)
O(1)–Zn(1)–O(3)	91·8 (3)	Zn(2)–N(3)–O(3)	111·3 (6)
O(2)–Zn(1)–O(3)	93·9 (3)	O(4)–N(4)–C(14)	114·3 (9)
O(2)–Zn(3)–O(4)	89·5 (3)	Zn(2)–N(4)–C(14)	131·6 (9)
O(3)–Zn(4)–O(4)	97·9 (3)	Zn(2)–N(4)–O(4)	112·7 (6)
O(1)–Zn(2)–N(3)	91·1 (3)	C(1)–Zn(1)–O(1)	125·1 (5)
O(1)–Zn(2)–N(4)	95·7 (3)	C(1)–Zn(1)–O(3)	119·8 (6)
O(2)–Zn(3)–N(1)	94·1 (4)	C(1)–Zn(1)–O(2)	122·3 (6)
O(4)–Zn(3)–N(1)	94·3 (4)	C(2)–Zn(2)–O(1)	115·6 (6)
O(3)–Zn(4)–N(2)	88·9 (4)	C(2)–Zn(2)–N(4)	122·3 (6)
O(4)–Zn(4)–N(2)	87·6 (4)	C(2)–Zn(2)–N(3)	123·9 (6)
Zn(1)–O(1)–N(1)	114·0 (6)	C(3)–Zn(3)–O(2)	116·8 (6)
Zn(1)–O(1)–Zn(2)	111·8 (4)	C(3)–Zn(3)–O(4)	119·7 (5)
Zn(2)–O(1)–N(1)	109·4 (6)	C(3)–Zn(3)–N(1)	132·3 (6)
Zn(1)–O(2)–N(2)	111·0 (6)	C(4)–Zn(4)–O(3)	120·5 (6)
Zn(1)–O(2)–Zn(3)	108·3 (4)	C(4)–Zn(4)–O(4)	119·9 (6)
Zn(3)–O(2)–N(2)	106·1 (6)	C(4)–Zn(4)–N(2)	132·8 (7)
Zn(1)–O(3)–N(3)	113·7 (6)	C(6)–C(5)–C(7)	119·1 (13)
Zn(1)–O(3)–Zn(4)	109·6 (3)	C(9)–C(8)–C(10)	120·0 (14)
Zn(4)–O(3)–N(3)	111·8 (6)	C(12)–C(11)–C(13)	117·8 (13)
Zn(3)–O(4)–N(4)	115·0 (6)	C(15)–C(14)–C(16)	117·9 (13)
Zn(3)–O(4)–Zn(4)	113·8 (4)	C(6)–C(5)–N(1)	117·9 (12)
Zn(4)–O(4)–N(4)	117·3 (6)	C(7)–C(5)–N(1)	122·9 (12)
O(1)–N(1)–C(5)	114·3 (10)	C(9)–C(8)–N(2)	121·0 (14)
Zn(3)–N(1)–C(5)	133·0 (9)	C(10)–C(8)–N(2)	118·9 (14)
Zn(3)–N(1)–O(1)	112·6 (7)	C(12)–C(11)–N(3)	122·8 (13)
O(2)–N(2)–C(8)	116·2 (10)	C(13)–C(11)–N(3)	119·4 (12)
Zn(4)–N(2)–C(8)	133·1 (10)	C(15)–C(14)–N(4)	123·5 (12)
Zn(4)–N(2)–O(2)	110·5 (6)	C(16)–C(14)–N(4)	118·6 (12)

variation is only from 2·042 (8) to 2·069 (8) Å. These seven Zn—O lengths may be taken to be the same within experimental error, with a mean value of 2·055 (8) Å. This distance is significantly shorter than the mean value of 2·078 (6) Å observed in $(MeZnOMe)_4$. In methylzinc acetoximate, because of the absence of four-membered rings, the angles at O do not depart appreciably from tetrahedral values. The strained environment in the methoxide is thus relaxed and atomic orbitals with greater '*s*' character will be used in bond formation with resultant shortening of the Zn—O bond.

For the extreme Zn—N distances of 2·047 (10) and 2·098 (10) Å the difference must be regarded as significant. Again, however, it is one distance, Zn(3)—N(1), which is appreciably shorter than the rest, the other three values agreeing very closely and having a mean value of 2·090 (10) Å. This mean value is the same as the distance observed in $ZnCl_2[SC(NH_2)NHNH_2]$ where Zn is both tetrahedrally coordinated and, together with N, is part of a five-membered ring (Cavalca, Nardelli & Branchi, 1960). The mean Zn—N distance in $(MeZnNPh_2)_2$ is 2·072 (8) Å (Bell *et al.*, 1983). By analogy with the Zn—O distances the Zn—N distances in the acetoximate would be expected to be shorter than 2·073 Å. However, in this case, the comparison is complicated by the differing hybrid states of both Zn and N in the two compounds.

In each of the four cases, the sum of the three angles at N is 360 ± 1°. This means that there are four individual zinc acetoximate '$Me_2C{=}NOZn$' units which are approximately planar. Mean-plane data have been deposited.* These involve Zn(2), Zn(3) and Zn(4) but not Zn(1) which is not bonded to N. The methyl C atoms attached to Zn also lie near to these planes. This arrangement brings each isopropylidene group into close proximity with the methyl C atoms on the adjacent Zn atom and results in the distortion of the four Zn—N=C angles to values >130°. The requirement of the five-membered rings necessitates a Zn—N—O angle of around 110°, leaving the third angle at N, the O—N=C angle, with a value of ~115°.

Thus in methylzinc acetoximate both N and O can take part in coordination to the metal and it is therefore not surprising that this molecule departs from the cubane arrangement in $(MeZnOMe)_4$ and so avoids the strain of four-membered rings. Using molecular models it is possible to fit together alternative arrangements for $(MeZnON{=}CMe_2)_4$ but all the isomeric structures which have been built retain the feature of two six-membered rings, though arrangements in which each Zn atom has a similar environment, being bonded to two O and one N atom, do seem feasible. The packing is shown in Fig. 2. There are no intermolecular contacts significantly less than the sum of the van der Waals radii.

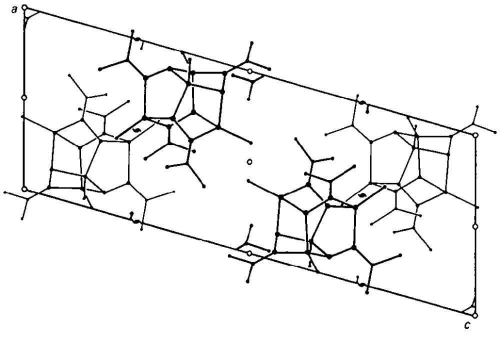

Fig. 2. $(MeZnON{=}CMe_2)_4$ – projection on [010].

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* See deposition footnote.

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Acta Cryst. (1984). C**40**, 616–618

Aquadichloro-μ-[3,6-di(2-pyridyl)pyridazine-*N*,*N'*,*N''*,*N'''*]-μ-hydroxo-nitrato-*O*-dicopper(II) Dichloro-μ-[3,6-di(2-pyridyl)pyridazine-*N*,*N'*,*N''*,*N'''*]-μ-hydroxo-μ-nitrato-*O*,*O'*-dicopper(II) Sesquihydrate, $\{[Cu_2Cl_2(OH)(NO_3)(C_{14}H_{10}N_4)(H_2O)][Cu_2Cl_2(OH)(NO_3)(C_{14}H_{10}N_4)]\}.1{\cdot}5H_2O$

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Abstract. M_r (title formula) = 1067·56, triclinic, $P\bar{1}$, a = 14·500 (3), b = 13·049 (3), c = 10·874 (2) Å, α = 84·8 (1), β = 110·1 (1), γ = 110·5 (1)°, V = 1809 (2) Å^3, Z = 2, D_m (by flotation) = 1·96, D_x = 1·959 g cm^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 26·46 cm^{-1}, $F(000)$ = 1066, T = 293 K, final conventional R is 0·050 over the 2114 unique observed reflections. The asymmetric unit consists of two different neutral dinuclear complexes and of 1·5 solvent molecules. Both complexes contain the tetradentate organic ligand, a bridging hydroxo group and two terminal chlorine ligands. In one complex the copper atoms are five-coordinate, in a distorted square-pyramidal geometry, the two apical sites being occupied by the oxygen atoms of a monodentate nitrato group and of a water molecule, respectively. In the second complex the copper atoms are bridged by a bidentate nitrato group. Such complexes form a dimeric structure in which one of the copper atoms achieves a distorted octahedral geometry, because of a long interaction with the μ-hydroxo oxygen of the second monomeric unit.

Introduction. Binuclear copper(II) complexes with ligands containing nitrogen donor atoms have received increasing attention as models for biological copper(II) systems, concerning the relationships between their structural features and physico-chemical properties. In this field we have already studied complexes obtained using the quadridentate ligand 3,6-di(2-pyridyl)pyridazine and carried out structural determinations of some of them (Ghedini, De Munno, Denti, Manotti Lanfredi & Tiripicchio, 1982; Dapporto, De Munno, Bruno & Romeo, 1983; De Munno, Denti & Dapporto, 1983; Dapporto, De Munno, Sega & Mealli, 1984). As a further contribution we report here the crystal structure of the title complex.

Experimental. Crystals were prepared by treating $[Cu_2Cl_4(C_{14}H_{10}N_4)(H_2O)].H_2O$ (De Munno, Denti & Dapporto, 1983) with hot aqueous 0·001 mol dm^{-3} nitric acid and by slowly evaporating the resulting solution in air. Green parallelepiped crystals, 0·30 × 0·40 × 0·25 mm, Philips PW 1100 diffractometer, graphite monochromator, 25 reflections used for measuring lattice parameters with 7° < θ < 15°, no systematic loss of intensity of the three standard reflections $\bar{5}12$, $\bar{2}2\bar{1}$, $\bar{2}02$, 6355 unique measured reflections with $\theta < 25°$, 2114 with $I > 3\sigma(I)$, θ–2θ scan, $-15 \leq h \leq 15$, $-15 \leq k \leq 15$, $0 \leq l \leq 12$ indices collected, absorption corrected with numerical method according to *SHELX* (Sheldrick, 1976), transmission factors between 0·44 and 0·50, Lp correction, scattering factors for non-hydrogen atoms (*International Tables for X-ray Crystallography,* 1974, p. 99), and for H atoms (Stewart, Davidson & Simpson, 1965), anomalous-dispersion corrections (*International Tables for X-ray Crystallography,* 1974, p. 149); direct methods, full matrix, $\sum w(|F_o| - |F_c|)^2$ minimized, anisotropic thermal parameters for Cu and Cl, isotropic for the other atoms, H of the hydroxy groups and linked molecule (from ΔF synthesis) and the other hydrogens (calculated) given isotropic U of 0·04 Å^2

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