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Dimeric Ethyl(2,6-di-*tert*-butylphenoxy)zinc

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Abstract. Bis(μ-2,6-di-*tert*-butylphenoxy)-bis(ethylzinc), $[Zn(\mu\text{-}C_{14}H_{21}O)(C_2H_5)]_2$, $M_r = 599.51$, monoclinic, $P2_1/n$, $a = 10.244\ (2)$, $b = 9.844\ (1)$, $c = 15.532\ (3)$ Å, $\beta = 92.10\ (1)^\circ$, $V = 1565.2\ (8)$ Å^3, $Z = 2$, $D_x = 1.272$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.594$ mm^{-1}, $F(000) = 640$, $T = 293\ (1)$ K, $R = 0.028$ for 2656 observed reflections with $I > 3\sigma(I)$. The centrosymmetric structure has tricoordinate Zn atoms. The two Zn atoms are bonded to the O atoms of the same two 2,6-di-*tert*-butylphenoxides to form a planar four-membered ring of alternating Zn and O atoms with Zn—O 1.970 (1) and 1.990 (1) Å, Zn···Zn 3.000 (1) Å, Zn—O—Zn 98.44 (5)°, and O—Zn—O 81.56 (5)°. Each Zn is also bonded to an ethyl group, Zn—C 1.949 (2) Å.

Introduction. As part of an effort to determine if organozinc cations could be synthesized by interaction of organozinc compounds with crown ethers or cryptands, we wanted to study the reaction of an alkyl(aryloxy)zinc compound (*R*ZnOAr) with crown ethers. To maximize the possibility that $^-$OAr would be lost [*e.g.* $R\text{ZnOAr} + \text{crown} \rightleftarrows R\text{Zn(crown}^+ + {}^-\text{OAr}]$ and not incorporated into a larger Zn-containing anion [*e.g.* $R\text{Zn(OAr)}_2^-$], we also wanted the O atom to be in a sterically hindered environment. Therefore we synthesized ethyl(2,6-di-*tert*-butylphenoxy)zinc. Because the structure of this phenoxide should be interesting and also because of possible comparisons with structures of some related compounds, we undertook the structural determination of this solid.

Experimental. A hexane solution of diethylzinc (5.0 mL, 1.0 *M*, 5.0 mmol) was added to a solution of 2,6-di-*tert*-butylphenol (1.03 g, 5.0 mmol) in hexane (10 mL). The solution was stirred for 5 min and then the vial left undisturbed. Within several hours, crystals suitable for single-crystal X-ray analysis had formed (1.42 g, 4.74 mmol, 95%): m.p. *ca* 363 K with decomposition. ^{1}H NMR (200 MHz, benzene-d_6, internal standard benzene-d_5 assumed to absorb at δ 7.15): δ 0.54 (q, 2 H, $J = 8.2$ Hz, CH_2), 1.07 (t, 3 H, $J = 8.2$ Hz, $C\mathit{H}_3CH_2$), 1.59 [s, 18 H, $(CH_3)_3C$], 6.91 (t, 1 H, $J = 8.2$ Hz, p-H), 7.31 (d, 2 H, $J = 8.2$ Hz, m-H atoms). ^{13}C NMR [50 MHz, EtOEt–benzene-d_6 (9:1, v:v), internal standard benzene-d_6 assumed to absorb at δ 128.00]: δ 2.08 (CH_2); 11.61 ($\mathit{C}H_3CH_2$); 32.73 [$(\mathit{C}H_3)_3C$]; 35.91 [$\mathit{C}(CH_3)_3$]; 121.10, 126.29, 140.41 and 160.27 (C atoms of aryl ring).

A prismatic crystal with approximate dimensions 0.30 × 0.45 × 0.50 mm, handled under a nitrogen atmosphere, was sealed in a glass capillary. Diffraction data were collected with an Enraf–Nonius CAD-4 automatic diffractometer equipped with a graphite monochromator. Unit-cell parameters indicated a monoclinic crystal system, and systematic absences indicated the space group. Lattice constants were obtained by least-squares refinement of the setting angles of 25 reflections with $10 < \theta < 15^\circ$. Intensity data were collected by the $\omega/2\theta$ scan technique, scan width $(0.80 + 0.35\tan\theta)^\circ$, using variable scan speed (1.5–5.5° min^{-1}) in the range $2 < \theta < 27^\circ$ with h 0→13, k 0→12, and l −19→19. The intensities of 3401 unique reflections (3806 total) were measured, of which 2656 had $I > 3\sigma(I)$ and were used; $R_f = 0.018$. The intensities of three reflections, chosen as standards and measured at regular intervals, decreased by 4.5%; the data were corrected for decay by appropriate scaling. Data were also corrected for Lorentz and polarization effects and for

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empirical absorption (North, Phillips & Mathews, 1968); maximum and minimum correction factors were 0.999 and 0.781, respectively.

The structure was solved by the heavy-atom method and refined by full-matrix least-squares calculations on F. Anisotropic thermal parameters were allowed for non-H atoms. Difference Fourier syntheses calculated at intermediate stages of refinement showed maxima consistent with the expected positions of H atoms. H atoms were included with isotropic temperature factors in subsequent refinement. The refinement converged with $R = 0.028$ and $wR = 0.036$ where $w = 1/[\sigma^2F_o + (0.040F_o)^2]^{1/2}$; 267 parameters were refined. At the conclusion of the refinement, $(\Delta/\sigma)_{\max} = 0.13$, the difference map had no peak larger than $0.24\ \mathrm{e\ Å^{-3}}$, and $S = 1.35$. Atomic scattering factors for non-H atoms were taken from Cromer & Mann (1968) and for H atoms from Stewart, Davidson & Simpson (1965); allowance was made for anomalous dispersion (Cromer & Liberman, 1970) but extinction corrections were ignored. The computer programs used in this study were from the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates, Inc., 1985).

Discussion. Fig. 1 shows a perspective drawing of the structure and indicates the atom-numbering scheme. Fig. 2 shows a stereoview of the crystal packing. Final fractional coordinates and equivalent isotropic thermal parameters for non-H atoms are given in Table 1 and molecular dimensions in Table 2.*

That Zn is only tricoordinate is the most notable feature of the structure. Unusually low coordination numbers have been found for compounds of other metals when bonded to the bulky 2,6-di-*tert*-butylphenoxy group, including Mg (Calabrese, Cushing & Ittel, 1988) and Li (Çetinkaya, Gümrükçü, Lappert, Atwood & Shakir, 1980; Huffman, Geerts & Caulton, 1984) compounds. We are aware of only one other structure having tricoordinate Zn in a four-membered ring with alternating Zn and O atoms: a dimer of EtZnO(Me)C=CHN(^{t}Bu)Et with each Zn also bonded to one N (van Vliet, van Koten, Buysingh, Jastrzebski & Spek, 1987). A related structure is that of a dimer of $MeZnNPh_2$, in which the Zn atoms are bridged by the N atoms to form a four-membered ring (Bell, Shearer & Spencer, 1983). In other structures with tricoordinate Zn, the bonds are to three Fe atoms which are surrounded by bulky groups (Petersen, Ragosta, Whitwell & Burlitch, 1983) or to W, N and C, with, however, an additional contact (2.61 Å) with an O atom (Budzelaar, Alberts-Jansen, Mollema, Boersma, van der Kerk, Spek & Duisenberg, 1983). Another structure with a four-membered ring of alternating Zn and O atoms is formally a dimer of $Et_2ZnO^tBu^-.K^+$; the tetracoordinate Zn atoms share two bridging ^{t}BuO groups, and each Zn is also bonded to two terminal ethyl groups (Fabicon, Parvez & Richey, 1991).

The Zn—C bond length [1.949 (2) Å] is similar to those found both in a variety of organozinc compounds in which Zn has higher coordination numbers (for example, Moseley & Shearer, 1973; Shearer & Spencer, 1980) and in monomeric dialkylzinc compounds in the gas phase (Almenningen, Helgaker, Haaland & Samdal, 1982). The Zn—O bond lengths are 1.970 (1) and 1.990 (1) Å, as would

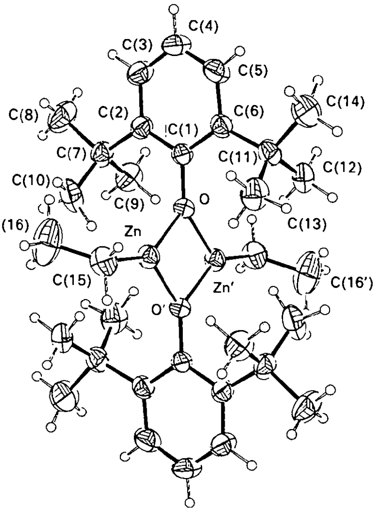

Fig. 1. *ORTEP* (Johnson, 1976) drawing of the dimer indicating the atom labeling. H atoms are assigned arbitary radii, other atoms are shown with 50% probability ellipsoids.

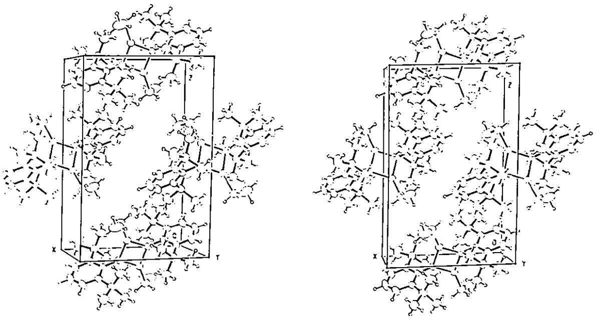

Fig. 2. Stereoscopic drawing of the crystal packing. H atoms are assigned arbitrary radii, other atoms are shown with 50% probability thermal ellipsoids.

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* Tables of H-atom coordinates, anisotropic temperature factors, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54693 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference CR0347]

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Zn	−0.06766 (2)	0.07663 (2)	0.42785 (1)	2.800 (4)
O	−0.1118 (1)	−0.0199 (1)	0.5340 (1)	2.44 (2)
C(1)	−0.2352 (2)	−0.0316 (2)	0.5668 (1)	2.48 (3)
C(2)	−0.2814 (2)	0.0709 (2)	0.6220 (1)	2.87 (4)
C(3)	−0.4085 (2)	0.0580 (2)	0.6496 (2)	3.94 (5)
C(4)	−0.4868 (2)	−0.0484 (3)	0.6262 (2)	4.59 (5)
C(5)	−0.4395 (2)	−0.1481 (3)	0.5744 (2)	4.02 (5)
C(6)	−0.3146 (2)	−0.1447 (2)	0.5437 (1)	2.90 (4)
C(7)	−0.1975 (2)	0.1910 (2)	0.6558 (1)	3.36 (4)
C(8)	−0.2754 (3)	0.2903 (3)	0.7099 (2)	5.31 (6)
C(9)	−0.0857 (2)	0.1377 (3)	0.7156 (2)	4.15 (5)
C(10)	−0.1438 (3)	0.2753 (2)	0.5833 (2)	4.84 (6)
C(11)	−0.2674 (2)	−0.2644 (2)	0.4892 (1)	3.50 (4)
C(12)	−0.1573 (2)	−0.3392 (2)	0.5380 (2)	4.13 (5)
C(13)	−0.2254 (3)	−0.2184 (3)	0.4003 (2)	4.56 (5)
C(14)	−0.3761 (3)	−0.3706 (3)	0.4730 (2)	6.12 (7)
C(15)	−0.1409 (2)	0.1749 (2)	0.3284 (1)	3.95 (5)
C(16)	−0.1972 (4)	0.3120 (3)	0.3429 (2)	6.92 (7)

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

The primed atoms are related to the unprimed atoms by the symmetry operation $-x$, $-y$, $1-z$.

Zn—O	1.970 (1)	C(5)—C(6)	1.382 (3)
Zn—O′	1.990 (1)	C(6)—C(11)	1.539 (3)
Zn—C(15)	1.949 (2)	C(7)—C(8)	1.533 (3)
O—C(1)	1.385 (2)	C(7)—C(9)	1.540 (3)
C(1)—C(2)	1.417 (2)	C(7)—C(10)	1.518 (3)
C(1)—C(6)	1.417 (2)	C(11)—C(12)	1.525 (3)
C(2)—C(3)	1.391 (3)	C(11)—C(13)	1.529 (3)
C(2)—C(7)	1.542 (3)	C(11)—C(14)	1.542 (3)
C(3)—C(4)	1.361 (3)	C(15)—C(16)	1.489 (4)
C(4)—C(5)	1.369 (3)		

O—Zn—O′	81.56 (5)	C(1)—C(6)—C(11)	123.5 (2)
O—Zn—C(15)	144.02 (9)	C(5)—C(6)—C(11)	119.0 (2)
O′—Zn—C(15)	134.49 (8)	C(2)—C(7)—C(8)	112.4 (2)
Zn—O—Zn′	98.44 (5)	C(2)—C(7)—C(9)	109.7 (2)
Zn—O—C(1)	126.0 (1)	C(2)—C(7)—C(10)	112.3 (2)
Zn—O′—C(1′)	135.5 (1)	C(8)—C(7)—C(9)	106.1 (2)
O—C(1)—C(2)	119.5 (2)	C(8)—C(7)—C(10)	105.6 (2)
O—C(1)—C(6)	119.7 (2)	C(9)—C(7)—C(10)	110.6 (2)
C(2)—C(1)—C(6)	120.7 (2)	C(6)—C(11)—C(12)	109.8 (2)
C(1)—C(2)—C(3)	117.3 (2)	C(6)—C(11)—C(13)	112.0 (2)
C(1)—C(2)—C(7)	123.9 (2)	C(6)—C(11)—C(14)	111.7 (2)
C(3)—C(2)—C(7)	118.8 (2)	C(12)—C(11)—C(13)	111.4 (2)
C(2)—C(3)—C(4)	122.5 (2)	C(12)—C(11)—C(14)	105.7 (2)
C(3)—C(4)—C(5)	119.5 (2)	C(13)—C(11)—C(14)	106.1 (2)
C(4)—C(5)—C(6)	122.5 (2)	Zn—C(15)—C(16)	118.1 (2)
C(1)—C(6)—C(5)	117.5 (2)		

be expected, considerably longer than the bonds from Zn to phenoxide O atoms [average 1.887 (6) Å] in bis(2,6-di-*tert*-butylphenoxy)Zn(THF)$_2$ (Geerts, Huffman & Caulton, 1986), in which the Zn is bonded to the O atoms of both phenoxide groups and of both THF (tetrahydrofuran) groups.

The Zn—O—Zn angle is 98.44 (5)°, and the O—Zn—O angle is 81.56 (5)°. The bond lengths and angles of the four-membered ring closely resemble those found in the four-membered ring of the dimer of (2,6-di-*tert*-butylphenoxy)$_2$Mg, whose overall structure is similar except for having terminal phenoxy groups instead of ethyl groups (Calabrese, Cushing & Ittel, 1988). In the four-membered ring of the Mg compound, the average value of Mg—O is 1.964 (3) Å, of Mg—O—Mg is 98.0 (1)°, and of O—Mg—O is 82.0 (1)°. The bond angles also resemble those in the $Et_2ZnO^tBu^-.K^+$ dimer [Zn—O—Zn 100.4 (3)°, O—Zn—O 79.56 (2)°] and in the EtZnO(Me)C=CHN(^{t}Bu)Et dimer [Zn—O—Zn 95.8 (5)°, O—Zn—O 84.2 (5)°]. As expected, however, bond lengths to Zn in the dimer of zincate ion $Et_2ZnO^tBu^-$ [average Zn—C 2.056 (8) Å and average Zn—O 2.094 (4) Å] are longer than those found in this work; bond lengths in the EtZnO(Me)C=CHN(^{t}Bu)Et dimer [Zn—C 1.99 (2) Å, average Zn—O 2.07 (1) Å] lie in between.

Two dimers of (2,6-di-*tert*-butylphenoxy)Li compounds have similar four-membered rings with Li instead of Zn atoms. Each Li is also bonded in one of these (Çentinkaya, Cümrükçü, Lappert, Atwood & Shakir, 1980) to a terminal diethyl ether (each aryl ring also has a 4-methyl group) and in the other (Huffman, Geerts & Caulton, 1984) to a terminal tetrahydrofuran. The *M*—O distances are shorter than in the Zn compound, as expected; moreover, the O—*M*—O angles are larger and the *M*—O—*M* angles correspondingly smaller.

The angle between normals to the planar four-membered ring and the mean plane of the aryl ring [largest deviation from plane is 0.014 Å for C(1)] is 89.01°. As expected for a 1,2,3-trisubstituted aryl ring with such bulky substituents, the atoms bonded to the ring are somewhat out of its mean plane [C(7) 0.097, C(11) 0.085, O −0.079 Å]. The CH$_2$ carbon [C(15)] of the ethyl group is slightly (0.043 Å) out of the plane of the four-membered ring. Torsion angles O—Zn—C(15)—C(16) and O′—Zn—C(15)—C(16) involving the ethyl group and the four-membered ring are 70.7 (1) and −112.3 (1)°, respectively.

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Structure of a New Polymorph of *cis*-$[(\mu\text{-}CH_2)(\mu\text{-}CO)\{Fe(\eta^5\text{-}C_5H_5)CO\}_2]$

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Abstract. μ-Carbonyl-μ-methylene-bis[carbonyl(η^5-cyclopentadienyl)iron](*Fe—Fe*), $[\{Fe(CO)(\eta^5\text{-}C_5H_5\}_2(\mu\text{-}CO)(\mu\text{-}CH_2)]$, $M_r = 339.9$, triclinic, $P\bar{1}$, $a = 6.618$ (2), $b = 9.0321$ (10), $c = 11.750$ (2) Å, $\alpha = 101.68$ (1), $\beta = 99.00$ (2), $\gamma = 103.52$ (2)°, $V = 653.2$ (5) Å^3, $Z = 2$, $D_x = 1.728$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 22.2$ cm^{-1}, $F(000) = 344$, $T = 292$ K, $R = 0.028$ for 1804 observed reflections. The structure reported herein is a different polymorph from the earlier reported monoclinic form [Korswagen, Alt, Speth & Ziegler (1981). *Angew. Chem. Int. Ed. Engl.* **20**, 1049–1051]. The molecular structure of the triclinic form agrees well with that of the monoclinic form, but more precise structural parameters are reported herein, and the bridging methylene H atoms are located. The Fe—Fe bond distance of 2.5196 (6) Å is regarded as a weak bond. The Fe—CH_2 bond distances of 1.965 (4) and 1.967 (3) Å are longer than the Fe—CO bond distances of 1.900 (3) and 1.913 (3) Å corresponding to the bridging carbonyl ligand. The Fe—CH_2—Fe bond angle of 79.7 (1)° is more acute than the Fe—CO—Fe bond angle of 82.7 (1)°. As predicted from molecular orbital calculations [Jemmis, Pinhas & Hoffmann (1980). *J. Am. Chem. Soc.* **102**, 2576–2585], the two bridging units are puckered along the Fe—Fe bond with a fold angle of 162.6 (4)°. The C—H bond lengths are 0.95 (3) and 1.00 (4) Å and the H—C—H bond angle is 110 (3)°. The molecular structure of *cis*-$[(\mu\text{-}CH_2)(\mu\text{-}CO)\{Fe(\eta^5\text{-}C_5H_5)CO\}_2]$ is comparable to the molecular structures of other bridging methylene metal dimers. Also, no statistically significant differences are found in the C—O bond distances of the terminal carbonyl ligands when the methylene bridge is replaced by a carbonyl bridge as in *cis*-$(\mu\text{-}CO)_2[Fe(\eta^5\text{-}C_5H_5)CO]_2$ [Bryan, Greene, Newlands & Field (1970). *J. Chem. Soc. A*, pp. 3068–3074], although the bridging methylene ligand is commonly regarded as a better donor and acceptor than the bridging carbonyl ligand.

Introduction. In assessing the charge density at the bridging C atom in bridging methylene metal dimers with solid-state deuterium NMR spectroscopy (Altbach, Hiyama, Gerson & Butler, 1987) and solid-state ^{13}C NMR spectroscopy (Kim, Altbach & Butler, 1991), we have prepared *cis*-$[(\mu\text{-}CH_2)(\mu\text{-}CO)\{Fe(\eta^5\text{-}C_5H_5)CO\}_2]$, (I). The crystal structure of (I) has been previously reported in brief by Korswagen, Alt, Speth & Ziegler (1981), but little information is presented in the literature. Having obtained a crystal with a different morphology from the previous reported structure we decided to determine the crystal structure of (I). The structure

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