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Bis(2,4,6-trimethylphenyl)zinc(II)

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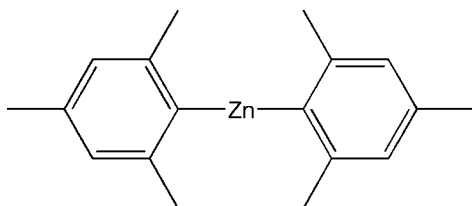
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Key indicators: single-crystal X-ray study; $T = 183$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; disorder in main residue; R factor = 0.071; wR factor = 0.270; data-to-parameter ratio = 19.2.

The title compound, $[\text{Zn}(\text{C}_9\text{H}_{11})_2]$ or Mes_2Zn (Mes = mesityl = 2,4,6-trimethylphenyl), crystallizes with a quarter of a molecule in the asymmetric unit. The Zn^{II} atom is in a strictly linear environment with a $\text{Zn}-\text{C}$ bond length of 1.951 (5) Å. Due to the imposed $2/m$ symmetry, both aromatic rings are coplanar. One of the methyl groups is disordered over two equally occupied positions.

Related literature

For the first synthesis of dimesitylzinc, see: Seidel & Bürger (1981). For related structures, see: Brooker *et al.* (1992); Cole *et al.* (2003); Markies *et al.* (1990); Sun *et al.* (1998); Weidenbruch *et al.* (1989); Westerhausen *et al.* (2005).



Experimental

Crystal data

$[\text{Zn}(\text{C}_9\text{H}_{11})_2]$
 $M_r = 303.73$

Tetragonal, $P4_2/ncm$
 $a = 18.3059$ (9) Å

$c = 5.0494$ (4) Å
 $V = 1692.08$ (18) Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 1.44$ mm⁻¹
 $T = 183$ K
 $0.05 \times 0.05 \times 0.04$ mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
10286 measured reflections
1016 independent reflections
685 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.071$
 $wR(F^2) = 0.270$
 $S = 1.13$
1016 reflections
53 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.60$ e Å⁻³

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2971).

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supplementary materials

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Bis(2,4,6-trimethylphenyl)zinc(II)

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Comment

After the first synthesis of dimesitylzinc by Seidel & Bürger (1981), its structure was determined more than 20 years later (Cole *et al.*, 2003). Here we present another modification of this diarylzinc compound.

Whereas dialkylzinc is monomeric diphenylzinc crystallizes as a loose and unsymmetric dimer (Markies *et al.* (1990)). A planar molecule with a strictly two coordinated zinc centre is observed for bis(2,4,6-trimethylphenyl)zinc (dimesitylzinc) by Cole *et al.* (2003). Other substitution patterns of the arene ring also lead to monomeric, but not strictly linear molecules in the solid state. Sun *et al.* (1998) published the structure of bis(pentafluorophenyl)zinc and Brooker *et al.* (1992) reported the structure of bis[2,4,6-tris(trifluoromethyl)phenyl]zinc with a C—Zn—C bond angle of 170°. The C—Zn—C angle decreases with increasing steric chain and a value of 165.9° was found in bis[2,4,6-tri(*tert*-butylphenyl)]zinc by Westerhausen *et al.* (2005).

Experimental

All manipulations were performed in an atmosphere of argon using standard Schlenk techniques. THF and toluene were dried (Na/benzophenone) and distilled prior to use. Mes₂Zn was prepared according to a literature procedure (Seidel & Bürger, 1981). Recrystallization of Mes₂Zn from toluene at +4°C led to the formation of single crystals of the title compound.

Refinement

All hydrogen atoms were set to idealized positions and were refined with 1.2 times (1.5 for methyl groups) the isotropic displacement parameter of the corresponding carbon atom. One of the methyl groups is disordered over two equally occupied positions. The structure contains solvent accessible voids. But the final difference peak of 1.33 e/Å³ is on a special position and could not be related to a solvent molecule.

Figures

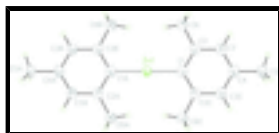


Fig. 1. Molecular structure of Mes₂Zn, showing 40% probability displacement ellipsoids and the atom numbering scheme.

Bis(2,4,6-trimethylphenyl)zinc(II)

Crystal data

[Zn(C₉H₁₁)₂]

Z = 4

supplementary materials

$$M_r = 303.73$$

Tetragonal, $P4_2/ncm$

Hall symbol: -P 4ac 2ac

$$a = 18.3059 (9) \text{ \AA}$$

$$b = 18.3059 (9) \text{ \AA}$$

$$c = 5.0494 (4) \text{ \AA}$$

$$\alpha = 90^\circ$$

$$\beta = 90^\circ$$

$$\gamma = 90^\circ$$

$$V = 1692.08 (18) \text{ \AA}^3$$

$$F_{000} = 640$$

$$D_x = 1.192 \text{ Mg m}^{-3}$$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 10286 reflections

$$\theta = 3.2\text{--}27.5^\circ$$

$$\mu = 1.44 \text{ mm}^{-1}$$

$$T = 183 \text{ K}$$

Octaeder, colourless

$$0.05 \times 0.05 \times 0.04 \text{ mm}$$

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$$T = 183 \text{ K}$$

φ and ω scans

Absorption correction: none

10286 measured reflections

1016 independent reflections

685 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.046$$

$$\theta_{\text{max}} = 27.5^\circ$$

$$\theta_{\text{min}} = 3.2^\circ$$

$$h = -22 \rightarrow 23$$

$$k = -23 \rightarrow 23$$

$$l = -6 \rightarrow 5$$

Refinement

Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.071$$

$$wR(F^2) = 0.270$$

$$S = 1.13$$

1016 reflections

53 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.1807P)^2 + 0.5133P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} < 0.001$$

$$\Delta\rho_{\text{max}} = 1.33 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.60 \text{ e \AA}^{-3}$$

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kF_c [1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.041 (11)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -

factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Zn1	0.0000	0.5000	0.0000	0.0388 (6)	
C1	-0.0524 (2)	0.5524 (2)	-0.2778 (9)	0.0397 (13)	
C2	-0.1176 (2)	0.5250 (2)	-0.3816 (7)	0.0413 (11)	
C3	-0.1542 (2)	0.5623 (2)	-0.5831 (8)	0.0424 (11)	
H3A	-0.1987	0.5431	-0.6500	0.051*	
C4	-0.1268 (2)	0.6268 (2)	-0.6875 (9)	0.0432 (14)	
C5	-0.1687 (2)	0.6687 (2)	-0.8986 (11)	0.0455 (14)	
H5A	-0.1418	0.7132	-0.9452	0.068*	0.50
H5B	-0.2170	0.6820	-0.8302	0.068*	0.50
H5C	-0.1743	0.6381	-1.0563	0.068*	0.50
C6	-0.1516 (2)	0.4566 (2)	-0.2699 (8)	0.0503 (12)	
H6A	-0.1933	0.4422	-0.3799	0.075*	
H6B	-0.1683	0.4659	-0.0886	0.075*	
H6C	-0.1153	0.4172	-0.2687	0.075*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0385 (7)	0.0385 (7)	0.0394 (9)	0.0034 (3)	-0.0015 (2)	0.0015 (2)
C1	0.0435 (19)	0.0435 (19)	0.032 (2)	0.008 (2)	0.0036 (14)	-0.0036 (14)
C2	0.043 (2)	0.044 (2)	0.038 (2)	0.0059 (17)	0.0021 (16)	-0.0033 (16)
C3	0.046 (2)	0.045 (2)	0.0353 (19)	0.0052 (16)	0.0002 (16)	-0.0052 (17)
C4	0.051 (2)	0.051 (2)	0.028 (2)	0.010 (3)	0.0028 (14)	-0.0028 (14)
C5	0.054 (2)	0.054 (2)	0.029 (3)	0.005 (3)	-0.0019 (16)	0.0019 (16)
C6	0.052 (3)	0.045 (2)	0.054 (2)	-0.0005 (18)	-0.0011 (19)	0.0024 (19)

Geometric parameters (\AA , $^\circ$)

Zn1—C1	1.951 (5)	C4—C3 ⁱⁱ	1.386 (5)
Zn1—C1 ⁱ	1.951 (5)	C4—C5	1.522 (7)
C1—C2 ⁱⁱ	1.396 (5)	C5—H5A	0.9800
C1—C2	1.396 (5)	C5—H5B	0.9800
C2—C3	1.397 (6)	C5—H5C	0.9800
C2—C6	1.509 (6)	C6—H6A	0.9800
C3—C4	1.386 (5)	C6—H6B	0.9800
C3—H3A	0.9500	C6—H6C	0.9800
C1—Zn1—C1 ⁱ	179.999 (1)	C4—C5—H5A	109.5
C2 ⁱⁱ —C1—C2	118.1 (5)	C4—C5—H5B	109.5
C2 ⁱⁱ —C1—Zn1	120.9 (2)	H5A—C5—H5B	109.5
C2—C1—Zn1	120.9 (2)	C4—C5—H5C	109.5
C1—C2—C3	120.6 (4)	H5A—C5—H5C	109.5

supplementary materials

C1—C2—C6	120.7 (4)	H5B—C5—H5C	109.5
C3—C2—C6	118.7 (3)	C2—C6—H6A	109.5
C4—C3—C2	121.3 (4)	C2—C6—H6B	109.5
C4—C3—H3A	119.4	H6A—C6—H6B	109.5
C2—C3—H3A	119.4	C2—C6—H6C	109.5
C3 ⁱⁱ —C4—C3	118.1 (5)	H6A—C6—H6C	109.5
C3 ⁱⁱ —C4—C5	120.9 (2)	H6B—C6—H6C	109.5
C3—C4—C5	120.9 (2)		

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-y+1/2, -x+1/2, z$.

Fig. 1

