

## [N-((E)-2-[[2-(Dimethylamino)ethyl]-iminomethyl]phenyl)-N-(2,6-dimethylphenyl)anilino- $\kappa^3$ N,N',N'']ethylzinc

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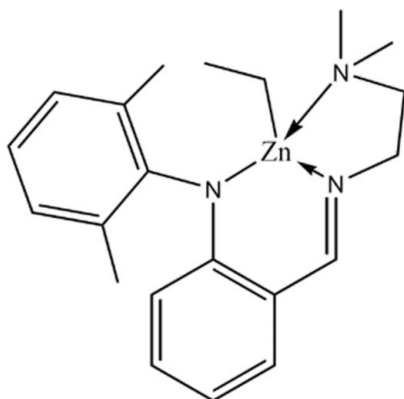
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å; R factor = 0.071; wR factor = 0.189; data-to-parameter ratio = 19.8.

The title ethyl-zinc complex,  $[\text{Zn}(\text{C}_2\text{H}_5)(\text{C}_{19}\text{H}_{24}\text{N}_3)]$ , bears a tridentate anilino-aldimine ligand and features one long Zn–N(amine) bond length, attributable to the crowded environment of the coordinated metal, arising from the dimethylphenyl group. The  $\text{Zn}^{\text{II}}$  ion adopts a distorted tetrahedral geometry, the dihedral angle between the two benzene rings being  $86.05$  ( $16$ )°.

### Related literature

For the synthesis, luminescent properties and applications in catalysis of complexes bearing anilido-aldimine ligands, see: Liu *et al.* (2005, 2006); Ren *et al.* (2007); Su *et al.* (2007); Yao *et al.* (2008).



### Experimental

#### Crystal data

$[\text{Zn}(\text{C}_2\text{H}_5)(\text{C}_{19}\text{H}_{24}\text{N}_3)]$

$M_r = 388.84$

Monoclinic,  $P2_1/c$

$a = 7.3664$  (15) Å

$b = 13.849$  (3) Å

$c = 20.296$  (4) Å

$\beta = 99.26$  (3)°

$V = 2043.5$  (7) Å<sup>3</sup>

$Z = 4$

Mo  $K\alpha$  radiation

$\mu = 1.21$  mm<sup>-1</sup>

$T = 293$  K

$0.23 \times 0.21 \times 0.19$  mm

#### Data collection

Rigaku R-Axis RAPID

diffractometer

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\text{min}} = 0.768$ ,  $T_{\text{max}} = 0.803$

18874 measured reflections

4571 independent reflections

2619 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.116$

#### Refinement

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

$wR(F^2) = 0.189$

$S = 0.96$

4571 reflections

231 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.98$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.63$  e Å<sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

Zn1–C20	1.991 (5)	Zn1–N2	2.487 (4)
Zn1–N1	2.009 (3)	Zn1–N3	2.004 (4)

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (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: BH2336).

### References

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**supplementary materials**

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**[*N*-((*E*)-2-{2-(Dimethylamino)ethyl}iminomethyl)phenyl)-*N*-(2,6-dimethylphenyl)anilido- $\kappa^3$ *N,N',N''*]ethylzinc**

**M. Issa-Madongo and Y. Mu**

**Comment**

Complexes bearing chelating anilido-alimine ligands have recently attracted attention because such ligands have similar framework and combine the steric and electronic characteristics of  $\beta$ -diketiminato and salicyaldiminato ligands, which have been extensively researched in coordination chemistry and catalysis. We have recently reported the luminescent properties of a series of Al(III) (Liu *et al.*, 2005, 2006), Zn<sup>II</sup> (Su *et al.*, 2007) and B(III) (Ren *et al.*, 2007) complexes with chelating anilido-alimine ligands, and catalytic properties of Al(III) complexes (Yao *et al.*, 2008) for the polymerization of  $\epsilon$ -caprolactone. Good results have been obtained. As a part of our study, the preparation and crystal structure of the title ethyl zinc complex bearing *N,N,N''*-tridentate anilido-alimine ligand is reported.

In the complex (Fig. 1), the C7=N1 [1.292 (6) Å] bond length is comparable to those found in similar anilido-imine compounds (Su *et al.*, 2007). The bond distances Zn—N1 [2.009 (3) Å] and Zn—N3 [2.004 (4) Å] are similar to those in other anilido-alimine zinc complexes. The pendant arm bond length Zn—N2 [2.487 (4) Å], in contrast, is longer than equivalent bonds found in other *N,N',O*-tridentate Schiff base Zn complexes. This may be due to the crowded environment in the metal center arising from the aryl group bonded to N3. The dihedral angle between the central benzene ring (C1...C6) and the benzene ring (C12...C17) is 86.05 (16)°.

**Experimental**

The complex was synthesized according to the literature method (Su *et al.*, 2007). A solution of ZnEt<sub>2</sub> (1.0 mol/L, 2.0 mmol) was added to a solution of the ligand (0.59 g, 2.0 mmol) in toluene (30 ml) at 273 K. After stirring for 24 h, the solution was evaporated to dryness and the resulting yellow powder was washed with hexane (Yield: 0.65 g, 90%). Anal. Calcd. for C<sub>21</sub>H<sub>29</sub>N<sub>3</sub>Zn (388.87): C 64.86, H 7.52, N 10.81%; Found: C 64.62, H 7.44, N 10.65%.

**Refinement**

The C-bound H atoms were positioned geometrically with C—H = 0.93 (aromatic and imine carbon), 0.97 (methylene) and 0.96 Å (methyl), and allowed to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = 1.2$  (1.5 for methyl)  $U_{\text{eq}}(\text{C})$ .

Figures

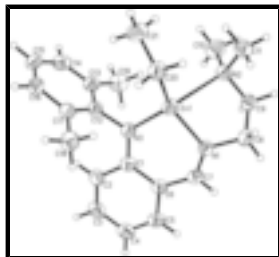


Fig. 1. View of the title complex, with displacement ellipsoids drawn at the 30% probability level.

[N-((E)-2-{[2-(Dimethylamino)ethyl]iminomethyl}phenyl)-N-(2,6-dimethylphenyl)anilino- $\kappa^3N,N',N''$ ]ethylzinc

Crystal data

[Zn(C<sub>2</sub>H<sub>5</sub>)(C<sub>19</sub>H<sub>24</sub>N<sub>3</sub>)]

*M<sub>r</sub>* = 388.84

Monoclinic, *P*2<sub>1</sub>/*c*

Hall symbol: -*P* 2ybc

*a* = 7.3664 (15) Å

*b* = 13.849 (3) Å

*c* = 20.296 (4) Å

β = 99.26 (3)°

*V* = 2043.5 (7) Å<sup>3</sup>

*Z* = 4

*F*(000) = 824

*D<sub>x</sub>* = 1.264 Mg m<sup>-3</sup>

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 10491 reflections

θ = 6.2–54.9°

μ = 1.21 mm<sup>-1</sup>

*T* = 293 K

Block, yellow

0.23 × 0.21 × 0.19 mm

Data collection

Rigaku R-Axis RAPID  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

ω scans

Absorption correction: multi-scan  
(*ABSCOR*; Higashi, 1995)

*T<sub>min</sub>* = 0.768, *T<sub>max</sub>* = 0.803

18874 measured reflections

4571 independent reflections

2619 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.116

θ<sub>max</sub> = 27.5°, θ<sub>min</sub> = 3.1°

*h* = -9→9

*k* = -16→17

*l* = -24→26

Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.071

*wR*(*F*<sup>2</sup>) = 0.189

*S* = 0.96

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/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.097*P*)<sup>2</sup>]

4571 reflections  
 231 parameters  
 0 restraints  
 0 constraints

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

$$(\Delta/\sigma)_{\text{max}} = 0.023$$

$$\Delta\rho_{\text{max}} = 0.98 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.63 \text{ e } \text{\AA}^{-3}$$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7787 (7)	0.8763 (3)	0.8847 (2)	0.0497 (10)
C2	0.7141 (7)	0.8858 (3)	0.8154 (2)	0.0558 (11)
H2	0.7400	0.8373	0.7866	0.067*
C3	0.6141 (8)	0.9649 (4)	0.7893 (3)	0.0672 (14)
H3	0.5740	0.9683	0.7435	0.081*
C4	0.5719 (8)	1.0393 (4)	0.8297 (3)	0.0671 (14)
H4	0.5068	1.0931	0.8115	0.080*
C5	0.6275 (7)	1.0321 (3)	0.8964 (3)	0.0580 (12)
H5	0.5944	1.0808	0.9237	0.070*
C6	0.7337 (6)	0.9537 (3)	0.9266 (2)	0.0483 (10)
C7	0.7748 (7)	0.9542 (3)	0.9974 (2)	0.0512 (11)
H7	0.7175	1.0017	1.0190	0.061*
C8	0.8819 (8)	0.8962 (3)	1.1069 (2)	0.0581 (12)
H8A	0.8122	0.9508	1.1194	0.070*
H8B	1.0066	0.9005	1.1310	0.070*
C9	0.7926 (8)	0.8012 (3)	1.1237 (2)	0.0594 (12)
H9A	0.7967	0.7965	1.1716	0.071*
H9B	0.6646	0.8003	1.1027	0.071*
C10	0.7658 (10)	0.6345 (4)	1.0890 (3)	0.090 (2)
H10A	0.8310	0.5805	1.0745	0.135*
H10B	0.6627	0.6499	1.0554	0.135*
H10C	0.7229	0.6183	1.1299	0.135*
C11	1.0496 (9)	0.6923 (4)	1.1502 (3)	0.0737 (16)
H11A	1.0089	0.6737	1.1910	0.111*
H11B	1.1306	0.7468	1.1584	0.111*
H11C	1.1138	0.6393	1.1339	0.111*
C12	0.9206 (7)	0.7257 (3)	0.8645 (2)	0.0505 (10)
C13	0.8071 (8)	0.6443 (3)	0.8554 (2)	0.0580 (12)
C14	0.8532 (8)	0.5707 (3)	0.8151 (2)	0.0662 (14)
H14	0.7762	0.5174	0.8068	0.079*
C15	1.0110 (9)	0.5750 (3)	0.7871 (3)	0.0701 (16)
H15	1.0435	0.5233	0.7621	0.084*
C16	1.1190 (9)	0.6548 (4)	0.7958 (2)	0.0667 (14)
H16	1.2234	0.6577	0.7755	0.080*
C17	1.0774 (8)	0.7330 (3)	0.8347 (2)	0.0579 (12)
C18	1.1964 (9)	0.8214 (4)	0.8426 (3)	0.0718 (15)
H18A	1.1236	0.8770	0.8275	0.108*
H18B	1.2934	0.8144	0.8165	0.108*
H18C	1.2486	0.8294	0.8887	0.108*

## supplementary materials

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C19	0.6367 (9)	0.6368 (4)	0.8876 (3)	0.0818 (17)
H19A	0.5704	0.5792	0.8724	0.123*
H19B	0.5598	0.6921	0.8758	0.123*
H19C	0.6714	0.6342	0.9353	0.123*
C20	1.2613 (7)	0.7185 (3)	1.0170 (3)	0.0611 (12)
H20A	1.3406	0.7502	0.9898	0.073*
H20B	1.3175	0.7252	1.0634	0.073*
C21	1.2474 (9)	0.6109 (3)	0.9989 (3)	0.0777 (16)
H21A	1.1714	0.5788	1.0263	0.117*
H21B	1.3680	0.5827	1.0062	0.117*
H21C	1.1941	0.6038	0.9528	0.117*
N1	0.8827 (6)	0.8962 (2)	1.03518 (17)	0.0499 (9)
N2	0.8894 (6)	0.7183 (2)	1.10019 (19)	0.0564 (10)
N3	0.8810 (6)	0.8002 (2)	0.91016 (18)	0.0520 (9)
Zn1	1.01743 (8)	0.78379 (3)	1.00320 (2)	0.0531 (2)

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.040 (3)	0.051 (2)	0.058 (3)	0.0013 (17)	0.007 (2)	0.0038 (17)
C2	0.052 (3)	0.063 (2)	0.053 (3)	0.007 (2)	0.010 (2)	0.0040 (19)
C3	0.055 (4)	0.080 (3)	0.066 (3)	0.017 (3)	0.008 (3)	0.021 (2)
C4	0.052 (4)	0.068 (3)	0.083 (4)	0.021 (2)	0.018 (3)	0.023 (2)
C5	0.045 (3)	0.052 (2)	0.079 (3)	0.0110 (19)	0.015 (2)	0.009 (2)
C6	0.039 (3)	0.043 (2)	0.064 (3)	0.0028 (16)	0.011 (2)	0.0048 (17)
C7	0.046 (3)	0.043 (2)	0.066 (3)	0.0022 (17)	0.012 (2)	-0.0009 (18)
C8	0.060 (4)	0.059 (3)	0.055 (3)	0.003 (2)	0.008 (2)	-0.0090 (19)
C9	0.060 (4)	0.064 (3)	0.056 (3)	0.006 (2)	0.015 (2)	0.002 (2)
C10	0.100 (6)	0.065 (3)	0.106 (5)	-0.013 (3)	0.016 (4)	-0.008 (3)
C11	0.076 (5)	0.084 (3)	0.058 (3)	0.022 (3)	-0.001 (3)	0.006 (2)
C12	0.051 (3)	0.050 (2)	0.048 (2)	0.0102 (18)	0.002 (2)	0.0017 (17)
C13	0.057 (4)	0.057 (2)	0.056 (3)	0.010 (2)	-0.001 (2)	0.0015 (19)
C14	0.067 (4)	0.055 (3)	0.069 (3)	0.003 (2)	-0.011 (3)	-0.003 (2)
C15	0.093 (5)	0.055 (3)	0.059 (3)	0.020 (3)	0.002 (3)	-0.010 (2)
C16	0.074 (4)	0.072 (3)	0.056 (3)	0.025 (3)	0.015 (3)	0.002 (2)
C17	0.064 (4)	0.058 (3)	0.050 (3)	0.010 (2)	0.004 (2)	0.0031 (18)
C18	0.065 (4)	0.073 (3)	0.078 (4)	-0.002 (3)	0.016 (3)	0.000 (2)
C19	0.065 (5)	0.081 (3)	0.096 (4)	-0.004 (3)	0.003 (3)	-0.001 (3)
C20	0.051 (3)	0.069 (3)	0.061 (3)	0.014 (2)	0.002 (2)	-0.004 (2)
C21	0.073 (5)	0.066 (3)	0.089 (4)	0.018 (3)	-0.001 (3)	-0.003 (2)
N1	0.050 (3)	0.0491 (18)	0.050 (2)	0.0020 (15)	0.0082 (17)	-0.0017 (14)
N2	0.056 (3)	0.056 (2)	0.056 (2)	0.0045 (17)	0.0019 (19)	-0.0002 (16)
N3	0.055 (3)	0.0526 (18)	0.047 (2)	0.0103 (16)	0.0023 (17)	-0.0017 (14)
Zn1	0.0500 (4)	0.0525 (3)	0.0541 (3)	0.0111 (2)	0.0003 (2)	-0.0051 (2)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

C1—N3	1.349 (5)	C12—C17	1.391 (7)
C1—C2	1.417 (6)	C12—C13	1.398 (7)

C1—C6	1.441 (6)	C12—N3	1.447 (5)
C2—C3	1.376 (6)	C13—C14	1.383 (6)
C2—H2	0.9300	C13—C19	1.510 (8)
C3—C4	1.384 (8)	C14—C15	1.375 (8)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.353 (7)	C15—C16	1.356 (8)
C4—H4	0.9300	C15—H15	0.9300
C5—C6	1.418 (6)	C16—C17	1.404 (6)
C5—H5	0.9300	C16—H16	0.9300
C6—C7	1.419 (6)	C17—C18	1.499 (7)
C7—N1	1.292 (6)	C18—H18A	0.9600
C7—H7	0.9300	C18—H18B	0.9600
C8—N1	1.457 (5)	C18—H18C	0.9600
C8—C9	1.533 (6)	C19—H19A	0.9600
C8—H8A	0.9700	C19—H19B	0.9600
C8—H8B	0.9700	C19—H19C	0.9600
C9—N2	1.471 (6)	C20—C21	1.534 (6)
C9—H9A	0.9700	Zn1—C20	1.991 (5)
C9—H9B	0.9700	C20—H20A	0.9700
C10—N2	1.470 (7)	C20—H20B	0.9700
C10—H10A	0.9600	C21—H21A	0.9600
C10—H10B	0.9600	C21—H21B	0.9600
C10—H10C	0.9600	C21—H21C	0.9600
C11—N2	1.472 (7)	Zn1—N1	2.009 (3)
C11—H11A	0.9600	Zn1—N2	2.487 (4)
C11—H11B	0.9600	Zn1—N3	2.004 (4)
C11—H11C	0.9600		
N3—C1—C2	122.1 (4)	C13—C14—H14	119.4
N3—C1—C6	121.5 (4)	C16—C15—C14	119.9 (4)
C2—C1—C6	116.4 (4)	C16—C15—H15	120.0
C3—C2—C1	122.0 (4)	C14—C15—H15	120.0
C3—C2—H2	119.0	C15—C16—C17	121.7 (5)
C1—C2—H2	119.0	C15—C16—H16	119.1
C2—C3—C4	121.5 (5)	C17—C16—H16	119.1
C2—C3—H3	119.3	C12—C17—C16	117.4 (5)
C4—C3—H3	119.3	C12—C17—C18	121.7 (4)
C5—C4—C3	118.5 (4)	C16—C17—C18	120.9 (5)
C5—C4—H4	120.8	C17—C18—H18A	109.5
C3—C4—H4	120.8	C17—C18—H18B	109.5
C4—C5—C6	123.1 (4)	H18A—C18—H18B	109.5
C4—C5—H5	118.5	C17—C18—H18C	109.5
C6—C5—H5	118.5	H18A—C18—H18C	109.5
C5—C6—C7	116.6 (4)	H18B—C18—H18C	109.5
C5—C6—C1	118.6 (4)	C13—C19—H19A	109.5
C7—C6—C1	124.7 (4)	C13—C19—H19B	109.5
N1—C7—C6	127.6 (4)	H19A—C19—H19B	109.5
N1—C7—H7	116.2	C13—C19—H19C	109.5
C6—C7—H7	116.2	H19A—C19—H19C	109.5
N1—C8—C9	107.1 (4)	H19B—C19—H19C	109.5

## supplementary materials

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N1—C8—H8A	110.3	C21—C20—Zn1	112.5 (4)
C9—C8—H8A	110.3	C21—C20—H20A	109.1
N1—C8—H8B	110.3	Zn1—C20—H20A	109.1
C9—C8—H8B	110.3	C21—C20—H20B	109.1
H8A—C8—H8B	108.6	Zn1—C20—H20B	109.1
N2—C9—C8	110.5 (4)	H20A—C20—H20B	107.8
N2—C9—H9A	109.6	C20—C21—H21A	109.5
C8—C9—H9A	109.6	C20—C21—H21B	109.5
N2—C9—H9B	109.6	H21A—C21—H21B	109.5
C8—C9—H9B	109.6	C20—C21—H21C	109.5
H9A—C9—H9B	108.1	H21A—C21—H21C	109.5
N2—C10—H10A	109.5	H21B—C21—H21C	109.5
N2—C10—H10B	109.5	C7—N1—C8	119.6 (4)
H10A—C10—H10B	109.5	C7—N1—Zn1	125.3 (3)
N2—C10—H10C	109.5	C8—N1—Zn1	113.8 (3)
H10A—C10—H10C	109.5	C10—N2—C9	110.2 (4)
H10B—C10—H10C	109.5	C10—N2—C11	108.7 (4)
N2—C11—H11A	109.5	C9—N2—C11	110.1 (4)
N2—C11—H11B	109.5	C10—N2—Zn1	118.1 (3)
H11A—C11—H11B	109.5	C9—N2—Zn1	103.7 (2)
N2—C11—H11C	109.5	C11—N2—Zn1	105.7 (3)
H11A—C11—H11C	109.5	C1—N3—C12	118.0 (4)
H11B—C11—H11C	109.5	C1—N3—Zn1	127.9 (3)
C17—C12—C13	121.4 (4)	C12—N3—Zn1	113.6 (3)
C17—C12—N3	119.6 (4)	C20—Zn1—N3	119.23 (18)
C13—C12—N3	118.8 (4)	C20—Zn1—N1	142.01 (18)
C14—C13—C12	118.3 (5)	N3—Zn1—N1	91.01 (14)
C14—C13—C19	120.3 (5)	C20—Zn1—N2	99.88 (18)
C12—C13—C19	121.3 (4)	N3—Zn1—N2	126.21 (16)
C15—C14—C13	121.1 (5)	N1—Zn1—N2	75.94 (13)
C15—C14—H14	119.4		



Fig. 1

