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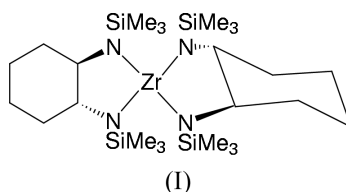
## Key indicators

Single-crystal X-ray study  
 $T = 298\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.012\text{ \AA}$   
 $R$  factor = 0.047  
 $wR$  factor = 0.109  
Data-to-parameter ratio = 14.9For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Bis[(1*R*,2*R*)-(–)-1,2-bis(trimethylsilylamino)-  
cyclohexane(2–)- $\kappa^2\text{N,N}'$ ]zirconium(IV)

The title complex, [(1*R*,2*R*)-(–)-1,2-(NSiMe<sub>3</sub>)<sub>2</sub>-C<sub>6</sub>H<sub>10</sub>]<sub>2</sub>Zr or [Zr(C<sub>12</sub>H<sub>28</sub>N<sub>2</sub>Si<sub>2</sub>)<sub>2</sub>], can be viewed as a tetrahedron located on a non-crystallographic twofold axis, which passes through the Zr atom and between the two N atoms of each cyclohexane-based ligand. The amide N atoms form two almost perpendicular planes with the central Zr atom, with a dihedral angle of 87.23 (18)°.

## Comment

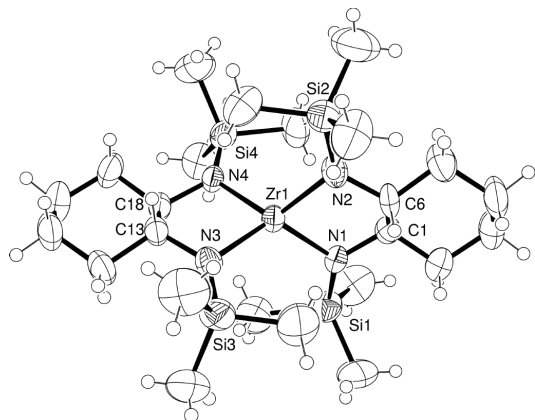
Group IV metal complexes containing bidentate bis(amide) or tetradentate bis(amidinate) ligands are promising systems for applications in catalysis because of their relationship to the well studied metallocene analogues Cp<sub>2</sub>MX<sub>2</sub>, bis(amide) (R<sub>2</sub>N)<sub>2</sub>MX<sub>2</sub> (Minhas *et al.*, 1996), and bis(amidinate) [N(R)C(R')N(R)]<sub>2</sub>MX<sub>2</sub> (Bambirra *et al.*, 2001; Foley *et al.*, 2000). An attractive goal in this area is to develop chiral metal complexes for exploitation in stereoselective catalysis (Hagadorn & Arnold, 1998). (1*R*,2*R*)-Diaminocyclohexane has proved a useful building block for a broad range of chiral reagents (Larrow *et al.*, 1994). Recently, we reported the chemistry of its lithium derivatives, including mono- and dilithium (1*R*,2*R*)-(–)-1,2-(NHSiMe<sub>3</sub>)<sub>2</sub>-C<sub>6</sub>H<sub>10</sub>, and grew single crystals suitable for X-ray diffraction analysis (Li *et al.*, 2002). Using this approach for the preparation of transition-metal complexes, we now report the synthesis and structure of the zirconium bis(bisamide) title complex, (I).



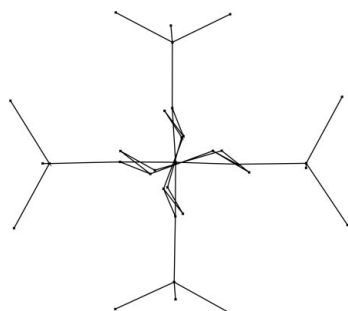
The molecular structure of (I) is shown in Fig. 1. The two cyclohexane rings adopt chair conformations. The molecule of (I) has a non-crystallographic twofold axis, which passes through the Zr atom and between the pairs of N atoms, N1 and N2, and N3 and N4 (Fig. 2). The structure can be viewed as a tetrahedron, with the metal ion in the center, bonded to two bidentate ligands.

All the amide N atoms are  $sp^2$ -hybridized (for example, the sum of the angles around N1 is 358.5°). The geometry around Zr is distorted tetrahedral, with the four Zr–N bond distances in the range 2.058 (4)–2.077 (5) Å and similar to literature values (Lee *et al.*, 2000). The five-membered Zr–N–C–C–N rings adopt an envelope conformation. From Table 1, we note that the intraannular N–Zr–N angles, N1–Zr1–N2 [86.8 (2)°] and N3–Zr1–N4 [86.72 (19)°], are significantly

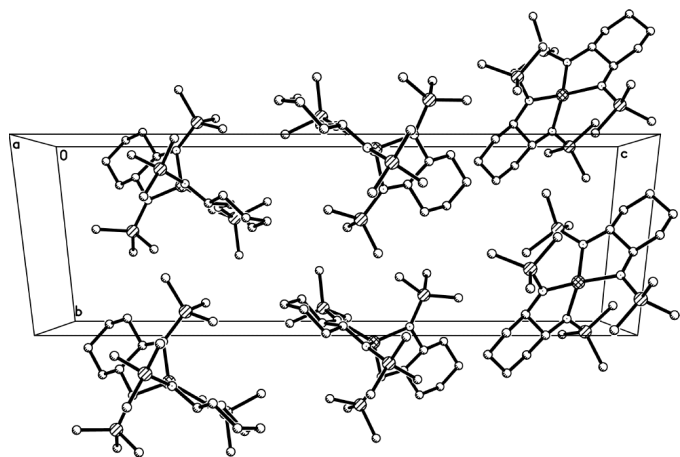
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**Figure 1**  
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by small spheres of arbitrary radii.



**Figure 2**  
A diagram of the molecule of (I), viewed along the non-crystallographic twofold axis. All H atoms have been omitted for clarity.



**Figure 3**  
A packing diagram of the title complex.

smaller than the ideal tetrahedral value due to chelation, while the other N—Zr—N angles between the two chelate rings are correspondingly larger. The N1—Zr1—N2 and N3—Zr1—N4 planes are almost perpendicular, the dihedral angle being 87.2 (2)°. In addition, the two NSiMe<sub>3</sub> groups related by the

approximate C<sub>2</sub> axis are symmetrically placed above and below the opposite N—Zr—N plane, as in the complex {(±)-*trans*-1,2-(NSiMe<sub>3</sub>)<sub>2</sub>-C<sub>6</sub>H<sub>10</sub>}TiI<sub>2</sub> (Tsuie *et al.*, 1997).

## Experimental

Li<sub>2</sub>[(1*R*,2*R*)-(–)-1,2-(NSiMe<sub>3</sub>)<sub>2</sub>-C<sub>6</sub>H<sub>10</sub>] can be converted to its sodium salt easily in hexane. When two equivalents of Na<sup>t</sup>Bu were added to the clear solution of the dilithium salt at room temperature, a white precipitate formed immediately. The mixture was stirred overnight and filtered. The white precipitate which was isolated was dried in a vacuum to give Na<sub>2</sub>[(1*R*,2*R*)-(–)-1,2-(NSiMe<sub>3</sub>)<sub>2</sub>-C<sub>6</sub>H<sub>10</sub>]. Treatment of Na<sub>2</sub>[(1*R*,2*R*)-(–)-1,2-(NSiMe<sub>3</sub>)<sub>2</sub>-C<sub>6</sub>H<sub>10</sub>] with a half equivalent of ZrCl<sub>4</sub> in toluene at low temperature gave a yellow solution. After filtering, the reaction afforded a colorless crystalline product in high yield. Spectroscopic analysis: <sup>1</sup>H NMR (CDCl<sub>3</sub>, δ, p.p.m.): 0.02 (*s*, 36H, SiMe<sub>3</sub>), 1.15 (*m*, 8H, CH), 1.59 (*m*, 8H, CH), 3.56 (*m*, 4H, CH); <sup>13</sup>C NMR (CDCl<sub>3</sub>, δ, p.p.m.): 2.42, 25.83, 36.26, 65.95.

## Crystal data

[Zr(C<sub>12</sub>H<sub>28</sub>N<sub>2</sub>Si<sub>2</sub>)<sub>2</sub>]  
M<sub>r</sub> = 604.31  
Trigonal, P3<sub>1</sub>  
a = 10.473 (5) Å  
c = 28.166 (18) Å  
V = 2676 (2) Å<sup>3</sup>  
Z = 3  
D<sub>x</sub> = 1.125 Mg m<sup>-3</sup>

Mo Kα radiation  
Cell parameters from 956 reflections  
θ = 2.2–27.0°  
μ = 0.46 mm<sup>-1</sup>  
T = 298 (2) K  
Block, colorless  
0.35 × 0.30 × 0.10 mm

## Data collection

Bruker SMART CCD area-detector diffractometer  
ω scans  
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)  
T<sub>min</sub> = 0.856, T<sub>max</sub> = 0.956  
11 157 measured reflections

4626 independent reflections  
4526 reflections with I > 2σ(I)  
R<sub>int</sub> = 0.038  
θ<sub>max</sub> = 25.0°  
h = -12 → 11  
k = -12 → 12  
l = -33 → 20

## Refinement

Refinement on F<sup>2</sup>  
R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.047  
wR(F<sup>2</sup>) = 0.109  
S = 1.28  
4626 reflections  
310 parameters  
H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0337P)<sup>2</sup> + 2.5967P]  
where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> = 0.003  
Δρ<sub>max</sub> = 0.48 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -1.04 e Å<sup>-3</sup>  
Absolute structure: Flack (1983),  
1477 Friedel pairs  
Flack parameter = 0.03 (6)

**Table 1**

Selected geometric parameters (Å, °).

Zr1—N4	2.058 (4)	Zr1—N1	2.064 (5)
Zr1—N2	2.064 (5)	Zr1—N3	2.077 (5)
N4—Zr1—N2	119.06 (18)	N4—Zr1—N3	86.72 (19)
N4—Zr1—N1	123.59 (19)	N2—Zr1—N3	123.3 (2)
N2—Zr1—N1	86.8 (2)	N1—Zr1—N3	121.71 (19)
N2—Zr1—N1—C1	-10.2 (4)	N3—Zr1—N4—C18	-10.5 (4)
N1—Zr1—N2—C6	-12.9 (4)	N1—C1—C6—N2	-44.0 (7)
N4—Zr1—N3—C13	-13.0 (4)	N3—C13—C18—N4	-45.9 (7)

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.98 Å and U<sub>iso</sub>(H) = 1.5U<sub>eq</sub>(C), but each group was allowed to rotate freely about its C—C bond. The position of the

amine H atom was refined freely along with an isotropic displacement parameter. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances in the range 0.95–1.00 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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