

{ μ -[Dimethyl(1,1,2-trimethylpropyl)-silyl](2-pyridylmethyl)amido}bis[methyl-zinc(II)]

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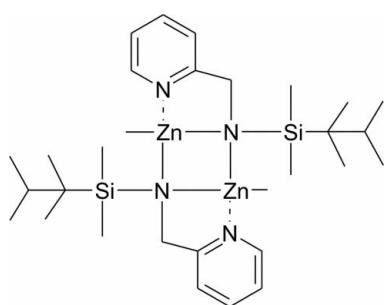
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Key indicators: single-crystal X-ray study; $T = 183$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.041; wR factor = 0.097; data-to-parameter ratio = 20.9.

Zincation of (dimethylhexylsilyl)(2-pyridylmethyl)amine (hexyl = 1,1,2-trimethylpropyl) with dimethylzinc in toluene yields the dimeric title compound, $[\text{Zn}_2(\text{CH}_3)_2(\text{C}_{14}\text{H}_{25}\text{N}_2\text{Si})_2]$. Each Zn atom is coordinated tetrahedrally by three N atoms and a methyl group. The molecule shows inversion symmetry. The central Zn_2N_2 ring is planar, with an $\text{N}-\text{Zn}-\text{N}$ angle of 95.33 (9)°. The exocyclic $\text{Zn}-\text{C}$ bond length of 1.995 (3) Å has a characteristic value.

Related literature

For related literature, see: Westerhausen *et al.* (2001, 2002).



Experimental

Crystal data

$[\text{Zn}_2(\text{CH}_3)_2(\text{C}_{14}\text{H}_{25}\text{N}_2\text{Si})_2]$	$V = 1641.2$ (6) Å ³
$M_r = 659.71$	$Z = 2$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 9.2939$ (19) Å	$\mu = 1.56$ mm ⁻¹
$b = 9.888$ (2) Å	$T = 183$ (2) K
$c = 18.124$ (4) Å	$0.05 \times 0.05 \times 0.04$ mm
$\beta = 99.83$ (3)°	

Data collection

Nonius KappaCCD diffractometer	3737 independent reflections
Absorption correction: none	2725 reflections with $I > 2\sigma(I)$
10869 measured reflections	$R_{\text{int}} = 0.061$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$	179 parameters
$wR(F^2) = 0.097$	H-atom parameters constrained
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.46$ e Å ⁻³
3737 reflections	$\Delta\rho_{\text{min}} = -0.46$ 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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); 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: DN2241).

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supplementary materials

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{ μ -[Dimethyl(1,1,2-trimethylpropyl)silyl](2-pyridylmethyl)amido}bis[methylzinc(II)]

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Comment

In the past, metallated (2-pyridylmethyl)(trialkylsilyl)amines were used for oxidative C–C coupling reactions. Zincation of (2-pyridylmethyl)(trialkylsilyl)amine (**A**; trialkylsilyl=Me₂tertBu, iPr₃) yields dimeric methylzinc-(2-pyridylmethyl)(trialkylsilyl)amide (**B**). Further addition of dimethylzinc to a toluene solution to **A** at raised temperatures yields the C–C coupling product bis(methylzinc)-[1,2-dipyridyl-1,2-bis(trialkylsilylamido)ethane] (Westerhausen *et al.*, 2002). The synthesis of methylzinc-(2-pyridylmethyl)(dimethylhexylsilyl)amide (**1**, thexyl=1,1,2-trimethylpropyl) is similar (Westerhausen *et al.*, 2002). Neither thermal decomposition nor the addition of an other equivalent of dimethyl zinc at elevated temperature initiates an oxidative C–C coupling reaction. Two (2-pyridylmethyl)(dimethylhexylsilyl)amines adopt bridging position between two methylzinc units forming a centrosymmetric four-membered ZnNⁱZnⁱNⁱ ring [symmetry code:(i) 1 – x , – y , – z]. The amine reacts as a bidentate ligand. The transannular Zn···Znⁱ distance of 2.8435 (9) Å compares as well to the transannular Zn···Znⁱ distance of 2.848 (1) Å in dimeric methylzinc-(2-pyridylmethyl)(triisopropylsilyl)amide (Westerhausen *et al.*, 2002). The zinc atoms are distorted tetrahedral coordinated by three nitrogen atoms and one methyl group. The Zn–N(1) distance of 2.100 (2) Å and Zn···N(2) of 2.113 (2) Å show characteristic values (Westerhausen *et al.*, 2001; Westerhausen *et al.*, 2002).

Experimental

All manipulations were carried out in an atmosphere of argon using standard Schlenk techniques. Toluene and pentane were dried (Na/benzophenone) and distilled prior to use. 2-Pyridylmethylamine and butyllithium were purchased from Aldrich. Dimethylhexylchlorosilane was purchased from Merck.¹H NMR and ¹³C NMR spectra were recorded at [C₆D₆]benzene solution at ambient temperature on a Bruker AC 400 MHz spectrometer and were referenced to deuterated benzene as an internal standard.

Methylzinc-(2-pyridylmethyl)(dimethylhexylsilyl)amide was prepared according to a literature procedure (Westerhausen *et al.* 2002) and recrystallized from pentane. After reduction of the volume to a third of the original volume crystals precipitated.

Physical data:

¹H NMR (200 MHz, [D₆]benzene) δ = 8.10 (d, ³J(H¹,H²) = 4.8, 1H, Pyr1); 6.79 (dt, ⁵J(H³,H¹) = 1.6, ³J(H³,H^{2/4}) = 7.8, 1H, Pyr3); 6.51 (d, ³J(H⁴,H³) = 8.0, 1H, Pyr4); 6.41 (t, ³J(H¹,H³) = 6.4, 1H, Pyr2); 4.64 (s, 2H, CH₂); 1.87 (m, 1H, CH); 1.13 (s, 6H, SiC(CH₃)₂); 1.02 (d, 6H, SiCCH(<u>CH₃</u>)₂<u>CH₃</u>); 0.15 (s, 6H, Si(CH₃)); –0.12 (s, 3H, ZnCH₃).

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^{13}C NMR (50 MHz, [D6]benzene) δ = 166.16 (Pyr5); 146.33 (Pyr1); 137.46 (Pyr3); 121.72 (Pyr2); 121.72 (Pyr4); 54.56 (2J , CH2); 34.75 (SiCC(CH₃)₂); 27.11 (SiC(CH₃)₂); 22.52 (SiCC(CH₃)₂); 19.09 (SiC(CH₃)₂)) −0.56 ((Si(CH₃)₂); −12.56 (ZnCH₃).

MS (DEI, m/z [%]): 644 (M—CH₄, 5), 575 (M—C(CH₃)₂CH(CH₃)₂), 477 (M-(C₆H₆N)₂, 100); 330 (M/2, 2); 245 (M/2-(C₆H₁₃), 10); 165 (C₈H₁₃N₂Si, 26).

IR (cm^{−1}): 3374, 3065, 3003, 2958, 2925, 2855, 1592, 1570, 1465, 1433, 1405, 1377, 1347, 1249, 1125, 1046, 995, 825, 774.

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. The methyl groups were allowed to rotate but not to tip.

Figures

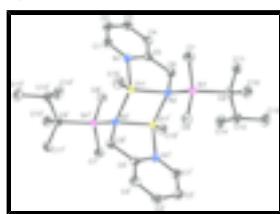


Fig. 1. The molecular structure of **1**, showing 40% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity. [Symmetry code: (i) $-x + 1, -y, -z$].

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Crystal data

[Zn ₂ (CH ₃) ₂ (C ₁₄ H ₂₅ N ₂ Si) ₂]	$F_{000} = 704$
$M_r = 659.71$	$D_x = 1.335 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
Hall symbol: -P 2yn	$\lambda = 0.71073 \text{ \AA}$
$a = 9.2939 (19) \text{ \AA}$	Cell parameters from 10869 reflections
$b = 9.888 (2) \text{ \AA}$	$\theta = 2.3\text{--}27.4^\circ$
$c = 18.124 (4) \text{ \AA}$	$\mu = 1.56 \text{ mm}^{-1}$
$\beta = 99.83 (3)^\circ$	$T = 183 (2) \text{ K}$
$V = 1641.2 (6) \text{ \AA}^3$	Prism, colourless
$Z = 2$	$0.05 \times 0.05 \times 0.04 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer	2725 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.061$
Monochromator: graphite	$\theta_{\text{max}} = 27.4^\circ$

$T = 183(2)$ K	$\theta_{\min} = 2.3^\circ$
φ and ω scans	$h = -12 \rightarrow 10$
Absorption correction: none	$k = -11 \rightarrow 12$
10869 measured reflections	$l = -23 \rightarrow 22$
3737 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.097$	$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{\max} = 0.013$
3737 reflections	$\Delta\rho_{\max} = 0.46 \text{ e } \text{\AA}^{-3}$
179 parameters	$\Delta\rho_{\min} = -0.46 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.65416 (3)	0.01284 (3)	0.007395 (18)	0.02359 (12)
Si1	0.56248 (8)	0.06666 (7)	0.16433 (4)	0.02352 (19)
N1	0.7051 (2)	-0.1940 (2)	0.01801 (13)	0.0246 (5)
N2	0.5159 (2)	-0.0342 (2)	0.08506 (13)	0.0230 (5)
C1	0.7981 (3)	-0.2629 (3)	-0.01697 (17)	0.0307 (7)
H1A	0.8574	-0.2143	-0.0456	0.037*
C2	0.8108 (3)	-0.4016 (3)	-0.01292 (17)	0.0354 (7)
H2A	0.8785	-0.4475	-0.0379	0.043*
C3	0.7235 (4)	-0.4732 (3)	0.02802 (19)	0.0356 (7)
H3A	0.7283	-0.5691	0.0307	0.043*
C4	0.6292 (3)	-0.4019 (3)	0.06490 (17)	0.0297 (7)
H4A	0.5689	-0.4487	0.0938	0.036*
C5	0.6226 (3)	-0.2616 (3)	0.05964 (15)	0.0232 (6)

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C6	0.5276 (3)	-0.1805 (3)	0.10344 (17)	0.0274 (6)
H6A	0.4282	-0.2199	0.0945	0.033*
H6B	0.5670	-0.1902	0.1575	0.033*
C7	0.7601 (3)	0.0335 (3)	0.20441 (19)	0.0376 (7)
H7A	0.8220	0.0674	0.1698	0.056*
H7B	0.7755	-0.0640	0.2116	0.056*
H7C	0.7855	0.0798	0.2527	0.056*
C8	0.5454 (3)	0.2484 (3)	0.13393 (17)	0.0299 (7)
H8A	0.5789	0.2583	0.0858	0.045*
H8B	0.6054	0.3051	0.1716	0.045*
H8C	0.4431	0.2766	0.1285	0.045*
C9	0.4582 (3)	0.0474 (3)	0.24740 (16)	0.0285 (6)
C10	0.5223 (4)	0.1584 (3)	0.30329 (18)	0.0397 (8)
H10A	0.4854	0.1465	0.3504	0.060*
H10B	0.4933	0.2475	0.2822	0.060*
H10C	0.6291	0.1516	0.3128	0.060*
C11	0.4913 (4)	-0.0908 (3)	0.28696 (18)	0.0410 (8)
H11A	0.4526	-0.0913	0.3340	0.062*
H11B	0.5971	-0.1054	0.2976	0.062*
H11C	0.4450	-0.1632	0.2543	0.062*
C12	0.2897 (3)	0.0733 (3)	0.22700 (18)	0.0337 (7)
H12A	0.2767	0.1667	0.2057	0.040*
C13	0.2119 (4)	0.0692 (4)	0.2954 (2)	0.0469 (9)
H13A	0.1095	0.0953	0.2802	0.070*
H13B	0.2598	0.1322	0.3336	0.070*
H13C	0.2170	-0.0226	0.3160	0.070*
C14	0.2123 (4)	-0.0213 (4)	0.1687 (2)	0.0487 (9)
H14A	0.1094	0.0051	0.1558	0.073*
H14B	0.2189	-0.1138	0.1884	0.073*
H14C	0.2580	-0.0171	0.1239	0.073*
C15	0.8221 (3)	0.1412 (3)	0.01791 (19)	0.0343 (7)
H15A	0.9128	0.0905	0.0183	0.051*
H15B	0.8280	0.1916	0.0649	0.051*
H15C	0.8079	0.2045	-0.0243	0.051*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.02182 (19)	0.02322 (18)	0.0269 (2)	-0.00257 (12)	0.00748 (13)	0.00065 (13)
Si1	0.0224 (4)	0.0245 (4)	0.0237 (4)	0.0010 (3)	0.0039 (3)	0.0013 (3)
N1	0.0205 (12)	0.0269 (12)	0.0263 (14)	-0.0001 (9)	0.0038 (10)	-0.0019 (9)
N2	0.0226 (12)	0.0198 (10)	0.0278 (13)	0.0020 (9)	0.0077 (10)	0.0032 (9)
C1	0.0237 (15)	0.0373 (16)	0.0313 (18)	0.0020 (12)	0.0048 (13)	-0.0014 (13)
C2	0.0340 (18)	0.0381 (17)	0.0344 (19)	0.0116 (13)	0.0063 (15)	-0.0060 (14)
C3	0.0442 (19)	0.0231 (14)	0.0371 (19)	0.0061 (13)	0.0005 (15)	-0.0014 (13)
C4	0.0310 (16)	0.0258 (14)	0.0310 (17)	-0.0020 (12)	0.0012 (13)	0.0015 (12)
C5	0.0193 (14)	0.0248 (13)	0.0242 (15)	-0.0003 (10)	0.0000 (12)	0.0002 (11)
C6	0.0296 (16)	0.0244 (13)	0.0300 (17)	0.0014 (11)	0.0104 (13)	0.0041 (12)

C7	0.0295 (17)	0.0455 (18)	0.0372 (19)	0.0048 (13)	0.0040 (15)	0.0052 (14)
C8	0.0304 (17)	0.0279 (14)	0.0308 (17)	-0.0051 (12)	0.0034 (14)	-0.0009 (12)
C9	0.0339 (17)	0.0307 (14)	0.0211 (16)	0.0044 (12)	0.0052 (13)	0.0014 (12)
C10	0.0398 (19)	0.0473 (19)	0.0318 (19)	0.0033 (14)	0.0050 (15)	-0.0065 (14)
C11	0.054 (2)	0.0410 (18)	0.0319 (19)	0.0110 (15)	0.0190 (17)	0.0105 (14)
C12	0.0299 (17)	0.0352 (16)	0.0390 (19)	-0.0007 (12)	0.0142 (15)	0.0026 (14)
C13	0.043 (2)	0.060 (2)	0.042 (2)	0.0032 (16)	0.0207 (17)	-0.0012 (17)
C14	0.0339 (19)	0.072 (2)	0.043 (2)	-0.0029 (17)	0.0149 (17)	-0.0074 (18)
C15	0.0277 (16)	0.0328 (15)	0.044 (2)	-0.0069 (12)	0.0093 (15)	-0.0019 (14)

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

Zn1—C15	1.995 (3)	C7—H7C	0.9800
Zn1—N1	2.100 (2)	C8—H8A	0.9800
Zn1—N2 ⁱ	2.109 (3)	C8—H8B	0.9800
Zn1—N2	2.113 (2)	C8—H8C	0.9800
Zn1—Zn1 ⁱ	2.8435 (8)	C9—C10	1.542 (4)
Si1—N2	1.742 (2)	C9—C11	1.550 (4)
Si1—C8	1.878 (3)	C9—C12	1.567 (4)
Si1—C7	1.885 (3)	C10—H10A	0.9800
Si1—C9	1.934 (3)	C10—H10B	0.9800
N1—C5	1.343 (3)	C10—H10C	0.9800
N1—C1	1.342 (3)	C11—H11A	0.9800
N2—C6	1.484 (3)	C11—H11B	0.9800
N2—Zn1 ⁱ	2.109 (3)	C11—H11C	0.9800
C1—C2	1.377 (4)	C12—C14	1.500 (4)
C1—H1A	0.9500	C12—C13	1.539 (4)
C2—C3	1.384 (4)	C12—H12A	1.0000
C2—H2A	0.9500	C13—H13A	0.9800
C3—C4	1.383 (4)	C13—H13B	0.9800
C3—H3A	0.9500	C13—H13C	0.9800
C4—C5	1.391 (4)	C14—H14A	0.9800
C4—H4A	0.9500	C14—H14B	0.9800
C5—C6	1.514 (4)	C14—H14C	0.9800
C6—H6A	0.9900	C15—H15A	0.9800
C6—H6B	0.9900	C15—H15B	0.9800
C7—H7A	0.9800	C15—H15C	0.9800
C7—H7B	0.9800		
C15—Zn1—N1	116.74 (10)	H7A—C7—H7C	109.5
C15—Zn1—N2 ⁱ	118.69 (11)	H7B—C7—H7C	109.5
N1—Zn1—N2 ⁱ	107.29 (8)	Si1—C8—H8A	109.5
C15—Zn1—N2	129.13 (11)	Si1—C8—H8B	109.5
N1—Zn1—N2	82.98 (8)	H8A—C8—H8B	109.5
N2 ⁱ —Zn1—N2	95.33 (9)	Si1—C8—H8C	109.5
C15—Zn1—Zn1 ⁱ	145.59 (9)	H8A—C8—H8C	109.5
N1—Zn1—Zn1 ⁱ	97.45 (6)	H8B—C8—H8C	109.5
N2 ⁱ —Zn1—Zn1 ⁱ	47.73 (6)	C10—C9—C11	107.5 (3)

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N2—Zn1—Zn1 ⁱ	47.60 (7)	C10—C9—C12	107.3 (2)
N2—Si1—C8	108.03 (12)	C11—C9—C12	111.4 (2)
N2—Si1—C7	107.85 (13)	C10—C9—Si1	104.6 (2)
C8—Si1—C7	107.90 (13)	C11—C9—Si1	111.16 (19)
N2—Si1—C9	119.94 (12)	C12—C9—Si1	114.4 (2)
C8—Si1—C9	107.10 (12)	C9—C10—H10A	109.5
C7—Si1—C9	105.51 (14)	C9—C10—H10B	109.5
C5—N1—C1	119.2 (2)	H10A—C10—H10B	109.5
C5—N1—Zn1	113.26 (17)	C9—C10—H10C	109.5
C1—N1—Zn1	127.22 (19)	H10A—C10—H10C	109.5
C6—N2—Si1	112.01 (18)	H10B—C10—H10C	109.5
C6—N2—Zn1 ⁱ	106.98 (17)	C9—C11—H11A	109.5
Si1—N2—Zn1 ⁱ	130.14 (11)	C9—C11—H11B	109.5
C6—N2—Zn1	109.58 (15)	H11A—C11—H11B	109.5
Si1—N2—Zn1	109.25 (11)	C9—C11—H11C	109.5
Zn1 ⁱ —N2—Zn1	84.67 (9)	H11A—C11—H11C	109.5
N1—C1—C2	122.5 (3)	H11B—C11—H11C	109.5
N1—C1—H1A	118.8	C14—C12—C13	108.4 (3)
C2—C1—H1A	118.8	C14—C12—C9	113.8 (2)
C1—C2—C3	119.0 (3)	C13—C12—C9	113.2 (3)
C1—C2—H2A	120.5	C14—C12—H12A	107.0
C3—C2—H2A	120.5	C13—C12—H12A	107.0
C4—C3—C2	118.4 (3)	C9—C12—H12A	107.0
C4—C3—H3A	120.8	C12—C13—H13A	109.5
C2—C3—H3A	120.8	C12—C13—H13B	109.5
C3—C4—C5	120.0 (3)	H13A—C13—H13B	109.5
C3—C4—H4A	120.0	C12—C13—H13C	109.5
C5—C4—H4A	120.0	H13A—C13—H13C	109.5
N1—C5—C4	120.8 (2)	H13B—C13—H13C	109.5
N1—C5—C6	118.1 (2)	C12—C14—H14A	109.5
C4—C5—C6	121.0 (2)	C12—C14—H14B	109.5
N2—C6—C5	115.2 (2)	H14A—C14—H14B	109.5
N2—C6—H6A	108.5	C12—C14—H14C	109.5
C5—C6—H6A	108.5	H14A—C14—H14C	109.5
N2—C6—H6B	108.5	H14B—C14—H14C	109.5
C5—C6—H6B	108.5	Zn1—C15—H15A	109.5
H6A—C6—H6B	107.5	Zn1—C15—H15B	109.5
Si1—C7—H7A	109.5	H15A—C15—H15B	109.5
Si1—C7—H7B	109.5	Zn1—C15—H15C	109.5
H7A—C7—H7B	109.5	H15A—C15—H15C	109.5
Si1—C7—H7C	109.5	H15B—C15—H15C	109.5

Symmetry codes: (i) $-x+1, -y, -z$.

Fig. 1

