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Crystal structure of *catena*-poly[[(3-*tert*-butylpyridine-κN)(4-*tert*-butylpyridine-κN)cadmium]-di-μ-thiocyanato-$\kappa^2 N{:}S;\kappa^2 S{:}N$]

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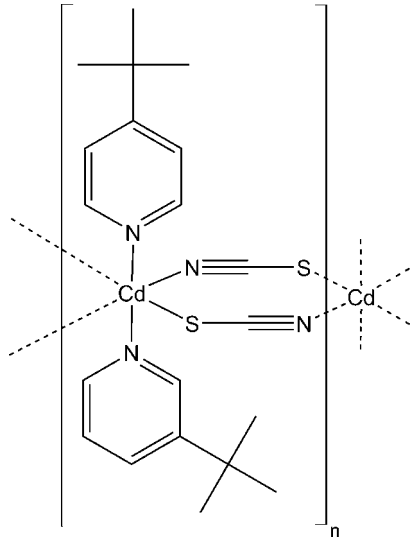
In the crystal structure of the title compound, $[Cd(NCS)_2(C_9H_{13}N)_2]_n$, the Cd^{II} cations are coordinated in a slightly distorted octahedral geometry by one 3-*tert*-butylpyridine ligand, one 4-*tert*-butylpyridine ligand and two pairs of translationally-equivalent μ-1,3-bridging thiocyanate ligands, all of which are in general positions. These μ-1,3-bridging thiocyante anions bridge the Cd^{II} cations, forming chains that propagate parallel to the *b* axis.

Keywords: crystal structure; cadmium coordination polymer; μ-1,3-thiocyanate anions.

CCDC reference: 1033510

1. Related literature

For related crystal structures with μ-1,3-bridging thiocyanate anions, see: Banerjee *et al.* (2005); Reinert *et al.* (2012*a*,*b*); Tahli *et al.* (2011).



2. Experimental

2.1. Crystal data

$[Cd(NCS)_2(C_9H_{13}N)_2]$
M_r = 498.97
Monoclinic, $P2_1/c$
a = 20.0917 (8) Å
b = 5.9503 (2) Å
c = 21.0198 (9) Å
β = 115.902 (3)°
V = 2260.51 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.16 mm^{-1}
T = 293 K
0.16 × 0.12 × 0.09 mm

2.2. Data collection

STOE IPDS-2 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
T_{min} = 0.760, T_{max} = 0.895
25365 measured reflections
5407 independent reflections
4159 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.084
S = 1.14
5407 reflections
244 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2008); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 2011); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: PK2535).

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supporting information

Acta Cryst. (2014). E**70**, m403–m404 [doi:10.1107/S1600536814024647]

Crystal structure of *catena*-poly[[(3-*tert*-butylpyridine-κN)(4-*tert*-butylpyridine-κN)cadmium]-di-μ-thiocyanato-$\kappa^2 N$:*S*;$\kappa^2 S$:*N*]

Julia Werner, Thorben Reinert, Inke Jess and Christian Näther

S1. Structural commentary

S2. Synthesis and crystallization

$CdSO_4 \cdot 8/3H_2O$ was purchased from Merck, and 4-*tert*-butylpyridine and $Ba(NCS)_2 \cdot 3H_2O$ were purchased from Alfa Aesar. The 4-*tert*-butylpyridine has a purity of only 97% and is contaminated with 3-*tert*-butylpyridine, which cannot be separated. $Cd(NCS)_2$ was synthesized by stirring 17.5 g (57.00 mmol) $Ba(NCS)_2 \cdot 3H_2O$ and 14.6 g (57.00 mmol) $CdSO_4 \cdot 8/3H_2O$ in 300 mL H_2O at RT for three hours. The white residue of $BaSO_4$ was filtered off and the solvent was evaporated by heating. The homogeneity of the product was investigated by X-ray powder diffraction and elemental analysis. The title compound was prepared by the reaction of (0.15 mmol) 34.3 mg $Cd(NCS)_2$ and (0.15 mmol) 22.2 μL 4-tert-butylpyridine in 1.0 mL H_2O at 80 °C in a closed 10 mL glass culture tube. After several days, colorless crystalline blocks were obtained.

S3. Refinement

Carbon-bound H atoms were positioned with idealized geometry and refined with $U_{iso}(H) = 1.2\ U_{eq}(C)$ using a riding model with C—H = 0.95 Å for aromatic and C—H = 0.99 Å for methyl H atoms.

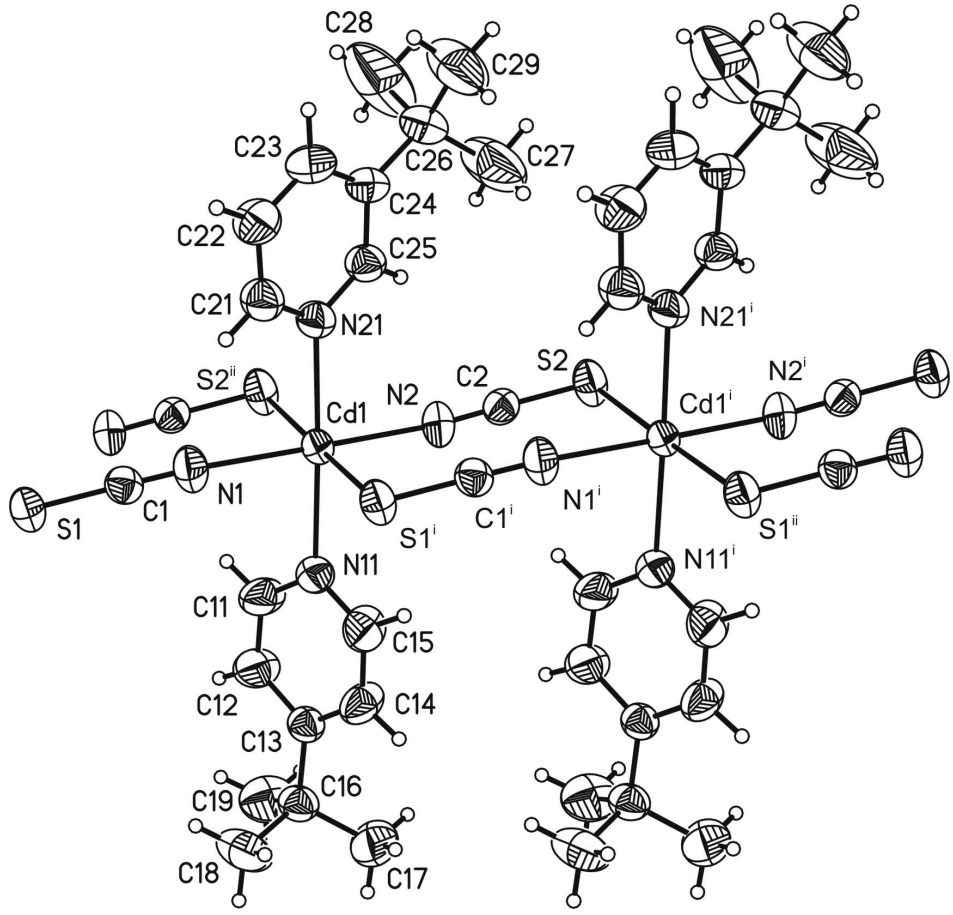

Figure 1

Crystal structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Symmetry code: i: (x, y+1, z); ii: (x, y-1, z).

***catena*-Poly[[(3-*tert*-butylpyridine-κN)(4-*tert*-butylpyridine-κN)cadmium]-di-μ-thiocyanato-$\kappa^2 N$:S;$\kappa^2 S$:N]**

Crystal data

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b = 5.9503 (2) Å
c = 21.0198 (9) Å
β = 115.902 (3)°
V = 2260.51 (15) Å^3
Z = 4
$F(000)$ = 1016
D_x = 1.466 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 25365 reflections
θ = 2.0–28.0°
μ = 1.16 mm^{-1}
T = 293 K
Block, colourless
0.16 × 0.12 × 0.09 mm

Data collection

STOE IPDS-2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
T_{min} = 0.760, T_{max} = 0.895
25365 measured reflections
5407 independent reflections

4159 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 28.0°$, $\theta_{min} = 2.0°$
$h = -26 \rightarrow 26$
$k = -7 \rightarrow 7$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.084$
$S = 1.14$
5407 reflections
244 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 0.8678P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.732991 (14)	0.24172 (4)	0.269228 (13)	0.04067 (8)
N1	0.67220 (19)	0.5638 (5)	0.20986 (17)	0.0535 (7)
C1	0.65872 (17)	0.7440 (6)	0.18809 (16)	0.0415 (6)
S1	0.63714 (5)	0.99805 (14)	0.15449 (5)	0.0484 (2)
N2	0.79392 (17)	−0.0756 (5)	0.32949 (17)	0.0514 (7)
C2	0.80555 (17)	−0.2557 (6)	0.35212 (16)	0.0421 (6)
S2	0.82427 (6)	−0.51157 (15)	0.38460 (5)	0.0554 (2)
N11	0.81232 (17)	0.2863 (5)	0.21360 (15)	0.0482 (7)
C11	0.8505 (3)	0.4716 (7)	0.2206 (2)	0.0724 (13)
H11	0.8473	0.5835	0.2501	0.087*
C12	0.8948 (3)	0.5105 (7)	0.1870 (2)	0.0713 (13)
H12	0.9209	0.6449	0.1950	0.086*
C13	0.9014 (2)	0.3547 (6)	0.14183 (19)	0.0443 (7)
C14	0.8606 (2)	0.1617 (7)	0.1342 (3)	0.0638 (11)
H14	0.8622	0.0474	0.1047	0.077*
C15	0.8174 (2)	0.1363 (7)	0.1698 (2)	0.0638 (11)
H15	0.7901	0.0047	0.1623	0.077*
C16	0.9479 (2)	0.3988 (7)	0.1019 (2)	0.0509 (8)
C17	0.9747 (3)	0.1778 (9)	0.0830 (3)	0.0847 (15)
H17A	1.0038	0.2105	0.0580	0.127*
H17B	0.9328	0.0882	0.0536	0.127*
H17C	1.0043	0.0967	0.1255	0.127*

C18	0.8990 (3)	0.5207 (10)	0.0334 (3)	0.0867 (16)
H18A	0.9269	0.5508	0.0072	0.130*
H18B	0.8821	0.6598	0.0444	0.130*
H18C	0.8571	0.4282	0.0056	0.130*
C19	1.0150 (3)	0.5424 (10)	0.1453 (3)	0.0862 (16)
H19A	1.0432	0.5674	0.1191	0.129*
H19B	1.0452	0.4664	0.1888	0.129*
H19C	0.9990	0.6840	0.1557	0.129*
N21	0.64660 (16)	0.2045 (5)	0.31532 (16)	0.0470 (7)
C21	0.5826 (2)	0.3142 (7)	0.2852 (2)	0.0546 (9)
H21	0.5733	0.4039	0.2459	0.066*
C22	0.5296 (2)	0.2999 (7)	0.3098 (2)	0.0629 (11)
H22	0.4855	0.3795	0.2879	0.076*
C23	0.5429 (2)	0.1660 (7)	0.3673 (2)	0.0573 (10)
H23	0.5075	0.1554	0.3846	0.069*
C24	0.60838 (19)	0.0467 (6)	0.39999 (18)	0.0454 (8)
C25	0.6582 (2)	0.0755 (6)	0.37141 (19)	0.0473 (8)
H25	0.7030	−0.0005	0.3928	0.057*
C26	0.6255 (2)	−0.0980 (7)	0.4648 (2)	0.0587 (10)
C27	0.6837 (4)	−0.2738 (12)	0.4745 (4)	0.133 (3)
H27A	0.6662	−0.3706	0.4339	0.200*
H27B	0.6933	−0.3611	0.5160	0.200*
H27C	0.7285	−0.2011	0.4797	0.200*
C28	0.6512 (6)	0.0518 (13)	0.5280 (3)	0.171 (4)
H28A	0.6627	−0.0373	0.5696	0.257*
H28B	0.6127	0.1569	0.5223	0.257*
H28C	0.6945	0.1319	0.5327	0.257*
C29	0.5577 (3)	−0.2282 (11)	0.4579 (4)	0.1055 (19)
H29A	0.5416	−0.3255	0.4175	0.158*
H29B	0.5188	−0.1249	0.4521	0.158*
H29C	0.5699	−0.3165	0.4997	0.158*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.04773 (12)	0.03305 (11)	0.04745 (12)	0.00141 (12)	0.02654 (10)	0.00495 (11)
N1	0.064 (2)	0.0352 (15)	0.0566 (18)	0.0003 (14)	0.0216 (16)	0.0037 (14)
C1	0.0434 (15)	0.0405 (15)	0.0407 (15)	−0.0062 (17)	0.0185 (13)	−0.0032 (17)
S1	0.0550 (5)	0.0374 (4)	0.0470 (5)	0.0007 (4)	0.0167 (4)	0.0063 (4)
N2	0.0536 (18)	0.0355 (14)	0.0603 (19)	0.0036 (13)	0.0204 (15)	0.0053 (14)
C2	0.0422 (15)	0.0421 (16)	0.0425 (15)	−0.0014 (17)	0.0190 (13)	−0.0044 (17)
S2	0.0675 (6)	0.0385 (4)	0.0481 (5)	−0.0007 (4)	0.0141 (5)	0.0058 (4)
N11	0.0579 (17)	0.0447 (16)	0.0456 (15)	−0.0034 (14)	0.0260 (14)	−0.0023 (13)
C11	0.108 (4)	0.055 (2)	0.084 (3)	−0.028 (2)	0.070 (3)	−0.024 (2)
C12	0.097 (3)	0.057 (2)	0.086 (3)	−0.032 (2)	0.065 (3)	−0.021 (2)
C13	0.0418 (18)	0.0475 (18)	0.0463 (19)	0.0013 (15)	0.0218 (16)	0.0042 (15)
C14	0.069 (3)	0.055 (2)	0.090 (3)	−0.017 (2)	0.055 (3)	−0.023 (2)
C15	0.063 (2)	0.052 (2)	0.088 (3)	−0.017 (2)	0.044 (2)	−0.011 (2)

C16	0.048 (2)	0.059 (2)	0.053 (2)	0.0025 (17)	0.0279 (17)	0.0068 (17)
C17	0.087 (3)	0.081 (3)	0.115 (4)	0.013 (3)	0.071 (3)	0.004 (3)
C18	0.082 (3)	0.117 (4)	0.074 (3)	0.019 (3)	0.047 (3)	0.038 (3)
C19	0.064 (3)	0.111 (4)	0.097 (4)	−0.026 (3)	0.047 (3)	−0.013 (3)
N21	0.0460 (15)	0.0488 (17)	0.0530 (16)	0.0030 (13)	0.0278 (14)	0.0065 (13)
C21	0.052 (2)	0.060 (2)	0.054 (2)	0.0076 (17)	0.0246 (18)	0.0103 (17)
C22	0.046 (2)	0.074 (3)	0.071 (3)	0.0149 (19)	0.0288 (19)	0.013 (2)
C23	0.048 (2)	0.075 (2)	0.058 (2)	−0.0019 (19)	0.0309 (19)	−0.0028 (19)
C24	0.0450 (18)	0.055 (2)	0.0395 (17)	−0.0068 (16)	0.0220 (15)	−0.0049 (15)
C25	0.0436 (18)	0.054 (2)	0.0477 (19)	0.0038 (16)	0.0230 (16)	0.0046 (16)
C26	0.063 (2)	0.071 (3)	0.049 (2)	−0.007 (2)	0.0303 (19)	0.0052 (19)
C27	0.118 (5)	0.159 (7)	0.151 (6)	0.059 (5)	0.084 (5)	0.103 (5)
C28	0.323 (12)	0.124 (6)	0.047 (3)	−0.086 (7)	0.063 (5)	−0.013 (3)
C29	0.094 (4)	0.110 (5)	0.124 (5)	−0.012 (4)	0.058 (4)	0.038 (4)

Geometric parameters (Å, º)

Cd1—N2	2.305 (3)	C18—H18A	0.9600
Cd1—N1	2.322 (3)	C18—H18B	0.9600
Cd1—N21	2.339 (3)	C18—H18C	0.9600
Cd1—N11	2.368 (3)	C19—H19A	0.9600
Cd1—S2^{i}	2.7412 (10)	C19—H19B	0.9600
Cd1—S1^{ii}	2.7513 (10)	C19—H19C	0.9600
N1—C1	1.151 (4)	N21—C21	1.330 (5)
C1—S1	1.644 (4)	N21—C25	1.339 (4)
S1—Cd1^{i}	2.7513 (10)	C21—C22	1.375 (5)
N2—C2	1.154 (4)	C21—H21	0.9300
C2—S2	1.644 (4)	C22—C23	1.374 (6)
S2—Cd1^{ii}	2.7412 (10)	C22—H22	0.9300
N11—C11	1.314 (5)	C23—C24	1.385 (5)
N11—C15	1.319 (5)	C23—H23	0.9300
C11—C12	1.378 (5)	C24—C25	1.384 (4)
C11—H11	0.9300	C24—C26	1.518 (5)
C12—C13	1.373 (5)	C25—H25	0.9300
C12—H12	0.9300	C26—C28	1.492 (7)
C13—C14	1.379 (5)	C26—C27	1.514 (7)
C13—C16	1.527 (5)	C26—C29	1.518 (6)
C14—C15	1.379 (5)	C27—H27A	0.9600
C14—H14	0.9300	C27—H27B	0.9600
C15—H15	0.9300	C27—H27C	0.9600
C16—C19	1.519 (6)	C28—H28A	0.9600
C16—C18	1.525 (6)	C28—H28B	0.9600
C16—C17	1.537 (6)	C28—H28C	0.9600
C17—H17A	0.9600	C29—H29A	0.9600
C17—H17B	0.9600	C29—H29B	0.9600
C17—H17C	0.9600	C29—H29C	0.9600
N2—Cd1—N1	179.28 (11)	H18A—C18—H18B	109.5

N2—Cd1—N21	90.40 (11)	C16—C18—H18C	109.5
N1—Cd1—N21	89.39 (11)	H18A—C18—H18C	109.5
N2—Cd1—N11	93.03 (11)	H18B—C18—H18C	109.5
N1—Cd1—N11	87.23 (11)	C16—C19—H19A	109.5
N21—Cd1—N11	175.34 (11)	C16—C19—H19B	109.5
N2—Cd1—S2^{i}	87.88 (8)	H19A—C19—H19B	109.5
N1—Cd1—S2^{i}	91.43 (8)	C16—C19—H19C	109.5
N21—Cd1—S2^{i}	90.86 (8)	H19A—C19—H19C	109.5
N11—Cd1—S2^{i}	92.42 (8)	H19B—C19—H19C	109.5
N2—Cd1—S1^{ii}	92.89 (8)	C21—N21—C25	117.4 (3)
N1—Cd1—S1^{ii}	87.79 (8)	C21—N21—Cd1	119.6 (2)
N21—Cd1—S1^{ii}	87.21 (8)	C25—N21—Cd1	123.0 (2)
N11—Cd1—S1^{ii}	89.46 (8)	N21—C21—C22	122.5 (4)
S2^{i}—Cd1—S1^{ii}	177.92 (3)	N21—C21—H21	118.8
C1—N1—Cd1	163.7 (3)	C22—C21—H21	118.8
N1—C1—S1	178.0 (3)	C23—C22—C21	118.8 (4)
C1—S1—Cd1^{i}	98.96 (11)	C23—C22—H22	120.6
C2—N2—Cd1	161.9 (3)	C21—C22—H22	120.6
N2—C2—S2	178.6 (3)	C22—C23—C24	120.7 (3)
C2—S2—Cd1^{ii}	100.68 (11)	C22—C23—H23	119.6
C11—N11—C15	115.2 (3)	C24—C23—H23	119.6
C11—N11—Cd1	121.6 (2)	C25—C24—C23	115.6 (3)
C15—N11—Cd1	122.9 (2)	C25—C24—C26	122.4 (3)
N11—C11—C12	124.1 (4)	C23—C24—C26	122.0 (3)
N11—C11—H11	118.0	N21—C25—C24	124.9 (3)
C12—C11—H11	118.0	N21—C25—H25	117.5
C13—C12—C11	121.3 (4)	C24—C25—H25	117.5
C13—C12—H12	119.3	C28—C26—C27	110.3 (6)
C11—C12—H12	119.3	C28—C26—C24	108.3 (4)
C12—C13—C14	114.3 (3)	C27—C26—C24	111.8 (3)
C12—C13—C16	122.0 (3)	C28—C26—C29	109.6 (5)
C14—C13—C16	123.6 (3)	C27—C26—C29	105.6 (5)
C13—C14—C15	120.7 (4)	C24—C26—C29	111.2 (4)
C13—C14—H14	119.6	C26—C27—H27A	109.5
C15—C14—H14	119.6	C26—C27—H27B	109.5
N11—C15—C14	124.3 (4)	H27A—C27—H27B	109.5
N11—C15—H15	117.8	C26—C27—H27C	109.5
C14—C15—H15	117.8	H27A—C27—H27C	109.5
C19—C16—C18	109.7 (4)	H27B—C27—H27C	109.5
C19—C16—C13	111.2 (3)	C26—C28—H28A	109.5
C18—C16—C13	107.7 (3)	C26—C28—H28B	109.5
C19—C16—C17	108.5 (4)	H28A—C28—H28B	109.5
C18—C16—C17	108.5 (4)	C26—C28—H28C	109.5
C13—C16—C17	111.3 (3)	H28A—C28—H28C	109.5
C16—C17—H17A	109.5	H28B—C28—H28C	109.5
C16—C17—H17B	109.5	C26—C29—H29A	109.5
H17A—C17—H17B	109.5	C26—C29—H29B	109.5
C16—C17—H17C	109.5	H29A—C29—H29B	109.5

H17A—C17—H17C	109.5	C26—C29—H29C	109.5
H17B—C17—H17C	109.5	H29A—C29—H29C	109.5
C16—C18—H18A	109.5	H29B—C29—H29C	109.5
C16—C18—H18B	109.5		

Symmetry codes: (i) x, y+1, z; (ii) x, y−1, z.