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Crystal structure of ethyl 4-(2-chlorophenyl)-2-methyl-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

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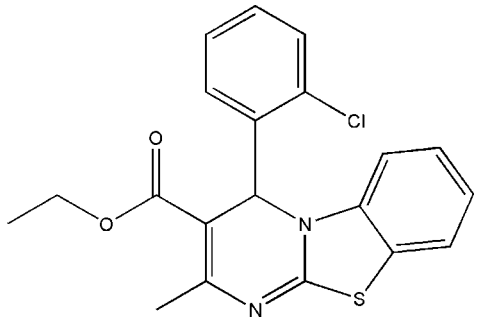
In the title compound, $C_{20}H_{17}ClN_2O_2S$, the dihedral angle between the planes of the benzothiazole fused ring system (r.m.s. deviation = 0.024 Å) and the chlorobenzene ring is 89.62 (12)°. The ester C—O—C—C side chain has an *anti* orientation [torsion angle = −155.2 (3)°]. In the crystal, weak aromatic π–π stacking interactions are observed between the phenyl and pyrimidine rings [centroid–centroid seperation = 3.666 (2) Å].

Keywords: crystal structure; pyrimido[2,1-*b*][1,3]benzothiazole; ester; biological activity.

CCDC reference: 1406433

1. Related literature

For biological activities of benzothiazoles, see: Landreau *et al.* (2002); Russo *et al.* (1985). For a related structure, see: Sankar *et al.* (2015).



2. Experimental

2.1. Crystal data

$C_{20}H_{17}ClN_2O_2S$
M_r = 384.87
Triclinic, $P\overline{1}$
a = 8.9049 (8) Å
b = 8.9275 (10) Å
c = 12.3564 (11) Å
α = 88.434 (8)°
β = 83.536 (7)°
γ = 66.201 (10)°
V = 892.90 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.20 mm

2.2. Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.880, T_{max} = 1.000
6440 measured reflections
3477 independent reflections
2275 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.142
S = 1.03
3477 reflections
237 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7475).

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supporting information

Acta Cryst. (2015). E**71**, o669 [doi:10.1107/S2056989015014905]

Crystal structure of ethyl 4-(2-chlorophenyl)-2-methyl-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

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S1. Experimental

To a mixture of ethylacetoacetate (1.0 mmol, 0.13 g), 2-chlorobenzadehyde (1.0 mmol, 0.14 g) and 2-aminobenzothiazole (1.0 mmol, 0.152 g) in a round bottom flask (25 ml), $C/TiO_2{\cdot}SO_3{\cdot}SbCl_2$ (0.1 g) was added and the reaction mixture was heated at 363 K under solvent-free conditions for 1 h. Hot ethanol (2 × 5 ml) was added to the reaction mixture and the catalyst was separated by simple filteration. Removal of the solvent under reduced pressure afforded the product, which was further crystallized from ethanol as yellow crystals (Yield: 88%).

S2. Refinement

All the H atoms were geometrically fixed and allowed to ride on their parent C atoms, with C—H distances of 0.93–0.96 Å; and with U_{iso}(H) = 1.2U_{eq}(C), except for the methyl group where U_{iso}(H) = 1.5U_{eq}(C).

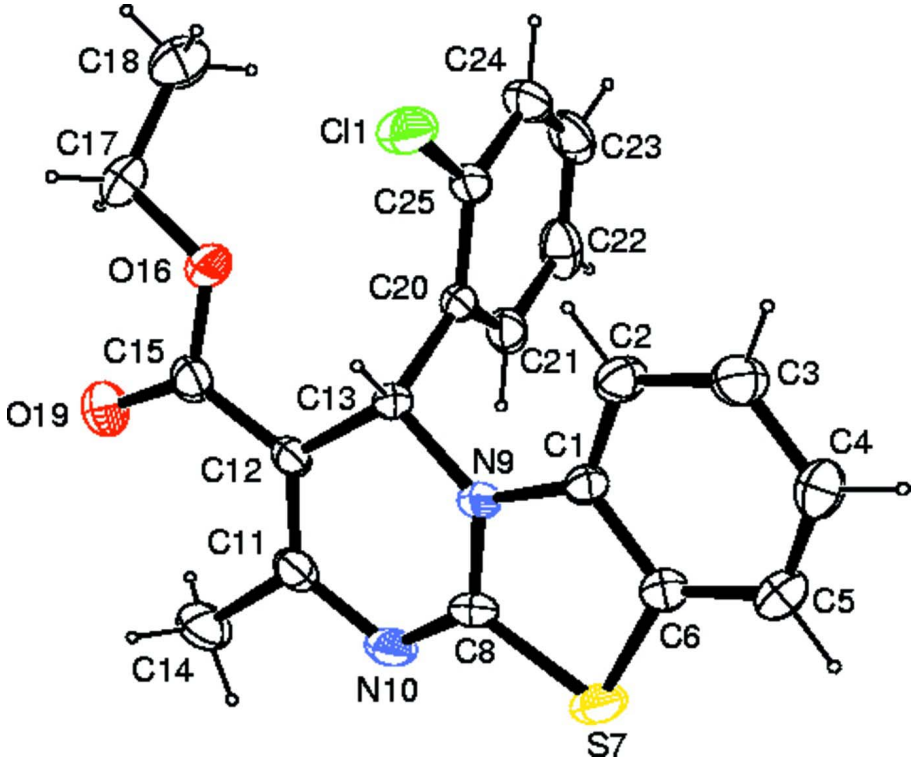

Figure 1

ORTEP view of the molecule with displacement ellipsoids drawn at the 40% probability level.

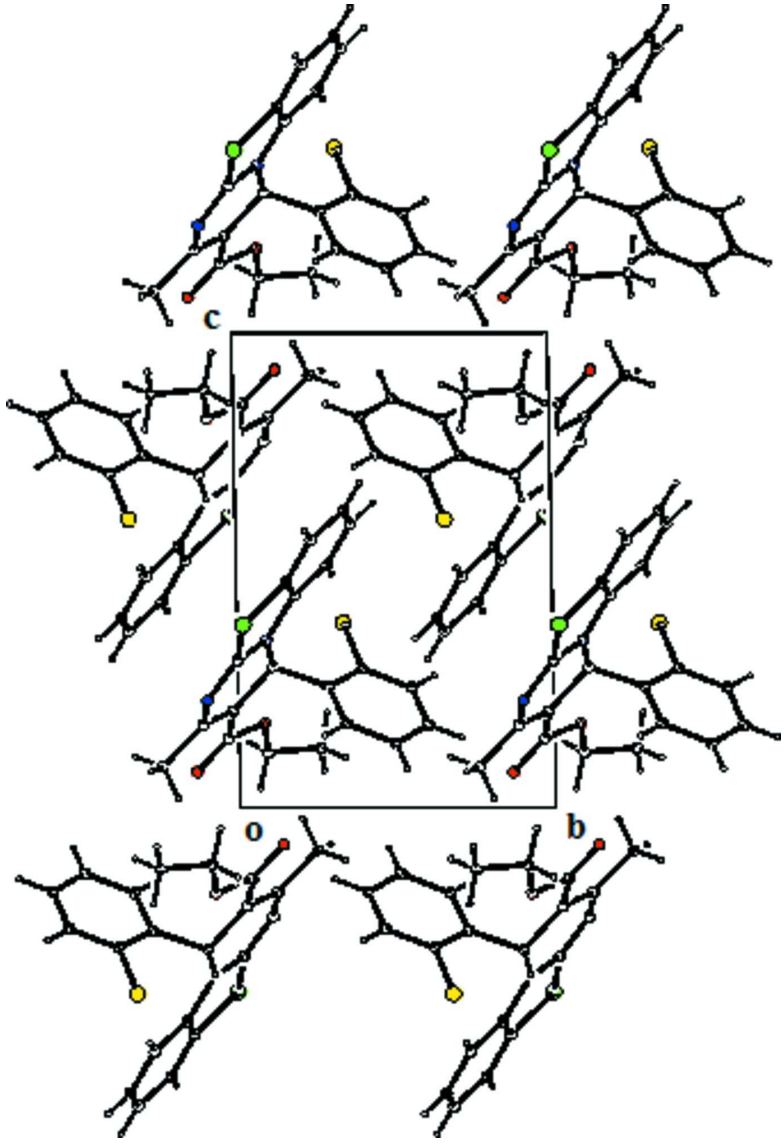

Figure 2

The packing arrangement of molecules viewed down the *a* axis.

Ethyl 4-(2-chlorophenyl)-2-methyl-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole- 3-carboxylate

Crystal data

$C_{20}H_{17}ClN_2O_2S$
M_r = 384.87
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.9049 (8) Å
b = 8.9275 (10) Å
c = 12.3564 (11) Å
α = 88.434 (8)°
β = 83.536 (7)°
γ = 66.201 (10)°
V = 892.90 (15) Å^3
Z = 2
F(000) = 400
D_x = 1.431 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1737 reflections
θ = 4.1–27.4°
μ = 0.35 mm^{-1}
T = 293 K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1049 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.880, T_{max} = 1.000
6440 measured reflections
3477 independent reflections
2275 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26.0°, θ_{min} = 3.9°
h = −10→10
k = −10→10
l = −9→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.142
S = 1.03
3477 reflections
237 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
$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 0.235P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S7	0.83804 (10)	0.01946 (11)	0.38623 (8)	0.0592 (3)
Cl1	0.02890 (10)	0.33670 (11)	0.39074 (8)	0.0648 (3)
N9	0.5363 (3)	0.0924 (2)	0.35456 (18)	0.0370 (6)
C25	0.1519 (4)	0.3941 (3)	0.2942 (2)	0.0431 (7)
C12	0.4497 (3)	−0.0172 (3)	0.2052 (2)	0.0368 (7)
O16	0.1721 (3)	0.0825 (2)	0.17927 (17)	0.0531 (6)
N10	0.7388 (3)	−0.0979 (3)	0.2270 (2)	0.0503 (7)
C21	0.4011 (4)	0.3408 (3)	0.1786 (2)	0.0445 (7)
H21	0.5087	0.2715	0.1531	0.053*
C8	0.6924 (3)	−0.0008 (3)	0.3117 (2)	0.0427 (7)
C20	0.3121 (3)	0.2857 (3)	0.2573 (2)	0.0345 (6)
C15	0.3219 (4)	−0.0328 (3)	0.1475 (2)	0.0446 (7)
O19	0.3434 (3)	−0.1335 (3)	0.0766 (2)	0.0719 (7)
C13	0.3919 (3)	0.1118 (3)	0.2977 (2)	0.0341 (6)
H13	0.3110	0.0911	0.3495	0.041*
C1	0.5264 (3)	0.1829 (3)	0.4491 (2)	0.0370 (7)

C11	0.6124 (4)	−0.1115 (3)	0.1764 (2)	0.0443 (7)
C3	0.4058 (4)	0.3661 (4)	0.6003 (3)	0.0553 (9)
H3	0.3125	0.4372	0.6429	0.066*
C24	0.0834 (4)	0.5496 (4)	0.2540 (3)	0.0612 (10)
H24	−0.0238	0.6201	0.2795	0.073*
C2	0.3873 (4)	0.2860 (4)	0.5114 (2)	0.0473 (8)
H2	0.2829	0.3019	0.4944	0.057*
C17	0.0396 (4)	0.0912 (4)	0.1179 (3)	0.0608 (9)
H17A	−0.0047	0.0128	0.1455	0.073*
H17B	0.0815	0.0642	0.0419	0.073*
C6	0.6823 (4)	0.1580 (3)	0.4765 (2)	0.0439 (7)
C14	0.6786 (4)	−0.2416 (4)	0.0879 (3)	0.0640 (10)
H14A	0.6468	−0.3300	0.1087	0.096*
H14B	0.7969	−0.2819	0.0772	0.096*
H14C	0.6343	−0.1959	0.0214	0.096*
C5	0.6991 (4)	0.2383 (4)	0.5662 (3)	0.0561 (9)
H5	0.8031	0.2214	0.5845	0.067*
C4	0.5604 (4)	0.3422 (4)	0.6264 (3)	0.0601 (9)
H4	0.5697	0.3978	0.6861	0.072*
C23	0.1744 (5)	0.5998 (4)	0.1761 (3)	0.0675 (11)
H23	0.1283	0.7051	0.1491	0.081*
C18	−0.0923 (5)	0.2586 (5)	0.1279 (4)	0.0812 (12)
H18A	−0.1419	0.2802	0.2021	0.122*
H18B	−0.1747	0.2677	0.0813	0.122*
H18C	−0.0457	0.3365	0.1069	0.122*
C22	0.3327 (5)	0.4969 (4)	0.1373 (3)	0.0594 (10)
H22	0.3932	0.5316	0.0839	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S7	0.0328 (4)	0.0641 (5)	0.0699 (6)	−0.0056 (4)	−0.0153 (4)	−0.0055 (5)
Cl1	0.0374 (5)	0.0726 (6)	0.0751 (6)	−0.0129 (4)	−0.0018 (4)	−0.0116 (5)
N9	0.0318 (13)	0.0316 (11)	0.0424 (14)	−0.0058 (10)	−0.0106 (11)	0.0003 (10)
C25	0.0382 (16)	0.0343 (14)	0.0534 (19)	−0.0077 (13)	−0.0169 (15)	−0.0053 (13)
C12	0.0402 (16)	0.0244 (12)	0.0425 (16)	−0.0089 (12)	−0.0075 (14)	−0.0002 (12)
O16	0.0401 (12)	0.0549 (12)	0.0632 (14)	−0.0144 (10)	−0.0165 (11)	−0.0126 (11)
N10	0.0361 (14)	0.0421 (13)	0.0579 (17)	−0.0002 (11)	−0.0040 (13)	−0.0080 (13)
C21	0.0514 (19)	0.0383 (15)	0.0456 (18)	−0.0177 (14)	−0.0141 (16)	−0.0004 (13)
C8	0.0324 (16)	0.0349 (14)	0.0517 (18)	−0.0028 (12)	−0.0107 (14)	0.0046 (14)
C20	0.0341 (15)	0.0295 (13)	0.0395 (16)	−0.0100 (12)	−0.0131 (13)	−0.0019 (12)
C15	0.0512 (19)	0.0349 (15)	0.0499 (18)	−0.0189 (14)	−0.0085 (16)	0.0017 (14)
O19	0.0711 (17)	0.0584 (14)	0.0851 (18)	−0.0210 (13)	−0.0161 (15)	−0.0285 (13)
C13	0.0299 (14)	0.0297 (13)	0.0421 (16)	−0.0097 (11)	−0.0104 (13)	0.0026 (12)
C1	0.0351 (16)	0.0344 (14)	0.0413 (16)	−0.0112 (12)	−0.0152 (13)	0.0058 (13)
C11	0.0492 (18)	0.0291 (14)	0.0487 (18)	−0.0092 (13)	−0.0068 (15)	−0.0011 (13)
C3	0.050 (2)	0.0590 (19)	0.0467 (19)	−0.0105 (16)	−0.0080 (16)	−0.0088 (16)
C24	0.053 (2)	0.0373 (17)	0.081 (3)	0.0000 (16)	−0.028 (2)	−0.0075 (17)

C2	0.0403 (17)	0.0554 (18)	0.0430 (17)	−0.0143 (15)	−0.0111 (15)	−0.0004 (15)
C17	0.052 (2)	0.068 (2)	0.074 (2)	−0.0304 (18)	−0.0224 (19)	−0.0030 (18)
C6	0.0400 (17)	0.0410 (15)	0.0484 (18)	−0.0121 (13)	−0.0129 (15)	0.0037 (14)
C14	0.058 (2)	0.0497 (18)	0.068 (2)	−0.0050 (17)	−0.0011 (19)	−0.0180 (18)
C5	0.048 (2)	0.064 (2)	0.060 (2)	−0.0208 (17)	−0.0246 (18)	0.0024 (18)
C4	0.065 (2)	0.066 (2)	0.053 (2)	−0.0261 (19)	−0.0207 (19)	−0.0087 (18)
C23	0.087 (3)	0.0312 (16)	0.079 (3)	−0.0107 (19)	−0.042 (2)	0.0046 (18)
C18	0.064 (3)	0.082 (3)	0.098 (3)	−0.022 (2)	−0.043 (2)	0.008 (2)
C22	0.092 (3)	0.0491 (19)	0.051 (2)	−0.039 (2)	−0.021 (2)	0.0095 (16)

Geometric parameters (Å, º)

S7—C8	1.741 (3)	C11—C14	1.505 (4)
S7—C6	1.744 (3)	C3—C4	1.379 (4)
Cl1—C25	1.735 (3)	C3—C2	1.388 (4)
N9—C8	1.353 (3)	C3—H3	0.9300
N9—C1	1.413 (3)	C24—C23	1.370 (5)
N9—C13	1.481 (3)	C24—H24	0.9300
C25—C24	1.376 (4)	C2—H2	0.9300
C25—C20	1.394 (4)	C17—C18	1.479 (5)
C12—C11	1.360 (4)	C17—H17A	0.9700
C12—C15	1.462 (4)	C17—H17B	0.9700
C12—C13	1.536 (3)	C6—C5	1.390 (4)
O16—C15	1.338 (3)	C14—H14A	0.9600
O16—C17	1.448 (3)	C14—H14B	0.9600
N10—C8	1.296 (4)	C14—H14C	0.9600
N10—C11	1.394 (4)	C5—C4	1.359 (4)
C21—C22	1.385 (4)	C5—H5	0.9300
C21—C20	1.389 (4)	C4—H4	0.9300
C21—H21	0.9300	C23—C22	1.374 (5)
C20—C13	1.521 (3)	C23—H23	0.9300
C15—O19	1.216 (3)	C18—H18A	0.9600
C13—H13	0.9800	C18—H18B	0.9600
C1—C2	1.370 (4)	C18—H18C	0.9600
C1—C6	1.394 (4)	C22—H22	0.9300
C8—S7—C6	91.01 (14)	C23—C24—C25	119.5 (3)
C8—N9—C1	114.1 (2)	C23—C24—H24	120.2
C8—N9—C13	121.6 (2)	C25—C24—H24	120.2
C1—N9—C13	123.8 (2)	C1—C2—C3	118.5 (3)
C24—C25—C20	121.5 (3)	C1—C2—H2	120.7
C24—C25—Cl1	117.2 (3)	C3—C2—H2	120.7
C20—C25—Cl1	121.3 (2)	O16—C17—C18	109.4 (3)
C11—C12—C15	121.0 (2)	O16—C17—H17A	109.8
C11—C12—C13	121.9 (2)	C18—C17—H17A	109.8
C15—C12—C13	117.1 (2)	O16—C17—H17B	109.8
C15—O16—C17	116.6 (2)	C18—C17—H17B	109.8
C8—N10—C11	115.9 (2)	H17A—C17—H17B	108.2

C22—C21—C20	121.3 (3)	C5—C6—C1	120.7 (3)
C22—C21—H21	119.3	C5—C6—S7	128.0 (2)
C20—C21—H21	119.3	C1—C6—S7	111.2 (2)
N10—C8—N9	127.7 (3)	C11—C14—H14A	109.5
N10—C8—S7	120.5 (2)	C11—C14—H14B	109.5
N9—C8—S7	111.8 (2)	H14A—C14—H14B	109.5
C21—C20—C25	117.5 (3)	C11—C14—H14C	109.5
C21—C20—C13	119.1 (2)	H14A—C14—H14C	109.5
C25—C20—C13	123.4 (3)	H14B—C14—H14C	109.5
O19—C15—O16	121.7 (3)	C4—C5—C6	118.6 (3)
O19—C15—C12	126.3 (3)	C4—C5—H5	120.7
O16—C15—C12	112.0 (2)	C6—C5—H5	120.7
N9—C13—C20	110.1 (2)	C5—C4—C3	120.9 (3)
N9—C13—C12	108.4 (2)	C5—C4—H4	119.6
C20—C13—C12	112.7 (2)	C3—C4—H4	119.6
N9—C13—H13	108.5	C24—C23—C22	120.9 (3)
C20—C13—H13	108.5	C24—C23—H23	119.5
C12—C13—H13	108.5	C22—C23—H23	119.5
C2—C1—C6	120.2 (2)	C17—C18—H18A	109.5
C2—C1—N9	128.0 (2)	C17—C18—H18B	109.5
C6—C1—N9	111.9 (2)	H18A—C18—H18B	109.5
C12—C11—N10	123.1 (2)	C17—C18—H18C	109.5
C12—C11—C14	125.1 (3)	H18A—C18—H18C	109.5
N10—C11—C14	111.8 (3)	H18B—C18—H18C	109.5
C4—C3—C2	121.1 (3)	C23—C22—C21	119.2 (4)
C4—C3—H3	119.5	C23—C22—H22	120.4
C2—C3—H3	119.5	C21—C22—H22	120.4

C11—N10—C8—N9	−2.2 (5)	C15—C12—C13—C20	−65.5 (3)
C11—N10—C8—S7	176.9 (2)	C8—N9—C1—C2	−179.2 (3)
C1—N9—C8—N10	178.7 (3)	C13—N9—C1—C2	8.4 (4)
C13—N9—C8—N10	−8.7 (5)	C8—N9—C1—C6	1.1 (3)
C1—N9—C8—S7	−0.4 (3)	C13—N9—C1—C6	−171.3 (2)
C13—N9—C8—S7	172.18 (18)	C15—C12—C11—N10	178.2 (3)
C6—S7—C8—N10	−179.5 (3)	C13—C12—C11—N10	−0.7 (4)
C6—S7—C8—N9	−0.3 (2)	C15—C12—C11—C14	−2.0 (5)
C22—C21—C20—C25	−0.7 (4)	C13—C12—C11—C14	179.1 (3)
C22—C21—C20—C13	178.5 (2)	C8—N10—C11—C12	6.8 (4)
C24—C25—C20—C21	0.4 (4)	C8—N10—C11—C14	−173.1 (3)
Cl1—C25—C20—C21	178.98 (19)	C20—C25—C24—C23	−0.2 (4)
C24—C25—C20—C13	−178.9 (2)	Cl1—C25—C24—C23	−178.8 (2)
Cl1—C25—C20—C13	−0.3 (4)	C6—C1—C2—C3	1.1 (4)
C17—O16—C15—O19	−5.8 (4)	N9—C1—C2—C3	−178.6 (3)
C17—O16—C15—C12	172.5 (2)	C4—C3—C2—C1	−0.5 (5)
C11—C12—C15—O19	5.4 (5)	C15—O16—C17—C18	−155.2 (3)
C13—C12—C15—O19	−175.7 (3)	C2—C1—C6—C5	−0.8 (4)
C11—C12—C15—O16	−172.8 (3)	N9—C1—C6—C5	179.0 (3)
C13—C12—C15—O16	6.1 (4)	C2—C1—C6—S7	179.0 (2)

C8—N9—C13—C20	−110.7 (3)	N9—C1—C6—S7	−1.3 (3)
C1—N9—C13—C20	61.2 (3)	C8—S7—C6—C5	−179.4 (3)
C8—N9—C13—C12	12.9 (3)	C8—S7—C6—C1	0.9 (2)
C1—N9—C13—C12	−175.2 (2)	C1—C6—C5—C4	−0.2 (5)
C21—C20—C13—N9	64.4 (3)	S7—C6—C5—C4	−179.9 (3)
C25—C20—C13—N9	−116.4 (3)	C6—C5—C4—C3	0.8 (5)
C21—C20—C13—C12	−56.7 (3)	C2—C3—C4—C5	−0.5 (5)
C25—C20—C13—C12	122.5 (3)	C25—C24—C23—C22	0.3 (5)
C11—C12—C13—N9	−8.7 (4)	C24—C23—C22—C21	−0.6 (5)
C15—C12—C13—N9	172.4 (2)	C20—C21—C22—C23	0.9 (4)
C11—C12—C13—C20	113.4 (3)		