Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Phenyl-5,6,7,8-tetrahydroimidazo-[2,1-*b*][1,3]benzothiazole

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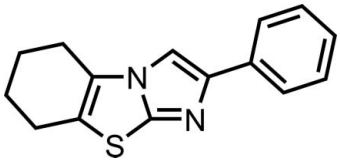
Received 5 May 2014; accepted 12 May 2014

Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.005 Å; R factor = 0.065; wR factor = 0.157; data-to-parameter ratio = 23.3.

The title compound, $C_{15}H_{14}N_2S$, crystallizes with two independent molecules in the asymmetric unit. The central imidazo[2,1-*b*][1,3]benzothiazole unit is planar (r.m.s. deviations of 0.010 and 0.008 Å for the two independent molecules). The fused tetrahydrohexane ring adopts a half-chair conformation. The phenyl substituent is twisted by 16.96 (13) and 22.89 (12)° relative to the central imidazo[2,1-*b*][1,3]benzo-][1,3]benzothiazole unit in the two molecules. In the crystal, there are no significant intermolecular interactions present.

Related literature

For applications of imidazo[2,1-*b*][1,3]benzothiazoles, see: Ager *et al.* (1988); Sanfilippo *et al.* (1988); Barchéchath *et al.* (2005); Andreani *et al.* (2008); Chao *et al.* (2009); Kumbhare *et al.* (2011); Chandak *et al.* (2013). For the crystal structures of related compounds, see: Landreau *et al.* (2002); Adib *et al.* (2008); Fun, Asik *et al.* (2011); Fun, Hemamalini *et al.* (2011); Ghabbour *et al.* (2012); Bunev *et al.* (2013*a*,*b*, 2014).



Experimental

Crystal data

$C_{15}H_{14}N_2S$
M_r = 254.34
Monoclinic, $P2_1/n$
a = 12.523 (3) Å
b = 10.699 (3) Å
c = 18.930 (5) Å
β = 102.291 (6)°
V = 2478.2 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 120 K
0.30 × 0.05 × 0.03 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.931, T_{max} = 0.993
33479 measured reflections
7558 independent reflections
3223 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.157
S = 0.93
7558 reflections
325 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Ministry of Education and Science of the Russian Federation (State contract No. 426).

Supporting information for this paper is available from the IUCr electronic archives (Reference: RK2428).

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supporting information

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2-Phenyl-5,6,7,8-tetrahydroimidazo[2,1-*b*][1,3]benzothiazole

Alexander S. Bunev, Elena V. Sukhonosova, Petr P. Purygin, Gennady I. Ostapenko and Victor N. Khrustalev

S1. Comment

Imidazo[2,1-*b*][1,3]benzothiazole are of great interest due to their biological properties. These compounds and their derivatives demonstrate the antitumor (Andreani *et al.*, 2008), antiallergic (Ager *et al.*, 1988), anesthetic (Sanfilippo *et al.*, 1988) and anti-cancer (Kumbhare *et al.*, 2011) activities as well as the inhibition activity of apoptosis in testiculargerm cells (Chandak *et al.*, 2013), lymphocytes (Barchéchath *et al.*, 2005), and FMS-like tyrosine kinase-3 (FLT3) (Chao *et al.*, 2009).

In this work, a 5,6,7,8-tetrahydroimidazo[2,1-*b*][1,3]benzothiazole, $C_{15}H_{14}N_2S$, (**I**) was prepared by the reaction of 5,6,7,8-tetrahydrobenzothiazole-2-amine with 2-bromo-1-phenylethanone (Fig. 1), and its structure was unambiguously established by the X-ray diffraction study (Fig. 2).

The bond lengths and angles within the molecule of **I** are in a good agreement with those found in the related compounds (Landreau *et al.*, 2002; Adib *et al.*, 2008; Fun, Asik *et al.*, 2011; Fun, Hemamalini *et al.*, 2011; Ghabbour *et al.*, 2012; Bunev *et al.*, 2013*a*,*b*; Bunev *et al.*, 2014).

The title compound, $C_{15}H_{14}N_2S$ (**I**), crystallizes with two crystallographically independent molecules in the asymmetric unit (Fig. 2). The central imidazo[2,1-*b*][1,3]benzothiazole moiety is planar (r.m.s. deviation = 0.010 and 0.008 Å, respectively, for the two crystallographically independent molecules). The fused tetrahydrohexane ring adopts a *half-chair* conformation (the C6, C7 and C21, C22 carbon atoms are out of the planes passed through the other atoms of the rings by 0.411 (6), -0.314 (6) and 0.387 (6), -0.355 (6) Å, respectively, for the two crystallographically independent molecules). The phenyl substituent is twisted by 16.96 (13) and 22.89 (12) ° (for the two crystallographically independent molecules, respectively) relative to the central imidazo[2,1-*b*][1,3]benzothiazole moiety.

In the crystal, the molecules of **I** are arrangement at van der Waals distances.

S2. Experimental

A mixture of 5,6,7,8-tetrahydrobenzothiazole–2–amine (1.54 g, 10 mmol) and 2–bromo–1–phenylethanone (1.99 g, 10 mmol) was dissolved in acetone (35 mL). The reaction mixture was stirred for 24 h. The resulting precipitate was collected, suspended in *Et*OH (50 mL) containing 6*N* HCl (5 mL) and heated under reflux. After cooling up to room temperature, the solution basified with 20% NH_4OH yielded the expected 5,6,7,8–tetrahydroimidazo[2,1–*b*][1,3]benzo-thiazole. The crude product was crystallized from EtOH. Yield is 82%. The single crystals of the product were obtained by slow crystallization from *Et*OH. M.p. = 440–442 K. IR (KBr), ν/cm^{-1}: 3137, 2933, 1602, 1539, 1465, 1438, 774, 718, 555. ^{1}H NMR (400 MHz, DMSO–d_6, 304 K): δ = 1.74–1.70 (m, 4H), 2.36–2.33 (m, 2H), 2.57–2.54 (m, 2H), 5.76 (s, 1H), 7.64 (t, 2H, J = 7.6), 7.78–7.75 (m, 1H), 8.06 (d, 2H, J = 7.9). Anal. Calcd for $C_{15}H_{14}N_2S$: C, 70.83; H, 5.55. Found: C, 70.91; H, 5.62.

S3. Refinement

All hydrogen atoms were placed in the calculated positions with C—H = 0.95 (aryl H) and 0.99 (methylene H) Å and refined in the riding model with fixed isotropic displacement parameters: U_{iso}(H) = 1.2U_{eq}(C).

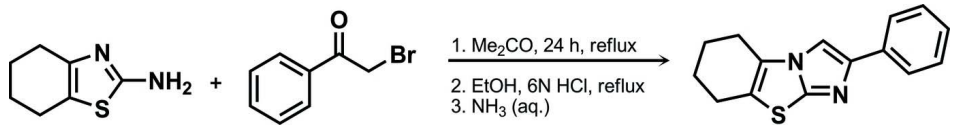

Figure 1

Synthesis of 2-phenyl-5,6,7,8-tetrahydroimidazo[2,1–*b*][1,3]benzothiazole.

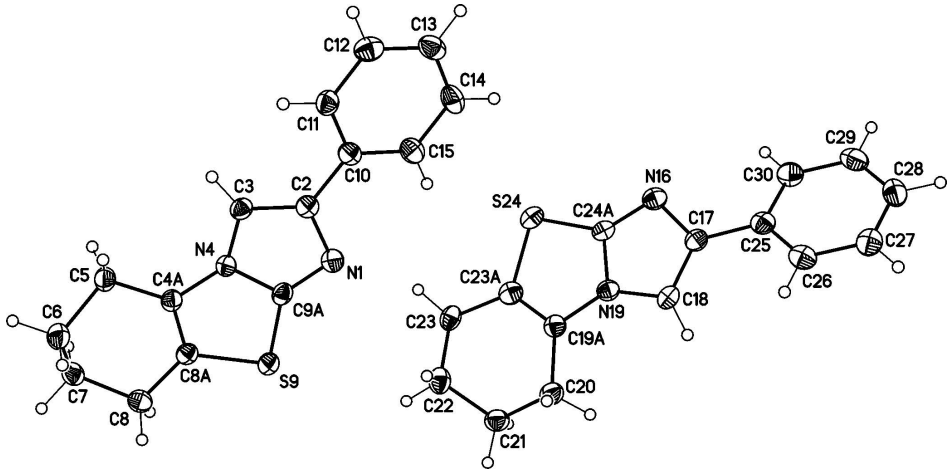

Figure 2

Molecular structure of **I** (two crystallographically independent molecules are presented). Displacement ellipsoids are shown at the 50% probability level. H atoms are depicted as small spheres of arbitrary radius.

2-Phenyl-5,6,7,8-tetrahydroimidazo[2,1-*b*][1,3]benzothiazole

Crystal data

$C_{15}H_{14}N_2S$
M_r = 254.34
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 12.523 (3) Å
b = 10.699 (3) Å
c = 18.930 (5) Å
β = 102.291 (6)°
V = 2478.2 (11) Å^3
Z = 8

$F(000)$ = 1072
D_x = 1.363 Mg m^{-3}
Melting point = 440–442 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 769 reflections
θ = 2.2–19.4°
μ = 0.24 mm^{-1}
T = 120 K
Needle, colourless
0.30 × 0.05 × 0.03 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.931, T_{max} = 0.993

33479 measured reflections
7558 independent reflections
3223 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 30.5°, θ_{min} = 1.8°
h = −17→17
k = −15→15
l = −27→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.157$
$S = 0.93$
7558 reflections
325 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
$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*–factor w*R* 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 $F^2 > 2\sigma(F^2)$ 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}
N1	0.3882 (2)	0.6184 (2)	0.11553 (14)	0.0237 (6)
C2	0.3823 (3)	0.5036 (3)	0.14946 (16)	0.0212 (7)
C3	0.4704 (3)	0.4301 (3)	0.14592 (16)	0.0219 (7)
H3	0.4850	0.3481	0.1647	0.026*
N4	0.5337 (2)	0.5003 (2)	0.10923 (14)	0.0205 (6)
C4A	0.6340 (3)	0.4902 (3)	0.08809 (16)	0.0209 (7)
C5	0.7062 (2)	0.3795 (3)	0.10652 (18)	0.0248 (7)
H5A	0.6796	0.3105	0.0725	0.030*
H5B	0.7049	0.3508	0.1560	0.030*
C6	0.8230 (3)	0.4141 (3)	0.10196 (18)	0.0277 (8)
H6A	0.8567	0.4646	0.1447	0.033*
H6B	0.8668	0.3370	0.1024	0.033*
C7	0.8246 (3)	0.4884 (3)	0.03323 (17)	0.0254 (7)
H7A	0.7920	0.4368	−0.0093	0.030*
H7B	0.9014	0.5058	0.0309	0.030*
C8	0.7619 (3)	0.6131 (3)	0.02925 (17)	0.0255 (7)
H8A	0.8072	0.6760	0.0605	0.031*
H8B	0.7463	0.6448	−0.0210	0.031*
C8A	0.6568 (2)	0.5938 (3)	0.05377 (16)	0.0212 (7)
S9	0.55424 (7)	0.70942 (8)	0.04714 (5)	0.0253 (2)
C9A	0.4804 (3)	0.6118 (3)	0.09283 (16)	0.0213 (7)
C10	0.2878 (3)	0.4714 (3)	0.18064 (17)	0.0229 (7)
C11	0.2908 (3)	0.3708 (3)	0.22804 (17)	0.0250 (7)
H11	0.3563	0.3241	0.2426	0.030*
C12	0.1987 (3)	0.3383 (3)	0.25409 (17)	0.0292 (8)

H12	0.2018	0.2695	0.2862	0.035*
C13	0.1024 (3)	0.4053 (3)	0.23360 (18)	0.0297 (8)
H13	0.0393	0.3822	0.2509	0.036*
C14	0.0992 (3)	0.5066 (3)	0.18754 (18)	0.0297 (8)
H14	0.0339	0.5539	0.1737	0.036*
C15	0.1911 (3)	0.5393 (3)	0.16149 (17)	0.0267 (8)
H15	0.1879	0.6091	0.1301	0.032*
N16	−0.1213 (2)	0.9124 (2)	0.11849 (14)	0.0252 (6)
C17	−0.1096 (3)	1.0225 (3)	0.15894 (17)	0.0224 (7)
C18	−0.0110 (3)	1.0785 (3)	0.15889 (16)	0.0230 (7)
H18	0.0156	1.1548	0.1818	0.028*
N19	0.0414 (2)	1.0016 (2)	0.11889 (14)	0.0218 (6)
C19A	0.1393 (3)	0.9974 (3)	0.09439 (17)	0.0230 (7)
C20	0.2241 (3)	1.0980 (3)	0.11148 (17)	0.0260 (8)
H20A	0.2640	1.0906	0.1624	0.031*
H20B	0.1887	1.1811	0.1049	0.031*
C21	0.3034 (3)	1.0841 (3)	0.06097 (18)	0.0278 (8)
H21A	0.3690	1.1358	0.0793	0.033*
H21B	0.2683	1.1156	0.0124	0.033*
C22	0.3384 (3)	0.9486 (3)	0.05454 (18)	0.0282 (8)
H22A	0.3731	0.9166	0.1031	0.034*
H22B	0.3934	0.9450	0.0239	0.034*
C23	0.2406 (3)	0.8651 (3)	0.02141 (17)	0.0252 (8)
H23A	0.2206	0.8784	−0.0315	0.030*
H23B	0.2608	0.7761	0.0304	0.030*
C23A	0.1451 (3)	0.8957 (3)	0.05424 (17)	0.0252 (7)
S24	0.02778 (7)	0.79992 (8)	0.04362 (5)	0.0269 (2)
C24A	−0.0288 (3)	0.9037 (3)	0.09640 (16)	0.0218 (7)
C25	−0.1985 (3)	1.0704 (3)	0.19086 (17)	0.0247 (7)
C26	−0.1767 (3)	1.1565 (3)	0.24797 (17)	0.0266 (8)
H26	−0.1036	1.1819	0.2669	0.032*
C27	−0.2608 (3)	1.2052 (3)	0.27725 (17)	0.0284 (8)
H27	−0.2448	1.2638	0.3158	0.034*
C28	−0.3676 (3)	1.1686 (3)	0.25042 (18)	0.0309 (8)
H28	−0.4250	1.2023	0.2704	0.037*
C29	−0.3911 (3)	1.0825 (3)	0.19427 (19)	0.0320 (8)
H29	−0.4645	1.0571	0.1760	0.038*
C30	−0.3071 (3)	1.0334 (3)	0.16478 (18)	0.0288 (8)
H30	−0.3236	0.9742	0.1266	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0236 (16)	0.0247 (16)	0.0223 (15)	0.0008 (12)	0.0038 (12)	0.0008 (11)
C2	0.0226 (18)	0.0226 (18)	0.0176 (16)	−0.0013 (13)	0.0027 (13)	−0.0033 (13)
C3	0.0264 (19)	0.0189 (17)	0.0217 (17)	0.0003 (13)	0.0082 (14)	0.0012 (13)
N4	0.0203 (15)	0.0194 (14)	0.0222 (15)	0.0007 (11)	0.0056 (11)	−0.0013 (11)
C4A	0.0207 (18)	0.0205 (17)	0.0207 (17)	0.0004 (13)	0.0025 (13)	−0.0032 (13)

C5	0.0242 (19)	0.0234 (18)	0.0280 (19)	0.0038 (14)	0.0087 (14)	0.0019 (14)
C6	0.0255 (19)	0.0277 (19)	0.0293 (19)	0.0043 (15)	0.0041 (15)	0.0022 (15)
C7	0.0232 (18)	0.0280 (19)	0.0267 (19)	−0.0010 (14)	0.0093 (14)	−0.0037 (14)
C8	0.0258 (19)	0.0275 (19)	0.0237 (18)	−0.0024 (14)	0.0065 (14)	0.0008 (14)
C8A	0.0233 (18)	0.0203 (17)	0.0194 (17)	−0.0003 (13)	0.0031 (13)	−0.0019 (13)
S9	0.0254 (5)	0.0210 (4)	0.0300 (5)	0.0024 (4)	0.0073 (4)	0.0033 (4)
C9A	0.0233 (18)	0.0189 (17)	0.0208 (17)	0.0013 (13)	0.0025 (13)	−0.0038 (13)
C10	0.0221 (18)	0.0250 (18)	0.0218 (17)	−0.0025 (13)	0.0053 (14)	−0.0044 (13)
C11	0.0261 (19)	0.0234 (18)	0.0262 (19)	0.0019 (14)	0.0071 (14)	−0.0031 (14)
C12	0.038 (2)	0.0266 (19)	0.0237 (19)	−0.0050 (15)	0.0087 (16)	−0.0005 (14)
C13	0.027 (2)	0.033 (2)	0.031 (2)	−0.0085 (16)	0.0101 (15)	−0.0087 (16)
C14	0.0192 (18)	0.040 (2)	0.029 (2)	0.0017 (15)	0.0030 (15)	−0.0046 (16)
C15	0.0261 (19)	0.033 (2)	0.0224 (18)	−0.0005 (15)	0.0074 (14)	−0.0006 (14)
N16	0.0287 (17)	0.0234 (15)	0.0229 (15)	−0.0014 (12)	0.0040 (12)	0.0014 (12)
C17	0.0249 (19)	0.0205 (17)	0.0209 (17)	0.0003 (13)	0.0032 (14)	0.0006 (13)
C18	0.0278 (19)	0.0188 (17)	0.0220 (18)	0.0022 (14)	0.0038 (14)	−0.0012 (13)
N19	0.0238 (15)	0.0209 (14)	0.0201 (15)	−0.0018 (11)	0.0035 (11)	−0.0012 (11)
C19A	0.0231 (18)	0.0224 (18)	0.0224 (18)	0.0016 (14)	0.0026 (14)	0.0025 (13)
C20	0.0263 (19)	0.0229 (18)	0.0274 (19)	−0.0010 (14)	0.0024 (14)	−0.0018 (14)
C21	0.027 (2)	0.0255 (19)	0.030 (2)	−0.0035 (15)	0.0035 (15)	−0.0016 (15)
C22	0.030 (2)	0.029 (2)	0.0267 (19)	0.0067 (15)	0.0085 (15)	0.0044 (14)
C23	0.031 (2)	0.0228 (18)	0.0215 (18)	0.0062 (14)	0.0054 (15)	0.0026 (13)
C23A	0.029 (2)	0.0241 (19)	0.0217 (18)	0.0014 (14)	0.0037 (14)	0.0059 (14)
S24	0.0317 (5)	0.0198 (4)	0.0286 (5)	−0.0002 (4)	0.0051 (4)	−0.0023 (4)
C24A	0.0260 (19)	0.0176 (17)	0.0213 (17)	−0.0016 (13)	0.0037 (14)	0.0000 (13)
C25	0.028 (2)	0.0225 (18)	0.0238 (18)	0.0006 (14)	0.0050 (14)	0.0070 (14)
C26	0.027 (2)	0.030 (2)	0.0217 (18)	−0.0011 (14)	0.0031 (14)	0.0086 (14)
C27	0.036 (2)	0.0271 (19)	0.0227 (18)	0.0041 (16)	0.0075 (15)	0.0067 (15)
C28	0.033 (2)	0.031 (2)	0.032 (2)	0.0081 (16)	0.0136 (16)	0.0109 (15)
C29	0.024 (2)	0.032 (2)	0.039 (2)	−0.0034 (15)	0.0054 (16)	0.0086 (17)
C30	0.029 (2)	0.030 (2)	0.0269 (19)	−0.0025 (15)	0.0032 (15)	0.0002 (14)

Geometric parameters (Å, º)

N1—C9A	1.317 (4)	N16—C24A	1.316 (4)
N1—C2	1.395 (4)	N16—C17	1.395 (4)
C2—C3	1.368 (4)	C17—C18	1.373 (4)
C2—C10	1.472 (4)	C17—C25	1.468 (4)
C3—N4	1.382 (4)	C18—N19	1.376 (4)
C3—H3	0.9500	C18—H18	0.9500
N4—C9A	1.370 (4)	N19—C24A	1.376 (4)
N4—C4A	1.400 (4)	N19—C19A	1.400 (4)
C4A—C8A	1.346 (4)	C19A—C23A	1.338 (4)
C4A—C5	1.486 (4)	C19A—C20	1.498 (4)
C5—C6	1.529 (4)	C20—C21	1.526 (4)
C5—H5A	0.9900	C20—H20A	0.9900
C5—H5B	0.9900	C20—H20B	0.9900
C6—C7	1.528 (4)	C21—C22	1.527 (4)

C6—H6A	0.9900	C21—H21A	0.9900
C6—H6B	0.9900	C21—H21B	0.9900
C7—C8	1.542 (4)	C22—C23	1.538 (4)
C7—H7A	0.9900	C22—H22A	0.9900
C7—H7B	0.9900	C22—H22B	0.9900
C8—C8A	1.500 (4)	C23—C23A	1.497 (4)
C8—H8A	0.9900	C23—H23A	0.9900
C8—H8B	0.9900	C23—H23B	0.9900
C8A—S9	1.769 (3)	C23A—S24	1.767 (3)
S9—C9A	1.742 (3)	S24—C24A	1.742 (3)
C10—C15	1.392 (4)	C25—C30	1.401 (4)
C10—C11	1.397 (4)	C25—C26	1.402 (4)
C11—C12	1.392 (4)	C26—C27	1.392 (4)
C11—H11	0.9500	C26—H26	0.9500
C12—C13	1.385 (5)	C27—C28	1.382 (5)
C12—H12	0.9500	C27—H27	0.9500
C13—C14	1.386 (5)	C28—C29	1.390 (5)
C13—H13	0.9500	C28—H28	0.9500
C14—C15	1.390 (4)	C29—C30	1.395 (5)
C14—H14	0.9500	C29—H29	0.9500
C15—H15	0.9500	C30—H30	0.9500

C9A—N1—C2	103.8 (3)	C24A—N16—C17	103.7 (3)
C3—C2—N1	111.2 (3)	C18—C17—N16	111.0 (3)
C3—C2—C10	127.7 (3)	C18—C17—C25	127.6 (3)
N1—C2—C10	121.1 (3)	N16—C17—C25	121.3 (3)
C2—C3—N4	105.5 (3)	C17—C18—N19	105.9 (3)
C2—C3—H3	127.2	C17—C18—H18	127.1
N4—C3—H3	127.2	N19—C18—H18	127.1
C9A—N4—C3	106.4 (3)	C18—N19—C24A	106.1 (3)
C9A—N4—C4A	115.2 (3)	C18—N19—C19A	139.1 (3)
C3—N4—C4A	138.3 (3)	C24A—N19—C19A	114.8 (3)
C8A—C4A—N4	111.7 (3)	C23A—C19A—N19	111.8 (3)
C8A—C4A—C5	126.0 (3)	C23A—C19A—C20	125.8 (3)
N4—C4A—C5	122.2 (3)	N19—C19A—C20	122.4 (3)
C4A—C5—C6	109.6 (3)	C19A—C20—C21	108.7 (3)
C4A—C5—H5A	109.7	C19A—C20—H20A	109.9
C6—C5—H5A	109.7	C21—C20—H20A	109.9
C4A—C5—H5B	109.7	C19A—C20—H20B	109.9
C6—C5—H5B	109.7	C21—C20—H20B	109.9
H5A—C5—H5B	108.2	H20A—C20—H20B	108.3
C7—C6—C5	111.1 (3)	C20—C21—C22	112.2 (3)
C7—C6—H6A	109.4	C20—C21—H21A	109.2
C5—C6—H6A	109.4	C22—C21—H21A	109.2
C7—C6—H6B	109.4	C20—C21—H21B	109.2
C5—C6—H6B	109.4	C22—C21—H21B	109.2
H6A—C6—H6B	108.0	H21A—C21—H21B	107.9
C6—C7—C8	113.1 (3)	C21—C22—C23	111.5 (3)

C6—C7—H7A	109.0	C21—C22—H22A	109.3
C8—C7—H7A	109.0	C23—C22—H22A	109.3
C6—C7—H7B	109.0	C21—C22—H22B	109.3
C8—C7—H7B	109.0	C23—C22—H22B	109.3
H7A—C7—H7B	107.8	H22A—C22—H22B	108.0
C8A—C8—C7	109.7 (3)	C23A—C23—C22	109.9 (3)
C8A—C8—H8A	109.7	C23A—C23—H23A	109.7
C7—C8—H8A	109.7	C22—C23—H23A	109.7
C8A—C8—H8B	109.7	C23A—C23—H23B	109.7
C7—C8—H8B	109.7	C22—C23—H23B	109.7
H8A—C8—H8B	108.2	H23A—C23—H23B	108.2
C4A—C8A—C8	123.6 (3)	C19A—C23A—C23	124.0 (3)
C4A—C8A—S9	112.6 (2)	C19A—C23A—S24	113.1 (3)
C8—C8A—S9	123.6 (2)	C23—C23A—S24	123.0 (2)
C9A—S9—C8A	89.97 (15)	C24A—S24—C23A	89.70 (15)
N1—C9A—N4	113.1 (3)	N16—C24A—N19	113.3 (3)
N1—C9A—S9	136.4 (3)	N16—C24A—S24	136.1 (3)
N4—C9A—S9	110.5 (2)	N19—C24A—S24	110.6 (2)
C15—C10—C11	118.2 (3)	C30—C25—C26	118.2 (3)
C15—C10—C2	120.2 (3)	C30—C25—C17	121.4 (3)
C11—C10—C2	121.6 (3)	C26—C25—C17	120.4 (3)
C12—C11—C10	120.6 (3)	C27—C26—C25	120.9 (3)
C12—C11—H11	119.7	C27—C26—H26	119.6
C10—C11—H11	119.7	C25—C26—H26	119.6
C13—C12—C11	120.6 (3)	C28—C27—C26	120.2 (3)
C13—C12—H12	119.7	C28—C27—H27	119.9
C11—C12—H12	119.7	C26—C27—H27	119.9
C12—C13—C14	119.2 (3)	C27—C28—C29	120.0 (3)
C12—C13—H13	120.4	C27—C28—H28	120.0
C14—C13—H13	120.4	C29—C28—H28	120.0
C13—C14—C15	120.4 (3)	C28—C29—C30	120.1 (3)
C13—C14—H14	119.8	C28—C29—H29	120.0
C15—C14—H14	119.8	C30—C29—H29	120.0
C14—C15—C10	121.1 (3)	C29—C30—C25	120.7 (3)
C14—C15—H15	119.5	C29—C30—H30	119.6
C10—C15—H15	119.5	C25—C30—H30	119.6

C9A—N1—C2—C3	−0.5 (3)	C24A—N16—C17—C18	1.2 (4)
C9A—N1—C2—C10	−177.9 (3)	C24A—N16—C17—C25	177.0 (3)
N1—C2—C3—N4	0.3 (4)	N16—C17—C18—N19	−1.1 (4)
C10—C2—C3—N4	177.5 (3)	C25—C17—C18—N19	−176.5 (3)
C2—C3—N4—C9A	0.0 (3)	C17—C18—N19—C24A	0.5 (3)
C2—C3—N4—C4A	177.0 (3)	C17—C18—N19—C19A	178.2 (3)
C9A—N4—C4A—C8A	−1.3 (4)	C18—N19—C19A—C23A	−178.6 (3)
C3—N4—C4A—C8A	−178.1 (3)	C24A—N19—C19A—C23A	−1.0 (4)
C9A—N4—C4A—C5	175.3 (3)	C18—N19—C19A—C20	0.5 (6)
C3—N4—C4A—C5	−1.5 (6)	C24A—N19—C19A—C20	178.1 (3)
C8A—C4A—C5—C6	17.0 (4)	C23A—C19A—C20—C21	14.3 (4)

N4—C4A—C5—C6	−159.1 (3)	N19—C19A—C20—C21	−164.7 (3)
C4A—C5—C6—C7	−45.4 (4)	C19A—C20—C21—C22	−45.1 (4)
C5—C6—C7—C8	61.3 (4)	C20—C21—C22—C23	62.7 (4)
C6—C7—C8—C8A	−42.5 (4)	C21—C22—C23—C23A	−43.5 (4)
N4—C4A—C8A—C8	175.9 (3)	N19—C19A—C23A—C23	−179.2 (3)
C5—C4A—C8A—C8	−0.6 (5)	C20—C19A—C23A—C23	1.7 (5)
N4—C4A—C8A—S9	0.6 (3)	N19—C19A—C23A—S24	0.8 (4)
C5—C4A—C8A—S9	−175.9 (3)	C20—C19A—C23A—S24	−178.3 (2)
C7—C8—C8A—C4A	12.9 (4)	C22—C23—C23A—C19A	13.0 (4)
C7—C8—C8A—S9	−172.2 (2)	C22—C23—C23A—S24	−167.0 (2)
C4A—C8A—S9—C9A	0.2 (3)	C19A—C23A—S24—C24A	−0.3 (3)
C8—C8A—S9—C9A	−175.1 (3)	C23—C23A—S24—C24A	179.7 (3)
C2—N1—C9A—N4	0.6 (3)	C17—N16—C24A—N19	−0.9 (3)
C2—N1—C9A—S9	−178.9 (3)	C17—N16—C24A—S24	−179.3 (3)
C3—N4—C9A—N1	−0.4 (4)	C18—N19—C24A—N16	0.3 (4)
C4A—N4—C9A—N1	−178.2 (3)	C19A—N19—C24A—N16	−178.0 (3)
C3—N4—C9A—S9	179.2 (2)	C18—N19—C24A—S24	179.1 (2)
C4A—N4—C9A—S9	1.5 (3)	C19A—N19—C24A—S24	0.8 (3)
C8A—S9—C9A—N1	178.6 (4)	C23A—S24—C24A—N16	178.2 (4)
C8A—S9—C9A—N4	−0.9 (2)	C23A—S24—C24A—N19	−0.3 (2)
C3—C2—C10—C15	−160.7 (3)	C18—C17—C25—C30	154.6 (3)
N1—C2—C10—C15	16.3 (5)	N16—C17—C25—C30	−20.4 (5)
C3—C2—C10—C11	17.3 (5)	C18—C17—C25—C26	−24.4 (5)
N1—C2—C10—C11	−165.7 (3)	N16—C17—C25—C26	160.6 (3)
C15—C10—C11—C12	1.2 (5)	C30—C25—C26—C27	−0.9 (5)
C2—C10—C11—C12	−176.8 (3)	C17—C25—C26—C27	178.1 (3)
C10—C11—C12—C13	−0.1 (5)	C25—C26—C27—C28	0.3 (5)
C11—C12—C13—C14	−0.9 (5)	C26—C27—C28—C29	0.3 (5)
C12—C13—C14—C15	0.9 (5)	C27—C28—C29—C30	−0.3 (5)
C13—C14—C15—C10	0.2 (5)	C28—C29—C30—C25	−0.3 (5)
C11—C10—C15—C14	−1.2 (5)	C26—C25—C30—C29	0.9 (5)
C2—C10—C15—C14	176.8 (3)	C17—C25—C30—C29	−178.1 (3)