

1-(10,11-Dihydrodibenz[*b,f*]azepin-5-yl)ethanone

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Key indicators

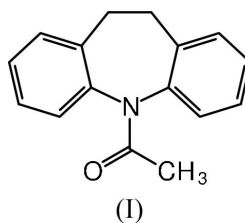
Single-crystal X-ray study
T = 150 K
Mean $\sigma(\text{C}—\text{C})$ = 0.002 Å
R factor = 0.031
wR factor = 0.076
Data-to-parameter ratio = 8.5

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The asymmetric unit of the title compound, C₁₆H₁₅NO, comprises two independent molecules (*A* and *B*), both adopting a half-boat conformation, or butterfly shape. The intramolecular dihedral angles between the benzene rings in *A* and *B* are 64.40 (4) and 65.24 (5)°, respectively.

Comment

The title compound, (I), is used as an intermediate for the synthesis of carbamazepine and oxcarbazepine (Kricka & Ledwith, 1974), two anticonvulsant drugs whose structures have been reported [Grzesiak *et al.*, 2003 (most recent form); Hempel *et al.*, 2005]. As part of a series of studies into the structural aspects of iminostilbene analogues, the structure of (I) was determined and is reported here. A search of the Cambridge Structural Database (November 2004 version; Allen, 2002) reveals that there are 27 compounds reported that contain a 10,11-dihydrodibenz[*b,f*]azepine moiety with only two containing an additional *N*-acetyl group, *viz.* the *N*-acetyldibenz[*b,f*]azepine dimer (Harding, 1983) and its hydrate structure (Taga *et al.*, 1986). The structure of (I) (Fig. 1) comprises two independent molecules, *A* and *B*, in the asymmetric unit, both of which adopt a half-boat conformation (Cremer & Pople, 1975) or butterfly shape. The intramolecular dihedral angles between the benzene rings in *A* and *B* are 64.40 (4) and 65.24 (5)°, respectively.



Experimental

The title compound was prepared by refluxing 10,11-dihydro-5*H*-dibenz[*b,f*]azepine (1.95 g, 10 mmol) in acetic anhydride (5 ml) for 6 h. Crystals were grown from methanol.

Crystal data

C₁₆H₁₅NO
M_r = 237.29
Orthorhombic, *P*2₁2₁2₁
a = 9.5674 (2) Å
b = 11.7020 (3) Å
c = 22.2785 (4) Å
V = 2494.25 (9) Å³
Z = 8
D_x = 1.264 Mg m^{−3}

Mo *K*α radiation
Cell parameters from 3179 reflections
 θ = 2.9–27.5°
 μ = 0.08 mm^{−1}
T = 150 (2) K
Prism, colourless
0.60 × 0.40 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2003)
 $T_{\min} = 0.954$, $T_{\max} = 0.992$
 19 122 measured reflections
 2778 independent reflections

2540 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
 $\theta_{\max} = 26.0^\circ$
 $h = -11 \rightarrow 11$
 $k = -14 \rightarrow 14$
 $l = -27 \rightarrow 27$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.076$
 $S = 1.02$
 2778 reflections
 328 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.3098P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0285 (14)

All H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.95 (ArH), 0.98 (CH₃) and 0.99 Å (CH₂). The isotropic displacement parameters for all H atoms were set equal to 1.25 U_{eq} of the carrier atom. The absolute configuration could not be accurately determined from the diffraction data, thus 1600 Friedel opposites were merged and the configuration arbitrarily assigned. The number of Friedel pairs is 1660.

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97.

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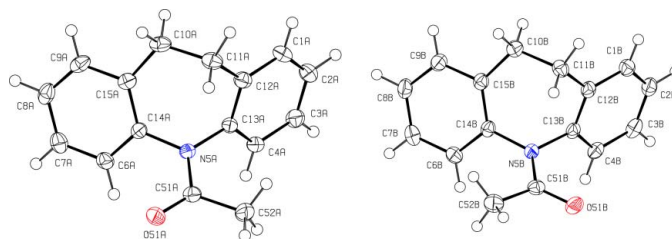


Figure 1

The molecular configuration and atom-numbering scheme for both independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius. The molecules are shown with similar view directions and not in their true relative orientations.

the EPSRC's Chemical Database Service at Daresbury (Fletcher *et al.*, 1996).

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supporting information

Acta Cryst. (2005). E61, o1609–o1610 [https://doi.org/10.1107/S1600536805013115]

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1-(10,11-Dihydrodibenz[*b,f*]azepin-5-yl)ethanone*Crystal data*

$C_{16}H_{15}NO$

$M_r = 237.29$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 9.5674$ (2) Å

$b = 11.7020$ (3) Å

$c = 22.2785$ (4) Å

$V = 2494.25$ (9) Å³

$Z = 8$

$F(000) = 1008$

$D_x = 1.264$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3179 reflections

$\theta = 2.9$ – 27.5°

$\mu = 0.08$ mm⁻¹

$T = 150$ K

Prism, colourless

$0.60 \times 0.40 \times 0.10$ mm

Data collection

Nonius KappaCCD

diffractometer

Radiation source: Bruker Nonius FR591

rotating anode

10 cm confocal mirrors monochromator

Detector resolution: 9.091 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 2003)

$T_{\min} = 0.954$, $T_{\max} = 0.992$

19122 measured reflections

2778 independent reflections

2540 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 3.3^\circ$

$h = -11 \rightarrow 11$

$k = -14 \rightarrow 14$

$l = -27 \rightarrow 27$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.076$

$S = 1.02$

2778 reflections

328 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

$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.3098P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.26$ e Å⁻³

$\Delta\rho_{\min} = -0.22$ e Å⁻³

Extinction correction: SHELXL97,

$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0285 (14)

Special details

Experimental. The minimum and maximum absorption values stated above are those calculated in *SHELXL97* from the given crystal dimensions. The ratio of minimum to maximum apparent transmission was determined experimentally as 0.800837.

Geometry. Least-squares planes (x, y, z in crystal coordinates) and deviations from them (* indicates atom used to define plane)

$2.9908 (0.0055) x + 5.0064 (0.0065) y + 18.8940 (0.0072) z = 7.7832 (0.0024)$

* $0.0017 (0.0011) \text{C1A}$ * $0.0033 (0.0011) \text{C2A}$ * $-0.0055 (0.0010) \text{C3A}$ * $0.0026 (0.0010) \text{C4A}$ * $0.0024 (0.0010) \text{C13A}$ * $-0.0045 (0.0010) \text{C12A}$

Rms deviation of fitted atoms = 0.0036

$6.8470 (0.0036) x - 2.5291 (0.0063) y - 14.7967 (0.0088) z = 0.8469 (0.0065)$

Angle to previous plane (with approximate e.s.d.) = 64.40 (0.04)

* $-0.0036 (0.0009) \text{C6A}$ * $0.0049 (0.0010) \text{C7A}$ * $-0.0019 (0.0010) \text{C8A}$ * $-0.0023 (0.0009) \text{C9A}$ * $0.0035 (0.0009) \text{C15A}$ * $-0.0006 (0.0009) \text{C14A}$

Rms deviation of fitted atoms = 0.0031

$7.3973 (0.0036) x - 3.9017 (0.0064) y - 12.0187 (0.0111) z = 5.2566 (0.0031)$

Angle to previous plane (with approximate e.s.d.) = 10.36 (0.08)

* $-0.0087 (0.0010) \text{C6B}$ * $0.0061 (0.0010) \text{C7B}$ * $0.0017 (0.0011) \text{C8B}$ * $-0.0067 (0.0010) \text{C9B}$ * $0.0039 (0.0009) \text{C15B}$ * $0.0038 (0.0009) \text{C14B}$

Rms deviation of fitted atoms = 0.0056

$3.4314 (0.0053) x - 10.3952 (0.0032) y + 6.3889 (0.0124) z = 2.8116 (0.0024)$

Angle to previous plane (with approximate e.s.d.) = 65.24 (0.05)

* $0.0055 (0.0010) \text{C1B}$ * $-0.0076 (0.0010) \text{C2B}$ * $0.0014 (0.0010) \text{C3B}$ * $0.0069 (0.0010) \text{C4B}$ * $-0.0091 (0.0010) \text{C13B}$ * $0.0028 (0.0010) \text{C12B}$

Rms deviation of fitted atoms = 0.0062

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O51A	0.82550 (11)	0.28256 (9)	0.15509 (5)	0.0286 (3)
N5A	0.68701 (11)	0.42139 (10)	0.19408 (5)	0.0200 (3)
C1A	0.40748 (16)	0.63632 (14)	0.17892 (7)	0.0303 (4)
H1A	0.3935	0.7098	0.1618	0.038*
C2A	0.29936 (16)	0.58411 (14)	0.20995 (7)	0.0322 (4)
H2A	0.2122	0.6223	0.2140	0.040*
C3A	0.31716 (15)	0.47727 (13)	0.23498 (7)	0.0273 (3)
H3A	0.2424	0.4413	0.2557	0.034*
C4A	0.44473 (14)	0.42304 (12)	0.22959 (6)	0.0218 (3)
H4A	0.4584	0.3499	0.2471	0.027*
C6A	0.85940 (14)	0.39654 (13)	0.27291 (6)	0.0228 (3)
H6A	0.8329	0.3184	0.2745	0.029*
C7A	0.95983 (15)	0.43693 (14)	0.31190 (7)	0.0269 (3)
H7A	1.0031	0.3868	0.3398	0.034*
C8A	0.99707 (15)	0.55152 (14)	0.31001 (7)	0.0277 (4)
H8A	1.0653	0.5807	0.3369	0.035*
C9A	0.93397 (15)	0.62276 (13)	0.26866 (7)	0.0253 (3)
H9A	0.9602	0.7010	0.2677	0.032*
C10A	0.77174 (16)	0.67239 (12)	0.18546 (7)	0.0262 (3)
H10A	0.8500	0.7052	0.1620	0.033*
H11A	0.7324	0.7350	0.2101	0.033*
C11A	0.65919 (16)	0.63516 (13)	0.14111 (7)	0.0270 (3)
H12A	0.6274	0.7023	0.1178	0.034*
H13A	0.6994	0.5791	0.1126	0.034*
C12A	0.53623 (15)	0.58235 (12)	0.17251 (6)	0.0231 (3)
C13A	0.55270 (14)	0.47529 (12)	0.19864 (6)	0.0195 (3)

C14A	0.79621 (13)	0.46853 (12)	0.23116 (6)	0.0191 (3)
C15A	0.83272 (14)	0.58400 (12)	0.22804 (6)	0.0210 (3)
C51A	0.71104 (15)	0.32820 (12)	0.15771 (6)	0.0223 (3)
C52A	0.58861 (16)	0.28645 (14)	0.12150 (7)	0.0307 (4)
H51A	0.6223	0.2388	0.0883	0.038*
H52A	0.5372	0.3520	0.1053	0.038*
H53A	0.5266	0.2413	0.1472	0.038*
O51B	0.84508 (11)	−0.15032 (9)	0.07124 (4)	0.0285 (2)
N5B	0.73882 (12)	−0.00453 (10)	0.02391 (5)	0.0204 (3)
C1B	0.38389 (15)	−0.12928 (13)	0.02440 (6)	0.0252 (3)
H1B	0.3225	−0.1672	−0.0025	0.031*
C2B	0.34099 (16)	−0.10637 (13)	0.08267 (7)	0.0265 (3)
H2B	0.2496	−0.1274	0.0952	0.033*
C3B	0.43030 (16)	−0.05315 (13)	0.12269 (7)	0.0272 (3)
H3B	0.4006	−0.0386	0.1627	0.034*
C4B	0.56308 (16)	−0.02100 (12)	0.10457 (6)	0.0242 (3)
H4B	0.6252	0.0149	0.1319	0.030*
C6B	0.81569 (15)	0.18997 (12)	0.00373 (6)	0.0235 (3)
H6B	0.8693	0.1861	0.0396	0.029*
C7B	0.81555 (16)	0.28942 (13)	−0.02988 (7)	0.0278 (3)
H7B	0.8705	0.3531	−0.0178	0.035*
C8B	0.73456 (16)	0.29516 (14)	−0.08122 (7)	0.0287 (4)
H8B	0.7330	0.3632	−0.1045	0.036*
C9B	0.65584 (16)	0.20186 (13)	−0.09869 (7)	0.0271 (3)
H9B	0.6000	0.2076	−0.1338	0.034*
C10B	0.56362 (16)	0.00460 (13)	−0.09084 (6)	0.0265 (3)
H10B	0.5908	−0.0089	−0.1331	0.033*
H11B	0.4661	0.0328	−0.0911	0.033*
C11B	0.56505 (15)	−0.11070 (12)	−0.05849 (6)	0.0243 (3)
H12B	0.5028	−0.1649	−0.0797	0.030*
H13B	0.6609	−0.1425	−0.0590	0.030*
C12B	0.51687 (14)	−0.09688 (12)	0.00527 (6)	0.0214 (3)
C13B	0.60392 (14)	−0.04200 (12)	0.04596 (6)	0.0204 (3)
C14B	0.73789 (14)	0.09537 (12)	−0.01449 (6)	0.0200 (3)
C15B	0.65551 (14)	0.09932 (12)	−0.06648 (6)	0.0217 (3)
C51B	0.85326 (15)	−0.06939 (13)	0.03633 (6)	0.0224 (3)
C52B	0.98762 (15)	−0.04118 (15)	0.00479 (7)	0.0317 (4)
H51B	1.0385	0.0169	0.0278	0.040*
H52B	0.9671	−0.0117	−0.0354	0.040*
H53B	1.0450	−0.1103	0.0015	0.040*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O51A	0.0244 (6)	0.0261 (6)	0.0353 (6)	0.0062 (5)	0.0005 (4)	−0.0072 (5)
N5A	0.0178 (5)	0.0166 (6)	0.0257 (6)	−0.0004 (5)	0.0001 (5)	−0.0018 (5)
C1A	0.0297 (8)	0.0201 (8)	0.0410 (9)	0.0054 (7)	−0.0050 (7)	0.0027 (7)
C2A	0.0228 (7)	0.0267 (9)	0.0471 (9)	0.0059 (7)	−0.0010 (7)	−0.0040 (7)

C3A	0.0211 (7)	0.0259 (9)	0.0348 (8)	−0.0013 (6)	0.0045 (6)	−0.0032 (7)
C4A	0.0229 (7)	0.0176 (8)	0.0247 (7)	0.0001 (6)	−0.0002 (6)	−0.0002 (6)
C6A	0.0192 (7)	0.0220 (8)	0.0273 (7)	0.0028 (6)	0.0039 (6)	−0.0014 (6)
C7A	0.0203 (7)	0.0335 (9)	0.0268 (7)	0.0047 (7)	0.0024 (6)	−0.0009 (6)
C8A	0.0186 (7)	0.0361 (9)	0.0284 (8)	−0.0026 (7)	0.0021 (6)	−0.0104 (7)
C9A	0.0238 (7)	0.0231 (8)	0.0290 (7)	−0.0057 (6)	0.0090 (6)	−0.0087 (6)
C10A	0.0302 (8)	0.0179 (8)	0.0304 (8)	−0.0020 (6)	0.0074 (7)	0.0017 (6)
C11A	0.0323 (8)	0.0215 (8)	0.0274 (8)	0.0003 (7)	0.0027 (6)	0.0060 (6)
C12A	0.0260 (7)	0.0204 (8)	0.0229 (7)	0.0015 (6)	−0.0013 (6)	0.0004 (6)
C13A	0.0184 (7)	0.0182 (8)	0.0220 (7)	0.0015 (6)	−0.0005 (6)	−0.0040 (6)
C14A	0.0156 (6)	0.0198 (7)	0.0219 (6)	−0.0002 (6)	0.0037 (6)	−0.0043 (6)
C15A	0.0200 (7)	0.0206 (7)	0.0224 (7)	−0.0014 (6)	0.0079 (6)	−0.0038 (6)
C51A	0.0237 (8)	0.0187 (8)	0.0245 (7)	−0.0003 (6)	0.0019 (6)	0.0002 (6)
C52A	0.0279 (8)	0.0268 (9)	0.0373 (9)	0.0014 (7)	−0.0027 (7)	−0.0098 (7)
O51B	0.0315 (6)	0.0245 (6)	0.0295 (5)	0.0037 (5)	−0.0040 (5)	0.0058 (4)
N5B	0.0189 (6)	0.0197 (6)	0.0225 (6)	−0.0005 (5)	−0.0011 (5)	0.0039 (5)
C1B	0.0246 (7)	0.0203 (8)	0.0306 (8)	0.0001 (6)	−0.0011 (6)	0.0038 (6)
C2B	0.0237 (7)	0.0249 (8)	0.0310 (8)	0.0025 (7)	0.0040 (6)	0.0079 (6)
C3B	0.0323 (8)	0.0253 (8)	0.0240 (7)	0.0071 (7)	0.0072 (7)	0.0063 (6)
C4B	0.0303 (8)	0.0211 (8)	0.0212 (7)	0.0017 (7)	−0.0017 (6)	0.0039 (6)
C6B	0.0198 (7)	0.0255 (8)	0.0253 (7)	−0.0005 (6)	0.0010 (6)	−0.0015 (6)
C7B	0.0269 (8)	0.0219 (8)	0.0347 (8)	−0.0041 (7)	0.0064 (7)	−0.0012 (6)
C8B	0.0272 (8)	0.0231 (8)	0.0359 (8)	0.0001 (7)	0.0045 (7)	0.0096 (6)
C9B	0.0242 (8)	0.0301 (9)	0.0271 (7)	0.0010 (7)	0.0005 (6)	0.0073 (6)
C10B	0.0291 (8)	0.0292 (8)	0.0214 (7)	−0.0045 (7)	−0.0015 (6)	0.0015 (6)
C11B	0.0245 (7)	0.0235 (8)	0.0250 (7)	−0.0032 (6)	0.0005 (6)	−0.0029 (6)
C12B	0.0236 (7)	0.0155 (7)	0.0251 (7)	0.0012 (6)	0.0002 (6)	0.0029 (6)
C13B	0.0216 (7)	0.0160 (7)	0.0237 (7)	0.0026 (6)	0.0002 (6)	0.0050 (6)
C14B	0.0200 (6)	0.0194 (7)	0.0206 (7)	0.0020 (6)	0.0034 (5)	0.0021 (5)
C15B	0.0204 (7)	0.0232 (8)	0.0215 (7)	0.0013 (6)	0.0033 (6)	0.0016 (6)
C51B	0.0245 (7)	0.0222 (8)	0.0206 (7)	0.0003 (6)	−0.0035 (6)	−0.0029 (6)
C52B	0.0238 (7)	0.0336 (9)	0.0377 (9)	0.0044 (7)	−0.0010 (7)	0.0029 (7)

Geometric parameters (Å, °)

O51A—C51A	1.2198 (17)	O51B—C51B	1.2279 (17)
N5A—C51A	1.3780 (18)	N5B—C51B	1.3607 (18)
N5A—C13A	1.4350 (17)	N5B—C13B	1.4489 (17)
N5A—C14A	1.4416 (17)	N5B—C14B	1.4486 (17)
C1A—C2A	1.386 (2)	C9B—C8B	1.382 (2)
C1A—C12A	1.392 (2)	C9B—C15B	1.398 (2)
C1A—H1A	0.95	C9B—H9B	0.95
C2A—C3A	1.379 (2)	C8B—C7B	1.383 (2)
C2A—H2A	0.95	C8B—H8B	0.95
C3A—C4A	1.381 (2)	C7B—C6B	1.384 (2)
C3A—H3A	0.95	C7B—H7B	0.95
C4A—C13A	1.3844 (19)	C6B—C14B	1.394 (2)
C4A—H4A	0.95	C6B—H6B	0.95

C6A—C7A	1.379 (2)	C4B—C13B	1.385 (2)
C6A—C14A	1.393 (2)	C4B—C3B	1.385 (2)
C6A—H6A	0.95	C4B—H4B	0.95
C7A—C8A	1.388 (2)	C3B—C2B	1.383 (2)
C7A—H7A	0.95	C3B—H3B	0.95
C8A—C9A	1.381 (2)	C2B—C1B	1.388 (2)
C8A—H8A	0.95	C2B—H2B	0.95
C9A—C15A	1.401 (2)	C1B—C12B	1.394 (2)
C9A—H9A	0.95	C1B—H1B	0.95
C10A—C15A	1.520 (2)	C11B—C12B	1.502 (2)
C10A—C11A	1.525 (2)	C11B—C10B	1.530 (2)
C10A—H10A	0.99	C11B—H12B	0.99
C10A—H11A	0.99	C11B—H13B	0.99
C11A—C12A	1.502 (2)	C10B—C15B	1.515 (2)
C11A—H12A	0.99	C10B—H10B	0.99
C11A—H13A	0.99	C10B—H11B	0.99
C12A—C13A	1.390 (2)	C15B—C14B	1.4017 (19)
C14A—C15A	1.397 (2)	C13B—C12B	1.388 (2)
C51A—C52A	1.504 (2)	C51B—C52B	1.502 (2)
C52A—H51A	0.98	C52B—H51B	0.98
C52A—H52A	0.98	C52B—H52B	0.98
C52A—H53A	0.98	C52B—H53B	0.98
C51A—N5A—C13A	122.60 (11)	C51B—N5B—C13B	118.62 (11)
C51A—N5A—C14A	121.26 (11)	C51B—N5B—C14B	125.12 (11)
C13A—N5A—C14A	116.12 (10)	C13B—N5B—C14B	116.04 (11)
C2A—C1A—C12A	120.78 (14)	C8B—C9B—C15B	122.31 (14)
C2A—C1A—H1A	119.6	C8B—C9B—H9B	118.8
C12A—C1A—H1A	119.6	C15B—C9B—H9B	118.8
C3A—C2A—C1A	120.59 (14)	C9B—C8B—C7B	119.98 (14)
C3A—C2A—H2A	119.7	C9B—C8B—H8B	120.0
C1A—C2A—H2A	119.7	C7B—C8B—H8B	120.0
C4A—C3A—C2A	119.37 (14)	C6B—C7B—C8B	119.26 (14)
C4A—C3A—H3A	120.3	C6B—C7B—H7B	120.4
C2A—C3A—H3A	120.3	C8B—C7B—H7B	120.4
C3A—C4A—C13A	119.99 (13)	C7B—C6B—C14B	120.64 (14)
C3A—C4A—H4A	120.0	C7B—C6B—H6B	119.7
C13A—C4A—H4A	120.0	C14B—C6B—H6B	119.7
C7A—C6A—C14A	121.05 (14)	C13B—C4B—C3B	119.04 (14)
C7A—C6A—H6A	119.5	C13B—C4B—H4B	120.5
C14A—C6A—H6A	119.5	C3B—C4B—H4B	120.5
C6A—C7A—C8A	119.40 (14)	C2B—C3B—C4B	120.07 (13)
C6A—C7A—H7A	120.3	C2B—C3B—H3B	120.0
C8A—C7A—H7A	120.3	C4B—C3B—H3B	120.0
C9A—C8A—C7A	119.41 (14)	C3B—C2B—C1B	120.49 (14)
C9A—C8A—H8A	120.3	C3B—C2B—H2B	119.8
C7A—C8A—H8A	120.3	C1B—C2B—H2B	119.8
C8A—C9A—C15A	122.52 (14)	C2B—C1B—C12B	120.22 (14)

C8A—C9A—H9A	118.7	C2B—C1B—H1B	119.9
C15A—C9A—H9A	118.7	C12B—C1B—H1B	119.9
C15A—C10A—C11A	118.75 (12)	C12B—C11B—C10B	110.36 (12)
C15A—C10A—H10A	107.6	C12B—C11B—H12B	109.6
C11A—C10A—H10A	107.6	C10B—C11B—H13B	109.6
C15A—C10A—H11A	107.6	C12B—C11B—H13B	109.6
C11A—C10A—H11A	107.6	C10B—C11B—H12B	109.6
H10A—C10A—H11A	107.1	H12B—C11B—H13B	108.1
C12A—C11A—C10A	111.64 (12)	C15B—C10B—C11B	118.11 (12)
C12A—C11A—H12A	109.3	C15B—C10B—H10B	107.8
C10A—C11A—H12A	109.3	C11B—C10B—H11B	107.8
C12A—C11A—H13A	109.3	C15B—C10B—H11B	107.8
C10A—C11A—H13A	109.3	C11B—C10B—H10B	107.8
H12A—C11A—H13A	108.0	H10B—C10B—H11B	107.1
C13A—C12A—C1A	117.80 (13)	C9B—C15B—C14B	116.82 (13)
C13A—C12A—C11A	118.49 (13)	C9B—C15B—C10B	116.44 (12)
C1A—C12A—C11A	123.67 (13)	C14B—C15B—C10B	126.73 (13)
C4A—C13A—C12A	121.46 (13)	C6B—C14B—C15B	120.96 (13)
C4A—C13A—N5A	120.62 (12)	C6B—C14B—N5B	117.74 (12)
C12A—C13A—N5A	117.90 (13)	C15B—C14B—N5B	121.20 (12)
C6A—C14A—C15A	120.63 (13)	C4B—C13B—C12B	121.89 (13)
C6A—C14A—N5A	117.76 (12)	C4B—C13B—N5B	121.15 (13)
C15A—C14A—N5A	121.51 (12)	C12B—C13B—N5B	116.94 (12)
C14A—C15A—C9A	116.98 (13)	C13B—C12B—C1B	118.27 (13)
C14A—C15A—C10A	126.38 (13)	C13B—C12B—C11B	118.89 (12)
C9A—C15A—C10A	116.63 (13)	C1B—C12B—C11B	122.67 (13)
O51A—C51A—N5A	121.63 (13)	O51B—C51B—N5B	120.51 (13)
O51A—C51A—C52A	122.10 (13)	O51B—C51B—C52B	121.37 (13)
N5A—C51A—C52A	116.27 (12)	N5B—C51B—C52B	118.10 (12)
C51A—C52A—H51A	109.5	C51B—C52B—H51B	109.5
C51A—C52A—H52A	109.5	C51B—C52B—H52B	109.5
H51A—C52A—H52A	109.5	H51B—C52B—H52B	109.5
C51A—C52A—H53A	109.5	C51B—C52B—H53B	109.5
H51A—C52A—H53A	109.5	H51B—C52B—H53B	109.5
H52A—C52A—H53A	109.5	H52B—C52B—H53B	109.5
C12A—C1A—C2A—C3A	−0.2 (2)	C15B—C9B—C8B—C7B	−0.7 (2)
C1A—C2A—C3A—C4A	0.9 (2)	C9B—C8B—C7B—C6B	−0.5 (2)
C2A—C3A—C4A—C13A	−0.8 (2)	C8B—C7B—C6B—C14B	1.5 (2)
C14A—C6A—C7A—C8A	0.9 (2)	C13B—C4B—C3B—C2B	0.6 (2)
C6A—C7A—C8A—C9A	−0.7 (2)	C4B—C3B—C2B—C1B	0.8 (2)
C7A—C8A—C9A—C15A	0.0 (2)	C3B—C2B—C1B—C12B	−1.2 (2)
C15A—C10A—C11A—C12A	56.69 (17)	C12B—C11B—C10B—C15B	60.81 (17)
C2A—C1A—C12A—C13A	−0.5 (2)	C8B—C9B—C15B—C14B	0.9 (2)
C2A—C1A—C12A—C11A	−178.13 (14)	C8B—C9B—C15B—C10B	179.74 (14)
C10A—C11A—C12A—C13A	−70.72 (17)	C11B—C10B—C15B—C9B	177.41 (13)
C10A—C11A—C12A—C1A	106.87 (16)	C11B—C10B—C15B—C14B	−3.9 (2)
C3A—C4A—C13A—C12A	0.1 (2)	C7B—C6B—C14B—C15B	−1.3 (2)

C3A—C4A—C13A—N5A	178.27 (13)	C7B—C6B—C14B—N5B	−177.85 (12)
C1A—C12A—C13A—C4A	0.6 (2)	C9B—C15B—C14B—C6B	0.12 (19)
C11A—C12A—C13A—C4A	178.33 (13)	C10B—C15B—C14B—C6B	−178.58 (13)
C1A—C12A—C13A—N5A	−177.65 (12)	C9B—C15B—C14B—N5B	176.52 (12)
C11A—C12A—C13A—N5A	0.09 (19)	C10B—C15B—C14B—N5B	−2.2 (2)
C51A—N5A—C13A—C4A	73.45 (18)	C51B—N5B—C14B—C6B	−64.33 (18)
C14A—N5A—C13A—C4A	−105.01 (14)	C13B—N5B—C14B—C6B	121.27 (14)
C51A—N5A—C13A—C12A	−108.30 (15)	C51B—N5B—C14B—C15B	119.16 (15)
C14A—N5A—C13A—C12A	73.24 (16)	C13B—N5B—C14B—C15B	−55.25 (17)
C7A—C6A—C14A—C15A	−0.4 (2)	C3B—C4B—C13B—C12B	−1.6 (2)
C7A—C6A—C14A—N5A	−176.84 (12)	C3B—C4B—C13B—N5B	176.65 (12)
C51A—N5A—C14A—C6A	−58.07 (17)	C51B—N5B—C13B—C4B	81.31 (17)
C13A—N5A—C14A—C6A	120.41 (13)	C14B—N5B—C13B—C4B	−103.90 (15)
C51A—N5A—C14A—C15A	125.49 (14)	C51B—N5B—C13B—C12B	−100.34 (15)
C13A—N5A—C14A—C15A	−56.03 (17)	C14B—N5B—C13B—C12B	74.45 (16)
C6A—C14A—C15A—C9A	−0.31 (19)	C4B—C13B—C12B—C1B	1.2 (2)
N5A—C14A—C15A—C9A	176.03 (11)	N5B—C13B—C12B—C1B	−177.12 (12)
C6A—C14A—C15A—C10A	−179.32 (13)	C4B—C13B—C12B—C11B	176.53 (13)
N5A—C14A—C15A—C10A	−3.0 (2)	N5B—C13B—C12B—C11B	−1.81 (19)
C8A—C9A—C15A—C14A	0.48 (19)	C2B—C1B—C12B—C13B	0.2 (2)
C8A—C9A—C15A—C10A	179.58 (13)	C2B—C1B—C12B—C11B	−174.92 (13)
C11A—C10A—C15A—C14A	0.1 (2)	C10B—C11B—C12B—C13B	−71.31 (16)
C11A—C10A—C15A—C9A	−178.89 (12)	C10B—C11B—C12B—C1B	103.78 (15)
C13A—N5A—C51A—O51A	−178.94 (13)	C13B—N5B—C51B—O51B	−8.42 (19)
C14A—N5A—C51A—O51A	−0.6 (2)	C14B—N5B—C51B—O51B	177.31 (12)
C13A—N5A—C51A—C52A	1.20 (19)	C13B—N5B—C51B—C52B	169.77 (13)
C14A—N5A—C51A—C52A	179.58 (13)	C14B—N5B—C51B—C52B	−4.5 (2)
