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

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Key indicators

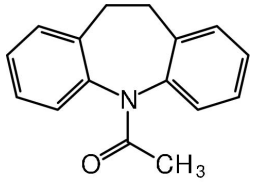
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.076
Data-to-parameter ratio = 8.5

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, $C_{16}H_{15}NO$, 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.



(I)

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

$C_{16}H_{15}NO$
M_r = 237.29
Orthorhombic, $P2_12_12_1$
a = 9.5674 (2) Å
b = 11.7020 (3) Å
c = 22.2785 (4) Å
V = 2494.25 (9) Å^3
Z = 8
D_x = 1.264 Mg m^{-3}
Mo $K\alpha$ 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	2540 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.037$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{max} = 26.0^\circ$
$T_{min} = 0.954$, $T_{max} = 0.992$	$h = -11 \rightarrow 11$
19 122 measured reflections	$k = -14 \rightarrow 14$
2778 independent reflections	$l = -27 \rightarrow 27$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.3098P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.076$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
2778 reflections	Extinction correction: *SHELXL97*
328 parameters	Extinction coefficient: 0.0285 (14)
H-atom parameters constrained	

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 (CH_3) and 0.99 Å (CH_2). The isotropic displacement parameters for all H atoms were set equal to $1.25U_{eq}$ 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: *SHELXS*97 (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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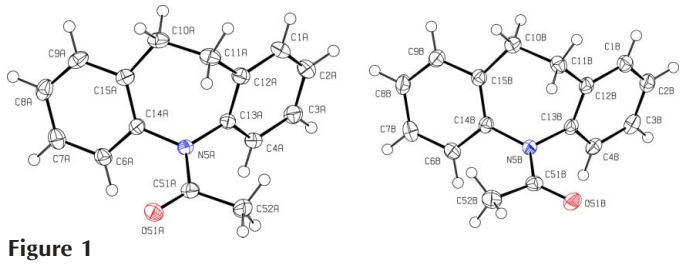

Figure 1
The molecular configuration and atom-numbering scheme for both independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius. The molecules are shown with similar view directions and not in their true relative orientations.

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supporting information

Acta Cryst. (2005). E**61**, o1609–o1610 [https://doi.org/10.1107/S1600536805013115]

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Crystal data

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$c = 22.2785$ (4) Å
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$F(000) = 1008$
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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 × 0.40 × 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 $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.3°$
$h = -11 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -27 \rightarrow 27$

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, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
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) C1A * 0.0033 (0.0011) C2A * -0.0055 (0.0010) C3A * 0.0026 (0.0010) C4A * 0.0024 (0.0010) C13A * -0.0045 (0.0010) 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) C6A * 0.0049 (0.0010) C7A * -0.0019 (0.0010) C8A * -0.0023 (0.0009) C9A * 0.0035 (0.0009) C15A * -0.0006 (0.0009) 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) C6B * 0.0061 (0.0010) C7B * 0.0017 (0.0011) C8B * -0.0067 (0.0010) C9B * 0.0039 (0.0009) C15B * 0.0038 (0.0009) 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) C1B * -0.0076 (0.0010) C2B * 0.0014 (0.0010) C3B * 0.0069 (0.0010) C4B * -0.0091 (0.0010) C13B * 0.0028 (0.0010) C12B
Rms deviation of fitted atoms = 0.0062

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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, º)

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)