Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Hydroxy-4-methyl-4-azatricyclo-[5.2.2.0^{2,6}]undec-8-en-3-one

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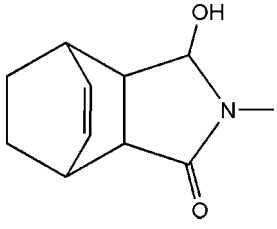
Received 23 October 2007; accepted 27 October 2007

Key indicators: single-crystal X-ray study; T = 203 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.084; data-to-parameter ratio = 24.8.

Two independent molecules (*A* and *B*) comprise the asymmetric unit in the crystal structure of the title compound, $C_{11}H_{15}NO_2$. The cyclohexane ring adopts a boat configuration, as does the fused cyclohexene ring that bridges the cyclohexane ring. The crystal packing is stabilized by intermolecular O—H···O hydrogen bonding between the hydroxyl H atom of molecule *A* and the ketone O atom of molecule *B* and *vice versa*. These link the molecules into chains running diagonally along the *bc* face of the unit cell.

Related literature

For related structures, see: Pollack *et al.* (1997); Monkman *et al.* (2002). For related literature, see: Birney *et al.* (2002); Stephan *et al.* (1988); Cremer & Pople (1975).



Experimental

Crystal data

$C_{11}H_{15}NO_2$
M_r = 193.24
Triclinic, $P\overline{1}$
a = 9.0426 (10) Å
b = 9.409 (3) Å
c = 12.260 (3) Å
α = 108.89 (2)°
β = 91.454 (13)°
γ = 91.817 (16)°
V = 985.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 203 K
0.55 × 0.46 × 0.36 mm

Data collection

Oxford Diffraction Gemini R CCD diffractometer
Absorption correction: none
12514 measured reflections
6368 independent reflections
2308 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.084
S = 0.73
6368 reflections
257 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2A—H2AA···O1B^{i}	0.83	1.90	2.7322 (16)	178
O2B—H2BA···O1A	0.83	1.86	2.6868 (16)	179

Symmetry code: (i) $x, y-1, z-1$.

Data collection: *CrysAlisPro* (Oxford Diffraction, 2007); cell refinement: *CrysAlisPro*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

RJB acknowledges the NSF MRI program (grant No. CHE-0619278) for funds to purchase the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2206).

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 doi:10.1107/S1600536807053779

supplementary materials

Acta Cryst. (2007). E**63**, o4565 [**doi:10.1107/S1600536807053779**]

5-Hydroxy-4-methyl-4-azatricyclo[5.2.2.0^{2,6}]undec-8-en-3-one

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Comment

5-Hydroxy-4-methyl-4-aza-tricyclo[5.2.2.0,2,6]undecan-3-one was synthesized to determine whether the cyclic imide functionality can be reduced from 4-methyl-4-aza-tricyclo[5.2.2.0,2,6]undec-8-ene-3,5-dione (Birney *et al.*, 2002) to a functionalized pyrrole using diisobutylaluminium hydride (DIBAL-H). This reaction was carried out to determine if a stepwise reduction of a cyclic imide into a pyrrole could use DIBAL-H instead of a more complex synthetic process (Stephan *et al.*, 1988). These pyrrole derivatives are used in a number of materials, especially intramolecularly hydrogen-bonded conjugated polymers (Pollack *et al.*, 1997). They are also used in the protonation and subsequent intramolecular hydrogen bonding as a method to control chain structure and to tune luminescence in heteroatomic conjugated polymers (Monkman *et al.*, 2002). A new pyrrole derivative (I), $C_{11}H_{15}NO_2$, was prepared and its crystal structure is reported herein.

Compound (I) crystallizes with two independent molecules (A & B) in the asymmetric unit (Fig. 1). The angle between the mean planes of cyclohexane and fused 4-methyl-4-aza-tricyclo-3-one group is 23.5 (4) ° [A] and 25.1 (2)° [B]. The cyclohexane ring is in a boat configuration with puckering parameters Q, θ and φ of 0.8771 (15) Å, 91.61 (10)° and 63.05 (10)°, respectively for C2A—C7A in A, and 0.8784 (16) Å, 90.79 (10)° and 61.45 (10)°, respectively for C2B—C7B in B (Cremer & Pople, 1975). The bridged, 6-membered [5.2.2.0,2,6] group is also in a boat configuration with puckering parameters Q, θ and φ of 0.7885 (16) Å, 90.14 (12)° and 358.87 (12)° for C3A–C6A,C10A,C11A in A; 0.7959 (16) Å, 90.24 (12)° and 359.24 (12)° for C3B—C6B,C10B,C11B in B. The crystal packing is stabilized by intermolecular O—H···O hydrogen bonding (Table 1) between the hydroxyl hydrogen atom of molecule A [O2A–H2AA] to the ketone oxygen atom of molecule B [O1B] and *vice versa*, which link the molecules into chains diagonal along the *bc* face of the unit cell (Fig. 2).

Experimental

4-Methyl-4-aza-tricyclo[5.2.2.0,2,6]undec-8-ene-3,5-dione (0.50 g), produced from the Diels–Alder synthesis of *N*-methyl maleimide and 1,3 cyclohexadiene, was dissolved in THF (30 ml) under nitrogen in an ice bath. DIBAL-H, (5.2 ml) was added dropwise for 5 min and allowed to stir for 3 h. The reaction was then stopped. The product was extracted using ethyl acetate and washed 3x with sat. $NaHCO_3$. This was dried over $MgSO_4$ to yield 0.29 g of product. Crystals were obtained from the slow evaporation of methanol solution of (I).

Refinement

The hydroxyl hydrogen atoms (H2AA & H2BA) were located in a difference Fourier map and along with all other H atoms were placed in their calculated positions and were then refined using the riding model approximation with O—H = 0.83 Å and C—H = 0.94 − 0.99 Å, and with U_{iso}(H) = 1.2–1.5U_{eq}(C, O).

Figures

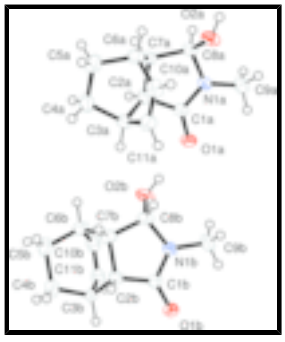

Fig. 1. Molecular structure of (I), showing atom labeling for the two independent molecules (A & B) and 50% probability displacement ellipsoids.

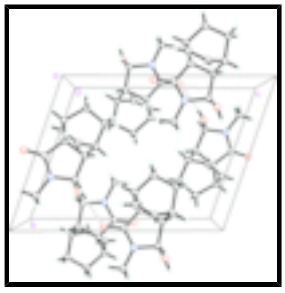

Fig. 2. Packing diagram of $C_{11}H_{15}NO_2$ viewed down the *a* axis. Dashed lines indicate O2A–H2AA···O1B and O2B–H2BA···O1A hydrogen bonds.

5-Hydroxy-4-methyl-4-azatricyclo[5.2.2.0^{2,6}]undec-8-en-3-one

Crystal data

$C_{11}H_{15}NO_2$	$Z = 4$
$M_r = 193.24$	$F_{000} = 416$
Triclinic, $P\overline{1}$	$D_x = 1.302$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.0426$ (10) Å	Cell parameters from 2564 reflections
$b = 9.409$ (3) Å	$\theta = 4.8$–32.4º
$c = 12.260$ (3) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 108.89$ (2)º	$T = 203$ K
$\beta = 91.454$ (13)º	Chunk, colourless
$\gamma = 91.817$ (16)º	0.55 × 0.46 × 0.36 mm
$V = 985.8$ (4) Å^3	

Data collection

Oxford Diffraction Gemini R CCD diffractometer	6368 independent reflections
Radiation source: fine-focus sealed tube	2308 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.048$
Detector resolution: 10.5081 pixels mm^{-1}	$\theta_{max} = 32.5$º
$T = 203$ K	$\theta_{min} = 4.8$º
φ and ω scans	$h = -13 \rightarrow 12$

Absorption correction: none
12514 measured reflections
$k = -13 \rightarrow 13$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.084$
$S = 0.73$
6368 reflections
257 parameters
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
$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: none

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 $F^2 > 2$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}
O1A	0.16069 (9)	0.52439 (10)	0.07775 (8)	0.0314 (2)
O2A	0.36646 (9)	0.25659 (10)	−0.24154 (8)	0.0366 (3)
H2AA	0.3548	0.1782	−0.2974	0.055*
O1B	0.33481 (10)	1.00085 (10)	0.57185 (8)	0.0333 (2)
O2B	0.12572 (10)	0.80442 (10)	0.22133 (9)	0.0387 (3)
H2BA	0.1357	0.7174	0.1777	0.058*
N1A	0.20075 (11)	0.34330 (12)	−0.09313 (9)	0.0276 (3)
N1B	0.28780 (11)	0.85683 (12)	0.38432 (10)	0.0284 (3)
C1A	0.18021 (13)	0.48518 (14)	−0.02746 (12)	0.0247 (3)
C2A	0.18082 (14)	0.58569 (14)	−0.10129 (11)	0.0267 (3)
H2AB	0.0796	0.6204	−0.1068	0.032*
C3A	0.28881 (15)	0.72297 (14)	−0.05591 (12)	0.0308 (3)
H3AA	0.2615	0.7893	0.0210	0.037*
C4A	0.28725 (17)	0.80663 (16)	−0.14502 (13)	0.0406 (4)
H4AA	0.1869	0.8383	−0.1546	0.049*
H4AB	0.3532	0.8967	−0.1177	0.049*

C5A	0.33856 (17)	0.70269 (16)	−0.26115 (13)	0.0411 (4)
H5AA	0.4280	0.7467	−0.2831	0.049*
H5AB	0.2612	0.6912	−0.3212	0.049*
C6A	0.37122 (15)	0.54791 (15)	−0.25038 (12)	0.0333 (4)
H6AA	0.4094	0.4802	−0.3230	0.040*
C7A	0.22374 (14)	0.48369 (14)	−0.22132 (11)	0.0268 (3)
H7AA	0.1461	0.4857	−0.2792	0.032*
C8A	0.22836 (14)	0.32299 (14)	−0.21366 (12)	0.0282 (3)
H8AA	0.1485	0.2580	−0.2639	0.034*
C9A	0.18835 (15)	0.21534 (15)	−0.05272 (13)	0.0356 (4)
H9AA	0.1873	0.2500	0.0309	0.053*
H9AB	0.2721	0.1525	−0.0777	0.053*
H9AC	0.0973	0.1577	−0.0842	0.053*
C10A	0.48098 (15)	0.57138 (15)	−0.15213 (14)	0.0351 (4)
H10A	0.5724	0.5249	−0.1612	0.042*
C11A	0.43988 (15)	0.66246 (15)	−0.05131 (13)	0.0343 (4)
H11A	0.4996	0.6869	0.0166	0.041*
C1B	0.31536 (14)	0.98754 (14)	0.46820 (12)	0.0257 (3)
C2B	0.32041 (15)	1.11280 (14)	0.41787 (11)	0.0282 (3)
H2BB	0.4225	1.1572	0.4259	0.034*
C3B	0.21215 (15)	1.23657 (14)	0.47498 (12)	0.0319 (4)
H3BA	0.2370	1.2838	0.5583	0.038*
C4B	0.22084 (17)	1.35259 (16)	0.41135 (13)	0.0416 (4)
H4BA	0.3224	1.3945	0.4170	0.050*
H4BB	0.1559	1.4351	0.4469	0.050*
C5B	0.17286 (18)	1.27657 (16)	0.28377 (13)	0.0438 (4)
H5BA	0.2532	1.2868	0.2343	0.053*
H5BB	0.0863	1.3252	0.2646	0.053*
C6B	0.13431 (15)	1.10831 (15)	0.26263 (13)	0.0362 (4)
H6BA	0.0980	1.0575	0.1821	0.043*
C7B	0.27679 (14)	1.03841 (15)	0.28943 (12)	0.0299 (3)
H7BA	0.3570	1.0593	0.2426	0.036*
C8B	0.26488 (15)	0.86870 (15)	0.26949 (12)	0.0297 (3)
H8BA	0.3442	0.8178	0.2189	0.036*
C9B	0.28810 (16)	0.71219 (15)	0.40165 (14)	0.0382 (4)
H9BA	0.2857	0.7261	0.4835	0.057*
H9BB	0.3771	0.6615	0.3707	0.057*
H9BC	0.2017	0.6518	0.3624	0.057*
C10B	0.01956 (16)	1.09860 (15)	0.34593 (15)	0.0379 (4)
H10B	−0.0733	1.0489	0.3223	0.045*
C11B	0.05950 (16)	1.16562 (15)	0.45542 (14)	0.0368 (4)
H11B	−0.0030	1.1684	0.5161	0.044*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0365 (6)	0.0309 (5)	0.0230 (6)	−0.0003 (4)	0.0021 (5)	0.0035 (4)
O2A	0.0326 (6)	0.0336 (6)	0.0340 (7)	0.0079 (4)	0.0020 (5)	−0.0032 (5)

O1B	0.0431 (6)	0.0312 (6)	0.0239 (6)	0.0069 (4)	−0.0006 (5)	0.0063 (4)
O2B	0.0352 (6)	0.0305 (6)	0.0381 (7)	0.0002 (4)	−0.0047 (5)	−0.0053 (5)
N1A	0.0320 (7)	0.0217 (6)	0.0265 (7)	0.0021 (5)	0.0005 (5)	0.0043 (5)
N1B	0.0332 (7)	0.0240 (6)	0.0250 (7)	0.0014 (5)	0.0023 (6)	0.0037 (6)
C1A	0.0184 (7)	0.0268 (8)	0.0262 (8)	−0.0002 (6)	−0.0007 (6)	0.0050 (7)
C2A	0.0236 (7)	0.0258 (7)	0.0292 (9)	0.0055 (6)	0.0002 (6)	0.0068 (7)
C3A	0.0400 (9)	0.0239 (8)	0.0261 (8)	−0.0020 (7)	0.0042 (7)	0.0047 (6)
C4A	0.0484 (10)	0.0314 (8)	0.0471 (11)	0.0024 (7)	0.0038 (8)	0.0194 (8)
C5A	0.0447 (9)	0.0449 (10)	0.0402 (10)	−0.0012 (8)	0.0016 (8)	0.0233 (8)
C6A	0.0316 (8)	0.0345 (8)	0.0304 (9)	0.0042 (7)	0.0042 (7)	0.0052 (7)
C7A	0.0241 (7)	0.0294 (8)	0.0234 (8)	0.0021 (6)	−0.0033 (6)	0.0038 (6)
C8A	0.0267 (8)	0.0286 (8)	0.0245 (8)	0.0006 (6)	0.0003 (6)	0.0019 (6)
C9A	0.0397 (9)	0.0277 (8)	0.0384 (9)	−0.0026 (6)	−0.0002 (7)	0.0101 (7)
C10A	0.0266 (8)	0.0352 (8)	0.0457 (10)	−0.0025 (7)	0.0038 (7)	0.0164 (8)
C11A	0.0304 (8)	0.0329 (8)	0.0387 (10)	−0.0094 (7)	−0.0071 (7)	0.0123 (8)
C1B	0.0232 (8)	0.0266 (8)	0.0260 (9)	0.0042 (6)	0.0025 (6)	0.0066 (7)
C2B	0.0274 (8)	0.0261 (8)	0.0284 (9)	−0.0022 (6)	−0.0022 (6)	0.0057 (6)
C3B	0.0487 (9)	0.0241 (8)	0.0227 (8)	0.0040 (7)	−0.0008 (7)	0.0074 (6)
C4B	0.0537 (10)	0.0295 (8)	0.0440 (10)	−0.0007 (7)	−0.0017 (8)	0.0158 (8)
C5B	0.0539 (10)	0.0421 (10)	0.0425 (11)	−0.0025 (8)	−0.0015 (8)	0.0244 (8)
C6B	0.0401 (9)	0.0369 (9)	0.0304 (9)	−0.0030 (7)	−0.0084 (7)	0.0103 (7)
C7B	0.0268 (8)	0.0374 (8)	0.0253 (8)	−0.0019 (6)	0.0035 (6)	0.0102 (7)
C8B	0.0250 (8)	0.0346 (8)	0.0249 (9)	0.0008 (6)	0.0014 (7)	0.0031 (7)
C9B	0.0466 (9)	0.0234 (8)	0.0445 (10)	0.0050 (7)	0.0067 (8)	0.0104 (7)
C10B	0.0294 (8)	0.0297 (8)	0.0555 (12)	0.0025 (7)	−0.0028 (8)	0.0154 (8)
C11B	0.0376 (9)	0.0323 (8)	0.0482 (11)	0.0119 (7)	0.0179 (8)	0.0216 (8)

Geometric parameters (Å, °)

O1A—C1A	1.2404 (15)	C9A—H9AA	0.9700
O2A—C8A	1.4140 (14)	C9A—H9AB	0.9700
O2A—H2AA	0.8300	C9A—H9AC	0.9700
O1B—C1B	1.2432 (15)	C10A—C11A	1.3259 (19)
O2B—C8B	1.4073 (16)	C10A—H10A	0.9400
O2B—H2BA	0.8300	C11A—H11A	0.9400
N1A—C1A	1.3387 (16)	C1B—C2B	1.4970 (19)
N1A—C9A	1.4451 (17)	C2B—C7B	1.5370 (19)
N1A—C8A	1.4565 (16)	C2B—C3B	1.5440 (17)
N1B—C1B	1.3345 (17)	C2B—H2BB	0.9900
N1B—C9B	1.4438 (17)	C3B—C11B	1.493 (2)
N1B—C8B	1.4581 (17)	C3B—C4B	1.5347 (19)
C1A—C2A	1.5060 (19)	C3B—H3BA	0.9900
C2A—C3A	1.5358 (19)	C4B—C5B	1.539 (2)
C2A—C7A	1.5412 (17)	C4B—H4BA	0.9800
C2A—H2AB	0.9900	C4B—H4BB	0.9800
C3A—C11A	1.5031 (18)	C5B—C6B	1.546 (2)
C3A—C4A	1.5396 (19)	C5B—H5BA	0.9800
C3A—H3AA	0.9900	C5B—H5BB	0.9800
C4A—C5A	1.5361 (19)	C6B—C10B	1.4954 (19)

C4A—H4AA	0.9800	C6B—C7B	1.5404 (19)
C4A—H4AB	0.9800	C6B—H6BA	0.9900
C5A—C6A	1.5413 (19)	C7B—C8B	1.5350 (19)
C5A—H5AA	0.9800	C7B—H7BA	0.9900
C5A—H5AB	0.9800	C8B—H8BA	0.9900
C6A—C10A	1.496 (2)	C9B—H9BA	0.9700
C6A—C7A	1.5436 (18)	C9B—H9BB	0.9700
C6A—H6AA	0.9900	C9B—H9BC	0.9700
C7A—C8A	1.5468 (18)	C10B—C11B	1.320 (2)
C7A—H7AA	0.9900	C10B—H10B	0.9400
C8A—H8AA	0.9900	C11B—H11B	0.9400
C8A—O2A—H2AA	109.5	C6A—C10A—H10A	122.4
C8B—O2B—H2BA	109.5	C10A—C11A—C3A	113.66 (14)
C1A—N1A—C9A	124.14 (11)	C10A—C11A—H11A	123.2
C1A—N1A—C8A	115.08 (11)	C3A—C11A—H11A	123.2
C9A—N1A—C8A	120.68 (11)	O1B—C1B—N1B	124.37 (13)
C1B—N1B—C9B	123.98 (12)	O1B—C1B—C2B	126.00 (13)
C1B—N1B—C8B	114.83 (12)	N1B—C1B—C2B	109.63 (12)
C9B—N1B—C8B	121.10 (11)	C1B—C2B—C7B	105.14 (11)
O1A—C1A—N1A	124.04 (13)	C1B—C2B—C3B	112.38 (10)
O1A—C1A—C2A	126.28 (12)	C7B—C2B—C3B	110.17 (11)
N1A—C1A—C2A	109.67 (12)	C1B—C2B—H2BB	109.7
C1A—C2A—C3A	113.55 (12)	C7B—C2B—H2BB	109.7
C1A—C2A—C7A	104.81 (10)	C3B—C2B—H2BB	109.7
C3A—C2A—C7A	110.95 (10)	C11B—C3B—C4B	108.09 (12)
C1A—C2A—H2AB	109.1	C11B—C3B—C2B	107.59 (11)
C3A—C2A—H2AB	109.1	C4B—C3B—C2B	107.31 (11)
C7A—C2A—H2AB	109.1	C11B—C3B—H3BA	111.2
C11A—C3A—C2A	106.35 (10)	C4B—C3B—H3BA	111.2
C11A—C3A—C4A	108.68 (11)	C2B—C3B—H3BA	111.2
C2A—C3A—C4A	107.56 (12)	C3B—C4B—C5B	109.51 (12)
C11A—C3A—H3AA	111.3	C3B—C4B—H4BA	109.8
C2A—C3A—H3AA	111.3	C5B—C4B—H4BA	109.8
C4A—C3A—H3AA	111.3	C3B—C4B—H4BB	109.8
C5A—C4A—C3A	109.68 (11)	C5B—C4B—H4BB	109.8
C5A—C4A—H4AA	109.7	H4BA—C4B—H4BB	108.2
C3A—C4A—H4AA	109.7	C4B—C5B—C6B	109.16 (12)
C5A—C4A—H4AB	109.7	C4B—C5B—H5BA	109.8
C3A—C4A—H4AB	109.7	C6B—C5B—H5BA	109.8
H4AA—C4A—H4AB	108.2	C4B—C5B—H5BB	109.8
C4A—C5A—C6A	109.33 (11)	C6B—C5B—H5BB	109.8
C4A—C5A—H5AA	109.8	H5BA—C5B—H5BB	108.3
C6A—C5A—H5AA	109.8	C10B—C6B—C7B	109.17 (12)
C4A—C5A—H5AB	109.8	C10B—C6B—C5B	107.80 (11)
C6A—C5A—H5AB	109.8	C7B—C6B—C5B	106.82 (12)
H5AA—C5A—H5AB	108.3	C10B—C6B—H6BA	111.0
C10A—C6A—C5A	108.20 (12)	C7B—C6B—H6BA	111.0
C10A—C6A—C7A	109.15 (12)	C5B—C6B—H6BA	111.0
C5A—C6A—C7A	106.43 (10)	C8B—C7B—C2B	105.91 (11)

C10A—C6A—H6AA	111.0	C8B—C7B—C6B	115.58 (11)
C5A—C6A—H6AA	111.0	C2B—C7B—C6B	108.49 (10)
C7A—C6A—H6AA	111.0	C8B—C7B—H7BA	108.9
C2A—C7A—C6A	107.76 (11)	C2B—C7B—H7BA	108.9
C2A—C7A—C8A	106.03 (10)	C6B—C7B—H7BA	108.9
C6A—C7A—C8A	115.39 (10)	O2B—C8B—N1B	110.62 (11)
C2A—C7A—H7AA	109.2	O2B—C8B—C7B	112.23 (11)
C6A—C7A—H7AA	109.2	N1B—C8B—C7B	104.12 (11)
C8A—C7A—H7AA	109.2	O2B—C8B—H8BA	109.9
O2A—C8A—N1A	109.21 (11)	N1B—C8B—H8BA	109.9
O2A—C8A—C7A	113.56 (10)	C7B—C8B—H8BA	109.9
N1A—C8A—C7A	103.97 (10)	N1B—C9B—H9BA	109.5
O2A—C8A—H8AA	110.0	N1B—C9B—H9BB	109.5
N1A—C8A—H8AA	110.0	H9BA—C9B—H9BB	109.5
C7A—C8A—H8AA	110.0	N1B—C9B—H9BC	109.5
N1A—C9A—H9AA	109.5	H9BA—C9B—H9BC	109.5
N1A—C9A—H9AB	109.5	H9BB—C9B—H9BC	109.5
H9AA—C9A—H9AB	109.5	C11B—C10B—C6B	114.46 (14)
N1A—C9A—H9AC	109.5	C11B—C10B—H10B	122.8
H9AA—C9A—H9AC	109.5	C6B—C10B—H10B	122.8
H9AB—C9A—H9AC	109.5	C10B—C11B—C3B	114.51 (13)
C11A—C10A—C6A	115.14 (12)	C10B—C11B—H11B	122.7
C11A—C10A—H10A	122.4	C3B—C11B—H11B	122.7
C9A—N1A—C1A—O1A	6.4 (2)	C9B—N1B—C1B—O1B	−4.9 (2)
C8A—N1A—C1A—O1A	−177.31 (11)	C8B—N1B—C1B—O1B	178.45 (11)
C9A—N1A—C1A—C2A	−172.17 (11)	C9B—N1B—C1B—C2B	174.51 (11)
C8A—N1A—C1A—C2A	4.11 (15)	C8B—N1B—C1B—C2B	−2.10 (14)
O1A—C1A—C2A—C3A	53.72 (16)	O1B—C1B—C2B—C7B	−175.46 (12)
N1A—C1A—C2A—C3A	−127.73 (11)	N1B—C1B—C2B—C7B	5.11 (13)
O1A—C1A—C2A—C7A	174.97 (12)	O1B—C1B—C2B—C3B	−55.60 (17)
N1A—C1A—C2A—C7A	−6.48 (14)	N1B—C1B—C2B—C3B	124.96 (12)
C1A—C2A—C3A—C11A	58.84 (13)	C1B—C2B—C3B—C11B	−60.52 (15)
C7A—C2A—C3A—C11A	−58.90 (14)	C7B—C2B—C3B—C11B	56.37 (14)
C1A—C2A—C3A—C4A	175.14 (10)	C1B—C2B—C3B—C4B	−176.62 (12)
C7A—C2A—C3A—C4A	57.39 (13)	C7B—C2B—C3B—C4B	−59.73 (14)
C11A—C3A—C4A—C5A	53.87 (15)	C11B—C3B—C4B—C5B	−54.41 (15)
C2A—C3A—C4A—C5A	−60.89 (14)	C2B—C3B—C4B—C5B	61.36 (15)
C3A—C4A—C5A—C6A	1.25 (16)	C3B—C4B—C5B—C6B	−0.81 (16)
C4A—C5A—C6A—C10A	−55.20 (14)	C4B—C5B—C6B—C10B	55.33 (15)
C4A—C5A—C6A—C7A	62.00 (15)	C4B—C5B—C6B—C7B	−61.89 (14)
C1A—C2A—C7A—C6A	−117.75 (12)	C1B—C2B—C7B—C8B	−6.01 (13)
C3A—C2A—C7A—C6A	5.20 (14)	C3B—C2B—C7B—C8B	−127.31 (11)
C1A—C2A—C7A—C8A	6.34 (13)	C1B—C2B—C7B—C6B	118.66 (11)
C3A—C2A—C7A—C8A	129.29 (11)	C3B—C2B—C7B—C6B	−2.65 (15)
C10A—C6A—C7A—C2A	51.26 (14)	C10B—C6B—C7B—C8B	65.93 (15)
C5A—C6A—C7A—C2A	−65.30 (14)	C5B—C6B—C7B—C8B	−177.76 (11)
C10A—C6A—C7A—C8A	−66.97 (14)	C10B—C6B—C7B—C2B	−52.80 (15)
C5A—C6A—C7A—C8A	176.47 (12)	C5B—C6B—C7B—C2B	63.52 (14)
C1A—N1A—C8A—O2A	121.68 (11)	C1B—N1B—C8B—O2B	−122.62 (12)

C9A—N1A—C8A—O2A	−61.90 (15)	C9B—N1B—C8B—O2B	60.66 (14)
C1A—N1A—C8A—C7A	0.15 (14)	C1B—N1B—C8B—C7B	−1.86 (14)
C9A—N1A—C8A—C7A	176.57 (11)	C9B—N1B—C8B—C7B	−178.57 (10)
C2A—C7A—C8A—O2A	−122.74 (11)	C2B—C7B—C8B—O2B	124.52 (11)
C6A—C7A—C8A—O2A	−3.56 (16)	C6B—C7B—C8B—O2B	4.39 (16)
C2A—C7A—C8A—N1A	−4.16 (13)	C2B—C7B—C8B—N1B	4.85 (13)
C6A—C7A—C8A—N1A	115.02 (12)	C6B—C7B—C8B—N1B	−115.29 (12)
C5A—C6A—C10A—C11A	57.17 (15)	C7B—C6B—C10B—C11B	58.18 (15)
C7A—C6A—C10A—C11A	−58.26 (15)	C5B—C6B—C10B—C11B	−57.51 (16)
C6A—C10A—C11A—C3A	0.68 (17)	C6B—C10B—C11B—C3B	−0.69 (17)
C2A—C3A—C11A—C10A	57.79 (15)	C4B—C3B—C11B—C10B	58.48 (15)
C4A—C3A—C11A—C10A	−57.75 (15)	C2B—C3B—C11B—C10B	−57.10 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2A—H2AA···O1B^{i}	0.83	1.90	2.7322 (16)	178
O2B—H2BA···O1A	0.83	1.86	2.6868 (16)	179

Symmetry codes: (i) *x*, *y*−1, *z*−1.

Fig. 1

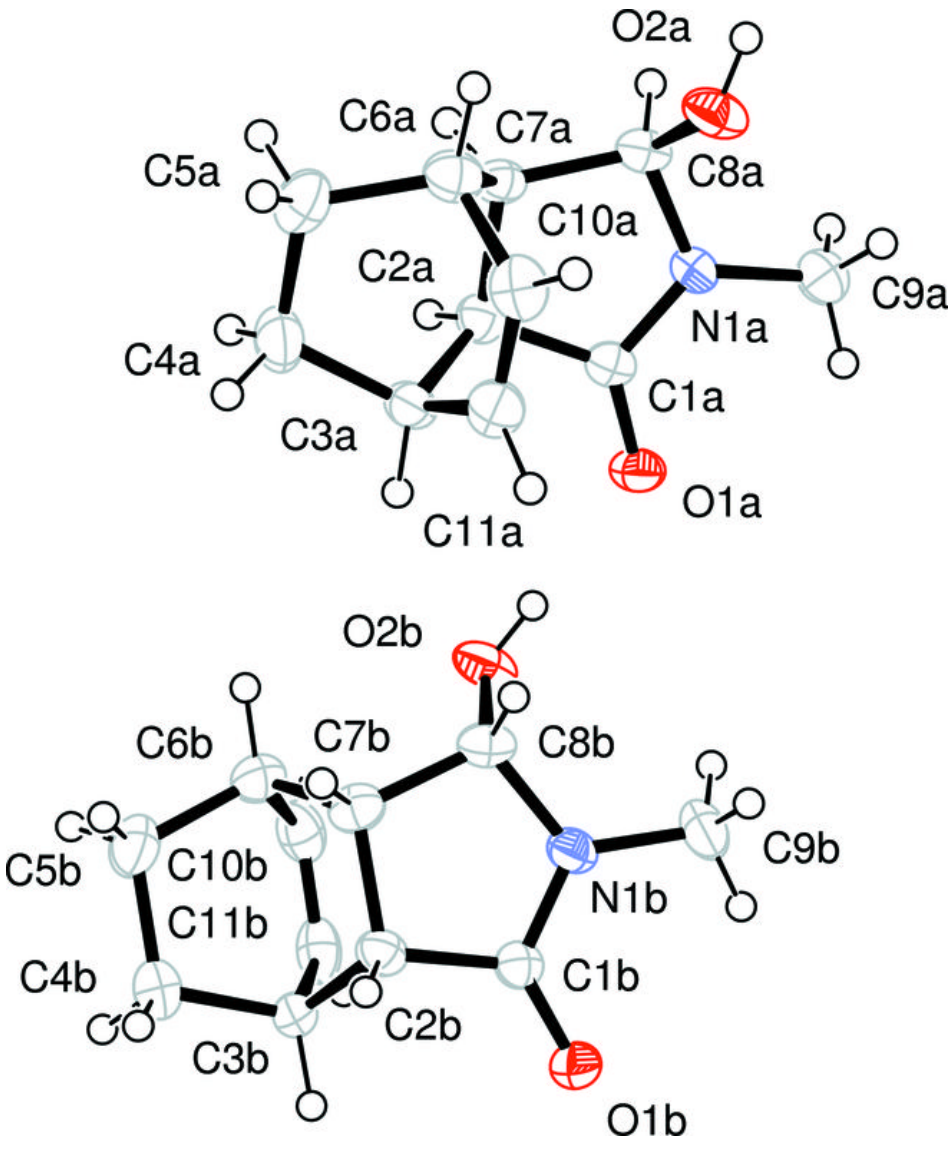
O2a
C6a
C7a
C8a
C5a
C10a
C2a
C9a
N1a
C4a
C1a
C3a
O1a
C11a
O2b
C6b
C7b
C8b
C9b
C5b
C10b
N1b
C11b
C4b
C1b
C2b
C3b
O1b

Fig. 2