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3a,4,10,10b-Tetrahydro-2*H*-furo[2,3-*a*]-carbazol-5(3*H*)-one

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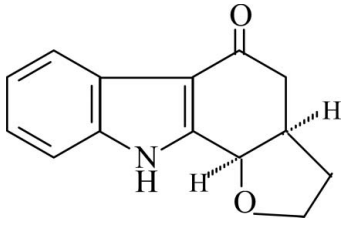
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.059; wR factor = 0.169; data-to-parameter ratio = 9.6.

The asymmetric unit of the title compound, $C_{14}H_{13}NO_2$, contains two independent molecules. In the indole ring system, the benzene and pyrrole rings are nearly coplanar, the dihedral angles being 0.48 (17) and 1.26 (17)°. The cyclohexenone and tetrahydrofuran rings have envelope conformations. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules into supramolecular chains nearly parallel to the c axis.

Related literature

For general background, see: Phillipson & Zenk (1980); Saxton (1983); Abraham (1975). For related structures, see: Hökelek *et al.* (1994, 1998, 1999, 2004, 2006); Patır *et al.* (1997); Hökelek & Patir (1999); Hökelek & Patır (2002). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{14}H_{13}NO_2$
M_r = 227.26
Triclinic, $P\overline{1}$
a = 9.5970 (2) Å
b = 10.0316 (3) Å
c = 12.4952 (3) Å
α = 98.498 (15)°
β = 107.499 (18)°
γ = 96.362 (19)°
V = 1119.21 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.25 × 0.20 × 0.15 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.961, T_{max} = 0.985
4127 measured reflections
3933 independent reflections
1657 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.169
S = 0.96
3933 reflections
411 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected torsion angles (°).

C10*B*—O1—C2—C3	8.7 (6)	C10′—O1′—C2′—C3′	0.3 (7)
C2—O1—C10*B*—C3*A*	−27.2 (5)	C2′—O1′—C10′—C3*A*′	−22.3 (6)
C3*A*—C3—C2—O1	12.9 (7)	C3*A*′—C3′—C2′—O1′	21.5 (7)
C10*B*—C3*A*—C3—C2	−28.0 (6)	C10′—C3*A*′—C4′—C5′	−39.9 (8)
C10*B*—C3*A*—C4—C5	−42.7 (6)	C10′—C3*A*′—C3′—C2′	−33.5 (6)
C5*A*—C5—C4—C3*A*	24.1 (6)	C3′—C3*A*′—C10′—O1′	34.5 (4)
C10*A*—C5*A*—C5—C4	−4.1 (6)	C4′—C3*A*′—C10′—C10	40.4 (6)
C5—C5*A*—C10*A*—C10*B*	2.8 (6)	C5*A*′—C5′—C4′—C3*A*′	17.8 (8)
C5*A*—C10*A*—C10*B*—C3*A*	−20.4 (6)	C10—C5*A*′—C5′—C4′	0.6 (7)
C10*A*—C10*B*—C3*A*—C4	38.6 (6)	C5′—C5*A*′—C10—N10′	−178.1 (4)
O1—C10*B*—C3*A*—C3	33.8 (5)	C5*A*′—C10—C10′—C3*A*′	−24.1 (6)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N10—H10···O2^{i}	0.96 (5)	1.98 (6)	2.876 (6)	155 (4)
N10′—H10′···O1^{ii}	0.83 (5)	2.01 (5)	2.829 (5)	169 (4)

Symmetry codes: (i) $x, y-1, z-1$; (ii) $-x+1, -y+1, -z+2$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2317).

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supplementary materials

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3a,4,10,10b-Tetrahydro-2*H*-furo[2,3-*a*]carbazol-5(3*H*)-one

N. Çaylak, T. Hökelek, N. Uludag and S. Patir

Comment

Tetrahydrocarbazole systems are present in the framework of a number of indole-type alkaloids of biological interest (Phillipson & Zenk, 1980; Saxton, 1983; Abraham, 1975). The structures of tricyclic, tetracyclic and pentacyclic ring systems with dithiolane and other substituents of the tetrahydrocarbazole core, have been the subject of much interest in our laboratory. These include 1,2,3,4-tetrahydrocarbazole-1-spiro-2'-[1,3]dithiolane, (II) (Hökelek *et al.*, 1994), *N*-(2-methoxyethyl)-*N*-{2,3,4,9-tetrahydrospiro[1*H*-carbazole-1, 2-(1,3)dithiolane]-4-yl}benzene-sulfonamide, (III) (Patır *et al.*, 1997), spiro[carbazole-1(2*H*),2'-[1,3]-dithiolan]-4(3*H*)-one, (IV) (Hökelek *et al.*, 1998), 9-acetonyl-3-ethylidene-1,2,3,4-tetrahydrospiro[carbazole-1,2'-[1,3] dithiolan]-4-one, (V) (Hökelek *et al.*, 1999), *N*-(2,2-dimethoxyethyl)-N -{9-methoxymethyl-1,2,3,4-tetrahydrospiro[carbazole-1,2'-[1,3]dithiolan] −4-yl}benzamide, (VI) (Hökelek & Patir, 1999); also the pentacyclic compounds 6-ethyl-4-(2-methoxyethyl)-2,6-methano-5-oxo-hexahydropyrrolo- (2,3 − d)carbazole-1-spiro-2'-(1,3)dithiolane, (VII) (Hökelek & Patır, 2002), *N*-(2-benzyloxyethyl)-4,7-dimethyl-6-(1,3-dithiolan-2-yl)-1,2,3,4, 5,6-hexahydro-1,5-methano-2-azocino[4,3-*b*]indol-2-one, (VIII) (Hökelek *et al.*, 2004) and 4-ethyl-6,6-ethylenedithio-2-(2-methoxyethyl)-7-methoxy- methylene-2,3,4,5,6,7-hexahydro-1,5-methano-1*H*-azocino[4,3-*b*]indol-3-one, (IX) (Hökelek *et al.*, 2006). The title compound, (I), may be considered as a synthetic precursor of tetracyclic indole alkaloids of biological interests. The present study was undertaken to ascertain its crystal structure.

The asymmetric unit of the title compound, (I), contains two independent molecules (Fig. 1). It consists of a carbazole skeleton with furan ring, in which the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The bonds N10—C9A [1.388 (5) Å], N10—C10A [1.357 (5) Å], N10'-C9A' [1.378 (5) Å] and N10'-C10 [1.362 (5) Å] generally agree with those in compounds (II)-(IX). In all structures atom N10 is substituted. The absolute configurations of C3a and C10*b* are *R* and *R*, respectively.

An examination of the deviations from the least-squares planes through individual rings shows that rings A (C5b/C6—C9/C9a), B (C5a/C5b/C9a/N10/C10*a*) and A' (C5b'/C6'-C9'/C9a'), B' (C5a'/C5b'/C9a'/N10'/C10) are planar. They are also nearly coplanar with dihedral angles of A/B = 0.48 (17)° and A'/B' = 1.26 (17)°. Rings C (C3a/C4/C5/C5a/C10*a*/C10*b*), D (O1/C2/C3/C3a/C10*b*) and C' (C3a'/C4'/C5'/C5a'/C10/C10'), D' (O1'/C2'/C3'/C3a'/C10') have envelope conformations with atoms C3a and C3a' displaced by −0.472 (5) Å (for ring C), 0.533 (5) Å (for ring D) and −0.491 (5) Å (for ring C'), −0.551 (5) Å (for ring D') from the planes of the other ring atoms, respectively. Rings C and C' have pseudo mirror planes running through atoms C3a and C5a (for ring C) and C3a' and C5a' (for ring C'), while rings D and D' have pseudo twofold axis and pseudo mirror plane, respectively, running through atom C2 and midpoint of C3a—C10*b* bond (for ring D) and atom C3a' and midpoint of O1'-C2'bond (for ring D'), as can be deduced from the torsion angles (Table 1).

In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 2) link the molecules into chains nearly parallel to *c* axis (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, (I), a solution of 2,3-dichloro −5,6-dicyano-*p*-benzoquine (4.25 g, 18.75 mmol) in tetrahydrofuran (30 ml) was added dropwise to an ice cold solution of (3aS,10bR)-3,3a,4,5,10,10*b* -hexahydro-2*H*-furo[2,3-*a*]carbazole (2.00 g, 9.37 mmol) in tetrahydrofuran -water (90:10, 50 ml). The mixture was stirred in an ice bath for 4 h, and then over night. The mixture was poured into sodium hydroxide (100 ml, 10%) and extracted with dichloromethane (25 ml). The organic layer was dried over anhydrous magnesium sulfate, and the solvent was evaporated under reduced pressure. The residue was crystallized from ethyl acetate (yield; 1.56 g, 73%).

Refinement

H atoms were located in difference syntheses and refined isotropically [N—H = 0.83 (4) and 0.96 (5) Å, U_{iso}(H) = 0.088 (17) and 0.062 (16) Å^2; C—H = 0.88 (4)–1.10 (6) Å, U_{iso}(H) = 0.038 (12)–0.16 (3) Å^2].

Figures

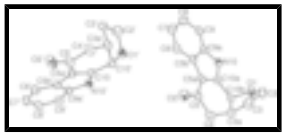

Fig. 1. The molecular structure of the title molecule with the atom-numbering scheme. The displacement ellipsoids are drawn at the 30% probability level. The hydrogen atoms are omitted for clarity.

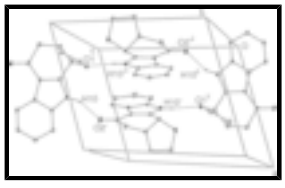

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted [symmetry code: (ii) $1-x$, $1-y$, $2-z$].

(3aR,10bR)-3a,4,10,10*b*-Tetrahydro-2*H*-furo[2,3-*a*]carbazol-5(3*H*)-one

Crystal data

$C_{14}H_{13}NO_2$	$Z = 4$
$M_r = 227.26$	$F_{000} = 480$
Triclinic, $P\overline{1}$	$D_x = 1.349$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 484 K
$a = 9.5970$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 10.0316$ (3) Å	Cell parameters from 25 reflections
$c = 12.4952$ (3) Å	$\theta = 3.5$–15.8º
$\alpha = 98.498$ (15)º	$\mu = 0.09$ mm^{-1}
$\beta = 107.499$ (18)º	$T = 298$ (2) K
$\gamma = 96.362$ (19)º	Block, colorless
$V = 1119.21$ (16) Å^3	0.25 × 0.20 × 0.15 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer	R_{int} = 0.032
Radiation source: fine-focus sealed tube	θ_{max} = 25.1º
Monochromator: graphite	θ_{min} = 3.2º
T = 298(2) K	h = −11→10
non–profiled ω scans	k = −11→11
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→14
T_{min} = 0.961, T_{max} = 0.985	3 standard reflections
4127 measured reflections	every 120 min
3933 independent reflections	intensity decay: 1%
1657 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.059	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.169	$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.96	$(\Delta/\sigma)_{max} < 0.001$
3933 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
411 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.3614 (4)	0.0625 (3)	0.8096 (2)	0.0575 (9)
O2	0.4088 (4)	0.4281 (4)	1.1854 (3)	0.0780 (11)

N10	0.6324 (4)	0.2534 (4)	0.9325 (3)	0.0453 (10)
H10	0.654 (5)	0.195 (5)	0.874 (4)	0.088 (17)*
C2	0.3106 (7)	−0.0309 (5)	0.8735 (5)	0.0624 (15)
H21	0.392 (6)	−0.087 (5)	0.903 (4)	0.097 (19)*
H22	0.245 (7)	−0.121 (6)	0.816 (5)	0.13 (2)*
C3	0.2490 (8)	0.0502 (6)	0.9539 (5)	0.0661 (16)
H31	0.325 (7)	0.061 (6)	1.033 (6)	0.13 (3)*
H32	0.150 (5)	0.010 (5)	0.952 (4)	0.077 (17)*
C3A	0.2338 (6)	0.1863 (5)	0.9132 (4)	0.0535 (13)
H3A	0.131 (5)	0.171 (4)	0.849 (4)	0.062 (14)*
C4	0.2435 (6)	0.3072 (6)	1.0061 (4)	0.0557 (14)
H41	0.213 (6)	0.398 (6)	0.971 (4)	0.108 (19)*
H42	0.178 (5)	0.286 (5)	1.042 (4)	0.065 (15)*
C5	0.3954 (5)	0.3658 (5)	1.0900 (4)	0.0483 (12)
C5A	0.5185 (5)	0.3427 (4)	1.0513 (3)	0.0393 (11)
C5B	0.6747 (5)	0.3850 (4)	1.1055 (4)	0.0424 (11)
C6	0.7658 (6)	0.4656 (5)	1.2100 (4)	0.0508 (13)
H6	0.720 (4)	0.503 (4)	1.259 (3)	0.038 (12)*
C7	0.9151 (7)	0.4841 (6)	1.2311 (5)	0.0622 (15)
H7	0.973 (5)	0.542 (4)	1.295 (4)	0.053 (14)*
C8	0.9794 (7)	0.4263 (5)	1.1547 (5)	0.0628 (15)
H8	1.086 (6)	0.443 (5)	1.167 (4)	0.093 (19)*
C9	0.8942 (5)	0.3469 (5)	1.0509 (4)	0.0524 (13)
H9	0.940 (5)	0.302 (5)	0.997 (4)	0.087 (17)*
C9A	0.7427 (5)	0.3281 (4)	1.0291 (3)	0.0409 (11)
C10A	0.4989 (5)	0.2641 (4)	0.9463 (3)	0.0383 (11)
C10B	0.3547 (5)	0.1990 (5)	0.8586 (4)	0.0441 (12)
H10B	0.328 (6)	0.255 (5)	0.803 (4)	0.096 (19)*
O1'	0.8017 (4)	0.7748 (3)	1.4224 (2)	0.0590 (9)
O2'	0.7739 (4)	1.0773 (4)	1.8104 (3)	0.0793 (12)
N10'	0.6708 (4)	1.0219 (4)	1.4240 (3)	0.0455 (10)
H10'	0.666 (5)	0.988 (4)	1.358 (4)	0.062 (16)*
C2'	0.9560 (7)	0.7825 (9)	1.4831 (5)	0.0769 (19)
H21'	1.001 (8)	0.847 (7)	1.452 (6)	0.16 (3)*
H22'	0.973 (6)	0.681 (6)	1.467 (5)	0.11 (2)*
C3'	0.9758 (6)	0.8239 (6)	1.6083 (4)	0.0574 (14)
H31'	1.015 (5)	0.926 (5)	1.631 (4)	0.087 (18)*
H32'	1.053 (5)	0.771 (4)	1.655 (4)	0.063 (14)*
C3A'	0.8212 (5)	0.7840 (5)	1.6150 (4)	0.0477 (12)
H3A'	0.807 (5)	0.682 (5)	1.615 (4)	0.076 (15)*
C4'	0.7920 (8)	0.8567 (6)	1.7204 (5)	0.0607 (15)
H41'	0.705 (6)	0.813 (5)	1.717 (4)	0.08 (2)*
H42'	0.873 (6)	0.848 (5)	1.793 (5)	0.090 (18)*
C5'	0.7615 (5)	1.0012 (5)	1.7200 (4)	0.0521 (13)
C5A'	0.7197 (4)	1.0437 (4)	1.6119 (3)	0.0399 (11)
C5B'	0.6913 (4)	1.1738 (4)	1.5828 (3)	0.0410 (11)
C6'	0.6891 (5)	1.3000 (5)	1.6424 (4)	0.0468 (12)
H6'	0.707 (4)	1.323 (4)	1.717 (4)	0.052 (14)*
C7'	0.6594 (5)	1.4060 (5)	1.5852 (4)	0.0524 (13)

H7'	0.651 (4)	1.498 (4)	1.619 (3)	0.048 (12)*
C8'	0.6326 (6)	1.3864 (5)	1.4668 (5)	0.0591 (14)
H8'	0.608 (4)	1.467 (4)	1.423 (3)	0.060 (13)*
C9'	0.6346 (5)	1.2632 (5)	1.4058 (4)	0.0520 (13)
H9'	0.615 (4)	1.246 (4)	1.329 (3)	0.042 (12)*
C9A'	0.6622 (4)	1.1557 (4)	1.4631 (3)	0.0401 (11)
C10	0.7070 (4)	0.9548 (4)	1.5132 (4)	0.0409 (11)
C10'	0.7247 (5)	0.8092 (5)	1.5011 (4)	0.0452 (12)
H10A	0.624 (5)	0.758 (4)	1.476 (3)	0.052 (13)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.079 (2)	0.050 (2)	0.0440 (19)	0.0092 (17)	0.0228 (17)	0.0041 (16)
O2	0.081 (3)	0.104 (3)	0.051 (2)	0.019 (2)	0.037 (2)	−0.014 (2)
N10	0.055 (3)	0.046 (2)	0.040 (2)	0.012 (2)	0.024 (2)	0.0056 (19)
C2	0.082 (4)	0.047 (3)	0.057 (3)	0.005 (3)	0.017 (3)	0.019 (3)
C3	0.072 (4)	0.063 (4)	0.069 (4)	0.001 (3)	0.032 (4)	0.020 (3)
C3A	0.052 (3)	0.061 (3)	0.050 (3)	0.011 (3)	0.017 (3)	0.017 (3)
C4	0.058 (4)	0.068 (4)	0.054 (3)	0.019 (3)	0.031 (3)	0.020 (3)
C5	0.061 (4)	0.054 (3)	0.042 (3)	0.023 (3)	0.027 (3)	0.013 (2)
C5A	0.054 (3)	0.038 (3)	0.033 (2)	0.014 (2)	0.023 (2)	0.007 (2)
C5B	0.055 (3)	0.040 (3)	0.040 (3)	0.016 (2)	0.024 (2)	0.010 (2)
C6	0.065 (4)	0.054 (3)	0.041 (3)	0.013 (3)	0.026 (3)	0.008 (2)
C7	0.066 (4)	0.064 (4)	0.046 (3)	0.008 (3)	0.011 (3)	−0.006 (3)
C8	0.055 (4)	0.067 (4)	0.066 (4)	0.013 (3)	0.020 (3)	0.006 (3)
C9	0.053 (4)	0.056 (3)	0.054 (3)	0.018 (3)	0.024 (3)	0.006 (3)
C9A	0.048 (3)	0.040 (3)	0.038 (3)	0.009 (2)	0.020 (2)	0.005 (2)
C10A	0.047 (3)	0.040 (3)	0.036 (3)	0.011 (2)	0.020 (2)	0.013 (2)
C10B	0.060 (3)	0.036 (3)	0.039 (3)	0.010 (2)	0.019 (3)	0.008 (2)
O1'	0.070 (3)	0.065 (2)	0.0415 (19)	0.0224 (18)	0.0193 (18)	−0.0018 (16)
O2'	0.134 (3)	0.083 (3)	0.045 (2)	0.050 (2)	0.049 (2)	0.0171 (19)
N10'	0.054 (3)	0.048 (3)	0.034 (2)	0.0126 (19)	0.016 (2)	0.004 (2)
C2'	0.067 (5)	0.110 (6)	0.055 (4)	0.028 (4)	0.024 (3)	0.004 (4)
C3'	0.052 (4)	0.059 (4)	0.060 (3)	0.017 (3)	0.017 (3)	0.003 (3)
C3A'	0.064 (4)	0.039 (3)	0.045 (3)	0.011 (2)	0.022 (2)	0.011 (2)
C4'	0.087 (5)	0.061 (4)	0.049 (3)	0.024 (4)	0.035 (3)	0.022 (3)
C5'	0.067 (3)	0.054 (3)	0.049 (3)	0.019 (3)	0.034 (3)	0.016 (3)
C5A'	0.049 (3)	0.038 (3)	0.035 (3)	0.005 (2)	0.019 (2)	0.003 (2)
C5B'	0.041 (3)	0.045 (3)	0.038 (3)	0.006 (2)	0.016 (2)	0.003 (2)
C6'	0.055 (3)	0.043 (3)	0.046 (3)	0.008 (2)	0.024 (3)	0.003 (3)
C7'	0.058 (3)	0.036 (3)	0.068 (4)	0.007 (2)	0.028 (3)	0.007 (3)
C8'	0.068 (4)	0.054 (4)	0.069 (4)	0.019 (3)	0.035 (3)	0.020 (3)
C9'	0.059 (3)	0.062 (4)	0.042 (3)	0.017 (3)	0.021 (3)	0.016 (3)
C9A'	0.044 (3)	0.045 (3)	0.036 (3)	0.012 (2)	0.018 (2)	0.008 (2)
C10	0.037 (3)	0.044 (3)	0.043 (3)	0.006 (2)	0.015 (2)	0.007 (2)
C10'	0.045 (3)	0.046 (3)	0.046 (3)	0.006 (3)	0.022 (3)	0.001 (2)

Geometric parameters (Å, °)

O1—C2	1.451 (5)	O1'—C2'	1.434 (6)
O1—C10B	1.432 (5)	O1'—C10'	1.424 (5)
O2—C5	1.225 (5)	O2'—C5'	1.232 (5)
N10—C9A	1.388 (5)	N10'—C9A'	1.378 (5)
N10—C10A	1.357 (5)	N10'—C10	1.362 (5)
N10—H10	0.96 (5)	N10'—H10'	0.83 (4)
C2—H21	1.03 (5)	C2'—H21'	0.93 (7)
C2—H22	1.07 (6)	C2'—H22'	1.05 (6)
C3—C2	1.498 (7)	C3'—C2'	1.507 (7)
C3—H31	1.02 (6)	C3'—H31'	1.02 (5)
C3—H32	0.98 (5)	C3'—H32'	1.04 (4)
C3A—C3	1.532 (7)	C3A'—C3'	1.524 (6)
C3A—H3A	1.05 (4)	C3A'—C4'	1.526 (6)
C3A—C4	1.524 (7)	C3A'—C10'	1.520 (6)
C4—H41	1.10 (6)	C3A'—H3A'	1.02 (5)
C4—H42	0.90 (4)	C4'—H41'	0.88 (5)
C5—C4	1.510 (7)	C4'—H42'	1.03 (5)
C5A—C5	1.433 (5)	C5'—C4'	1.510 (7)
C5A—C5B	1.432 (6)	C5A'—C5'	1.432 (6)
C5A—C10A	1.375 (5)	C5A'—C10	1.375 (5)
C5B—C6	1.403 (6)	C5B'—C5A'	1.439 (6)
C6—C7	1.362 (7)	C5B'—C6'	1.376 (6)
C6—H6	0.91 (4)	C5B'—C9A'	1.416 (5)
C7—C8	1.378 (7)	C6'—H6'	0.88 (4)
C7—H7	0.90 (4)	C7'—C6'	1.376 (6)
C8—H8	0.98 (5)	C7'—C8'	1.403 (6)
C9—C8	1.377 (6)	C7'—H7'	0.98 (4)
C9—H9	0.99 (5)	C8'—H8'	1.05 (4)
C9A—C5B	1.399 (5)	C9A'—C9'	1.387 (6)
C9A—C9	1.382 (6)	C9'—C8'	1.358 (6)
C10A—C10B	1.490 (6)	C9'—H9'	0.90 (4)
C10B—C3A	1.515 (6)	C10—C10'	1.481 (6)
C10B—H10B	0.95 (5)	C10'—H10A	0.98 (4)
C10B—O1—C2	108.7 (3)	C9A'—N10'—H10'	126 (3)
C10A—N10—C9A	108.6 (3)	O1'—C2'—C3'	106.8 (4)
C10A—N10—H10	129 (3)	O1'—C2'—H22'	103 (3)
C9A—N10—H10	122 (3)	C3'—C2'—H22'	109 (3)
O1—C2—C3	107.2 (4)	O1'—C2'—H21'	103 (5)
O1—C2—H21	109 (3)	C3'—C2'—H21'	115 (5)
C3—C2—H21	121 (3)	H22'—C2'—H21'	118 (6)
O1—C2—H22	109 (3)	C2'—C3'—C3A'	103.6 (5)
C3—C2—H22	120 (3)	C2'—C3'—H32'	110 (2)
H21—C2—H22	89 (4)	C3A'—C3'—H32'	112 (2)
C2—C3—C3A	105.1 (4)	C2'—C3'—H31'	108 (3)
C2—C3—H32	115 (3)	C3A'—C3'—H31'	114 (3)
C3A—C3—H32	107 (3)	H32'—C3'—H31'	110 (4)

C2—C3—H31	106 (4)	C10'—C3A'—C3'	101.4 (4)
C3A—C3—H31	113 (4)	C10'—C3A'—C4'	115.4 (4)
H32—C3—H31	111 (4)	C3'—C3A'—C4'	115.6 (5)
C10B—C3A—C4	114.9 (4)	C10'—C3A'—H3A'	109 (3)
C10B—C3A—C3	101.8 (4)	C3'—C3A'—H3A'	107 (3)
C4—C3A—C3	114.8 (4)	C4'—C3A'—H3A'	108 (3)
C10B—C3A—H3A	109 (2)	C5'—C4'—C3A'	116.7 (4)
C4—C3A—H3A	110 (2)	C5'—C4'—H42'	113 (3)
C3—C3A—H3A	106 (2)	C3A'—C4'—H42'	110 (3)
C5—C4—C3A	116.7 (4)	C5'—C4'—H41'	102 (4)
C5—C4—H42	112 (3)	C3A'—C4'—H41'	104 (3)
C3A—C4—H42	109 (3)	H42'—C4'—H41'	111 (4)
C5—C4—H41	100 (3)	O2'—C5'—C5A'	122.7 (4)
C3A—C4—H41	113 (3)	O2'—C5'—C4'	120.0 (4)
H42—C4—H41	106 (4)	C5A'—C5'—C4'	117.2 (4)
O2—C5—C5A	123.5 (4)	C10—C5A'—C5'	120.6 (4)
O2—C5—C4	120.2 (4)	C10—C5A'—C5B'	108.1 (4)
C5A—C5—C4	116.3 (4)	C5'—C5A'—C5B'	131.2 (4)
C10A—C5A—C5B	106.9 (3)	C6'—C5B'—C9A'	119.1 (4)
C10A—C5A—C5	121.5 (4)	C6'—C5B'—C5A'	135.4 (4)
C5B—C5A—C5	131.5 (4)	C9A'—C5B'—C5A'	105.4 (3)
C9A—C5B—C6	118.0 (4)	C5B'—C6'—C7'	119.7 (5)
C9A—C5B—C5A	106.5 (4)	C5B'—C6'—H6'	127 (3)
C6—C5B—C5A	135.5 (4)	C7'—C6'—H6'	114 (3)
C7—C6—C5B	118.1 (5)	C6'—C7'—C8'	120.4 (5)
C7—C6—H6	125 (2)	C6'—C7'—H7'	126 (2)
C5B—C6—H6	117 (2)	C8'—C7'—H7'	113 (2)
C6—C7—C8	122.8 (5)	C9—C8—C7	121.1 (5)
C6—C7—H7	118 (3)	C9—C8—H8	115 (3)
C8—C7—H7	119 (3)	C7—C8—H8	124 (3)
C8—C9—C9A	116.4 (5)	C9'—C8'—C7'	121.2 (5)
C8—C9—H9	121 (3)	C9'—C8'—H8'	119 (2)
C9A—C9—H9	122 (3)	C7'—C8'—H8'	120 (2)
C9—C9A—N10	128.2 (4)	C8'—C9'—C9A'	118.5 (5)
C9—C9A—C5B	123.7 (4)	C8'—C9'—H9'	124 (2)
N10—C9A—C5B	108.1 (4)	C9A'—C9'—H9'	117 (2)
N10—C10A—C5A	109.9 (4)	N10'—C9A'—C9'	131.0 (4)
N10—C10A—C10B	123.7 (4)	N10'—C9A'—C5B'	107.8 (4)
C5A—C10A—C10B	126.4 (4)	C9'—C9A'—C5B'	121.1 (4)
O1—C10B—C10A	110.8 (4)	N10'—C10—C5A'	108.6 (4)
O1—C10B—C3A	105.8 (4)	N10'—C10—C10'	124.0 (4)
C10A—C10B—C3A	110.3 (4)	C5A'—C10—C10'	127.4 (4)
O1—C10B—H10B	113 (3)	O1'—C10'—C10	111.3 (4)
C10A—C10B—H10B	109 (3)	O1'—C10'—C3A'	105.5 (4)
C3A—C10B—H10B	107 (3)	C10—C10'—C3A'	109.1 (4)
C10'—O1'—C2'	109.8 (4)	O1'—C10'—H10A	112 (2)
C10—N10'—C9A'	110.0 (4)	C10—C10'—H10A	105 (2)
C10—N10'—H10'	123 (3)	C3A'—C10'—H10A	113 (2)
C10B—O1—C2—C3	8.7 (6)	C10'—O1'—C2'—C3'	0.3 (7)

C2—O1—C10B—C10A	92.5 (4)	C2'—O1'—C10'—C10	96.0 (5)
C2—O1—C10B—C3A	−27.2 (5)	C2'—O1'—C10'—C3A'	−22.3 (6)
C10A—N10—C9A—C9	−179.1 (4)	C10—N10'—C9A'—C9'	177.3 (4)
C10A—N10—C9A—C5B	0.9 (5)	C10—N10'—C9A'—C5B'	−1.3 (5)
C9A—N10—C10A—C5A	−1.0 (5)	C3A'—C3'—C2'—O1'	21.5 (7)
C9A—N10—C10A—C10B	178.8 (4)	C10'—C3A'—C4'—C5'	−39.9 (8)
C3A—C3—C2—O1	12.9 (7)	C3'—C3A'—C4'—C5'	78.2 (7)
C4—C3A—C3—C2	−152.8 (5)	C10'—C3A'—C3'—C2'	−33.5 (6)
C10B—C3A—C3—C2	−28.0 (6)	C4'—C3A'—C3'—C2'	−159.1 (5)
C10B—C3A—C4—C5	−42.7 (6)	C3'—C3A'—C10'—O1'	34.5 (4)
C3—C3A—C4—C5	74.9 (6)	C4'—C3A'—C10'—O1'	160.1 (4)
O2—C5—C4—C3A	−155.4 (4)	C3'—C3A'—C10'—C10	−85.3 (4)
C5A—C5—C4—C3A	24.1 (6)	C4'—C3A'—C10'—C10	40.4 (6)
C10A—C5A—C5—O2	175.4 (4)	O2'—C5'—C4'—C3A'	−161.7 (5)
C5B—C5A—C5—O2	−2.1 (7)	C5A'—C5'—C4'—C3A'	17.8 (8)
C10A—C5A—C5—C4	−4.1 (6)	C10—C5A'—C5'—O2'	−179.9 (5)
C5B—C5A—C5—C4	178.4 (4)	C5B'—C5A'—C5'—O2'	3.3 (8)
C10A—C5A—C5B—C9A	−0.2 (4)	C10—C5A'—C5'—C4'	0.6 (7)
C5—C5A—C5B—C9A	177.6 (4)	C5B'—C5A'—C5'—C4'	−176.2 (5)
C10A—C5A—C5B—C6	179.2 (5)	C5'—C5A'—C10—N10'	−178.1 (4)
C5—C5A—C5B—C6	−3.0 (8)	C5B'—C5A'—C10—N10'	−0.6 (5)
C5B—C5A—C10A—N10	0.7 (4)	C5'—C5A'—C10—C10'	3.5 (7)
C5—C5A—C10A—N10	−177.3 (4)	C5B'—C5A'—C10—C10'	−179.1 (4)
C5B—C5A—C10A—C10B	−179.1 (4)	C6'—C5B'—C5A'—C10	−179.1 (5)
C5—C5A—C10A—C10B	2.8 (6)	C9A'—C5B'—C5A'—C10	−0.2 (5)
C9A—C5B—C6—C7	0.1 (6)	C6'—C5B'—C5A'—C5'	−2.0 (8)
C5A—C5B—C6—C7	−179.3 (5)	C9A'—C5B'—C5A'—C5'	176.9 (4)
C5B—C6—C7—C8	−0.5 (8)	C9A'—C5B'—C6'—C7'	−0.2 (7)
C6—C7—C8—C9	0.7 (8)	C5A'—C5B'—C6'—C7'	178.6 (4)
C9A—C9—C8—C7	−0.5 (7)	C6'—C5B'—C9A'—N10'	−180.0 (4)
C9—C9A—C5B—C6	0.1 (6)	C5A'—C5B'—C9A'—N10'	0.9 (4)
N10—C9A—C5B—C6	−179.9 (4)	C6'—C5B'—C9A'—C9'	1.3 (6)
C9—C9A—C5B—C5A	179.6 (4)	C5A'—C5B'—C9A'—C9'	−177.9 (4)
N10—C9A—C5B—C5A	−0.4 (4)	C8'—C7'—C6'—C5B'	−0.5 (7)
N10—C9A—C9—C8	−179.9 (4)	C6'—C7'—C8'—C9'	0.3 (7)
C5B—C9A—C9—C8	0.2 (7)	C9A'—C9'—C8'—C7'	0.7 (7)
N10—C10A—C10B—O1	42.9 (6)	N10'—C9A'—C9'—C8'	−180.0 (4)
C5A—C10A—C10B—O1	−137.3 (4)	C5B'—C9A'—C9'—C8'	−1.5 (7)
N10—C10A—C10B—C3A	159.8 (4)	N10'—C10—C10'—O1'	41.6 (6)
C5A—C10A—C10B—C3A	−20.4 (6)	C5A'—C10—C10'—O1'	−140.2 (4)
O1—C10B—C3A—C4	158.5 (4)	N10'—C10—C10'—C3A'	157.7 (4)
C10A—C10B—C3A—C4	38.6 (6)	C5A'—C10—C10'—C3A'	−24.1 (6)
O1—C10B—C3A—C3	33.8 (5)	C9A'—N10'—C10—C5A'	1.2 (5)
C10A—C10B—C3A—C3	−86.1 (5)	C9A'—N10'—C10—C10'	179.7 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N10—H10···O2^{i}	0.96 (5)	1.98 (6)	2.876 (6)	155 (4)

N10'—H10'···O1^{ii}	0.83 (5)	2.01 (5)	2.829 (5)	169 (4)

Symmetry codes: (i) *x*, *y*−1, *z*−1; (ii) −*x*+1, −*y*+1, −*z*+2.

Fig. 1

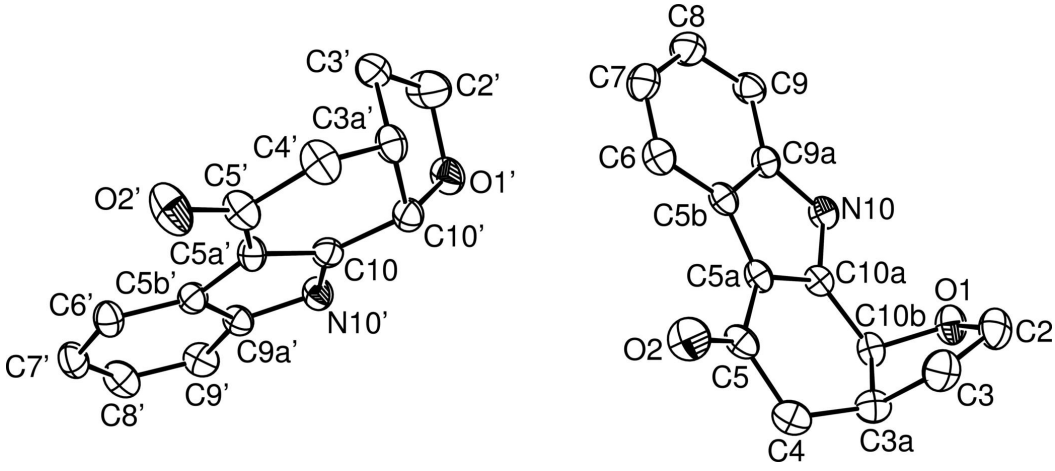

Fig. 2