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1-Isopropyl-6,6,8a-trimethyl-1,3a,5,6,7,8a-hexahydro-3*H*-1-benzofuro[2,3-*b*]pyrrole-2,4-dione

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.101
Data-to-parameter ratio = 10.7

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

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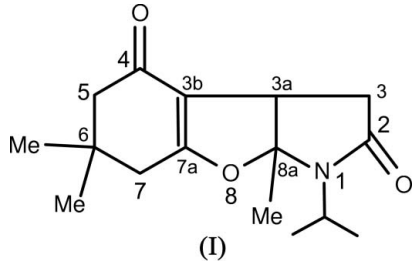
The crystal structure of the title benzofuran derivative, $C_{16}H_{23}NO_3$, has been elucidated. The tricyclic core, *i.e.* the tetrahydrobenzo–dihydrofuro–pyrrolidine ring system, is non-planar owing to the folding of the five-membered rings at their *cis* junction. The cyclohexene ring assumes a half-chair conformation, while the dihydrofuran and pyrrolidine rings each adopt an envelope conformation. Intramolecular C—H···O hydrogen bonds form *S*(6) closed patterns.

Comment

The title compound, (I), has been shown to exhibit a moderate hypoglycemic activity in a previous structure–activity relationship study (Nagarajan *et al.*, 1988). Compound (I) is a new tricyclic benzofuran derivative containing linearly fused tetrahydrobenzo–dihydrofuro–pyrrolidine (*A*–*B*–*C*) rings. This chiral molecule formally derives from a perhydro–furo (or –pyrrolo)–benzofuran system (Nagarajan *et al.*, 1988) and is structurally related to a structure containing a tetrahydrobenzo–furo–furan ring system, which we recently published (Nagaraj *et al.*, 2005).



(I)

The molecular structure is shown in Fig. 1. The *BC* ring-junction is *cis* (Bucourt, 1974). The shape of the tricyclic core is non-planar owing to the folding at the *BC* junction. The torsion angles at this junction, namely N1—C1—C4—C5 and O1—C1—C4—C3, are −99.69 (11) and 132.78 (11)°, respectively. The structure of the analogous molecule based on a chiral tetrahydrobenzo–furo–furan core (Nagaraj *et al.*, 2005) also has a non-planar shape for its tricyclic core, and the equivalent torsion angles are 103.98 (10) and −127.17 (10)°, respectively. The torsion angle C1—N1—C14—C15 in (I), describing the conformation of the *N*-isopropyl substituent, is 121.51 (14)°. The internal torsion angles of individual rings are shown in Fig. 1. Ring *A* (cyclohexene) adopts a half-chair (C_2) conformation (Bucourt, 1974) with the following values of puckering parameters (Cremer & Pople, 1975): q_2 = 0.347 (2), q_3 = −0.275 (2) Å, φ_2 = 345.7 (3), θ_2 = 128.4 (2)° and Q = 0.443 (2) Å. Rings *B* and *C* adopt envelope (C_s) conforma-

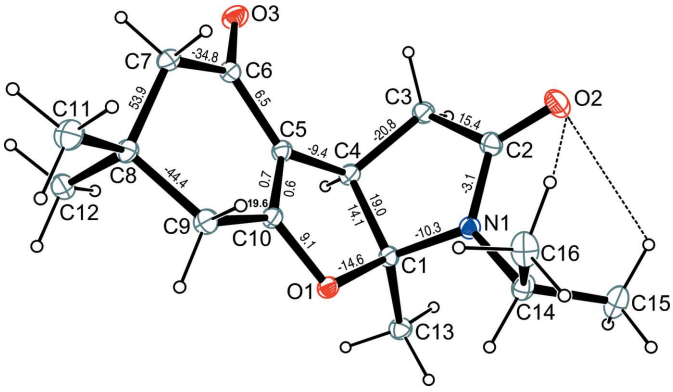


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and numerical values refer to the internal torsion angles (°) of individual rings (s.u. values lie in the range 0.1–0.2°). Intramolecular C—H···O hydrogen bonds are displayed with dashed lines.

tions (Fuchs, 1978) with atoms C1 and C4, respectively, at the flap positions. Atoms C1 and C4 are 0.24 (1) and 0.33 (1) Å out of the mean planes formed by the remaining ring atoms. The puckering amplitudes q (Å) and the phase angles φ (°) of the five-membered rings are 0.146 (2) and 39.7 (7), and 0.210 (2) and 80.3 (5), respectively. Two $S(6)$ hydrogen-bonded closed patterns (Bernstein *et al.*, 1995) are formed by C15—H···O2 and C16—H···O2 intramolecular contacts (Table 1). The lengths and directionality suggest that these hydrogen bonds are very weak. The crystal packing is entirely due to van der Waals interactions.

Experimental

The synthesis of (I) was described by Nagarajan *et al.* (1988). Suitable single crystals were obtained by slow evaporation of a benzene–hexane (1:1) solution.

Crystal data

$C_{16}H_{23}NO_3$
M_r = 277.35
Triclinic, $P\overline{1}$
a = 8.9725 (3) Å
b = 10.2472 (3) Å
c = 10.4372 (3) Å
α = 101.733 (2)°
β = 109.290 (1)°
γ = 115.765 (1)°
V = 744.67 (4) Å^3
Z = 2
D_x = 1.237 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3368 reflections
θ = 2.9–27.5°
μ = 0.09 mm^{-1}
T = 120 (2) K
Plate, colourless
0.22 × 0.18 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.890, T_{max} = 0.996
14657 measured reflections
2911 independent reflections
2548 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 26.0°
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.101
S = 1.09
2911 reflections
273 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 0.3291P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H153···O2	0.99 (3)	2.54 (2)	3.122 (2)	118 (2)
C16—H161···O2	1.02 (3)	2.58 (2)	3.186 (2)	118 (1)

Larger than expected values of residual electron density were observed and attributed to the presence of a few poorly fitting reflections ($\overline{1}22$, $1\overline{1}1$, 011, $2\overline{2}1$, $\overline{2}22$ and $\overline{1}\overline{1}1$). The application of an extinction correction [extinction parameter = 0.57 (3)] further degraded the model quality. In the absence of any obvious cause, these reflections were omitted during the last cycles of refinement. Residual electron density was then featureless and the residual factor R dropped from 0.056 to 0.039 for observed data. H atoms were located in a difference map and were refined freely [C—H = 0.97 (2)–1.02 (2) Å].

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: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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