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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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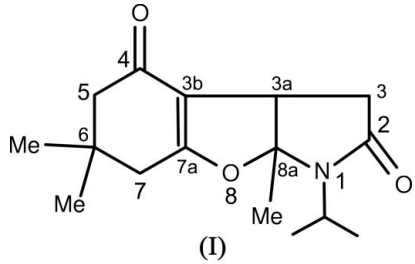
The crystal structure of the title benzofuran derivative, $C_{16}H_{23}NO_3$, has been elucidated. The tricyclic core, *i.e.* the tetrahydrobenzo–dihydrofuro–pyrrolidine ring system, is non-planar owing to the folding of the five-membered rings at their *cis* junction. The cyclohexene ring assumes a 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-

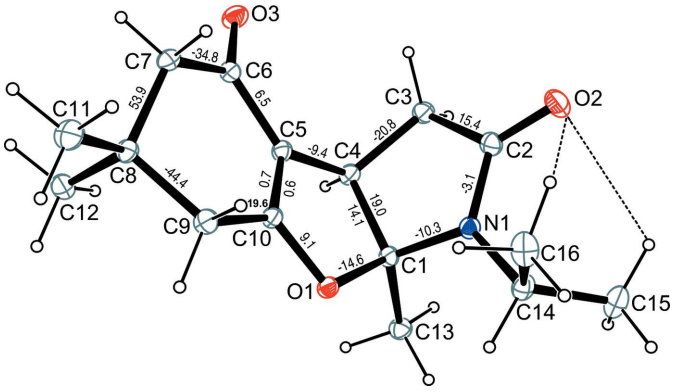


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and numerical values refer to the internal torsion angles (°) of individual rings (s.u. values lie in the range 0.1–0.2°). Intramolecular C—H···O hydrogen bonds are displayed with dashed lines.

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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supporting information

Acta Cryst. (2006). E**62**, o1328–o1329 [https://doi.org/10.1107/S1600536806006180]

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Plate, colourless
0.22 × 0.18 × 0.05 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)
$T_{min} = 0.890$, $T_{max} = 0.996$
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$\theta_{max} = 26.0°$, $\theta_{min} = 3.2°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.101$
$S = 1.09$
2911 reflections
273 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
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}

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 0.872591.

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.
========== Weighted least-squares planes through the starred atoms (Nardelli, Musatti, Domiano & Andreetti Ric·Sci.(1965),15(II—A),807). Equation of the plane: m1*X+m2*Y+m3*Z=d
==========
Plane 1 m1 = 0.48034(0.00080) m2 = -0.51073(0.00089) m3 = -0.71305(0.00054) D = -3.47648(0.00103) Atom d s d/s (d/s)**2 O1 * 0.0009 0.0010 0.840 0.705 C4 * -0.0017 0.0015 - 1.094 1.196 C5 * 0.0030 0.0015 1.999 3.995 C10 * -0.0033 0.0015 - 2.183 4.764 C1 0.2352 0.0015 155.518 24185.846 C6 0.0321 0.0016 20.563 422.838 C9 0.0025 0.0016 1.520 2.310 ========== Sum((d/s)**2) for starred atoms 10.660 Chi-squared at 95% for 1 degrees of freedom: 3.84 The group of atoms deviates significantly from planarity
Plane 2 m1 = -0.57685(0.00075) m2 = -0.81274(0.00052) m3 = -0.08184(0.00118) D = -2.53847(0.00205) Atom d s d/s (d/s)**2 C1 * 0.0096 0.0016 5.952 35.423 N1 * -0.0121 0.0014 - 8.837 78.089 C2 * 0.0182 0.0017 10.568 111.686 C3 * -0.0118 0.0019 - 6.308 39.797 C4 0.3292 0.0017 196.068 38442.645 O2 0.0241 0.0014 17.584 309.209 C14 0.0026 0.0018 1.426 2.033 ========== Sum((d/s)**2) for starred atoms 264.994 Chi-squared at 95% for 1 degrees of freedom: 3.84 The group of atoms deviates significantly from planarity
Plane 3 m1 = 0.50473(0.00072) m2 = -0.44860(0.00067) m3 = -0.73757(0.00047) D = -3.49574(0.00100) Atom d s d/s (d/s)**2 C5 * 0.0160 0.0015 10.544 111.186 C6 * -0.0513 0.0016 - 32.865 1080.080 C7 * 0.0504 0.0017 29.536 872.347 C9 * -0.0388 0.0016 - 23.749 564.027 C10 * 0.0268 0.0015 17.427 303.694 C8 - 0.6054 0.0016 - 375.708 141156.844 O1 0.1249 0.0010 120.888 14613.903 O3 - 0.1396 0.0012 - 120.991 14638.831 C1 0.4114 0.0015 271.503 73713.656 C4 0.1036 0.0015 67.343 4535.050 ========== Sum((d/s)**2) for starred atoms 2931.333 Chi-squared at 95% for 2 degrees of freedom: 5.99 The group of atoms deviates significantly from planarity
Plane 4 m1 = 0.43339(0.00065) m2 = -0.56971(0.00061) m3 = -0.69829(0.00046) D = -3.49262(0.00082) Atom d s d/s (d/s)**2 O1 * -0.0444 0.0010 - 42.960 1845.563 C4 * -0.0857 0.0015 - 55.889 3123.553 C5 * 0.0320 0.0015 21.356 456.076 C10 * 0.0398 0.0015 26.204 686.636 C1 * 0.1049 0.0015 69.890 4884.553 C6 0.1476 0.0016 95.146 9052.818 C9 0.1452 0.0016 89.106 7939.827 ========== Sum((d/s)**2) for starred atoms 10996.382 Chi-squared at 95% for 2 degrees of freedom: 5.99 The group of atoms deviates significantly from planarity
Plane 5 m1 = -0.54504(0.00072) m2 = -0.81842(0.00049) m3 = -0.18198(0.00081) D = -2.64088(0.00166) Atom d s d/s (d/s)**2 C1 * -0.0918 0.0016 - 57.193 3271.052 N1 * 0.0233 0.0014 17.159 294.425 C2 * 0.0494 0.0017 28.976 839.585 C3 * -0.1326 0.0019 - 71.591 5125.335 C4 * 0.1238 0.0017 74.360 5529.379 O2 0.1575 0.0014 115.889 13430.213 C14 0.1679 0.0018 92.604 8575.446 ========== Sum((d/s)**2) for starred atoms 15059.777 Chi-squared at 95% for 2 degrees of freedom: 5.99 The group of atoms deviates significantly from planarity
Dihedral angles formed by LSQ-planes Plane - plane angle (s.u.) angle (s.u.) 1 2 78.68 (0.07) 101.32 (0.07) 1 3 4.08 (0.06) 175.92 (0.06) 1 4 4.40 (0.06) 175.60 (0.06) 1 5 73.38 (0.07) 106.62 (0.07) 2 3 82.31 (0.07) 97.69 (0.07) 2 4 74.33 (0.07) 105.67 (0.07) 2 5 6.03 (0.08) 173.97 (0.08) 3 4 8.37 (0.05) 171.63 (0.05) 3 5 76.92 (0.06) 103.08 (0.06) 4 5 69.08 (0.05) 110.92 (0.05)

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.01944 (12)	0.39092 (11)	0.14638 (10)	0.0189 (2)
O2	0.32915 (15)	0.18884 (13)	0.02664 (12)	0.0305 (3)
O3	0.24363 (14)	0.22511 (12)	0.50240 (11)	0.0263 (3)
N1	0.21495 (15)	0.34804 (13)	0.06151 (12)	0.0185 (3)

C1	0.21660 (18)	0.45088 (15)	0.18038 (15)	0.0171 (3)
C2	0.31312 (19)	0.28088 (16)	0.10736 (16)	0.0213 (3)
C3	0.4043 (2)	0.34509 (18)	0.27510 (16)	0.0218 (3)
H31	0.540 (2)	0.427 (2)	0.3151 (19)	0.030 (4)*
H32	0.392 (2)	0.257 (2)	0.3074 (19)	0.029 (4)*
C4	0.30327 (18)	0.42019 (16)	0.31682 (15)	0.0183 (3)
H4	0.388 (2)	0.519 (2)	0.4074 (19)	0.025 (4)*
C5	0.12821 (18)	0.30714 (15)	0.31940 (15)	0.0177 (3)
C6	0.10779 (19)	0.21344 (16)	0.40668 (15)	0.0191 (3)
C7	−0.0935 (2)	0.09516 (17)	0.36745 (17)	0.0216 (3)
H71	−0.093 (2)	0.0685 (19)	0.4518 (19)	0.025 (4)*
H72	−0.142 (2)	−0.004 (2)	0.282 (2)	0.032 (4)*
C8	−0.22872 (19)	0.15110 (16)	0.31966 (16)	0.0199 (3)
C9	−0.21885 (19)	0.20124 (17)	0.19147 (16)	0.0218 (3)
H91	−0.284 (2)	0.106 (2)	0.100 (2)	0.029 (4)*
H92	−0.283 (2)	0.261 (2)	0.1752 (19)	0.031 (4)*
C10	−0.02110 (19)	0.29910 (15)	0.22264 (15)	0.0178 (3)
C11	−0.4302 (2)	0.01507 (19)	0.2635 (2)	0.0295 (4)
H111	−0.444 (3)	−0.018 (2)	0.346 (2)	0.042 (5)*
H112	−0.464 (3)	−0.075 (2)	0.181 (2)	0.041 (5)*
H113	−0.520 (3)	0.049 (2)	0.229 (2)	0.038 (5)*
C12	−0.1750 (2)	0.28916 (18)	0.45332 (18)	0.0254 (3)
H121	−0.255 (3)	0.331 (2)	0.426 (2)	0.035 (5)*
H122	−0.040 (2)	0.377 (2)	0.4999 (19)	0.028 (4)*
H123	−0.195 (2)	0.252 (2)	0.531 (2)	0.036 (5)*
C13	0.3084 (2)	0.62117 (17)	0.19824 (17)	0.0217 (3)
H131	0.442 (2)	0.6640 (19)	0.2287 (17)	0.023 (4)*
H132	0.296 (2)	0.685 (2)	0.2756 (19)	0.030 (4)*
H133	0.250 (2)	0.632 (2)	0.106 (2)	0.030 (4)*
C14	0.10618 (19)	0.31569 (18)	−0.09504 (15)	0.0226 (3)
H14	0.052 (2)	0.3839 (19)	−0.0917 (18)	0.026 (4)*
C15	0.2334 (2)	0.3652 (2)	−0.16630 (18)	0.0307 (4)
H151	0.335 (3)	0.478 (2)	−0.108 (2)	0.040 (5)*
H152	0.161 (3)	0.352 (2)	−0.266 (2)	0.044 (5)*
H153	0.295 (3)	0.305 (2)	−0.166 (2)	0.036 (5)*
C16	−0.0585 (2)	0.1430 (2)	−0.17838 (18)	0.0301 (4)
H161	−0.012 (3)	0.069 (2)	−0.171 (2)	0.038 (5)*
H162	−0.133 (3)	0.124 (2)	−0.285 (2)	0.044 (5)*
H163	−0.143 (2)	0.122 (2)	−0.134 (2)	0.033 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0163 (5)	0.0238 (5)	0.0216 (5)	0.0117 (4)	0.0105 (4)	0.0135 (4)
O2	0.0337 (6)	0.0330 (6)	0.0332 (6)	0.0232 (5)	0.0188 (5)	0.0113 (5)
O3	0.0252 (5)	0.0326 (6)	0.0258 (6)	0.0171 (5)	0.0114 (5)	0.0180 (5)
N1	0.0183 (6)	0.0206 (6)	0.0170 (6)	0.0107 (5)	0.0083 (5)	0.0078 (5)
C1	0.0133 (6)	0.0194 (7)	0.0188 (7)	0.0083 (5)	0.0083 (5)	0.0087 (5)

C2	0.0181 (7)	0.0217 (7)	0.0262 (8)	0.0106 (6)	0.0123 (6)	0.0105 (6)
C3	0.0194 (7)	0.0277 (8)	0.0252 (8)	0.0151 (6)	0.0123 (6)	0.0149 (6)
C4	0.0159 (6)	0.0199 (7)	0.0179 (7)	0.0088 (6)	0.0074 (6)	0.0090 (6)
C5	0.0168 (6)	0.0183 (7)	0.0187 (7)	0.0092 (6)	0.0092 (6)	0.0082 (5)
C6	0.0222 (7)	0.0192 (7)	0.0195 (7)	0.0127 (6)	0.0112 (6)	0.0085 (6)
C7	0.0240 (7)	0.0194 (7)	0.0273 (8)	0.0122 (6)	0.0150 (6)	0.0138 (6)
C8	0.0177 (7)	0.0184 (7)	0.0249 (7)	0.0086 (6)	0.0118 (6)	0.0111 (6)
C9	0.0163 (7)	0.0239 (7)	0.0242 (8)	0.0099 (6)	0.0089 (6)	0.0113 (6)
C10	0.0197 (7)	0.0175 (6)	0.0183 (7)	0.0100 (6)	0.0106 (6)	0.0085 (5)
C11	0.0214 (8)	0.0246 (8)	0.0401 (10)	0.0086 (7)	0.0160 (7)	0.0156 (7)
C12	0.0266 (8)	0.0262 (8)	0.0302 (8)	0.0164 (7)	0.0168 (7)	0.0130 (7)
C13	0.0207 (7)	0.0201 (7)	0.0250 (8)	0.0104 (6)	0.0116 (6)	0.0110 (6)
C14	0.0196 (7)	0.0284 (8)	0.0170 (7)	0.0120 (6)	0.0075 (6)	0.0088 (6)
C15	0.0260 (8)	0.0403 (10)	0.0227 (8)	0.0138 (8)	0.0132 (7)	0.0140 (7)
C16	0.0196 (7)	0.0330 (9)	0.0244 (8)	0.0088 (7)	0.0084 (7)	0.0046 (7)

Geometric parameters (Å, º)

O1—C10	1.3599 (16)	C8—C9	1.5428 (19)
O1—C1	1.4780 (15)	C9—C10	1.4828 (18)
O2—C2	1.2226 (17)	C9—H91	0.984 (18)
O3—C6	1.2321 (17)	C9—H92	1.009 (18)
N1—C2	1.3642 (18)	C11—H111	1.02 (2)
N1—C1	1.4465 (17)	C11—H112	0.98 (2)
N1—C14	1.4754 (18)	C11—H113	1.001 (19)
C1—C13	1.5083 (19)	C12—H121	0.984 (19)
C1—C4	1.5475 (18)	C12—H122	1.001 (18)
C2—C3	1.511 (2)	C12—H123	1.003 (19)
C3—C4	1.5276 (19)	C13—H131	0.981 (16)
C3—H31	0.999 (18)	C13—H132	0.995 (18)
C3—H32	1.000 (17)	C13—H133	0.993 (18)
C4—C5	1.5062 (18)	C14—C16	1.524 (2)
C4—H4	0.981 (17)	C14—C15	1.524 (2)
C5—C10	1.3409 (19)	C14—H14	1.015 (17)
C5—C6	1.4486 (18)	C15—H151	0.99 (2)
C6—C7	1.5182 (19)	C15—H152	0.97 (2)
C7—C8	1.5397 (19)	C15—H153	0.991 (19)
C7—H71	0.972 (17)	C16—H161	1.012 (19)
C7—H72	1.001 (18)	C16—H162	1.01 (2)
C8—C11	1.5269 (19)	C16—H163	0.986 (19)
C8—C12	1.530 (2)		
C10—O1—C1	106.56 (9)	C10—C9—H91	107.3 (10)
C2—N1—C1	114.23 (11)	C8—C9—H91	109.1 (10)
C2—N1—C14	124.67 (11)	C10—C9—H92	110.5 (10)
C1—N1—C14	121.03 (11)	C8—C9—H92	110.1 (10)
N1—C1—O1	107.93 (10)	H91—C9—H92	108.8 (14)
N1—C1—C13	113.64 (11)	C5—C10—O1	114.25 (11)

O1—C1—C13	107.74 (10)	C5—C10—C9	126.89 (12)
N1—C1—C4	103.99 (10)	O1—C10—C9	118.86 (11)
O1—C1—C4	106.33 (10)	C8—C11—H111	110.5 (11)
C13—C1—C4	116.70 (11)	C8—C11—H112	109.8 (11)
O2—C2—N1	125.69 (13)	H111—C11—H112	109.6 (15)
O2—C2—C3	126.18 (13)	C8—C11—H113	110.7 (11)
N1—C2—C3	108.09 (11)	H111—C11—H113	107.4 (15)
C2—C3—C4	104.74 (11)	H112—C11—H113	108.8 (15)
C2—C3—H31	107.7 (10)	C8—C12—H121	111.5 (11)
C4—C3—H31	111.0 (10)	C8—C12—H122	112.0 (10)
C2—C3—H32	109.6 (10)	H121—C12—H122	109.2 (14)
C4—C3—H32	114.0 (10)	C8—C12—H123	110.1 (10)
H31—C3—H32	109.5 (13)	H121—C12—H123	106.2 (14)
C5—C4—C3	114.25 (11)	H122—C12—H123	107.7 (14)
C5—C4—C1	100.56 (10)	C1—C13—H131	108.0 (9)
C3—C4—C1	104.48 (11)	C1—C13—H132	110.3 (10)
C5—C4—H4	112.6 (9)	H131—C13—H132	109.6 (13)
C3—C4—H4	112.7 (9)	C1—C13—H133	112.8 (10)
C1—C4—H4	111.2 (9)	H131—C13—H133	109.5 (13)
C10—C5—C6	121.02 (12)	H132—C13—H133	106.7 (14)
C10—C5—C4	110.05 (11)	N1—C14—C16	109.81 (12)
C6—C5—C4	128.93 (12)	N1—C14—C15	111.05 (12)
O3—C6—C5	122.42 (12)	C16—C14—C15	114.05 (13)
O3—C6—C7	122.39 (12)	N1—C14—H14	105.4 (9)
C5—C6—C7	115.15 (11)	C16—C14—H14	107.3 (9)
C6—C7—C8	114.64 (11)	C15—C14—H14	108.9 (9)
C6—C7—H71	108.4 (10)	C14—C15—H151	109.7 (11)
C8—C7—H71	110.7 (9)	C14—C15—H152	109.3 (11)
C6—C7—H72	107.7 (10)	H151—C15—H152	108.4 (16)
C8—C7—H72	107.4 (10)	C14—C15—H153	110.6 (11)
H71—C7—H72	107.7 (14)	H151—C15—H153	106.4 (15)
C11—C8—C12	109.17 (12)	H152—C15—H153	112.3 (16)
C11—C8—C7	109.80 (11)	C14—C16—H161	110.5 (10)
C12—C8—C7	109.23 (12)	C14—C16—H162	108.6 (11)
C11—C8—C9	108.16 (12)	H161—C16—H162	112.9 (15)
C12—C8—C9	111.04 (11)	C14—C16—H163	110.2 (10)
C7—C8—C9	109.42 (11)	H161—C16—H163	107.2 (14)
C10—C9—C8	110.89 (11)	H162—C16—H163	107.4 (15)

C2—N1—C1—O1	−122.94 (11)	C1—C4—C5—C6	169.67 (13)
C14—N1—C1—O1	54.09 (15)	C10—C5—C6—O3	−175.76 (13)
C2—N1—C1—C13	117.63 (13)	C4—C5—C6—O3	5.3 (2)
C14—N1—C1—C13	−65.34 (15)	C10—C5—C6—C7	6.57 (19)
C2—N1—C1—C4	−10.29 (14)	C4—C5—C6—C7	−172.36 (13)
C14—N1—C1—C4	166.74 (11)	O3—C6—C7—C8	147.54 (13)
C10—O1—C1—N1	96.49 (11)	C5—C6—C7—C8	−34.79 (17)
C10—O1—C1—C13	−140.41 (11)	C6—C7—C8—C11	172.47 (12)
C10—O1—C1—C4	−14.58 (13)	C6—C7—C8—C12	−67.85 (15)

C1—N1—C2—O2	179.05 (13)	C6—C7—C8—C9	53.91 (16)
C14—N1—C2—O2	2.1 (2)	C11—C8—C9—C10	−163.97 (12)
C1—N1—C2—C3	−3.13 (15)	C12—C8—C9—C10	76.27 (15)
C14—N1—C2—C3	179.97 (12)	C7—C8—C9—C10	−44.39 (15)
O2—C2—C3—C4	−166.83 (14)	C6—C5—C10—O1	−178.50 (11)
N1—C2—C3—C4	15.37 (14)	C4—C5—C10—O1	0.61 (16)
C2—C3—C4—C5	88.10 (13)	C6—C5—C10—C9	0.7 (2)
C2—C3—C4—C1	−20.80 (14)	C4—C5—C10—C9	179.80 (13)
N1—C1—C4—C5	−99.69 (11)	C1—O1—C10—C5	9.08 (15)
O1—C1—C4—C5	14.11 (13)	C1—O1—C10—C9	−170.17 (12)
C13—C1—C4—C5	134.30 (12)	C8—C9—C10—C5	19.6 (2)
N1—C1—C4—C3	18.97 (13)	C8—C9—C10—O1	−161.21 (11)
O1—C1—C4—C3	132.78 (11)	C2—N1—C14—C16	65.27 (17)
C13—C1—C4—C3	−107.04 (13)	C1—N1—C14—C16	−111.43 (14)
C3—C4—C5—C10	−120.63 (13)	C2—N1—C14—C15	−61.79 (18)
C1—C4—C5—C10	−9.35 (14)	C1—N1—C14—C15	121.51 (14)
C3—C4—C5—C6	58.39 (19)		

Hydrogen-bond geometry (Å, º)

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)