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(1S^*,2S^*,4S^*)-3,3-Difluoro-2,4-dihydroxy-5,5-dimethylcyclooct-5(*Z*)-en-1-yl *N*,*N*-diethylcarbamate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.013 Å
R factor = 0.082
wR factor = 0.230
Data-to-parameter ratio = 6.9

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

The structure of the title compound, $C_{15}H_{25}F_2NO_4$, is reported and reveals a pseudorotational relationship between the ring conformation of this compound and that of an isomeric by-product reported in the following paper.

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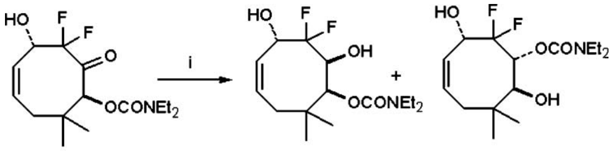
Comment

Conformational equilibria in eight-membered carbocycles occur *via* two main processes, pseudorotation and ring inversion. The latter exchanges substituent groups between equatorial and axial environments in a pseudo-enantiomeric relationship. Ring inversion is usually the more energetically demanding process; barriers to inversion exchange of 7.3–8.5 kcal mol^{-1} have been reported, with smaller barriers (*ca* 5 kcal mol^{-1}) (Servis & Noe, 1973) for the pseudorotation. [For early attempts to apply variable-temperature NMR to these phenomena, see Anderson *et al.* (1969) and St Jacques *et al.* (1966).] Recent work from our group has attempted to define these processes for a trio of difluorinated cyclooctenyl systems (Fawcett, Griffith *et al.*, 2005). We were interested in observing a pseudorotational relationship between the ring conformations in the pair of reduction products (1) and (2), obtained upon treatment of a precursor ketone with sodium borohydride.



(1) (2)

Product (1) (the major product) arises from the opposite sense of hydride attack, with the *N*,*N*-diethylcarbamoyl group retaining its original location (Fig. 1). Product (2), reported in the following paper (Fawcett, Percy *et al.*, 2005), arises from reagent attack on the ring face which bears the hydroxyl group, followed by migration of the *N*,*N*-diethylcarbamoyl group on to the newly formed hydroxyl group (Balnaves *et al.*, 1999). A comparison of the two molecules is shown in Fig. 2. O—H···O hydrogen bonding links molecules of (1) into chains along the *b* axis (Table 1).

Experimental

The precursor ketone was prepared as described in the literature (Fawcett, Griffith *et al.*, 2005). Sodium borohydride (1.8 mmol, 70 mg) was added in five portions to a cold (273 K) solution of the ketone (1.8 mmol, 0.59 g) in ethanol (10 ml). After completion of the addition, the reaction mixture was allowed to warm to room

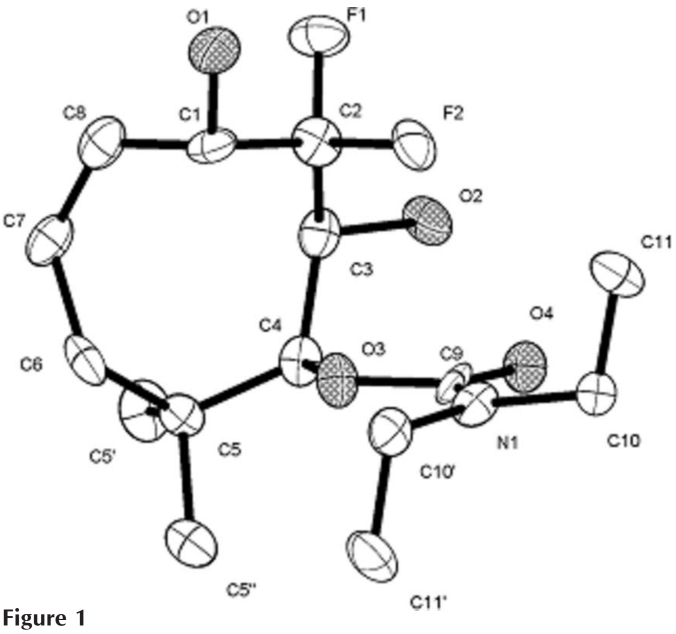


Figure 1
The molecular structure of (1), showing the atom-numbering scheme and 50% displacement ellipsoids. H atoms have been omitted for clarity.

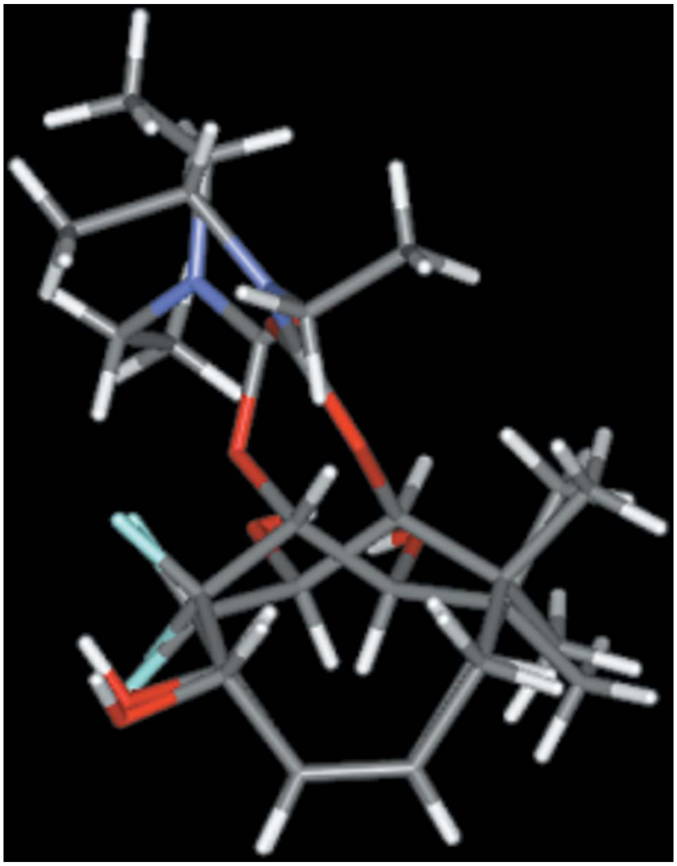


Figure 2
An overlay showing the relationship between the structures of compounds (1) and (2).

temperature, stirred for 2 h at this temperature and poured over a mixture of ice and water (25 ml). HCl (10 ml of a 1 *N* solution) was added cautiously and the mixture was extracted with diethyl ether (3 × 25 ml). The combined organic extracts were dried ($MgSO_4$), filtered and concentrated under reduced pressure to leave a white solid (0.51 g). Purification by column chromatography (40% ethyl acetate in light petroleum) afforded the desired diol (1) as a white solid (0.43 g, 72%). R_F (40% ethyl acetate in light petroleum) 0.29; m.p. 388–389 K (found: C 56.17, H 7.71, N 4.29%; $C_{15}H_{25}F_2NO_4$ requires: C 56.06, H 7.84, N, 4.36%); ν_{max}(KBr)/cm^{-1} 3460 (*s br*, O—H), 3356 (*s br*, O—H), 2977 (*m*, =C—H), 2877 (*m*, C—H), 1671 (*s*, C=O); ^{1}H NMR (250 MHz, $CDCl_3$): δ 5.83 (1H, *dd*, J = 18.5, 9.0 Hz, H-5), 5.53 (1H, *t*, J = 9.0, 9.0 Hz, H-4), 4.84 (1H, *ddd*, $^3J_{HF}$ = 21.3, 8.0, 4.1 Hz, H-3), 4.48 (1H, *d*, J = 5.7 Hz, H-8), 4.18–4.04 (1H, *m*, H-1), 3.42–3.10 [5H, *m*, —OH and —N(CH_2CH_3)$_2$], 2.45 (1H, *dd*, J_{gem} = 13.8, J = 8.5 Hz, H-6a), 2.17 (1H, *br s*, —OH), 1.77 (1H, *dd*, J_{gem} = 13.8 Hz, J = 8.3 Hz, H-6b), 1.18–0.93 [12H, *m*, —N(CH_2CH_3)$_2$ and 2 × —CH_3]; ^{13}C NMR (63 MHz, $CDCl_3$): δ 157.4, 131.4, 131.3 (*d*, $^3J_{CF}$ = 6.6 Hz), 122.8 (*dd*, $^1J_{CF}$ = 253.1, 246.9 Hz), 87.4 (*d*, $^3J_{CF}$ = 9.2 Hz), 70.8 (*dd*, $^2J_{CF}$ = 23.9, 19.8 Hz), 68.4 (*dd*, $^2J_{CF}$ = 23.9, 20.9 Hz), 42.8, 42.0, 39.8, 34.9, 30.4, 24.3, 14.5, 13.4; ^{19}F NMR (235 MHz, $CDCl_3$): δ −118.5 (1F, *dddd*, J_{gem} = 241.5, $^3J_{HF}$ = 21.2, 10.6, $^4J_{FH}$ = 6.6 Hz), −122.1 (1F, *dd*, J_{gem} = 241.5, $^3J_{FH}$ = 16.6 Hz); [HRMS (FAB, [*M*+H]$^+$) Found: 322.18293, calculated for $C_{15}H_{26}F_2NO_4$: 322.18299]; *m*/*z* (FAB): 322 (100%, [*M*+H]$^+$). An analytical sample was recrystallized by vapour diffusion (ethyl acetate/light petroleum) to afford colourless needles.

Crystal data

$C_{15}H_{25}F_2NO_4$
M_r = 321.36
Monoclinic, *Pn*
a = 7.9651 (14) Å
b = 6.4632 (12) Å
c = 15.445 (3) Å
β = 90.136 (3)°
V = 795.1 (2) Å^3
Z = 2
D_x = 1.342 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 2104 reflections
θ = 2.6–24.7°
μ = 0.11 mm^{-1}
T = 150 (2) K
Block cut from needle, colourless
0.24 × 0.18 × 0.12 mm

Data collection

Bruker APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
5283 measured reflections
1403 independent reflections
1332 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 25.0°
$h = -9 \rightarrow 9$
$k = -7 \rightarrow 7$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.082
$wR(F^2)$ = 0.230
S = 1.13
1403 reflections
203 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1065P)^2 + 3.2641P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$O2^{i}	0.84	2.18	2.816 (10)	133

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

H atoms were positioned geometrically, with C—H = 0.95–1.00 Å and O—H = 0.84 Å, and treated as riding, with U_{iso}(H) = 1.2 or 1.5 (methyl and OH) times U_{eq} of the parent atom.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine

o3320 Fawcett *et al.* • $C_{15}H_{25}F_2NO_4$ *Acta Cryst.* (2005). E**61**, o3319–o3321

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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supporting information

Acta Cryst. (2005). E**61**, o3319–o3321 [doi:10.1107/S1600536805024839]

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S1. Comment

Conformational equilibria in eight-membered carbocycles occur *via* two main processes, pseudorotation and ring inversion. The latter exchanges substituent groups between equatorial and axial environments in a pseudo-enantiomeric relationship. Ring inversion is usually the more energetically demanding process; barriers to inversion exchange of 7.3–8.5 kcal mol^{-1} have been reported, with smaller barriers (*ca* 5 kcal mol^{-1}) (Servis & Noe, 1973) for the pseudorotation. [For early attempts to apply variable-temperature NMR to these phenomena, see Anderson *et al.* (1969) and St Jacques *et al.* (1966).] Recent work from our group has attempted to define these processes for a trio of difluorinated cyclooctenyl systems (Griffith *et al.*, 2005). We were interested to observe a pseudorotational relationship between the ring conformations in the pair of reduction products (1) and (2), obtained upon treatment of a precursor ketone with sodium borohydride.

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S2. Experimental

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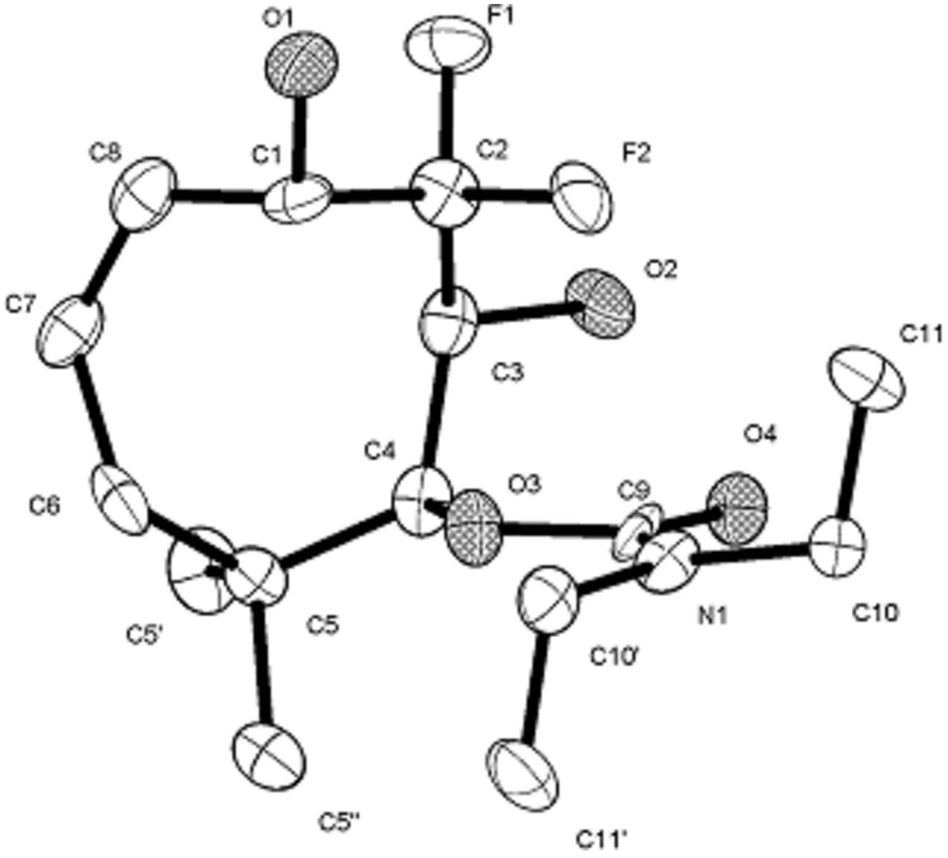

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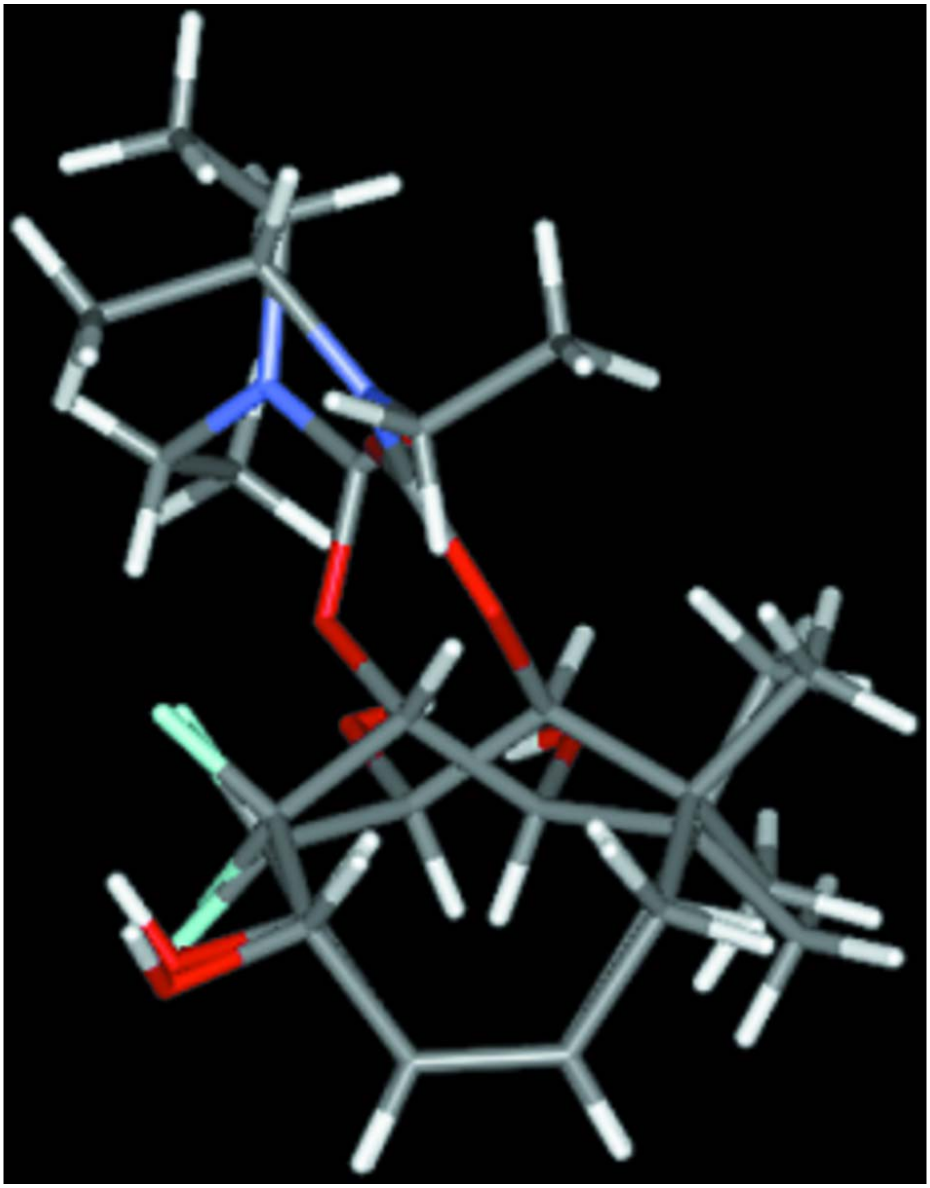

Figure 2

An overlay showing the relationship between the structures of compounds (1) and (2).

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Hall symbol: P -2yac	Cell parameters from 2104 reflections
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$c = 15.445$ (3) Å	$T = 150$ K
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Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
5283 measured reflections
1403 independent reflections
1332 reflections with $I > 2\sigma(I)$
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$\theta_{\text{max}} = 25.0°$, $\theta_{\text{min}} = 2.6°$
$h = -9\rightarrow9$
$k = -7\rightarrow7$
$l = -18\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.082$
$wR(F^2) = 0.230$
$S = 1.13$
1403 reflections
203 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.1065P)^2 + 3.2641P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}} = 0.003$
$\Delta\rho_{\text{max}} = 0.37$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.45$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	$U_{\text{iso}}*/U_{\text{eq}}$
F1	0.6752 (7)	0.0571 (9)	0.2519 (4)	0.0344 (14)
F2	0.6142 (6)	0.1799 (8)	0.1254 (4)	0.0293 (12)
O1	0.7967 (8)	0.4476 (10)	0.2290 (4)	0.0285 (15)
H1	0.7144	0.4897	0.1998	0.043*
O2	0.6712 (8)	−0.2340 (10)	0.1225 (4)	0.0291 (15)
H2	0.7050	−0.3474	0.1024	0.044*
O3	0.8718 (8)	0.0997 (9)	0.0008 (4)	0.0249 (14)
O4	0.6287 (8)	−0.0565 (9)	−0.0385 (4)	0.0253 (14)
N1	0.6973 (9)	0.2608 (11)	−0.0879 (5)	0.0228 (16)
C1	0.8721 (11)	0.2781 (14)	0.1864 (6)	0.0243 (19)
H1A	0.9126	0.3248	0.1284	0.029*
C2	0.7432 (12)	0.1048 (15)	0.1735 (6)	0.026 (2)
C3	0.8118 (11)	−0.0981 (14)	0.1351 (6)	0.025 (2)
H3	0.8849	−0.1626	0.1805	0.030*
C4	0.9179 (11)	−0.0828 (13)	0.0516 (6)	0.0227 (19)
H4	0.8865	−0.2054	0.0155	0.027*
C5	1.1090 (11)	−0.0844 (14)	0.0577 (6)	0.0218 (19)

C5′	1.1659 (12)	−0.2867 (15)	0.1012 (7)	0.033 (2)
H5′1	1.1501	−0.2759	0.1640	0.050*
H5′2	1.2848	−0.3109	0.0887	0.050*
H5′3	1.0990	−0.4022	0.0788	0.050*
C5"	1.1791 (12)	−0.0842 (16)	−0.0346 (7)	0.032 (2)
H5"1	1.1315	−0.2007	−0.0670	0.047*
H5"2	1.3016	−0.0975	−0.0324	0.047*
H5"3	1.1490	0.0458	−0.0634	0.047*
C6	1.1867 (11)	0.1057 (15)	0.1063 (7)	0.029 (2)
H6A	1.3097	0.1034	0.0973	0.034*
H6B	1.1433	0.2334	0.0788	0.034*
C7	1.1546 (12)	0.1196 (14)	0.2009 (6)	0.026 (2)
H7	1.2371	0.0628	0.2386	0.032*
C8	1.0180 (12)	0.2062 (14)	0.2379 (6)	0.029 (2)
H8	1.0155	0.2214	0.2991	0.035*
C9	0.7223 (11)	0.0905 (15)	−0.0430 (5)	0.0227 (19)
C10	0.5307 (11)	0.2911 (17)	−0.1285 (6)	0.028 (2)
H10C	0.4777	0.1548	−0.1391	0.034*
H10D	0.5444	0.3612	−0.1850	0.034*
C10′	0.8155 (11)	0.4301 (14)	−0.0983 (6)	0.024 (2)
H10A	0.8889	0.4363	−0.0465	0.029*
H10B	0.7523	0.5617	−0.1014	0.029*
C11	0.4178 (12)	0.4201 (16)	−0.0708 (6)	0.030 (2)
H11D	0.4013	0.3486	−0.0156	0.045*
H11E	0.3090	0.4402	−0.0994	0.045*
H11F	0.4703	0.5550	−0.0603	0.045*
C11′	0.9211 (13)	0.411 (2)	−0.1756 (8)	0.041 (3)
H11A	0.9914	0.2876	−0.1708	0.061*
H11B	0.9927	0.5341	−0.1807	0.061*
H11C	0.8495	0.4002	−0.2271	0.061*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.044 (3)	0.033 (3)	0.027 (3)	−0.008 (3)	0.013 (2)	0.000 (2)
F2	0.018 (2)	0.031 (3)	0.039 (3)	0.006 (2)	−0.001 (2)	−0.002 (3)
O1	0.033 (4)	0.024 (3)	0.029 (3)	0.010 (3)	−0.002 (3)	−0.006 (3)
O2	0.026 (3)	0.021 (3)	0.040 (4)	−0.010 (3)	0.008 (3)	−0.007 (3)
O3	0.022 (3)	0.017 (3)	0.036 (3)	0.002 (3)	−0.007 (3)	0.001 (3)
O4	0.026 (3)	0.021 (3)	0.030 (3)	−0.002 (3)	−0.003 (3)	0.001 (3)
N1	0.030 (4)	0.015 (4)	0.024 (4)	0.006 (3)	−0.005 (3)	−0.003 (3)
C1	0.033 (5)	0.022 (4)	0.018 (4)	−0.004 (4)	0.006 (4)	−0.006 (4)
C2	0.028 (5)	0.019 (5)	0.030 (5)	0.005 (4)	−0.001 (4)	0.005 (4)
C3	0.025 (5)	0.022 (5)	0.029 (5)	0.000 (4)	−0.007 (4)	−0.001 (4)
C4	0.024 (4)	0.015 (4)	0.029 (5)	0.000 (3)	−0.004 (4)	0.003 (4)
C5	0.021 (4)	0.021 (4)	0.023 (4)	0.002 (4)	0.001 (4)	0.006 (4)
C5′	0.032 (5)	0.026 (5)	0.042 (6)	0.006 (4)	−0.007 (4)	0.013 (4)
C5"	0.025 (5)	0.033 (5)	0.036 (5)	0.000 (4)	0.005 (4)	−0.001 (4)

C6	0.014 (4)	0.029 (5)	0.044 (6)	−0.002 (4)	0.005 (4)	−0.002 (4)
C7	0.037 (5)	0.021 (4)	0.021 (5)	0.001 (4)	−0.009 (4)	0.003 (4)
C8	0.037 (5)	0.021 (5)	0.028 (5)	−0.007 (4)	−0.006 (4)	−0.002 (4)
C9	0.025 (4)	0.032 (5)	0.011 (4)	−0.004 (4)	−0.007 (3)	−0.004 (4)
C10	0.021 (4)	0.040 (6)	0.024 (4)	−0.002 (4)	−0.001 (4)	−0.006 (4)
C10′	0.024 (5)	0.023 (5)	0.027 (5)	0.008 (4)	−0.005 (4)	0.002 (4)
C11	0.025 (5)	0.033 (5)	0.032 (5)	0.006 (4)	0.008 (4)	0.009 (4)
C11′	0.023 (5)	0.051 (7)	0.047 (6)	−0.007 (5)	0.010 (5)	−0.009 (5)

Geometric parameters (Å, º)

F1—C2	1.363 (11)	C5′—H5′2	0.980
F2—C2	1.356 (11)	C5′—H5′3	0.980
O1—C1	1.412 (10)	C5"—H5"1	0.980
O1—H1	0.840	C5"—H5"2	0.980
O2—C3	1.437 (11)	C5"—H5"3	0.980
O2—H2	0.840	C6—C7	1.487 (13)
O3—C9	1.369 (11)	C6—H6A	0.990
O3—C4	1.463 (10)	C6—H6B	0.990
O4—C9	1.210 (11)	C7—C8	1.351 (14)
N1—C9	1.315 (12)	C7—H7	0.950
N1—C10′	1.453 (12)	C8—H8	0.950
N1—C10	1.480 (11)	C10—C11	1.517 (13)
C1—C8	1.482 (13)	C10—H10C	0.990
C1—C2	1.533 (13)	C10—H10D	0.990
C1—H1A	1.000	C10′—C11′	1.468 (14)
C2—C3	1.540 (12)	C10′—H10A	0.990
C3—C4	1.548 (13)	C10′—H10B	0.990
C3—H3	1.000	C11—H11D	0.980
C4—C5	1.524 (12)	C11—H11E	0.980
C4—H4	1.000	C11—H11F	0.980
C5—C5"	1.532 (13)	C11′—H11A	0.980
C5—C5′	1.538 (12)	C11′—H11B	0.980
C5—C6	1.567 (13)	C11′—H11C	0.980
C5′—H5′1	0.980		
C1—O1—H1	109.5	H5"1—C5"—H5"2	109.5
C3—O2—H2	109.5	C5—C5"—H5"3	109.5
C9—O3—C4	116.6 (7)	H5"1—C5"—H5"3	109.5
C9—N1—C10′	126.2 (7)	H5"2—C5"—H5"3	109.5
C9—N1—C10	117.9 (8)	C7—C6—C5	116.7 (8)
C10′—N1—C10	115.7 (7)	C7—C6—H6A	108.1
O1—C1—C8	109.1 (7)	C5—C6—H6A	108.1
O1—C1—C2	110.0 (7)	C7—C6—H6B	108.1
C8—C1—C2	111.4 (8)	C5—C6—H6B	108.1
O1—C1—H1A	108.8	H6A—C6—H6B	107.3
C8—C1—H1A	108.8	C8—C7—C6	125.5 (8)
C2—C1—H1A	108.8	C8—C7—H7	117.2

F2—C2—F1	105.4 (7)	C6—C7—H7	117.2
F2—C2—C1	108.4 (7)	C7—C8—C1	122.3 (8)
F1—C2—C1	108.5 (7)	C7—C8—H8	118.9
F2—C2—C3	111.3 (7)	C1—C8—H8	118.9
F1—C2—C3	106.9 (7)	O4—C9—N1	126.5 (8)
C1—C2—C3	115.7 (7)	O4—C9—O3	122.7 (8)
O2—C3—C2	107.2 (7)	N1—C9—O3	110.7 (8)
O2—C3—C4	110.6 (7)	N1—C10—C11	110.8 (8)
C2—C3—C4	117.5 (8)	N1—C10—H10C	109.5
O2—C3—H3	107.0	C11—C10—H10C	109.5
C2—C3—H3	107.0	N1—C10—H10D	109.5
C4—C3—H3	107.0	C11—C10—H10D	109.5
O3—C4—C5	106.7 (7)	H10C—C10—H10D	108.1
O3—C4—C3	111.2 (7)	N1—C10′—C11′	113.6 (8)
C5—C4—C3	119.7 (7)	N1—C10′—H10A	108.8
O3—C4—H4	106.1	C11′—C10′—H10A	108.8
C5—C4—H4	106.1	N1—C10′—H10B	108.8
C3—C4—H4	106.1	C11′—C10′—H10B	108.8
C4—C5—C5"	107.9 (7)	H10A—C10′—H10B	107.7
C4—C5—C5′	109.0 (7)	C10—C11—H11D	109.5
C5"—C5—C5′	107.4 (8)	C10—C11—H11E	109.5
C4—C5—C6	114.7 (7)	H11D—C11—H11E	109.5
C5"—C5—C6	107.5 (7)	C10—C11—H11F	109.5
C5′—C5—C6	110.0 (8)	H11D—C11—H11F	109.5
C5—C5′—H5′1	109.5	H11E—C11—H11F	109.5
C5—C5′—H5′2	109.5	C10′—C11′—H11A	109.5
H5′1—C5′—H5′2	109.5	C10′—C11′—H11B	109.5
C5—C5′—H5′3	109.5	H11A—C11′—H11B	109.5
H5′1—C5′—H5′3	109.5	C10′—C11′—H11C	109.5
H5′2—C5′—H5′3	109.5	H11A—C11′—H11C	109.5
C5—C5"—H5"1	109.5	H11B—C11′—H11C	109.5
C5—C5"—H5"2	109.5		

O1—C1—C2—F2	59.4 (9)	C3—C4—C5—C5′	59.3 (11)
C8—C1—C2—F2	−179.4 (7)	O3—C4—C5—C6	62.8 (9)
O1—C1—C2—F1	−54.5 (9)	C3—C4—C5—C6	−64.5 (11)
C8—C1—C2—F1	66.6 (9)	C4—C5—C6—C7	68.2 (10)
O1—C1—C2—C3	−174.7 (7)	C5"—C5—C6—C7	−171.7 (8)
C8—C1—C2—C3	−53.6 (10)	C5′—C5—C6—C7	−55.0 (10)
F2—C2—C3—O2	−51.6 (9)	C5—C6—C7—C8	−86.0 (12)
F1—C2—C3—O2	63.0 (9)	C6—C7—C8—C1	7.2 (15)
C1—C2—C3—O2	−176.0 (7)	O1—C1—C8—C7	−149.3 (9)
F2—C2—C3—C4	73.6 (10)	C2—C1—C8—C7	89.1 (10)
F1—C2—C3—C4	−171.7 (7)	C10′—N1—C9—O4	−175.1 (9)
C1—C2—C3—C4	−50.8 (11)	C10—N1—C9—O4	8.3 (13)
C9—O3—C4—C5	153.8 (7)	C10′—N1—C9—O3	5.7 (12)
C9—O3—C4—C3	−74.1 (9)	C10—N1—C9—O3	−170.9 (7)
O2—C3—C4—O3	96.4 (8)	C4—O3—C9—O4	2.5 (12)

C2—C3—C4—O3	−27.1 (11)	C4—O3—C9—N1	−178.3 (7)
O2—C3—C4—C5	−138.4 (8)	C9—N1—C10—C11	95.1 (10)
C2—C3—C4—C5	98.1 (10)	C10′—N1—C10—C11	−81.9 (10)
O3—C4—C5—C5"	−57.0 (9)	C9—N1—C10′—C11′	92.9 (11)
C3—C4—C5—C5"	175.8 (8)	C10—N1—C10′—C11′	−90.4 (10)
O3—C4—C5—C5′	−173.4 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.84	2.18	2.816 (10)	133

Symmetry code: (i) *x*, *y*+1, *z*.