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(1*R**,3*S**,8*S**)-2,2-Difluoro-3,8-dihydroxy-5,5-dimethylcyclooct-4(*Z*)-en-1-yl *N*,*N*-diethylcarbamate

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Key indicators

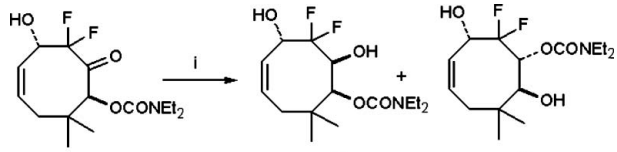
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.095
Data-to-parameter ratio = 13.7

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 presented. Comparison of this minor product with the isomeric major product of the synthesis is made in the previous paper.

Comment

The pseudorotational relationship between the ring conformations of the title compound, (2), and diol (1), which was presented in the previous paper (Fawcett *et al.*, 2005), are discussed in the *Comment* of that paper.



(1) (2)

Hydrogen bonding (Table 1) links molecules of (2) into sheets perpendicular to the *c* axis.

Experimental

Compound (2) was obtained as the minor product during the preparation of diol (1), as described in the previous paper (Fawcett *et al.*, 2005). A sample was recrystallized by vapour diffusion (ethyl acetate/light petroleum) to afford colourless crystals.

Crystal data

$C_{15}H_{25}F_2NO_4$
M_r = 321.36
Monoclinic, $P2_1/c$
a = 20.062 (14) Å
b = 6.433 (4) Å
c = 12.424 (9) Å
β = 97.346 (12)°
V = 1590.4 (19) Å^3
Z = 4
D_x = 1.342 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3558 reflections
θ = 3.1–28.1°
μ = 0.11 mm^{-1}
T = 150 (2) K
Block, colourless
0.28 × 0.22 × 0.15 mm

Data collection

Bruker APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
10968 measured reflections
2805 independent reflections
2413 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 25.0°
$h = -23 \rightarrow 23$
$k = -7 \rightarrow 7$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.095
S = 1.05
2805 reflections
205 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.1172P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

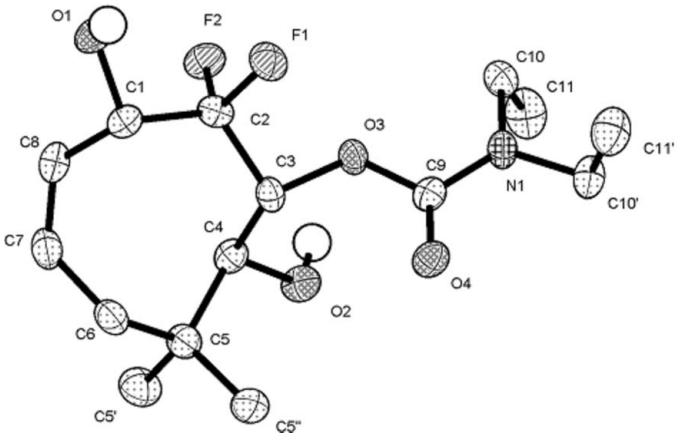


Figure 1
The molecular structure of (2), showing the atom-numbering scheme and 50% displacement ellipsoids. H atoms have been omitted.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O4^{i}	0.84	1.92	2.7598 (19)	173
O2—H2···O1^{ii}	0.84	2.01	2.827 (2)	163

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

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

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