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Ethyl 2-(1,2,3,4-tetrahydrospiro[carbazole-3,2′-[1,3]dioxolan]-9-yl)acetate

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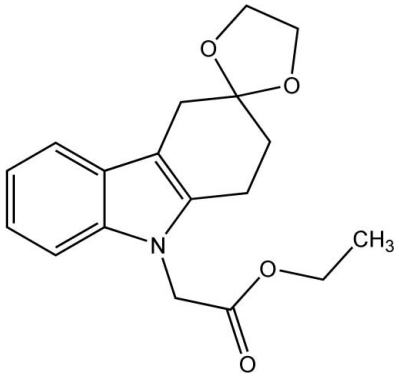
Key indicators: single-crystal X-ray study; T = 180 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.112; data-to-parameter ratio = 18.5.

In the title compound, $C_{18}H_{21}NO_4$, the hydrogenated six-membered ring of the carbazole unit adopts a half-chair conformation. The dioxolane ring and ethylacetate substituent point to opposite sides of the carbazole plane. The ethylacetate substituent adopts an essentially fully extended conformation, and its mean plane forms a dihedral angle of 83.8 (1)° with respect to the carbazole mean plane. The molecules are arranged into stacks in which the carbazole planes form a dihedral angle of 4.4 (1)° and have an approximate interplanar separation of 3.6 Å.

Related literature

For background literature and synthesis details, see: Ulven & Kostenis (2005, 2006). For a related structure, see: Bjerrum *et al.* (2009).



Experimental

Crystal data

$C_{18}H_{21}NO_4$
M_r = 315.36
Monoclinic, $P2_1/c$
a = 10.5533 (4) Å
b = 17.3773 (6) Å
c = 8.9637 (3) Å
β = 105.629 (1)°
V = 1583.05 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 180 K
0.50 × 0.50 × 0.10 mm

Data collection

Bruker-Nonius X8 APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.847, T_{max} = 0.991
25055 measured reflections
3851 independent reflections
3174 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.112
S = 1.04
3851 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We are grateful to the Danish Natural Sciences Research Council and the Carlsberg Foundation for provision of the X-ray equipment.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2485).

References

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supplementary materials

Acta Cryst. (2009). E**65**, o685 [**doi:10.1107/S160053680900748X**]

Ethyl 2-(1,2,3,4-tetrahydrospiro[carbazole-3,2'-[1,3]dioxolan]-9-yl)acetate

P. M. G. Löffler, T. Ulven and A. D. Bond

Comment

The title compound is useful as an intermediate in the synthesis of antagonists of the prostaglandin D_2 receptor CRTH2 (DP_2) (Ulven & Kostenis, 2006).

Experimental

The compound was synthesized as described in Ulven & Kostenis (2005).

Refinement

H atoms bound to C atoms were placed in idealized positions with C—H = 0.95–0.99 Å and refined as riding with U_{iso}(H) = 1.2 or 1.5U_{eq}(C). The methyl group was allowed to rotate about its local threefold axis.

Figures

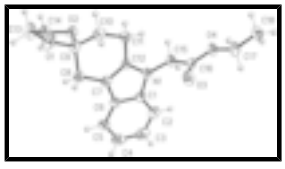

Fig. 1. Molecular structure of the title compound with displacement ellipsoids shown at 50% probability for non-H atoms.

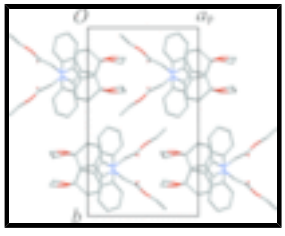

Fig. 2. Packing diagram viewed along the *c* axis, showing stacked carbazole units. H atoms are omitted.

Ethyl 2-(1,2,3,4-tetrahydrospiro[carbazole-3,2'-[1,3]dioxolan]-9-yl)acetate

Crystal data

$C_{18}H_{21}NO_4$	F_{000} = 672
M_r = 315.36	D_x = 1.323 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 7203 reflections
a = 10.5533 (4) Å	θ = 2.0–28.2°
b = 17.3773 (6) Å	μ = 0.09 mm^{-1}

c = 8.9637 (3) Å	T = 180 K
β = 105.629 (1)º	Plate, brown
V = 1583.05 (10) Å^3	0.50 × 0.50 × 0.10 mm
Z = 4	

Data collection

Bruker-Nonius X8 APEXII CCD diffractometer	3851 independent reflections
Radiation source: fine-focus sealed tube	3174 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025
T = 180 K	θ_{max} = 28.3º
thin–slice ω and φ scans	θ_{min} = 3.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −13→13
T_{min} = 0.847, T_{max} = 0.991	k = −23→21
25055 measured reflections	l = −11→11

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H-atom parameters constrained
$wR(F^2)$ = 0.112	$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.4296P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
3851 reflections	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
208 parameters	$\Delta\rho_{min}$ = −0.25 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 $F^2 > \sigma(F^2)$ 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	1.21234 (9)	0.15818 (7)	1.12008 (10)	0.0460 (3)

O2	1.17774 (8)	0.16828 (5)	0.85930 (9)	0.0310 (2)
O3	0.54620 (9)	0.18009 (6)	0.72948 (11)	0.0431 (2)
O4	0.48419 (8)	0.12028 (5)	0.50032 (10)	0.0319 (2)
N1	0.76683 (9)	0.24098 (6)	0.66065 (11)	0.0269 (2)
C1	0.76918 (11)	0.32014 (7)	0.67550 (13)	0.0271 (2)
C2	0.68683 (13)	0.37599 (8)	0.58858 (16)	0.0375 (3)
H2A	0.6137	0.3624	0.5048	0.045*
C3	0.71574 (15)	0.45191 (8)	0.62915 (18)	0.0459 (3)
H3A	0.6611	0.4912	0.5719	0.055*
C4	0.82262 (15)	0.47257 (8)	0.75148 (18)	0.0428 (3)
H4A	0.8399	0.5254	0.7756	0.051*
C5	0.90393 (13)	0.41716 (7)	0.83833 (15)	0.0339 (3)
H5A	0.9766	0.4316	0.9219	0.041*
C6	0.87768 (11)	0.33951 (6)	0.80135 (13)	0.0263 (2)
C7	0.93961 (10)	0.26886 (6)	0.86264 (13)	0.0256 (2)
C8	1.06186 (12)	0.25587 (8)	0.99027 (14)	0.0324 (3)
H8A	1.0427	0.2630	1.0916	0.039*
H8B	1.1296	0.2940	0.9825	0.039*
C9	1.11382 (11)	0.17469 (7)	0.98003 (13)	0.0305 (3)
C10	1.00563 (12)	0.11489 (7)	0.95185 (14)	0.0334 (3)
H10A	0.9625	0.1170	1.0371	0.040*
H10B	1.0448	0.0631	0.9526	0.040*
C11	0.90183 (12)	0.12724 (7)	0.79701 (14)	0.0311 (3)
H11A	0.9359	0.1086	0.7109	0.037*
H11B	0.8213	0.0977	0.7958	0.037*
C12	0.86989 (10)	0.21090 (6)	0.77637 (13)	0.0255 (2)
C13	1.33628 (14)	0.16069 (11)	1.09153 (17)	0.0486 (4)
H13A	1.3774	0.1090	1.1043	0.058*
H13B	1.3950	0.1968	1.1636	0.058*
C14	1.31189 (12)	0.18790 (9)	0.92705 (15)	0.0398 (3)
H14A	1.3257	0.2441	0.9228	0.048*
H14B	1.3704	0.1612	0.8740	0.048*
C15	0.67326 (11)	0.19717 (7)	0.54503 (13)	0.0290 (2)
H15A	0.7192	0.1537	0.5107	0.035*
H15B	0.6361	0.2304	0.4540	0.035*
C16	0.56219 (11)	0.16596 (6)	0.60498 (13)	0.0264 (2)
C17	0.37837 (12)	0.08256 (8)	0.54942 (15)	0.0373 (3)
H17A	0.3230	0.1215	0.5826	0.045*
H17B	0.4156	0.0477	0.6377	0.045*
C18	0.29786 (13)	0.03789 (8)	0.41509 (16)	0.0407 (3)
H18A	0.2259	0.0120	0.4450	0.061*
H18B	0.3534	−0.0006	0.3835	0.061*
H18C	0.2613	0.0729	0.3285	0.061*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0307 (5)	0.0794 (7)	0.0284 (5)	0.0149 (5)	0.0090 (4)	0.0144 (4)

O2	0.0270 (4)	0.0428 (5)	0.0261 (4)	0.0009 (3)	0.0119 (3)	0.0002 (3)
O3	0.0339 (5)	0.0658 (6)	0.0338 (5)	−0.0159 (4)	0.0163 (4)	−0.0132 (4)
O4	0.0255 (4)	0.0388 (5)	0.0317 (4)	−0.0088 (3)	0.0081 (3)	−0.0039 (3)
N1	0.0220 (5)	0.0306 (5)	0.0275 (5)	−0.0043 (4)	0.0057 (4)	−0.0010 (4)
C1	0.0241 (5)	0.0308 (6)	0.0295 (5)	−0.0023 (4)	0.0126 (4)	0.0011 (4)
C2	0.0323 (6)	0.0439 (7)	0.0371 (6)	0.0055 (5)	0.0106 (5)	0.0077 (5)
C3	0.0507 (8)	0.0371 (7)	0.0540 (8)	0.0124 (6)	0.0211 (7)	0.0119 (6)
C4	0.0532 (8)	0.0284 (6)	0.0553 (8)	0.0005 (6)	0.0294 (7)	−0.0007 (6)
C5	0.0356 (6)	0.0329 (6)	0.0385 (6)	−0.0058 (5)	0.0193 (5)	−0.0072 (5)
C6	0.0247 (5)	0.0302 (6)	0.0280 (5)	−0.0022 (4)	0.0138 (4)	−0.0017 (4)
C7	0.0227 (5)	0.0307 (6)	0.0256 (5)	−0.0016 (4)	0.0100 (4)	−0.0027 (4)
C8	0.0264 (6)	0.0425 (7)	0.0270 (6)	0.0004 (5)	0.0051 (4)	−0.0068 (5)
C9	0.0264 (6)	0.0444 (7)	0.0221 (5)	0.0054 (5)	0.0092 (4)	0.0046 (5)
C10	0.0331 (6)	0.0362 (6)	0.0350 (6)	0.0046 (5)	0.0160 (5)	0.0083 (5)
C11	0.0299 (6)	0.0284 (6)	0.0361 (6)	−0.0026 (4)	0.0109 (5)	−0.0010 (5)
C12	0.0216 (5)	0.0300 (6)	0.0263 (5)	−0.0019 (4)	0.0088 (4)	−0.0005 (4)
C13	0.0301 (7)	0.0766 (11)	0.0369 (7)	−0.0055 (7)	0.0054 (5)	−0.0011 (7)
C14	0.0288 (6)	0.0552 (8)	0.0383 (7)	−0.0054 (6)	0.0142 (5)	−0.0041 (6)
C15	0.0241 (5)	0.0381 (6)	0.0252 (5)	−0.0066 (5)	0.0073 (4)	−0.0031 (5)
C16	0.0204 (5)	0.0315 (6)	0.0265 (5)	−0.0005 (4)	0.0050 (4)	0.0002 (4)
C17	0.0277 (6)	0.0442 (7)	0.0404 (7)	−0.0096 (5)	0.0099 (5)	0.0018 (6)
C18	0.0340 (7)	0.0403 (7)	0.0445 (7)	−0.0114 (5)	0.0049 (5)	0.0023 (6)

Geometric parameters (Å, °)

O1—C13	1.3995 (17)	C8—H8A	0.990
O1—C9	1.4268 (14)	C8—H8B	0.990
O2—C14	1.4233 (15)	C9—C10	1.5140 (18)
O2—C9	1.4254 (13)	C10—C11	1.5340 (17)
O3—C16	1.1982 (14)	C10—H10A	0.990
O4—C16	1.3311 (14)	C10—H10B	0.990
O4—C17	1.4613 (14)	C11—C12	1.4926 (16)
N1—C1	1.3817 (15)	C11—H11A	0.990
N1—C12	1.3872 (14)	C11—H11B	0.990
N1—C15	1.4412 (14)	C13—C14	1.503 (2)
C1—C2	1.3926 (17)	C13—H13A	0.990
C1—C6	1.4146 (16)	C13—H13B	0.990
C2—C3	1.381 (2)	C14—H14A	0.990
C2—H2A	0.950	C14—H14B	0.990
C3—C4	1.392 (2)	C15—C16	1.5154 (15)
C3—H3A	0.950	C15—H15A	0.990
C4—C5	1.382 (2)	C15—H15B	0.990
C4—H4A	0.950	C17—C18	1.4910 (18)
C5—C6	1.3993 (16)	C17—H17A	0.990
C5—H5A	0.950	C17—H17B	0.990
C6—C7	1.4293 (16)	C18—H18A	0.980
C7—C12	1.3591 (16)	C18—H18B	0.980
C7—C8	1.4929 (15)	C18—H18C	0.980
C8—C9	1.5252 (18)		

C13—O1—C9	109.16 (9)	H10A—C10—H10B	107.9
C14—O2—C9	106.09 (9)	C12—C11—C10	109.32 (10)
C16—O4—C17	115.65 (9)	C12—C11—H11A	109.8
C1—N1—C12	108.24 (9)	C10—C11—H11A	109.8
C1—N1—C15	125.89 (10)	C12—C11—H11B	109.8
C12—N1—C15	125.87 (10)	C10—C11—H11B	109.8
N1—C1—C2	130.37 (11)	H11A—C11—H11B	108.3
N1—C1—C6	107.66 (10)	C7—C12—N1	109.97 (10)
C2—C1—C6	121.97 (11)	C7—C12—C11	125.57 (10)
C3—C2—C1	117.28 (13)	N1—C12—C11	124.41 (10)
C3—C2—H2A	121.4	O1—C13—C14	105.53 (11)
C1—C2—H2A	121.4	O1—C13—H13A	110.6
C2—C3—C4	121.92 (13)	C14—C13—H13A	110.6
C2—C3—H3A	119.0	O1—C13—H13B	110.6
C4—C3—H3A	119.0	C14—C13—H13B	110.6
C5—C4—C3	120.83 (13)	H13A—C13—H13B	108.8
C5—C4—H4A	119.6	O2—C14—C13	103.35 (10)
C3—C4—H4A	119.6	O2—C14—H14A	111.1
C4—C5—C6	119.03 (12)	C13—C14—H14A	111.1
C4—C5—H5A	120.5	O2—C14—H14B	111.1
C6—C5—H5A	120.5	C13—C14—H14B	111.1
C5—C6—C1	118.98 (11)	H14A—C14—H14B	109.1
C5—C6—C7	134.14 (11)	N1—C15—C16	112.31 (9)
C1—C6—C7	106.87 (10)	N1—C15—H15A	109.1
C12—C7—C6	107.24 (10)	C16—C15—H15A	109.1
C12—C7—C8	123.20 (11)	N1—C15—H15B	109.1
C6—C7—C8	129.47 (10)	C16—C15—H15B	109.1
C7—C8—C9	110.13 (10)	H15A—C15—H15B	107.9
C7—C8—H8A	109.6	O3—C16—O4	124.27 (10)
C9—C8—H8A	109.6	O3—C16—C15	124.99 (10)
C7—C8—H8B	109.6	O4—C16—C15	110.74 (9)
C9—C8—H8B	109.6	O4—C17—C18	107.80 (10)
H8A—C8—H8B	108.1	O4—C17—H17A	110.1
O2—C9—O1	105.70 (9)	C18—C17—H17A	110.1
O2—C9—C10	108.03 (10)	O4—C17—H17B	110.1
O1—C9—C10	110.29 (10)	C18—C17—H17B	110.1
O2—C9—C8	111.67 (10)	H17A—C17—H17B	108.5
O1—C9—C8	108.76 (10)	C17—C18—H18A	109.5
C10—C9—C8	112.19 (10)	C17—C18—H18B	109.5
C9—C10—C11	112.18 (10)	H18A—C18—H18B	109.5
C9—C10—H10A	109.2	C17—C18—H18C	109.5
C11—C10—H10A	109.2	H18A—C18—H18C	109.5
C9—C10—H10B	109.2	H18B—C18—H18C	109.5
C11—C10—H10B	109.2		

C12—N1—C1—C2	−179.74 (11)	C7—C8—C9—O2	76.13 (12)
C15—N1—C1—C2	0.15 (19)	C7—C8—C9—O1	−167.60 (9)
C12—N1—C1—C6	1.15 (12)	C7—C8—C9—C10	−45.32 (13)
C15—N1—C1—C6	−178.96 (9)	O2—C9—C10—C11	−61.20 (12)
N1—C1—C2—C3	−178.46 (12)	O1—C9—C10—C11	−176.27 (9)

C6—C1—C2—C3	0.54 (18)	C8—C9—C10—C11	62.32 (13)
C1—C2—C3—C4	0.1 (2)	C9—C10—C11—C12	−43.12 (13)
C2—C3—C4—C5	−0.5 (2)	C6—C7—C12—N1	0.87 (12)
C3—C4—C5—C6	0.20 (19)	C8—C7—C12—N1	−176.05 (9)
C4—C5—C6—C1	0.41 (16)	C6—C7—C12—C11	178.28 (10)
C4—C5—C6—C7	179.11 (12)	C8—C7—C12—C11	1.36 (17)
N1—C1—C6—C5	178.40 (10)	C1—N1—C12—C7	−1.28 (12)
C2—C1—C6—C5	−0.80 (16)	C15—N1—C12—C7	178.83 (10)
N1—C1—C6—C7	−0.62 (11)	C1—N1—C12—C11	−178.72 (10)
C2—C1—C6—C7	−179.82 (10)	C15—N1—C12—C11	1.39 (16)
C5—C6—C7—C12	−178.95 (12)	C10—C11—C12—C7	12.73 (15)
C1—C6—C7—C12	−0.15 (12)	C10—C11—C12—N1	−170.22 (10)
C5—C6—C7—C8	−2.3 (2)	C9—O1—C13—C14	5.60 (16)
C1—C6—C7—C8	176.51 (11)	C9—O2—C14—C13	31.96 (14)
C12—C7—C8—C9	14.73 (15)	O1—C13—C14—O2	−23.07 (16)
C6—C7—C8—C9	−161.45 (11)	C1—N1—C15—C16	−97.30 (13)
C14—O2—C9—O1	−29.21 (13)	C12—N1—C15—C16	82.58 (13)
C14—O2—C9—C10	−147.26 (10)	C17—O4—C16—O3	−3.87 (17)
C14—O2—C9—C8	88.91 (12)	C17—O4—C16—C15	176.02 (10)
C13—O1—C9—O2	14.11 (14)	N1—C15—C16—O3	5.41 (17)
C13—O1—C9—C10	130.64 (12)	N1—C15—C16—O4	−174.47 (9)
C13—O1—C9—C8	−105.93 (13)	C16—O4—C17—C18	176.84 (10)

Fig. 1

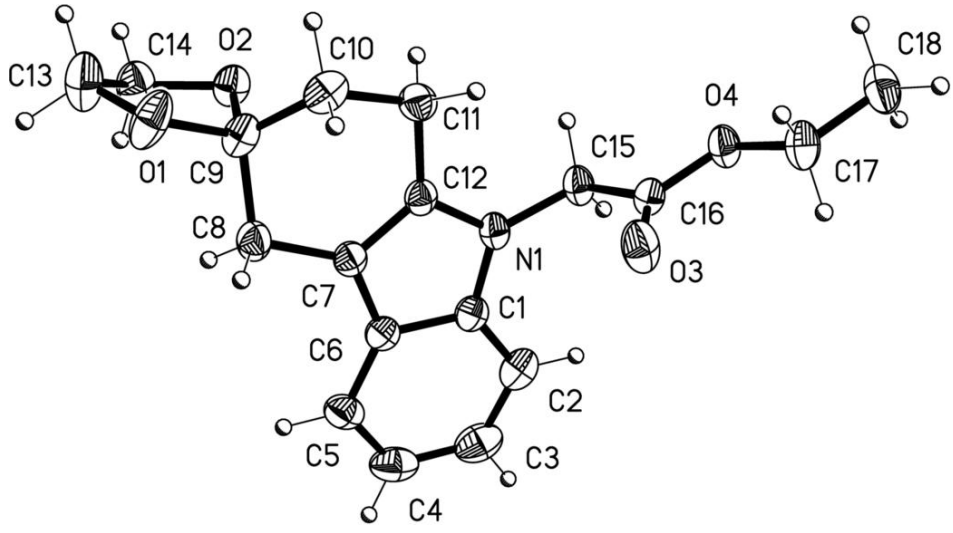
C13
C14
O2
C10
C18
O4
C11
C15
O1
C9
C12
C17
C16
C8
N1
O3
C7
C1
C6
C2
C5
C3
C4

Fig. 2