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Methyl 9*H*-carbazole-9-acetate

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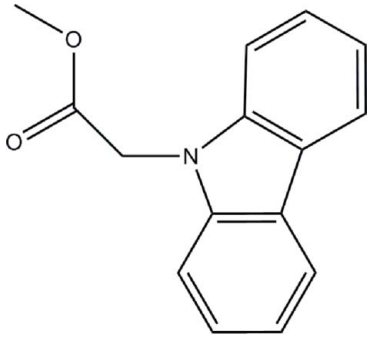
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.003 Å; R factor = 0.055; wR factor = 0.143; data-to-parameter ratio = 15.9.

The title compound, $C_{15}H_{13}NO_2$, was synthesized by *N*-alkylation of methyl bromoacetate with 9*H*-carbazole. The carbazole ring system is essentially planar (mean atomic deviation = 0.0346 Å) and makes a dihedral angle of 86.5 (7)° with the methyl acetate group. Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure.

Related literature

The title compound is an intermediate in the synthesis of -(9-carbazole) acetyl chloride, a novel fluorescence derivatization reagent, see: Xie *et al.* (2006); Bong *et al.* (1992). For bond distances, see: Allen *et al.* (1987). For the synthesis, see: Xie *et al.* (2006).



Experimental

Crystal data

$C_{15}H_{13}NO_2$
M_r = 239.26
Monoclinic, $P2_1/c$
a = 10.875 (3) Å
b = 5.8773 (12) Å
c = 18.608 (4) Å
β = 103.599 (3)°
V = 1155.9 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 93 K
0.43 × 0.33 × 0.27 mm

Data collection

Rigaku SPIDER diffractometer
Absorption correction: none
8735 measured reflections
2615 independent reflections
1587 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.143
S = 1.00
2615 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15B···O2^{i}	0.98	2.43	3.374 (3)	161

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2513).

References

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Methyl 9*H*-carbazole-9-acetate

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S1. Comment

The title compound is useful as an intermediate in the synthesis of 2-(9-carbazole) acetyl chloride, a novel fluorescence derivatization reagent (Xie *et al.*, 2006; Bong *et al.*, 1992). We report here the crystal structure of (I), which is of interest to us in the field. The molecular structure of(I) is showed in Fig.1. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The carbazole ring system is essentially planar with mean deviation of 0.0346 Å. The methylacetate substituent adopts a fully extended conformation, and its mean plane forms a dihedral angle of 93.5 (7)° with the carbazole mean plane. In the crystal structure weak C—H···O hydrogen bonding in present (Table 1).

S2. Experimental

The title compound was prepared by the method reported in literature (Xie *et al.*, 2006). The crystals were obtained by dissolving the title compound (0.1 g) in methanol (20 ml), and evaporating the solvent slowly at room temperature. Colorless prism-shaped crystals were formed after 3 d.

S3. Refinement

H atoms were placed in calculated positions and refined in ride mode with C—H = 0.95, 0.99 and 0.98 Å for aromatic, methylene and methyl H atoms. U_{iso}(H) = 1.2U_{eq}(C).

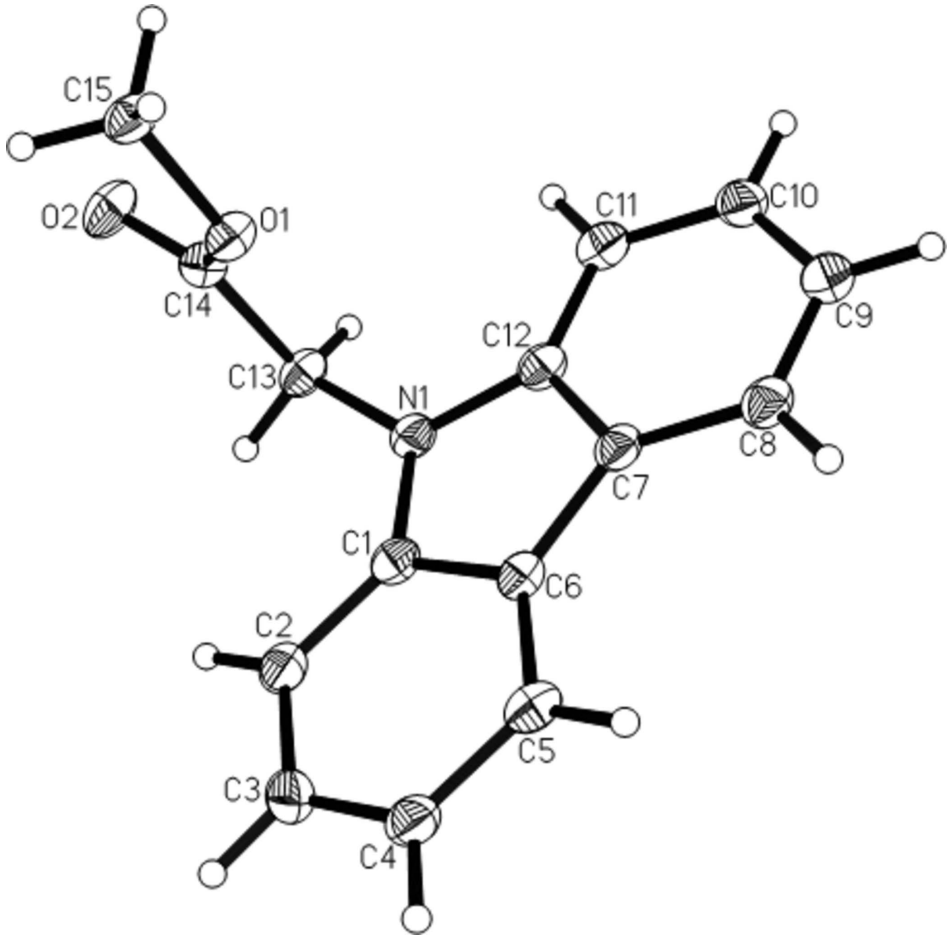

Figure 1

The molecular structure of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

Methyl 9*H*-carbazole-9-acetate

Crystal data

$C_{15}H_{13}NO_2$
$M_r = 239.26$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.875$ (3) Å
$b = 5.8773$ (12) Å
$c = 18.608$ (4) Å
$\beta = 103.599$ (3)°
$V = 1155.9$ (5) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3382 reflections
$\theta = 3.3$–27.5°
$\mu = 0.09$ mm^{-1}
$T = 93$ K
Prism, colorless
0.43 × 0.33 × 0.27 mm

Data collection

Rigaku SPIDER diffractometer
Radiation source: Rotating Anode
Graphite monochromator
ω scans
8735 measured reflections
2615 independent reflections
1587 reflections with $I > 2\sigma(I)$

R_{int} = 0.041
θ_{max} = 27.5°, θ_{min} = 3.3°
h = −14→14
k = −7→7
l = −24→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.143
S = 1.00
2615 reflections
164 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2 + 1.86P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.15039 (15)	0.6886 (3)	0.49433 (9)	0.0250 (4)
O2	0.11310 (18)	1.0517 (3)	0.45700 (10)	0.0349 (5)
N1	0.31648 (18)	0.7955 (3)	0.62011 (11)	0.0239 (5)
C1	0.4231 (2)	0.6749 (4)	0.61296 (13)	0.0242 (5)
C2	0.5141 (2)	0.7323 (4)	0.57434 (13)	0.0272 (6)
H2	0.5081	0.8696	0.5468	0.033*
C3	0.6132 (2)	0.5828 (5)	0.57759 (13)	0.0295 (6)
H3	0.6767	0.6191	0.5520	0.035*
C4	0.6228 (2)	0.3793 (5)	0.61751 (13)	0.0290 (6)
H4	0.6912	0.2784	0.6179	0.035*
C5	0.5332 (2)	0.3246 (4)	0.65647 (13)	0.0268 (6)
H5	0.5400	0.1872	0.6840	0.032*
C6	0.4324 (2)	0.4737 (4)	0.65483 (13)	0.0234 (5)
C7	0.3267 (2)	0.4732 (4)	0.68959 (13)	0.0237 (5)
C8	0.2899 (2)	0.3290 (4)	0.74042 (13)	0.0276 (6)
H8	0.3364	0.1942	0.7568	0.033*
C9	0.1848 (2)	0.3858 (5)	0.76644 (14)	0.0309 (6)
H9	0.1602	0.2914	0.8021	0.037*
C10	0.1143 (2)	0.5807 (5)	0.74077 (14)	0.0304 (6)
H10	0.0412	0.6141	0.7585	0.036*
C11	0.1483 (2)	0.7260 (5)	0.69037 (14)	0.0289 (6)
H11	0.0994	0.8574	0.6730	0.035*

C12	0.2565 (2)	0.6735 (4)	0.66592 (13)	0.0247 (5)
C13	0.2590 (2)	0.9803 (4)	0.57265 (13)	0.0263 (6)
H13A	0.3273	1.0713	0.5597	0.032*
H13B	0.2146	1.0802	0.6012	0.032*
C14	0.1662 (2)	0.9128 (4)	0.50173 (13)	0.0230 (5)
C15	0.0599 (2)	0.6135 (4)	0.42803 (13)	0.0284 (6)
H15A	−0.0252	0.6634	0.4301	0.034*
H15B	0.0617	0.4471	0.4250	0.034*
H15C	0.0821	0.6794	0.3844	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0257 (9)	0.0222 (9)	0.0240 (9)	−0.0003 (7)	−0.0001 (7)	0.0000 (7)
O2	0.0394 (11)	0.0295 (10)	0.0297 (10)	0.0044 (8)	−0.0043 (8)	0.0064 (8)
N1	0.0227 (10)	0.0239 (11)	0.0238 (10)	0.0030 (8)	0.0031 (8)	0.0028 (9)
C1	0.0241 (12)	0.0244 (13)	0.0217 (12)	0.0004 (10)	0.0005 (9)	−0.0026 (10)
C2	0.0275 (13)	0.0284 (13)	0.0236 (12)	−0.0001 (11)	0.0021 (10)	0.0023 (10)
C3	0.0298 (13)	0.0358 (15)	0.0233 (12)	−0.0010 (11)	0.0073 (10)	−0.0008 (11)
C4	0.0246 (12)	0.0358 (14)	0.0244 (12)	0.0034 (11)	0.0014 (10)	−0.0012 (11)
C5	0.0253 (12)	0.0237 (13)	0.0279 (13)	0.0033 (10)	−0.0006 (10)	0.0003 (11)
C6	0.0232 (12)	0.0247 (12)	0.0204 (11)	0.0005 (10)	0.0012 (9)	0.0003 (10)
C7	0.0226 (12)	0.0251 (12)	0.0204 (11)	−0.0018 (10)	−0.0007 (9)	0.0004 (10)
C8	0.0273 (13)	0.0290 (13)	0.0237 (12)	−0.0021 (11)	0.0001 (10)	−0.0008 (11)
C9	0.0271 (13)	0.0361 (15)	0.0278 (13)	−0.0034 (12)	0.0031 (10)	0.0010 (12)
C10	0.0246 (12)	0.0402 (16)	0.0258 (13)	−0.0006 (11)	0.0049 (10)	−0.0023 (12)
C11	0.0266 (13)	0.0310 (14)	0.0268 (13)	0.0031 (11)	0.0016 (10)	−0.0032 (11)
C12	0.0244 (12)	0.0244 (13)	0.0226 (12)	−0.0010 (10)	0.0000 (10)	−0.0024 (10)
C13	0.0281 (13)	0.0227 (13)	0.0250 (12)	0.0032 (10)	0.0001 (10)	0.0020 (10)
C14	0.0234 (12)	0.0191 (12)	0.0266 (12)	0.0016 (10)	0.0059 (10)	−0.0003 (10)
C15	0.0258 (12)	0.0310 (13)	0.0249 (12)	−0.0038 (11)	−0.0010 (10)	−0.0034 (11)

Geometric parameters (Å, º)

O1—C14	1.332 (3)	C7—C8	1.397 (3)
O1—C15	1.454 (3)	C7—C12	1.416 (3)
O2—C14	1.211 (3)	C8—C9	1.383 (3)
N1—C12	1.388 (3)	C8—H8	0.9500
N1—C1	1.392 (3)	C9—C10	1.399 (4)
N1—C13	1.446 (3)	C9—H9	0.9500
C1—C2	1.394 (3)	C10—C11	1.381 (4)
C1—C6	1.407 (3)	C10—H10	0.9500
C2—C3	1.381 (4)	C11—C12	1.393 (3)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.398 (4)	C13—C14	1.514 (3)
C3—H3	0.9500	C13—H13A	0.9900
C4—C5	1.383 (3)	C13—H13B	0.9900
C4—H4	0.9500	C15—H15A	0.9800

C5—C6	1.398 (3)	C15—H15B	0.9800
C5—H5	0.9500	C15—H15C	0.9800
C6—C7	1.445 (3)		

C14—O1—C15	115.67 (18)	C8—C9—C10	120.7 (2)
C12—N1—C1	108.52 (19)	C8—C9—H9	119.7
C12—N1—C13	124.4 (2)	C10—C9—H9	119.7
C1—N1—C13	125.0 (2)	C11—C10—C9	121.7 (2)
N1—C1—C2	129.4 (2)	C11—C10—H10	119.1
N1—C1—C6	109.1 (2)	C9—C10—H10	119.1
C2—C1—C6	121.5 (2)	C10—C11—C12	117.8 (2)
C3—C2—C1	117.7 (2)	C10—C11—H11	121.1
C3—C2—H2	121.2	C12—C11—H11	121.1
C1—C2—H2	121.2	N1—C12—C11	129.9 (2)
C2—C3—C4	121.8 (2)	N1—C12—C7	108.9 (2)
C2—C3—H3	119.1	C11—C12—C7	121.2 (2)
C4—C3—H3	119.1	N1—C13—C14	116.1 (2)
C5—C4—C3	120.2 (2)	N1—C13—H13A	108.3
C5—C4—H4	119.9	C14—C13—H13A	108.3
C3—C4—H4	119.9	N1—C13—H13B	108.3
C4—C5—C6	119.3 (2)	C14—C13—H13B	108.3
C4—C5—H5	120.4	H13A—C13—H13B	107.4
C6—C5—H5	120.4	O2—C14—O1	124.5 (2)
C5—C6—C1	119.5 (2)	O2—C14—C13	122.4 (2)
C5—C6—C7	133.7 (2)	O1—C14—C13	113.1 (2)
C1—C6—C7	106.8 (2)	O1—C15—H15A	109.5
C8—C7—C12	119.8 (2)	O1—C15—H15B	109.5
C8—C7—C6	133.5 (2)	H15A—C15—H15B	109.5
C12—C7—C6	106.6 (2)	O1—C15—H15C	109.5
C9—C8—C7	118.8 (2)	H15A—C15—H15C	109.5
C9—C8—H8	120.6	H15B—C15—H15C	109.5
C7—C8—H8	120.6		

C12—N1—C1—C2	−178.1 (2)	C6—C7—C8—C9	176.1 (3)
C13—N1—C1—C2	17.5 (4)	C7—C8—C9—C10	1.8 (4)
C12—N1—C1—C6	−0.3 (3)	C8—C9—C10—C11	−1.6 (4)
C13—N1—C1—C6	−164.7 (2)	C9—C10—C11—C12	−0.5 (4)
N1—C1—C2—C3	178.6 (2)	C1—N1—C12—C11	178.8 (2)
C6—C1—C2—C3	1.0 (4)	C13—N1—C12—C11	−16.7 (4)
C1—C2—C3—C4	0.5 (4)	C1—N1—C12—C7	1.0 (3)
C2—C3—C4—C5	−1.3 (4)	C13—N1—C12—C7	165.5 (2)
C3—C4—C5—C6	0.6 (4)	C10—C11—C12—N1	−175.1 (2)
C4—C5—C6—C1	0.8 (4)	C10—C11—C12—C7	2.5 (4)
C4—C5—C6—C7	−178.1 (2)	C8—C7—C12—N1	175.7 (2)
N1—C1—C6—C5	−179.7 (2)	C6—C7—C12—N1	−1.2 (3)
C2—C1—C6—C5	−1.6 (4)	C8—C7—C12—C11	−2.3 (3)
N1—C1—C6—C7	−0.5 (3)	C6—C7—C12—C11	−179.3 (2)
C2—C1—C6—C7	177.6 (2)	C12—N1—C13—C14	−77.4 (3)

C5—C6—C7—C8	3.7 (5)	C1—N1—C13—C14	84.6 (3)
C1—C6—C7—C8	−175.3 (3)	C15—O1—C14—O2	−1.6 (4)
C5—C6—C7—C12	−180.0 (3)	C15—O1—C14—C13	178.6 (2)
C1—C6—C7—C12	1.0 (3)	N1—C13—C14—O2	−179.0 (2)
C12—C7—C8—C9	0.2 (3)	N1—C13—C14—O1	0.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15*B*···O2^{i}	0.98	2.43	3.374 (3)	161

Symmetry code: (i) *x*, *y*−1, *z*.