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# Ethyl (3-ethyl-1-oxo-1,2,3,4-tetrahydrocarbazol-2-yl)acetate

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**Key indicators**

Single-crystal X-ray study
$T$ = 295 K
Mean $\sigma$(C–C) = 0.004 Å
$R$ factor = 0.047
$wR$ factor = 0.145
Data-to-parameter ratio = 12.9

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

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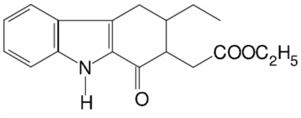
The title compound, $C_{18}H_{21}NO_3$, consists of a carbazole skeleton with carboxyethyl and ethyl groups at positions 2 and 3, respectively. The heterocyclic ring and the benzene ring fused to it are nearly planar, while the cyclohexenone ring adopts a distorted sofa conformation. Molecules are linked in pairs about inversion centers by N—H···O hydrogen bonds [N···O = 2.831 (3) Å and N—H···O = 158 (3)°].

## Comment

Tricyclic ring systems such as tetrahydrocarbazole with side chains are present in the framework of a number of indole-type alkaloids of biological interest. The introduction of the ketone on atom C4 (according to the carbazole numbering) and conversion of the ester group into amide following the ring cyclization could constitute a new entry into the total synthesis of (±)-dasycarpidone and (±)-uleine.



(I)

In the molecule of the title compound, (I), rings $A$ and $B$ are almost planar [maximum deviations are 0.008 (2) Å for C8$A$ and 0.005 (3) Å for C9$A$] and the dihedral angle between these two rings is 1.3 (2)°. Rings $A$ and $C$ in the carbazole moiety are slightly bent away from the central five-membered ring in the same direction, with torsion angles of 1.6 (4) (C5—C5$A$—C8$A$—C8) and 3.0 (4)° (C4—C4$A$—C9$A$—C1). Ring $C$ is not planar and adopts a distorted sofa conformation. The ring puckering parameters (Cremer & Pople, 1975) for this ring are $Q$ = 0.472 (3) Å, $\theta$ = 52.7 (4)° and $\varphi$ = 100.6 (4)°.

In the title compound, the bond lengths of the central ring of carbazole are shortened [N9—C8$A$ = 1.360 (4) Å and N9—

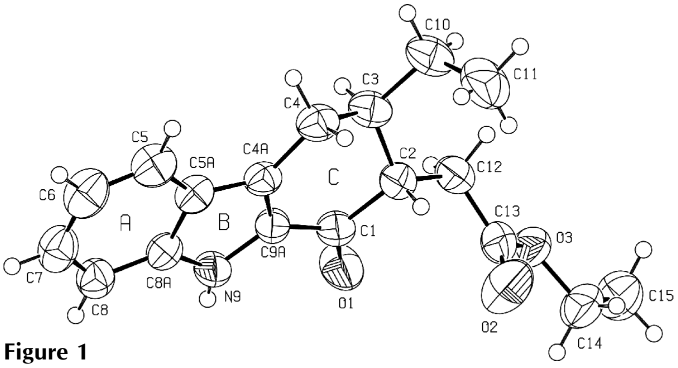


**Figure 1**
*ORTEP* (Johnson, 1965) drawing of the molecule of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

C9*A* = 1.381 (3) Å] compared with the corresponding values in ethyl 4-methyl-9*H*-carbazole-3-carboxylate (Hökelek *et al.*, 2002). The ethyl and carboxyethyl groups in the molecule are oriented out of the mean plane of ring *C*, with torsion angles of 70.6 (4) (C4—C3—C10—C11) and −75.2 (3)° (C1—C2—C12—C13).

The crystal structure of the title compound is stabilized by intermolecular hydrogen bonds. The hydrogen-bonding geometry is given in Table 2. The intermolecular hydrogen bonds between the ketone O atoms and the indole N—H groups of neighbouring molecules causes dimerization of the substituted carbazole molecules. As can be seen from the packing diagram (Fig. 2), the molecules are in a head-to-tail arrangement. In addition, there is an intermolecular C—H···*π* interaction between the H atom of C3 and the *B* ring of a symmetry-related molecule. The distance between atom H3 and the centroid of the *B* ring is 2.598 Å (symmetry code: $1 - x$, $2 - y$, $-z$) and the C3—H3···centroid angle is 167° (Spek, 2000). No significant intermolecular *π*–*π* interactions involving the aromatic rings are found in the crystal structure.

## Experimental

The title compound was prepared from 3-ethyl-2-ethoxalyl-1,2,3,4-tetrahydrocarbazol-1-one (4 g, 12.76 mmol), zinc dust (5 g, 61.17 mmol) and acetic acid (40 ml). The resulting mixture was stirred at room temperature for 16 h. The mixture was diluted with ethyl acetate and filtered. The filtrate was poured into NaOH solution (150 ml, 10%), extracted with ethyl acetate, dried over $MgSO_4$ and evaporated. The compound was obtained by column chromatography, using silica gel and dichloromethane/ethyl acetate (1:1) according to the literature (Wenkert & Dave, 1962). It was recrystallized from diethyl ether [m.p. 431 K, 2.1 g (55%) yield].

*Crystal data*

$C_{18}H_{21}NO_3$
$M_r = 299.36$
Monoclinic, $C2/c$
$a = 15.0923$ (11) Å
$b = 8.0510$ (5) Å
$c = 26.928$ (2) Å
$\beta = 97.535$ (8)°
$V = 3243.7$ (4) Å$^3$
$Z = 8$

$D_x = 1.226$ Mg m$^{-3}$
Mo *Kα* radiation
Cell parameters from 22 reflections
$\theta = 9.3$–18.1°
$\mu = 0.08$ mm$^{-1}$
$T = 295$ (2) K
Prism, colorless
0.48 × 0.42 × 0.12 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min} = 0.961$, $T_{max} = 0.990$
5078 measured reflections
2619 independent reflections
1344 reflections with $I > 2\sigma(I)$

$R_{int} = 0.052$
$\theta_{max} = 24.3°$
$h = -17 \rightarrow 17$
$k = 0 \rightarrow 9$
$l = -31 \rightarrow 31$
3 standard reflections
frequency: 120 min
intensity decay: none

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.145$
$S = 0.99$
2619 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0725P)^2 + 0.4277P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ e Å$^{-3}$

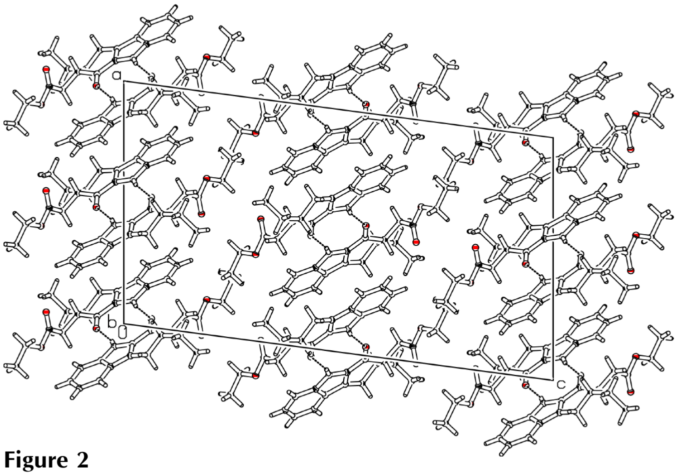


**Figure 2**
The crystal packing, viewed along the *a* axis. Dotted lines represent N—H···O interactions.

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O3—C13 | 1.324 (3) | C2—C1 | 1.529 (4) |
| O3—C14 | 1.458 (3) | C5*A*—C8*A* | 1.414 (4) |
| N9—C8*A* | 1.360 (3) | C5*A*—C4*A* | 1.412 (4) |
| N9—C9*A* | 1.381 (3) | C4*A*—C4 | 1.487 (4) |
| O1—C1 | 1.228 (3) | O2—C13 | 1.194 (3) |
| C9*A*—C4*A* | 1.367 (3) | C4—C3 | 1.525 (4) |
| C9*A*—C1 | 1.437 (4) | C12—C13 | 1.498 (4) |
| C2—C12 | 1.528 (4) | C3—C10 | 1.533 (4) |
| C2—C3 | 1.528 (4) | | |
| C8A—N9—C9*A* | 108.0 (2) | O1—C1—C2 | 122.0 (2) |
| C4*A*—C9*A*—C1 | 124.5 (3) | C9*A*—C1—C2 | 114.3 (2) |
| C12—C2—C3 | 115.9 (2) | C4*A*—C4—C3 | 112.4 (2) |
| C12—C2—C1 | 109.2 (2) | C13—C12—C2 | 111.9 (2) |
| C8*A*—C5*A*—C4*A* | 107.0 (2) | C4—C3—C2 | 110.5 (2) |
| N9—C8*A*—C5*A* | 108.2 (2) | C2—C3—C10 | 114.1 (2) |
| C9*A*—C4*A*—C4 | 121.6 (2) | | |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N9—H9···O1$^{i}$ | 0.94 (3) | 1.94 (3) | 2.831 (3) | 158 (3) |
| C14—H14*B*···O2$^{ii}$ | 0.97 | 2.60 | 3.566 (4) | 174 |

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

The H atom of N9 was located in a difference Fourier map and refined freely. All other H atoms were positioned geometrically, with C—H = 0.93, 0.96, 0.98 and 0.97 Å for aromatic, methyl, methine and methylene H atoms, respectively, and refined riding on their parent atoms, with $U_{iso}(H) = 1.3U_{eq}(C)$.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1965); software used to prepare material for publication: *PARST* (Nardelli, 1995).

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