Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(4-Carboxyphenylmethylene)-2-tetralone

Eliud O. Oloo,[a] J. Wilson Quail,[a]* Maniyan P. Padmanilayam[b] and Jonathan R. Dimmock[b]

[a]Department of Chemistry, University of Saskatchewan, 110 Science Place, Saskatoon, Saskatchewan, Canada S7N 5C9, and [b]College of Pharmacy and Nutrition, University of Saskatchewan, 110 Science Place, Saskatoon, Saskatchewan, Canada S7N 5C9

Correspondence e-mail: quail@sask.usask.ca

Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.154
Data-to-parameter ratio = 13.5

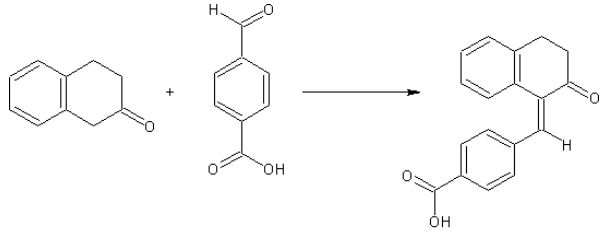
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 10 May 2002
Accepted 14 May 2002
Online 24 May 2002

The title compound, $C_{18}H_{14}O_3$, crystallizes in space group $P2_1/c$ as hydrogen-bonded dimers with crystallographic inversion symmetry.

Comment

One of the major interests in this laboratory is the preparation of novel α,β-unsaturated ketones and related compounds as candidate cytotoxic and anticancer agents (Dimmock *et al.*, 1999, 2000). In a recent study, compound (*E*)-1-(4-carboxyphenylmethylene)-2-tetralone, (I), was prepared as a prototypic candidate cytotoxic agent by the condensation between 4-carboxybenzaldehyde and 2-tetralone. X-ray crystallography reveals that the aryl aldehyde condensed at position 1 of 2-tetralone, and the configuration of the olefinic double bond adopts *E* stereochemistry. In the bicyclic tetralone part of (I), atoms C2 to C9 are planar within 0.021 Å. Atoms C1 and C10 lie at distances of 0.639 and 0.959 Å, respectively, on the same side of this plane.



(I)

Compound (I) possesses an IC_{50} figure of 19.3 μ*M* against murine P388 cells and no toxicity was noted when a dose of 300 mg kg^{-1} was administered to mice. The development of analogues of (I) is currently underway, with a view to producing compounds with increased cytotoxicity unaccompanied by murine toxicity.

Hydrogen bonds for compound (I) are shown in Table 1. (I) is a carboxylic acid, and its structure shows crystallographically centrosymmetric hydrogen-bonded dimers. The O···O distances are quite short at 2.64 (2) Å. Based on a normalized H-atom position (O—H = 0.84 Å), the H···O distance is 1.81 Å and the O—H···O angle is 170°. In addition, there are non-classical hydrogen bonds from C5 to O1 between molecules, linking them into chains parallel to the *c* axis.

Experimental

Compound (I) (m.p. 504–506 K) was prepared in 40% yield by a literature method (Jha *et al.*, 2002). The single-crystal used in the X-ray crystallographic determination was obtained from the reaction product. The evaluation using P388 cells was carried out by a

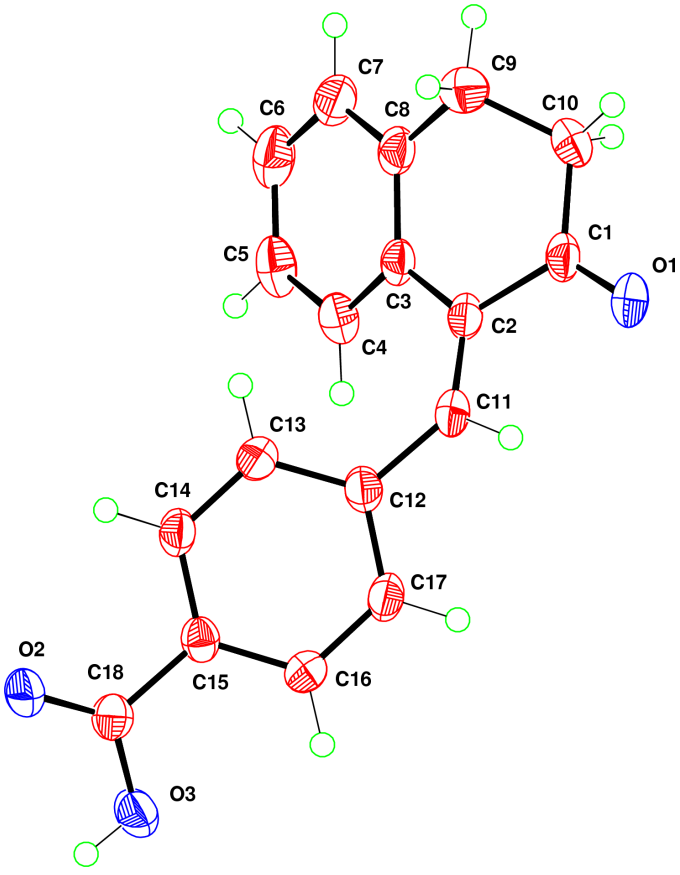


Figure 1
A general view of (I), with ellipsoids drawn at the 50% probability level.

literature procedure (Phillips *et al.*, 1989), and neurotoxicity was determined at the end of 0.5 and 4 h using a reported method (Stables & Kupferberg, 1997).

Crystal data

$C_{18}H_{14}O_3$
M_r = 278.29
Monoclinic, $P2_1/c$
a = 6.435 (2) Å
b = 14.604 (6) Å
c = 16.388 (6) Å
β = 118.641 (7)°
V = 1351.6 (9) Å^3
Z = 4
D_x = 1.368 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2457 reflections
θ = 2.5–25°
μ = 0.09 mm^{-1}
T = 193 (2) K
Fragment, yellow
0.35 × 0.22 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
6165 measured reflections
2571 independent reflections
1652 reflections with $I > 2\sigma(I)$
R_{int} = 0.078
θ_{max} = 25.9°
$h = -7 \rightarrow 6$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.154
S = 0.94
2571 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0842P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{i}	0.84	1.81	2.643 (2)	170
C5—H5···O1^{ii}	0.95	2.52	3.321 (3)	142
C11—H11···O1	0.95	2.30	2.735 (3)	107

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

All H atoms were placed in calculated positions (O—H = 0.84 Å, C—H = 0.99 Å on aliphatic C atoms and 0.95 Å on aromatic C atoms). U_{iso}(H) values were assigned as 1.2U_{eq} of the parent atom.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *Xtal*3.7 (Hall *et al.*, 2000); software used to prepare material for publication: *SHELXL*97.

Financial assistance for this project was provided by the Natural Sciences and Engineering Research Council of Canada for an operating grant to JWQ. This work was also supported by a grant to JRD from Purdue Neuroscience Co., USA. We thank Dr R. MacDonald, University of Alberta, for collecting the diffraction data. Appreciation is extended to Dr T. M. Allen, University of Alberta, Canada, and Mr J. P. Stables, National Institute of Neurological Disorders and Stroke, Bethesda, USA, who supervised the P388 and murine toxicity evaluations, respectively.

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dimmock, J. R., Elias, D. W., Beazely, M. A. & Kandepu, N. M. (1999). *Curr. Med. Chem.* **6**, 1125–1149.
Dimmock, J. R., Kumar, P., Nazarali, A. J., Motaganahalli, N. L., Kowalchuk, T. P., Beazely, M. A., Quail, J. W., Oloo, E. O., Allen, T. M., Szydlowski, J., DeClercq, E. & Balzarini, J. (2000). *Eur. J. Med. Chem.* **35**, 967–977.
Hall, S. R., du Boulay, D. J. & Olthof-Hazekamp, R. (2000). Editors. *The Xtal*3.7 *System*. University of Western Australia, Australia.
Jha, A., Padmanilayam, M. P. & Dimmock, J. R. (2002). *Synthesis*, pp. 463–465.
Phillips, O. A., Nelson, L. A., Knaus, E. E., Allen, T. M. & Fathi-Afshar, F. (1989). *Drug Des. Deliv.* **4**, 121–127.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Stables, J. P. & Kupferberg, H. J. (1997). *Molecular and Cellular Targets for Antiepileptic Drugs*, edited by G. Avanzini, P. Tanganelli and M. Avoli, pp. 191–198. London: John Libbey and Company Ltd.