Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*R*,*S*)-Methyl 3-methyl-5-oxo-1-phenylcyclohex-3-ene-1-carboxylate

Li-Ying Yao, Chen Zhang* and Bi-Song Wang

Zhejiang University, Department of Medicinal Chemistry, College of Pharmaceutical Science, Hangzhou 310031, Zhejiang, People's Republic of China

Correspondence e-mail: chenzhang@zju.edu.cn

Received 11 May 2006
Accepted 6 June 2006

The title compound, $C_{15}H_{16}O_3$, has two molecules in the asymmetric unit. The cyclohex-2-enone ring has an envelope conformation in both molecules. The crystal exhibits twinning. The molecular packing in the crystal structure is determined by weak intermolecular C—H···O interactions.

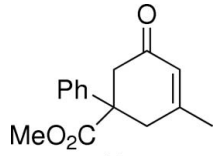
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.122
Data-to-parameter ratio = 17.6

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

Comment

Derivatives of the highly reactive unsaturated compounds with the cyclohex-2-enone ring can be used as precursors in the syntheses of various compounds such as vitamin E, amino acids, terpenes and others (Hu *et al.*, 2003). In addition, cyclohex-2-enone derivatives have shown a wide range of biological activities, such as antimicrobial (Li & Strobel, 2001), protecting cerebral neurocytes (Luu *et al.*, 2004) and many others. We are interested in their pharmaceutical properties. In this paper, we present the X-ray crystal structure analysis of the title compound, (I) (Fig. 1). The crystal packing is defined by weak hydrogen bonds [C27—H271 = 0.97 Å, H271···O2^{i} = 2.617 Å, C27···O2^{i} = 3.204 (2) Å, C27—H271···O2^{i} = 119° where (i): $1-x, 1-y, 2-z$; Fig. 2].



(I)

Experimental

A solution of 4-carboxymethyl-4-phenyl-2,6-heptanedione (262 mg, 1 mmol) and sodium methoxide (54 mg, 1 mmol) in methanol (10 ml) was heated at 323 K for 4 h. The reaction mixture was acidified with dilute aqueous HCl, then concentrated and partitioned between water and dichloromethane. The pure product was obtained through silica gel chromatography, and diffraction quality crystals were obtained by slow evaporation of a diethyl ether/hexane (1.1) solution at room temperature.

Crystal data

$C_{15}H_{16}O_3$
M_r = 244.29
Triclinic, $P\overline{1}$
a = 9.317 (4) Å
b = 12.332 (5) Å
c = 12.734 (5) Å
α = 67.495 (16)°
β = 84.826 (16)°
γ = 74.278 (16)°
V = 1300.9 (9) Å^3
Z = 4
D_x = 1.247 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (1) K
Block, colourless
0.27 × 0.26 × 0.17 mm

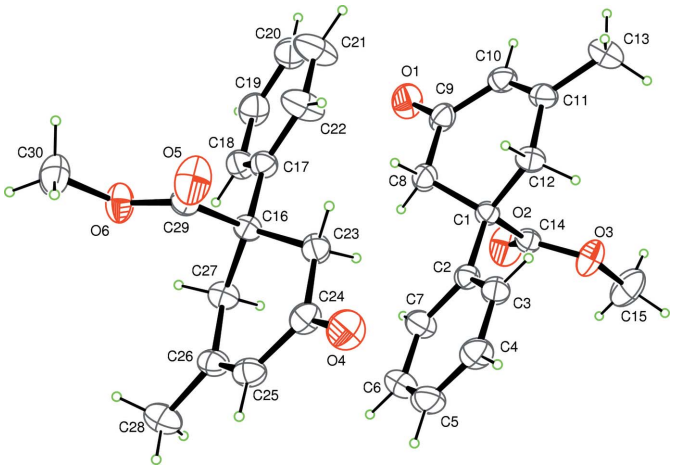


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level.

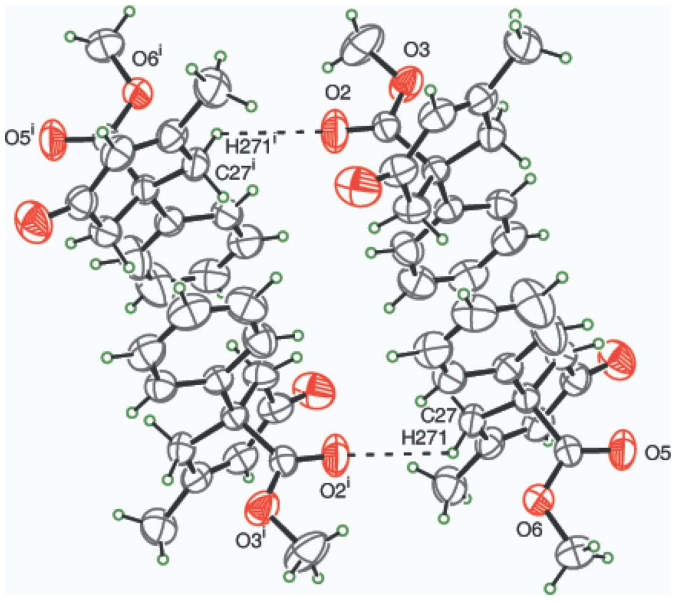


Figure 2
View of the hydrogen bonds between neighbouring molecules. Hydrogen bonds are indicated by dashed lines. Symmetry code (i): $1-x$, $1-y$, $2-z$.

Data collection

Rigaku R-AXIS RAPID diffractometer	5900 independent reflections
ω scans	3491 reflections with $F^2 > 2\sigma(F^2)$
Absorption correction: none	$R_{int} = 0.023$
12919 measured reflections	$\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[0.0008F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.122$	$\Delta\rho_{max} = 0.22$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.15$ e Å^{-3}
5900 reflections	Extinction correction: Larson (1970), equation 22
336 parameters	Extinction coefficient: 66 (15)
H-atom parameters constrained	

The methyl H atoms were positioned with idealized geometry and allowed to rotate but not to tip, with C—H distances of 0.96 Å, and refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C). All other H atoms were placed in geometrically idealized positions with C—H distances of 0.93 Å (sp^2) or 0.97 Å (sp^3), and were refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

X-ray data were collected at the analysis centre of Zhejiang University.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, C. K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Hu, B. C., Lv, C. X. & Liu, Z. L. (2003). *Yingyong Huaxue*, **20**, 1012–1014. (In Chinese.)
Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, pp. 291–294. Copenhagen: Munksgaard.
Li, J. Y. & Strobel, G. A. (2001). *Phytochemistry*, **57**, 261–265.
Luu, B., Kudo, Y., Yamada, M., Uchida, M., Suma, Y. & Suzuki, H. (2004). US Patent 0 152 786.
Rigaku (1998). *PROCESS-AUTO*. Version 1.06. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.7.0. Rigaku/MSC, The Woodlands, Texas, USA.