Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(4*R*)-5,5-Dimethyl-4-phenyl-3-[(*E*)-3-phenylacryloyl]-1,3-oxazolidin-2-one

Rui Yang, Mei-Su Zhang, Lei Nie, Jing Chen and Jun Lin*‡

School of Chemical Science and Technology, Key Laboratory of Medicinal Chemistry of Natural Resources, Ministry of Education, Yunnan University, Kunming 650091, People's Republic of China

‡ Alternative address: State Key Laboratory of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China.

Correspondence e-mail: linjun@ynu.edu.cn

Received 15 August 2006
Accepted 3 December 2006

The title compound, $C_{20}H_{19}NO_3$, shows a planar Ph—C=C—C(O) fragment. The molecules are linked through C—H···O interactions into a chain running in the *b* direction.

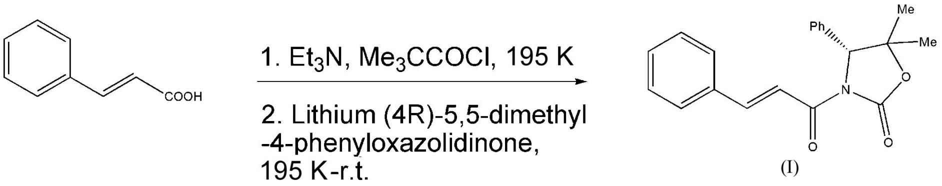
Key indicators

Single-crystal X-ray study
T = 273 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.084
Data-to-parameter ratio = 9.5

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

Comment

The title compound, (I), is used as a precursor in various of asymmetric reactions, for example alkylation (Evans *et al.*, 1992), aldol reactions (Evans *et al.*, 1995), conjugate addition (Lander & Hegedus, 1994) and Diels–Alder reactions (Marchand *et al.*, 1995). The crystal structure of (I) has been determined using X-ray diffraction and the results are shown in Fig. 1.



(I)

Conjugation effects ensure that the O3/C12/C13/C14 fragment and the phenyl ring (C15/C16/C17/C18/C19/C20) are almost coplanar. The substituents of the C13=C14 double bond adopt a *trans*-configuration. The five-membered oxazolidinone ring is almost planar [maximum deviation from planarity is 0.104 (3) Å for atom C3]. The dihedral angle between the N1/C1/C3/O2/C2 mean plane and the C6–C11 phenyl ring attached to C1 is 74.89 (8)°.

In the crystal structure, adjacent molecules are linked through C—H···O hydrogen-bonding interactions (Table 2), forming a chain running in the *b* direction (Fig. 2).

Experimental

A solution of (2*E*)-cinnamic acid (1.836 g, 12.4 mmol) in tetrahydrofuran (100 ml) was stirred under N_2 at 195 K for 15 min, and then triethylamine (1.65 ml, 12.4 mmol) was added *via* a syringe, followed by trimethylacetyl chloride (1.65 g, 12.4 mmol). The resulting suspension was stirred at 195 K for 15 min, then at 273 K for 1 h and finally at 195 K for 15 min, before being transferred *via* a cannula into a slurry of lithium 5,5-dimethyl-4-phenyl-2-oxazolidinone at 273 K, which was prepared 10 min in advance at 195 K by addition of *n*-butyllithium (3.15 ml, 2.2 *M*, 7 mmol) to a solution of 5,5-dimethyl-4-phenyl-2-oxazolidinone (1.847 g, 6.9 mmol) in tetrahydrofuran (100 ml) at 195 K. The resulting slurry was stirred at 195 K for 1 h and then at room temperature for 2 h. The reaction was quenched with a saturated solution of ammonium chloride in water (150 ml). The organic phase was separated off and the aqueous phase was extracted with ethyl acetate. The combined organic phases were

doi:10.1107/S160053680605224X

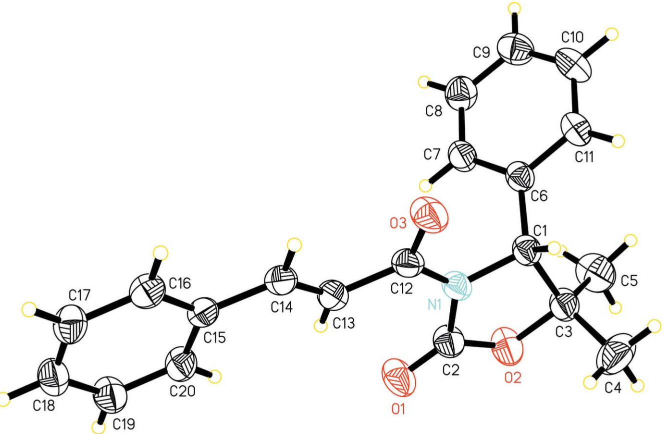


Figure 1
The molecular structure of (I), with the numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

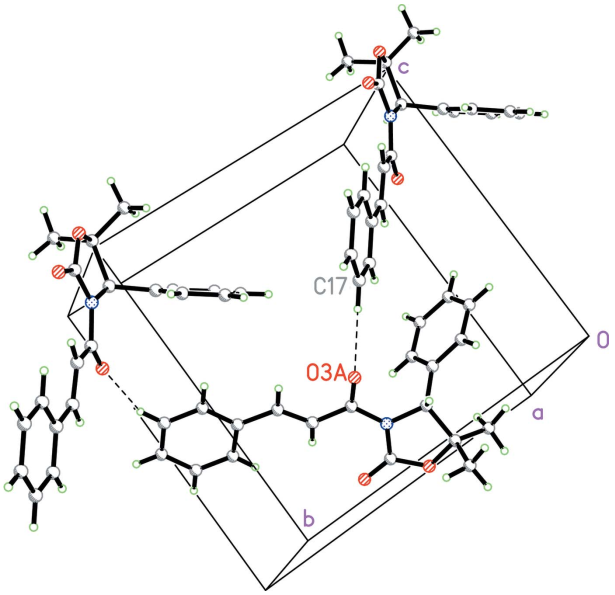


Figure 2
A view of the one-dimensional network in the crystal packing of compound (I), showing the hydrogen bonds as dashed lines. [Symmetry code: (A) $-x, y+\frac{1}{2}, -z+1$.]

washed with saturated sodium bicarbonate, brine and water, dried over anhydrous magnesium sulfate and rotary-evaporated; the crude product was purified by recrystallization from ethyl acetate and hexane (1:8) to give the pure product (2.2 g) as crystals suitable for X-ray diffraction. High-resolution MS for $C_{20}H_{19}NO_3$: $[M+\mathrm{Na}]^+$ calculated 344.1262; found: 344.1341; m.p. 426.5–427.0 K; $[\alpha]_D^{20}$ = 23.243 (c = 0.37, $CHCl_3$). An alternative synthesis of the title compound was described by Davies & Sanganee (1995).

Crystal data

$C_{20}H_{19}NO_3$
$M_r = 321.36$
Monoclinic, $P2_1$
$a = 5.7455$ (7) Å
$b = 12.8894$ (16) Å
$c = 11.6696$ (14) Å
$\beta = 94.317$ (2)°
$V = 861.75$ (18) Å^3
$Z = 2$
$D_x = 1.238$ Mg m$^{-3$}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 273$ (2) K
Block, colourless
0.31 × 0.28 × 0.25 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min} = 0.974$, $T_{\max} = 0.978$
8211 measured reflections
2057 independent reflections
1561 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.084$
$S = 1.05$
2057 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.0378P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.09$ e Å^{-3}
$\Delta\rho_{\min} = -0.11$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.041 (6)

Table 1
Selected torsion angles (°).

O3—C12—C13—C14	−6.6 (3)	C13—C14—C15—C20	2.1 (3)
C12—C13—C14—C15	−177.12 (19)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C17—H17A···O3$^{\rm i}$	0.93	2.42	3.272 (3)	153

Symmetry code: (i) $-x, y+\frac{1}{2}, -z+1$.

In the absence of significant anomalous dispersion effects, Friedel pairs were merged; the absolute configuration was assigned on the basis of the known configuration of the starting material. All H atoms were placed in idealized positions and refined with riding constraints, with C—H distances in the range 0.93–0.96 Å and with $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

This project was supported by the National Natural Science Foundation of China (grant Nos. 20472071 and 20562014), and by the Open Foundation of the State Key Laboratory of Elemento-Organic Chemistry, Nankai University (grant No. 0405).

References

Bruker (1998). *SMART* (Version 5.628) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
Davies, S. G. & Sanganee, H. J. (1995). *Tetrahedron Asymmetry* **6**, 671–674.
Evans, D. A., Gage, J. R., Leighton, J. L. & Kim, A. S. (1992). *J. Org. Chem.* **57**, 1961–1963.
Evans, D. A., Ratz, A. M., Huff, B. E. & Sheppard, G. S. (1995). *J. Am. Chem. Soc.* **117**, 3448–3467.
Lander, P. A. & Hegedus, L. S. (1994). *J. Am. Chem. Soc.* **116**, 8126–8132.
Marchand, A., Mauger, C., Guingant, A. & Pradère, J.-P. (1995). *Tetrahedron Asymmetry* **6**, 853–856.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.