

**2(R)-Hydroxy-4-(4-methoxybenzoyl)-2-methoxycarbonylmethyl-5-(4-methoxyphenyl)-1-(2-methylphenyl)-2,3-dihydro-1H-3-pyrrolone**Sema Öztürk,<sup>a</sup> Mustafa Saçmacı,<sup>b</sup> Şevket Hakan Üngören,<sup>b</sup> Mehmet Akkurt,<sup>a\*</sup> Hoong-Kun Fun<sup>c</sup> and Yunus Akçamur<sup>b</sup><sup>a</sup>Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, <sup>b</sup>Department of Chemistry, Yozgat Faculty of Arts and Sciences, Erciyes University, Yozgat, Turkey, and <sup>c</sup>X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

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**Key indicators**

Single-crystal X-ray study

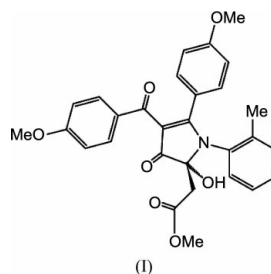
T = 293 K

Mean  $\sigma(C-C) = 0.006 \text{ \AA}$ 

R factor = 0.078

wR factor = 0.257

Data-to-parameter ratio = 16.7

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, C<sub>29</sub>H<sub>27</sub>NO<sub>7</sub>, has a non-planar configuration. The methoxycarbonylmethyl group exhibits an *E* configuration. The crystal structure is stabilized by intermolecular O—H...O and C—H...O contacts.**Comment**The title compound, (I), is a derivative of 2,3-dihydro-1H-3-pyrrolone, which exhibits antimicrobiological and pharmacological activity (Koz'minykh *et al.*, 2002). The bond lengths and angles in (I) (Table 1) are within normal ranges (Allen *et al.*, 1987). The methoxycarbonylmethyl group exhibits an *E* configuration. Compound (I) contains four planar rings, *viz* A, B, C and D (Fig. 1). In the pyrrolone ring A, the maximum deviation from planarity is 0.028 (3) Å for C2. The dihedral angles between rings A/B, A/C, A/D, B/C, B/D and C/D are 34.6 (1), 72.0 (2), 74.2 (1), 59.6 (2), 80.3 (1) and 40.1 (2)°, respectively.

The crystal structure of (I) is stabilized by O—H...O and C—H...O intermolecular contacts (Table 2).

**Experimental**A mixture of 4-(4-methoxybenzoyl)-2-[(*Z*)-methoxycarbonylmethylene]-5-(4-methoxyphenyl)-2,3-dihydro-3-furanone (2.213 g, 5 mmol) and *o*-toluidine (0.536 g, 5 mmol) in dry benzene (25 ml) was refluxed for 0.5 h. The solid obtained after evaporation of the solvent was recrystallized from ethanol to give yellow crystals of 2(*R*)-hydroxy-4-(4-methoxybenzoyl)-2-methoxycarbonylmethyl-5-(4-methoxyphenyl)-1-(2-methylphenyl)-2,3-dihydro-1H-3-pyrrolone; yield: 1.574 g (59%), m.p: 442 K. Solvents were dried by refluxing with the appropriate drying agent and distilled before use. Melting points were determined on an Electrothermal 9200 apparatus and were uncorrected. Elemental analysis was performed with a Carlo Erba Elemental Analyzer 1108. An FT-IR spectrum was measured on a Jasco-Plus Model 460 spectrometer, using a potassium bromide pellet. The <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Gemini-Varian 200 MHz instrument. The chemical shifts are reported in p.p.m. referenced to tetramethylsilane. FT-IR (KBr, cm<sup>-1</sup>):  $\nu = 3200.29$  (broad OH), 1740.92 (C2=O), 1653.66 (C22=O), 1633.41

Received 12 May 2003

Accepted 19 May 2003

Online 31 May 2003

(C2=O), 1257.84 (C20—O4—C21); <sup>1</sup>H NMR (CDCl<sub>3</sub>, p.p.m.): δ = 2.181 (s, 3H, Ph—CH<sub>3</sub>), 2.806–3.081 (q, 2H, CH<sub>2</sub>), 3.528, 3.666, 3.813 (s, 9H, OCH<sub>3</sub>), 5.901 (s, 1H, OH), 6.885–7.942 (m, 12H, Ar—H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, p.p.m.): δ = 20.895 (C18), 41.927 (C19), 54.230, 57.180, 57.384 (C21, C29 and C11), 92.432 (C1), 110.032–165.105 (C=C, aromatic and aliphatic), 181.055 (C20=O), 190.213 (C22=O), 198.213 (C2=O). Analysis calculated for C<sub>29</sub>H<sub>27</sub>NO<sub>7</sub>: C 69.78, N 2.62, H 6.61%; found: C 69.65, N 2.56, H 6.59%.

Crystal data

C<sub>29</sub>H<sub>27</sub>NO<sub>7</sub>  
*M<sub>r</sub>* = 501.52  
 Triclinic, *P* $\bar{1}$   
*a* = 8.948 (5) Å  
*b* = 11.254 (5) Å  
*c* = 13.724 (5) Å  
 α = 110.167 (5)°  
 β = 93.755 (5)°  
 γ = 90.106 (5)°  
*V* = 1294.1 (10) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.287 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 2345 reflections  
 θ = 5.7–53.7°  
 μ = 0.09 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, yellow  
 0.28 × 0.24 × 0.18 mm

Data collection

Siemens SMART CCD area-detector diffractometer  
 ω scans  
 Absorption correction: refined from Δ*F* (Parkin *et al.*, 1995)  
*T<sub>min</sub>* = 0.975, *T<sub>max</sub>* = 0.984  
 8181 measured reflections  
 5384 independent reflections  
 2742 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.023  
 θ<sub>max</sub> = 27.3°  
*h* = -11 → 11  
*k* = -15 → 14  
*l* = -18 → 18

Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.078  
*wR* (*F*<sup>2</sup>) = 0.257  
*S* = 1.00  
 5384 reflections  
 322 parameters  
 H-atom parameters not refined  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.1411*P*)<sup>2</sup>]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> < 0.001  
 Δρ<sub>max</sub> = 0.54 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.29 e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

O1—C1	1.413 (4)	O6—C29	1.409 (8)
O2—C2	1.232 (4)	O7—C8	1.367 (5)
O3—C20	1.194 (5)	O7—C11	1.401 (6)
O4—C20	1.318 (5)	N1—C4	1.351 (4)
O4—C21	1.444 (7)	N1—C12	1.411 (4)
O5—C22	1.223 (4)	N1—C1	1.488 (4)
O6—C26	1.347 (6)		
C20—O4—C21	115.9 (4)	N1—C4—C3	113.1 (2)
C26—O6—C29	119.2 (4)	N1—C4—C5	121.0 (3)
C8—O7—C11	118.6 (3)	O7—C8—C7	116.0 (4)
C1—N1—C4	109.0 (2)	O7—C8—C9	124.8 (4)
C1—N1—C12	122.3 (3)	N1—C12—C13	117.4 (4)
C4—N1—C12	127.6 (3)	N1—C12—C17	122.6 (4)
O1—C1—C2	108.7 (3)	O3—C20—O4	122.9 (4)
O1—C1—C19	109.4 (2)	O4—C20—C19	111.5 (3)
O1—C1—N1	108.6 (3)	O3—C20—C19	125.6 (4)
N1—C1—C19	113.3 (3)	O5—C22—C23	121.0 (3)
N1—C1—C2	102.0 (2)	O5—C22—C3	121.1 (3)
O2—C2—C3	130.1 (3)	O6—C26—C25	123.7 (4)
O2—C2—C1	121.6 (2)	O6—C26—C27	116.5 (4)
C21—O4—C20—O3	0.8 (7)	O1—C1—C2—O2	64.7 (4)
C21—O4—C20—C19	-177.2 (5)	O1—C1—C2—C3	-109.9 (3)
C4—N1—C1—O1	111.9 (3)		

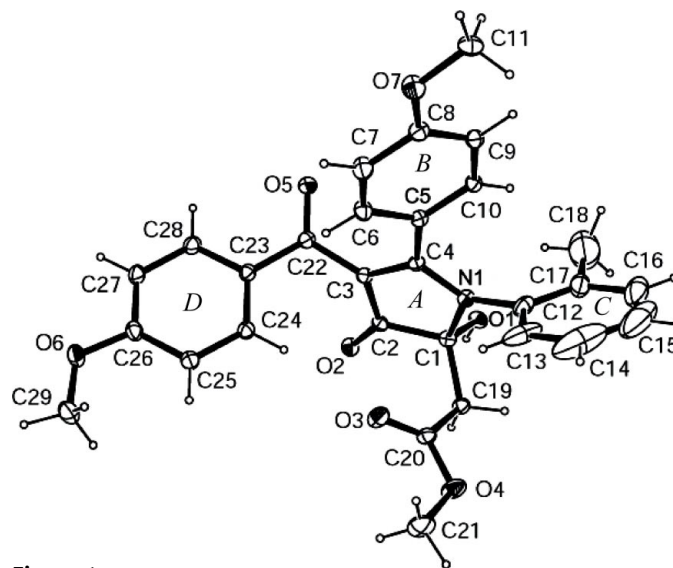


Figure 1

An ORTEP drawing of (I), showing the labelling of the non-H atoms. Displacement ellipsoids are drawn at the 10% probability level.

Table 2

Intermolecular contacts (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1...O2 <sup>i</sup>	0.82	1.98	2.681 (4)	143
C9—H9...O5 <sup>ii</sup>	0.93	2.58	3.485 (6)	165
C19—H19A...O2 <sup>i</sup>	0.97	2.59	3.282 (5)	128

Symmetry codes: (i) 1 - *x*, -*y*, 1 - *z*; (ii) 1 - *x*, 1 - *y*, 2 - *z*.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: PARST (Nardelli, 1995) and WinGX (Farrugia, 1999).

This study was financially supported by the Research Center of Erciyes University. MS and HU thank Dr Hasan Seçen and Dr Cavit Kazaz for helpful discussions.

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