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Methyl (±)-1-ethyl-2-hydroxy-4-(4-methoxybenzoyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1*H*-pyrrole-2-acetate

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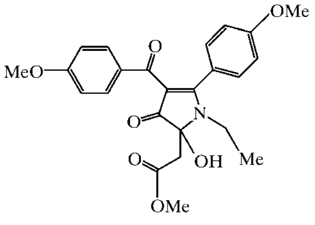
The title compound, $C_{24}H_{25}NO_7$, is a racemic mixture of 2,3-dihydro-1*H*-pyrrol-3-ones. It crystallizes in the triclinic system, space group *P*1, with *Z* = 2. The asymmetric unit contains two enantiomorphic molecules and the structure is stabilized by hydrogen-bond contacts.

Comment

Pyrrolones have received considerable attention as a result of the presence of the lactam ring in some antibiotics, in bile pigments and in the natural alkaloid jatropham, which shows inhibitory activity towards P-338 lymphocytic leukaemia (Jimenez *et al.*, 1988; Gossauer, 1983; Wiedhopf *et al.*, 1973). Among these chemical structures, 2,3-dihydro-1*H*-pyrrol-3-one derivatives (Weigele *et al.*, 1972, 1976; Gelin & Gelin, 1979; Chantegrel & Gelin, 1978) exhibit remarkable anti-microbiological and pharmacological activities (Koz'minykh *et al.*, 1992, 1996, 2002). In the light of this, we have synthesized the title compound, (I), which consists of two enantiomers, and present its X-ray crystal structure.



(I)

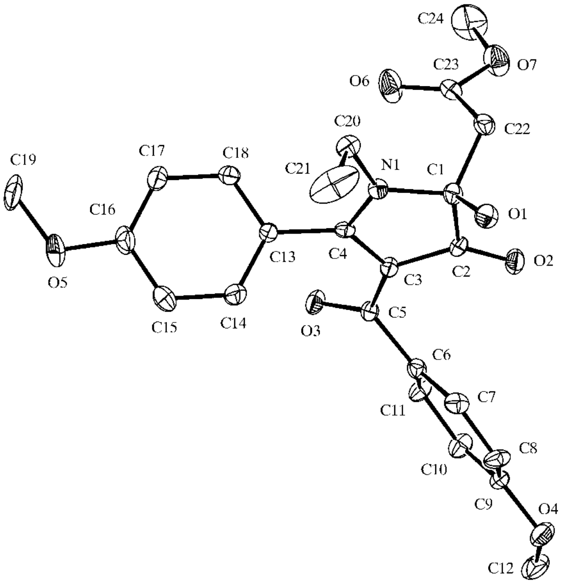


Figure 1
The molecular structure of molecule (I*a*), showing the atom-numbering scheme and with displacement ellipsoids drawn at the 20% probability level. H atoms have been omitted for clarity.

The two molecules in the asymmetric unit of (I), (I*a*) and (I*b*), are enantiomorphs of each other, as shown in Figs. 1 and 2 and *via* the torsion angles in Table 1. The bond lengths are almost identical in both molecules and are comparable with those in related structures (Allen *et al.*, 1987). In molecule (I*a*), the angles between planes *A* (N1/C1–C4), *B* (C6–C11) and *C* (C13–C18) are *A*/*B* = 57.4 (2), *A*/*C* = 60.0 (2) and *B*/*C* = 85.5 (2)°. In molecule (I*b*), the equivalent planes are *D* (N2/C25–C28), *E* (C30–C35) and *F* (C37–C42), and the angles between them are *D*/*E* = 69.4 (2), *D*/*F* = 54.7 (2) and *E*/*F* = 89.0 (2)°. These values are similar to those in 2(*R*)-hydroxy-4-(4-methoxybenzoyl)-2-methoxycarbonylmethyl-5-(4-methoxyphenyl)-1-(2-methylphenyl)-2,3-dihydro-1*H*-3-pyrrolone (Öztürk *et al.*, 2003). The angles between the planes of

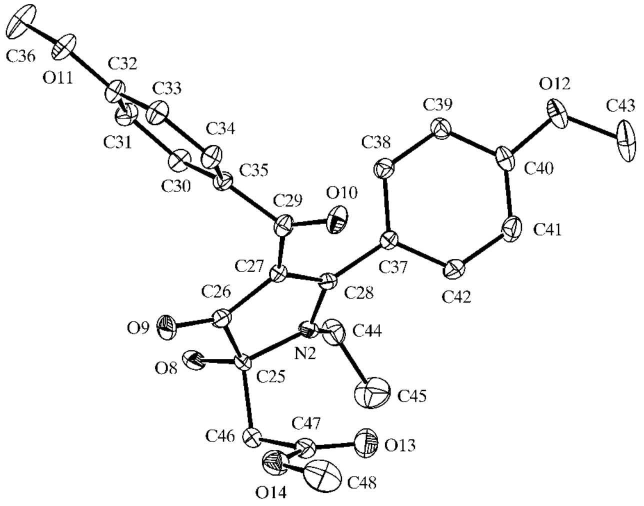


Figure 2
The molecular structure of molecule (I*b*), showing the atom-numbering scheme and displacement ellipsoids drawn at the 20% probability level. H atoms have been omitted for clarity.

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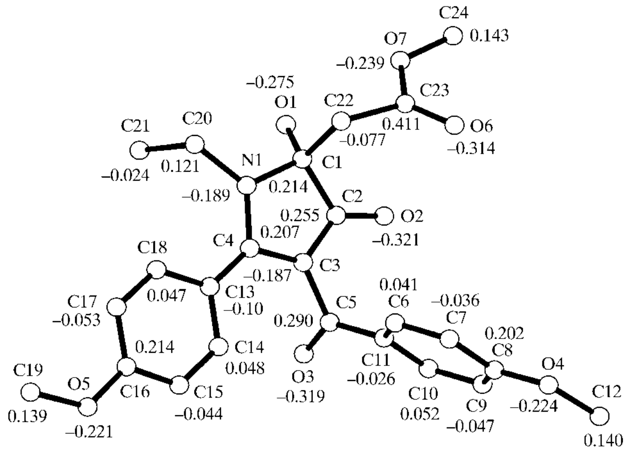

Figure 3
The CNDO-calculated charge distribution for (I).

similar rings of the two enantiomorphic molecules of (I) are not identical, with values of A/D = 41.6 (2), B/E = 68.2 (2) and C/F = 51.1 (2)°.

We have carried out CNDO (complete neglect of differential overlap) quantum mechanical calculations on (I), and the calculated charge distribution on the molecule of (I) in the gas phase is shown in Fig. 3. The dipole moment of (I) is ~17.51 $\times 10^{-30}$ Cm. The highest occupied and lowest unoccupied molecular orbital energy levels are −10.1370 and 1.1187 eV, respectively.

The crystal structure of (I) reveals a number of three-dimensional O—H···O and C—H···O short hydrogen-bond contacts, details of which are given in 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 ethylamine (70% in water; 0.322 g, 0.40 ml, 5 mmol) in benzene (25 ml) was refluxed for 30 min. The solid remaining after evaporation of the solvent was recrystallized from ethanol to give white crystals of (I) (yield: 1.54 g, 70%; m.p. 435 K). Solvents were dried by refluxing with the appropriate drying agent and were distilled before use. Melting points were determined on an Electrothermal 9200 apparatus and are uncorrected. Elemental analysis was performed with a Carlo–Erba 1108 Elemental Analyzer. The FT–IR spectrum was measured on a Jasco Plus Model 460 spectrometer. The ^{1}H and ^{13}C NMR spectra were obtained on a Gemini–Varian 200 MHz instrument. Spectroscopic analysis, FT–IR (KBr pellet, ν, cm^{-1}): 3212.34 (broad OH), 1733.69 (C12═O), 1678.69 (C8═O), 1674.39 (C4═O), 1251.58 (C12—O—C13); ^{1}H NMR (CDCl$_3$, p.p.m.): 1.095–1.167 (*s*, 3H, CH$_3$), 2.940–3.211 (*q*, 2H, CH$_2$C═O), 3.300–3.604 (*q*, 2H, NCH$_2$), 3.884, 3.780, 3.797 (*s*, 9H, OCH$_3$), 5.942 (*s*, 1H, OH), 6.731–7.700 (*m*, 8H, Ar-H); ^{13}C NMR (CDCl$_3$, p.p.m.): 17.474 (C7), 40.230 (C11), 42.588 (C6), 53.904, 57.216 (C13, C10, C9), 90.746 (C5), 112.392–164.865 (C═C, aromatic and aliphatic), 182.945 (C12═O), 188.924 (C8═O), 196.037 (C4═O). Analysis calculated for $C_{24}H_{25}NO_7$: C 65.59, N 3.19, H 5.73%; found: C 65.41, N 3.30, H 5.72%.

Crystal data

$C_{24}H_{25}NO_7$
M_r = 439.45
Triclinic, $P1$
a = 10.160 (5) Å
b = 10.785 (5) Å
c = 11.942 (5) Å
α = 66.369 (5)°
β = 75.928 (5)°
γ = 72.005 (5)°
V = 1129.4 (9) Å^3
Z = 2
D_x = 1.292 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2644 reflections
θ = 4.6–49.1°
μ = 0.10 mm^{-1}
T = 293 K
Block, colourless
0.52 × 0.26 × 0.20 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: refined on ΔF [Parkin *et al.*, 1995; cubic fit to $\sin(\theta)/\lambda$ with 24 parameters]
$T_{\min}$ = 0.952, $T_{\max}$ = 0.981
6035 measured reflections
6035 independent reflections
4369 reflections with $I > 2\sigma(I)$
$\theta_{\max}$ = 28.3°
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 11$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R(F)$ = 0.066
$wR(F^2)$ = 0.127
S = 1.11
6035 reflections
577 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0372P)^2 + 0.2621P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.002
$\Delta\rho_{\max}$ = 0.22 e Å^{-3}
$\Delta\rho_{\min}$ = −0.19 e Å^{-3}

Table 1
Selected torsion angles (°).

C4—N1—C1—O1	−121.1 (4)	C28—N2—C25—O8	127.2 (4)

Table 2
Hydrogen-bonding and short intra/intermolecular contact geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O10^{i}	0.82	2.04	2.844 (5)	168
O8—H8A···O3	0.82	1.95	2.754 (5)	169
C12—H12C···O1^{ii}	0.96	2.54	3.434 (7)	155
C19—H19A···O9^{iii}	0.96	2.48	3.211 (7)	133
C24—H24A···O4^{iv}	0.96	2.54	3.440 (10)	156
C44—H44A···O8	0.97	2.45	2.869 (6)	106
C45—H45B···O11^{v}	0.96	2.56	3.471 (10)	159

Symmetry codes: (i) $1+x, y, z$; (ii) $x, y-1, z$; (iii) $x, y, 1+z$; (iv) $x-1, 1+y, z$; (v) $x, y-1, 1+z$.

H atoms were introduced at calculated positions and refined as riding atoms, with bond lengths of 0.82 (O—H), 0.93 (C—H aromatic), 0.96 (CH$_3$) and 0.97 Å (CH$_2$), and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(parent atom) or $1.5U_{\rm eq}$(methyl C and hydroxyl O).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *PARST* (Nardelli, 1995) and *WinGX* (Farrugia, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TY1001). Services for accessing these data are described at the back of the journal.

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