Table 2. *Selected geometric parameters* (Å, °)

S—C23	1.669 (3)	C7—C8	1.384 (7)
O1—C14	1.216 (3)	C8—C9	1.380 (5)
O2—C13	1.418 (3)	C9—C10	1.399 (4)
N1—N2	1.376 (3)	C11—C12	1.521 (3)
N1—C1	1.285 (3)	C12—C13	1.534 (3)
N2—C23	1.354 (3)	C13—C14	1.519 (3)
N3—C23	1.320 (3)	C13—C22	1.519 (4)
C1—C2	1.505 (3)	C14—C15	1.475 (4)
C1—C10	1.469 (3)	C15—C16	1.396 (4)
C2—C3	1.522 (3)	C15—C20	1.394 (4)
C2—C11	1.528 (3)	C16—C17	1.371 (5)
C3—C4	1.498 (4)	C17—C18	1.366 (6)
C4—C5	1.502 (4)	C18—C19	1.364 (6)
C5—C6	1.381 (4)	C19—C20	1.394 (5)
C5—C10	1.395 (4)	C20—C21	1.509 (5)
C6—C7	1.363 (6)	C21—C22	1.509 (5)
N2—N1—C1	118.15 (18)	O2—C13—C14	110.03 (19)
N1—N2—C23	118.92 (19)	O2—C13—C22	110.83 (22)
N1—C1—C2	127.11 (21)	C12—C13—C14	106.93 (20)
N1—C1—C10	116.02 (20)	C12—C13—C22	113.80 (20)
C2—C1—C10	116.79 (20)	C14—C13—C22	107.40 (22)
C1—C2—C3	107.76 (19)	O1—C14—C13	119.66 (25)
C1—C2—C11	109.57 (18)	O1—C14—C15	122.67 (23)
C3—C2—C11	114.74 (18)	C13—C14—C15	117.66 (22)
C2—C3—C4	111.82 (21)	C14—C15—C16	119.22 (23)
C3—C4—C5	113.53 (23)	C14—C15—C20	120.4 (3)
C4—C5—C6	119.3 (3)	C16—C15—C20	120.4 (3)
C4—C5—C10	121.07 (23)	C15—C16—C17	120.2 (3)
C6—C5—C10	119.6 (3)	C16—C17—C18	119.5 (3)
C5—C6—C7	120.7 (3)	C17—C18—C19	121.1 (3)
C6—C7—C8	120.5 (3)	C18—C19—C20	121.1 (3)
C7—C8—C9	119.9 (3)	C15—C20—C19	117.6 (3)
C8—C9—C10	119.8 (3)	C15—C20—C21	121.0 (3)
C1—C10—C5	119.92 (22)	C19—C20—C21	121.4 (3)
C1—C10—C9	120.65 (24)	C20—C21—C22	113.78 (23)
C5—C10—C9	119.38 (25)	C13—C22—C21	112.54 (24)
C2—C11—C12	112.40 (19)	S—C23—N2	119.31 (18)
C11—C12—C13	114.60 (19)	S—C23—N3	124.06 (19)
O2—C13—C12	107.76 (21)	N2—C23—N3	116.63 (23)

H atoms were placed in calculated positions on the corresponding O atom (O—H = 0.85 Å), C atoms (C—H = 1.00 Å) and N atoms (N—H = 1.00 Å), and were not refined. The U_{iso} of each H atom was assigned to be U_{eq} + 0.01 of the corresponding O, N or C atom.

The ω-scan width was $(1.10 + 0.35\tan\theta)°$ with a θ-scan rate of 0.69–5.49° min^{-1}. The scan angle was extended 25% on each side of each peak for background measurement. Refinement was by full-matrix least-squares methods.

Data collection and cell refinement: Enraf–Nonius CAD-4 software. Data reduction: *DATRD2 NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER NRCVAX*. Program(s) used to refine structure: *LSTSQ NRCVAX*. Molecular graphics: *ORTEP NRCVAX* (Johnson, 1976). Software used to prepare material for publication: *TABLES NRCVAX*; *UTILITY NRCVAX*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and torsion angles have been deposited with the IUCr (Reference: CR1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N,*N*′-Carbonylbis(L-phenylalanine ethyl ester)

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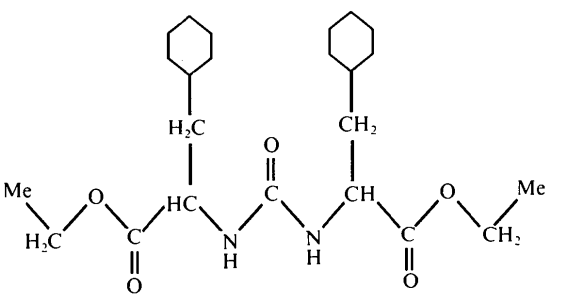
Abstract

The title compound, $C_{23}H_{28}N_2O_5$, is a symmetrical urea derivative with a twofold rotation axis coincident with the carbonyl group [C(1)=O(1)]. Its structure is characterized by a long planar region (14 Å) from which two phenyl rings project out at about 45°. In addition, two O atoms are positioned between the phenyl rings and are separated by about 7 Å. The orientation of the phenyl rings may explain the moderate inhibitory effect of this derivative.

Comment

The HIV-1 protease is a potential target for AIDS chemotherapy. It is an aspartyl protease, a class of enzymes which includes such physiologically important enzymes as pepsin and renin. This means that drugs designed to combat the HIV-1 protease must be very specific as well as potent. The *C*2 symmetrical active site of the HIV-1 protease is a key structural feature, which can be exploited in an attempt to design more specific inhibitors (Kempf *et al.*, 1990). The title compound (I) is the first of a series of symmetrical urea derivatives that are being investigated. Such studies should prove invaluable in rational drug design against this enzyme. Fig. 1 displays the title compound using an *ORTEP*II (Johnson, 1976) representation and Fig. 2 shows the crystal packing arrangement using *PLUTO* (Motherwell & Clegg, 1978).



(I)

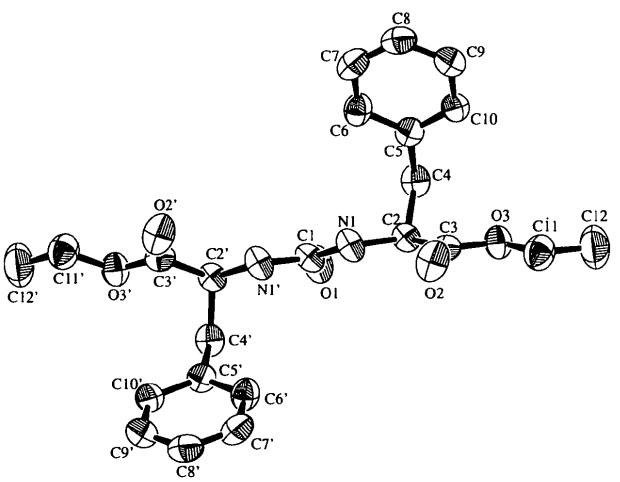


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the molecular structure of, *N*,*N*′-carbonylbis(L-phenylalanine ethyl ester) with coordinate atom labeling.

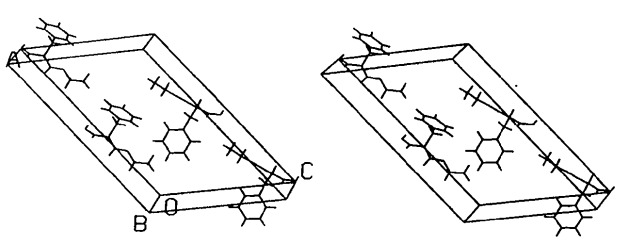


Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) stereo plot of the packing; the unit-cell axes and origin are labeled.

Experimental

The title compound was synthesized *via* a nucleophilic displacement by L-phenylalanine ethyl ester on *N*,*N*′-carbonyldiimidazole (Staab, 1957), m.p. 413–415 K. Crystals were obtained from a 7:3 acetonitrile/water solution by slow evaporation at 298 K.

Crystal data

$C_{23}H_{28}N_2O_5$
$M_r = 412.48$
Monoclinic
*C*2
$a = 20.093\ (4)$ Å
$b = 4.897\ (2)$ Å
$c = 13.808\ (2)$ Å
$\beta = 124.06\ (1)°$
$V = 1125.6$ Å^3
$Z = 2$
$D_x = 1.217$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 23 reflections
$\theta = 25.05–40.0°$
$\mu = 0.666$ mm^{-1}
$T = 296$ K
Rectangular
0.25 × 0.07 × 0.05 mm
Colorless

Data collection

AFC-6*R* diffractometer
ω–2θ scans
Absorption correction: empirical (North, Phillips & Mathews, 1968)
$T_{min} = 0.81$, $T_{max} = 1.00$
991 measured reflections
960 independent reflections
808 observed reflections [$I > 3\sigma(I)$]
$R_{int} = 0.031$
$\theta_{max} = 60.1°$
$h = 0 \rightarrow 22$
$k = 0 \rightarrow 5$
$l = -15 \rightarrow 12$
3 standard reflections monitored every 150 reflections
intensity variation: none

Refinement

Refinement on *F*
$R = 0.039$
$wR = 0.049$
$S = 1.68$
808 reflections
137 parameters
H-atom parameters not refined
$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: secondary (*MITHRIL*; Gilmore, 1984)
Extinction coefficient: 0.50077×10^{-4}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
O(1)	1	−0.3159	0	4.5 (2)
O(2)	0.8630 (2)	0.3597	0.0698 (3)	5.5 (1)
O(3)	0.8474 (1)	0.024 (1)	0.1627 (2)	4.4 (1)

N(1)	0.9703 (2)	0.085 (1)	0.0503 (3)	3.9 (1)
C(1)	1	−0.064 (1)	0	3.6 (2)
C(2)	0.9383 (2)	−0.050 (1)	0.1089 (3)	3.5 (1)
C(3)	0.8799 (2)	0.136 (1)	0.1111 (3)	3.5 (1)
C(4)	1.0044 (2)	−0.157 (1)	0.2324 (3)	3.8 (1)
C(5)	1.0548 (2)	0.063 (1)	0.3196 (3)	3.6 (1)
C(6)	1.1200 (2)	0.178 (1)	0.3248 (3)	4.4 (2)
C(7)	1.1664 (2)	0.377 (1)	0.4065 (4)	5.1 (2)
C(8)	1.1490 (2)	0.465 (1)	0.4835 (3)	5.2 (2)
C(9)	1.0853 (2)	0.355 (1)	0.4801 (3)	5.3 (2)
C(10)	1.0381 (2)	0.157 (1)	0.3983 (3)	4.3 (2)
C(11)	0.7894 (2)	0.185 (1)	0.1669 (4)	5.6 (2)
C(12)	0.7567 (3)	0.023 (2)	0.2218 (5)	7.9 (3)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(1)	1.234 (6)	C(4)—C(5)	1.508 (5)
O(2)—C(3)	1.195 (5)	C(5)—C(6)	1.393 (5)
O(3)—C(3)	1.324 (4)	C(5)—C(10)	1.383 (5)
O(3)—C(11)	1.437 (5)	C(6)—C(7)	1.381 (6)
N(1)—C(1)	1.354 (4)	C(7)—C(8)	1.362 (6)
N(1)—C(2)	1.443 (4)	C(8)—C(9)	1.363 (5)
C(2)—C(3)	1.498 (5)	C(9)—C(10)	1.386 (6)
C(2)—C(4)	1.553 (5)	C(11)—C(12)	1.481 (7)
C(3)—O(3)—C(11)	116.7 (3)	C(2)—C(4)—C(5)	114.4 (3)
C(1)—N(1)—C(2)	120.3 (3)	C(4)—C(5)—C(6)	121.6 (3)
O(1)—C(1)—N(1)	122.5 (2)	C(4)—C(5)—C(10)	121.0 (3)
N(1)—C(1)—N(1)	115.1 (5)	C(6)—C(5)—C(10)	117.4 (4)
N(1)—C(2)—C(3)	109.5 (3)	C(5)—C(6)—C(7)	120.8 (4)
N(1)—C(2)—C(4)	113.3 (3)	C(6)—C(7)—C(8)	120.6 (4)
C(3)—C(2)—C(4)	112.4 (3)	C(7)—C(8)—C(9)	119.6 (4)
O(2)—C(3)—O(3)	122.9 (4)	C(8)—C(9)—C(10)	120.4 (4)
O(2)—C(3)—C(2)	124.3 (3)	C(5)—C(10)—C(9)	121.0 (4)
O(3)—C(3)—C(2)	112.8 (3)	O(3)—C(11)—C(12)	109.1 (5)

The data were corrected for Lp effects. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984). The origin was defined by fixing the *y*-axis position of O(1). H atoms were generated using optimum bonding geometry and included in F_c, but their displacement parameters and positions were not refined; non-H atoms were refined anisotropically. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$. Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964). This compound has two chiral centres, therefore we collected all Friedel pairs and switched the signs of the coordinates in an attempt to determine the absolute configuration. We were unable to distinguish between the two enantiomers with our data; we expect that the chirality of the starting compound was maintained under the reaction conditions. All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1985).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: BR1060). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Ethoxy-6-[1-(4-methoxyphenyl)ethyl]-1,3-benzodioxole

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Abstract

The title compound, $C_{18}H_{20}O_4$, is structurally similar to podophyllotoxin. It contains a fused dioxole and phenyl ring system and an unfused phenyl ring. However, this compound lacks the cyclohexyl and lactone rings which complete the four-membered ring system of podophyllotoxin. In addition, the unfused phenyl ring in podophyllotoxin contains three methoxy groups in the *para* and *meta* positions, whereas the title compound contains only a methoxy group in the *para* position.

Comment

The title compound (I) belongs to a series of 6-benzyl-1,3-benzodioxole derivatives which have podophyllotoxin-like antimitotic activity (Batra, Jurd & Hamel 1985). These derivatives are structurally similar to podophyllotoxin, which is characterized by a four-membered fused ring system and an unfused phenyl ring, and may be used to probe the structure–activity relationships of podophyllotoxin. A recent crystal structure determination for a 6-benzyl-1,3-benzodioxole derivative has shown that an intact fused ring system may not be necessary for antimitotic activity (Sicheri, Derry, Gupta & Yang,