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Key indicators

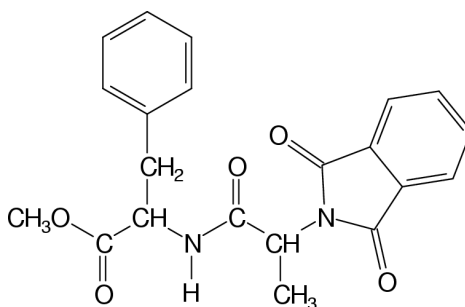
Single-crystal X-ray study
 $T = 149\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.038
 wR factor = 0.086
Data-to-parameter ratio = 6.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.***N*-Phthaloyl-L-alanyl-L-phenylalanine methyl ester**

The title compound, $\text{C}_{21}\text{H}_{20}\text{N}_2\text{O}_5$, is a terminally protected dipeptide. The backbone adopts an extended conformation, with the phthaloyl group orthogonal to the plane of the *trans* peptide link. The phenylalanine side chain adopts the g^- conformation. In the crystal lattice, the molecules are stacked with a strong intermolecular hydrogen bond between the H atom of the amide link and the amide carbonyl group of a neighbour.

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Comment

The crystal packing of small unprotected peptides is largely dominated by electrostatic and hydrogen bonding interactions of the terminal functional groups (Görbitz & Gundersen, 1996). Small protected peptides, which are less likely to form hydrogen bonds, are thus useful models for the study of relatively weak interactions. The title compound, (I), was prepared from the corresponding protected amino acids, phthaloylalanine and phenylalanine methyl ester. The conformation of the dipeptide and the numbering scheme are shown in Fig. 1. The peptide link is in a *trans* configuration and shows a slight deviation from planarity [$\omega_1 = -174.2(2)^\circ$]. The backbone adopts an extended conformation with torsion angles $\Psi_1 = -146.9(2)$, $\varphi_2 = 153.8(2)^\circ$ and $\Psi_2 = 166.5(2)^\circ$. The phthaloyl group is approximately perpendicular [$\varphi_1^1 = 77.6(3)^\circ$, $\varphi_1^2 = -93.4(3)^\circ$] to the plane of the peptide link. The side chain of phenylalanine adopts the g^- conformation [$\chi_2^1 = -64.4(3)^\circ$], which is most frequently found for this residue (Ashida *et al.*, 1987).



(I)

The molecules stack in a head-to-head fashion along the *b* axis (Fig. 2), with a repeat distance between individual molecules in the stack of 4.8481(2) Å, the length of the unit-cell edge in the *b* direction. Packing is stabilized by an intermolecular hydrogen bond formed between the amide H2 atom of the peptide and carbonyl atom O3 of an adjacent molecule within the stack; $\text{N2} \cdots \text{O3}$ 2.983(3) Å, $\text{N2}-\text{H2} \cdots \text{O3}$ 164° for symmetry operation $x, 1+y, z$.

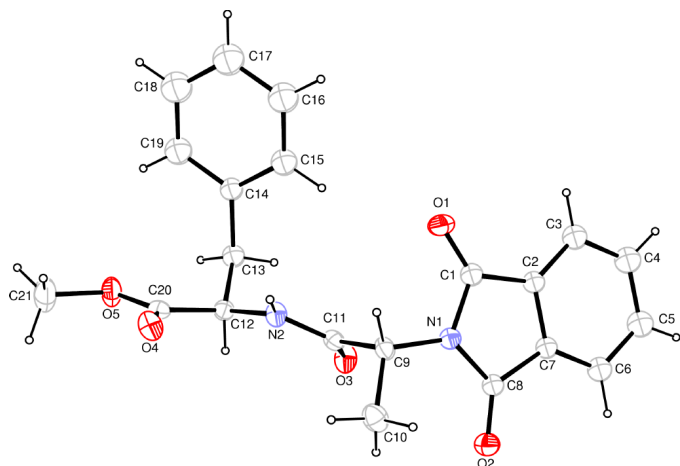


Figure 1
Perspective drawing (ORTEP-3; Farrugia, 1997) of the molecule of (I), showing the atom-numbering scheme, with displacement ellipsoids drawn at the 50% probability level.

Experimental

N-Phthaloyl-L-alanyl-L-phenylalanine methyl ester was synthesized by the coupling of *N*-phthaloyl-L-alanine acid chloride with L-phenylalanine methyl ester. Crystals of the dipeptide were obtained by slow evaporation of a solution of the dipeptide in isopropylether/methanol/ water (1:1:2). If racemic alanine is used instead of the L-isomer then a mixture of diastereomers is obtained, from which the title compound crystallizes preferentially. ^1H NMR (200 MHz) in CDCl_3 , δ (p.p.m.): 1.66 (3H, *d*, 7.4 Hz), 3.11 (2H, AMX system $\delta_A = 3.13$, $\delta_M = 3.08$, $J_{AM} = 13.8$ Hz, $J_{AX} = J_{MX} = 5.8$ Hz), 3.82 (3H, *s*), 4.86 (1H, *m*), 4.90 (1H, *q*, 7.4 Hz), 6.37 (1H, *d*, 7.2 Hz), 7.07 (5H, *m*) 7.81 (4H, *m*).

Crystal data

$\text{C}_{21}\text{H}_{20}\text{N}_2\text{O}_5$	$D_x = 1.335 \text{ Mg m}^{-3}$
$M_r = 380.39$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 3956 reflections
$a = 10.9627(5) \text{ \AA}$	$\theta = 1.1\text{--}24.7^\circ$
$b = 4.8481(2) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 18.5150(9) \text{ \AA}$	$T = 149(2) \text{ K}$
$\beta = 105.991(1)^\circ$	Needle, colourless
$V = 945.96(7) \text{ \AA}^3$	$0.65 \times 0.10 \times 0.05 \text{ mm}$
$Z = 2$	

Data collection

Bruker CCD area-detector diffractometer	1635 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.055$
Absorption correction: none	$\theta_{\text{max}} = 24.7^\circ$
3956 measured reflections	$h = -11 \rightarrow 12$
1765 independent reflections	$k = -5 \rightarrow 2$
	$l = -21 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0271P)^2 + 0.4579P]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.086$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.13$	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
1765 reflections	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$
255 parameters	
H-atom parameters constrained	

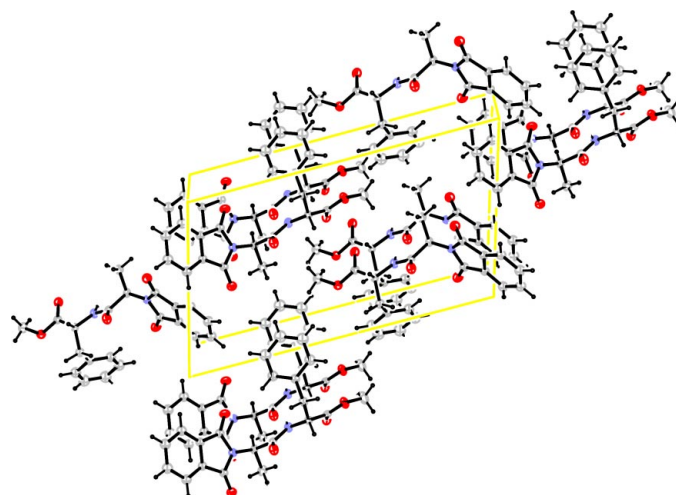


Figure 2
A view of the unit cell of (I) (ORTEP-3; Farrugia, 1997).

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
$\text{N2--H2}\cdots\text{O3}^i$	0.88	2.13	2.983 (3)	164

Symmetry code: (i) $x, 1 + y, z$.

The absolute configuration could not be determined reliably and the corresponding Flack parameter is not quoted. Freidel pairs were averaged, as the molecule contains no atoms heavier than O. The structure shown in Fig. 1 is consistent with the preparation from L-phenylalanine. H atoms were included in calculated positions using a riding model.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997) and *TITAN2000* (Hunter & Simpson, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997) and *TITAN2000*; molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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