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L-Tyrosine *n*-butyl ester

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.133
Data-to-parameter ratio = 8.7

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

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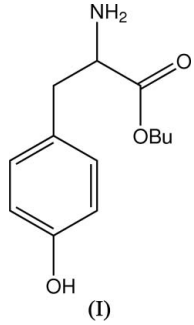
The title compound, $C_{13}H_{19}NO_3$, adopts a folded conformation. Molecules are connected into a three-dimensional array *via* O—H···N and N—H···O hydrogen bonding.

Comment

L-Tyrosine *n*-butyl ester was initially investigated for its antibacterial and antifungal activities (Allen *et al.*, 1960), but more recent interest has revolved around its applications in materials science (Suzuki *et al.*, 2001; Ye *et al.*, 2004). It is the former, *i.e.* potential pharmaceutical applications, that motivated the present study of the title ester, (I).



(I)

The molecular structure of (I) (Fig. 1 and Table 1) shows the expected geometric parameters (Allen *et al.*, 1987). The molecule adopts a somewhat folded or U-shaped conformation as evidenced in the C1/C2/C3/C4 torsion angle of 55.6 (4)°. Despite the adoption of this conformation, there is no evidence for significant intramolecular C—H···π interactions. In terms of geometric parameters and overall conformation, the structure of (I) resembles that of the ethyl analogue (Pieret *et al.*, 1970).

Hydrogen bonding plays a significant role in stabilizing the crystal structure; see Table 2 for geometric parameters and symmetry operations. The most prominent link occurs between the phenol H and the amine N atoms, to form chains along the *c* axis. The chains are linked into undulating layers *via* N1—H2n···O3 interactions. A three-dimensional array is then generated *via* a bifurcated hydrogen bond formed between N1/H1n and O1 and O3 atoms derived from two different molecules of two chains.

Experimental

L-Tyrosine *n*-butyl ester was prepared by reacting equimolar L-tyrosine and thionyl chloride in *n*-butanol. Thionyl chloride (11.9 g, 0.1 mol) was added dropwise to *n*-butanol (200 ml) at 273 K and L-tyrosine (18.1 g, 0.1 mol) was then added. The mixture was heated to 323 K and stirred for 30 min, then cooled to ambient temperature and

stirred for a further 12 h. Volatiles were removed in a vacuum and the residue was dissolved in water (150 ml). The pH of the solution was adjusted to 8.5 with 2 mol l^{-1} NaOH to precipitate L-tyrosine *n*-butyl ester (I, 20.4 g, yield 86%). Compound (I) (0.1 g) was dissolved in ethanol (20 ml) and single crystals were obtained by slow evaporation.

Crystal data

$C_{13}H_{19}NO_3$
M_r = 237.29
Orthorhombic, $P2_12_12_1$
a = 5.5380 (11) Å
b = 14.508 (3) Å
c = 15.908 (3) Å
V = 1278.1 (4) Å^3
Z = 4
D_x = 1.233 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 650 reflections
θ = 5.6–24.8°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colourless
0.40 × 0.20 × 0.20 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
3802 measured reflections
1341 independent reflections
1111 reflections with $I > \sigma(I)$
R_{int} = 0.027
θ_{max} = 25.0°
$h = 0 \rightarrow 6$
$k = -6 \rightarrow 17$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.133
S = 1.08
1341 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0813P)^2 + 0.1625P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.029 (6)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.209 (4)	O3—C7	1.362 (4)
O2—C1	1.314 (4)	N1—C2	1.458 (4)
O2—C10	1.456 (4)		
C1—O2—C10	118.8 (3)	C7—O3—H3*O*	113.8

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3o···N1^{i}	0.82	1.98	2.793 (3)	169
N1—H1n···O1^{ii}	0.89	2.49	3.183 (4)	136
N1—H1n···O3^{iii}	0.89	2.52	3.273 (3)	143
N1—H2n···O3^{iv}	0.89	2.53	3.371 (3)	158

Symmetry codes: (i) $-x+\frac{3}{2}, -y, z-\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+\frac{1}{2}, -y, z+\frac{1}{2}$.

All H atoms were allowed to ride on their parent atoms at distances of 0.93 (aromatic H), 0.96 (methyl H), 0.97 (methylene H), 0.98 (methine H), 0.82 (O—H) and 0.89 Å (N—H), and with U_{iso}(H) values of 1.2U_{eq}(parent atom) for aromatic, methine and methylene H atoms, and 1.5U_{eq}(parent atom) for methyl, phenol and amine H atoms. In the absence of significant anomalous scattering effects,-Friedel pairs were averaged in the final refinement.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

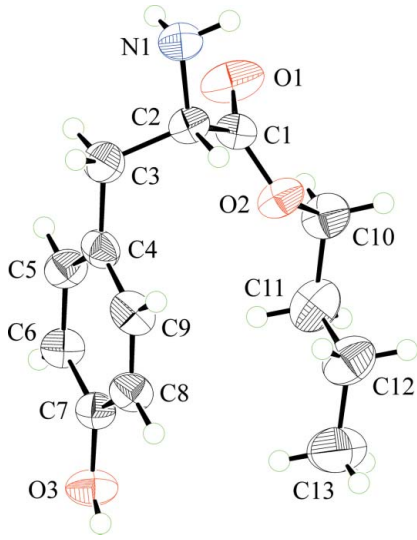


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

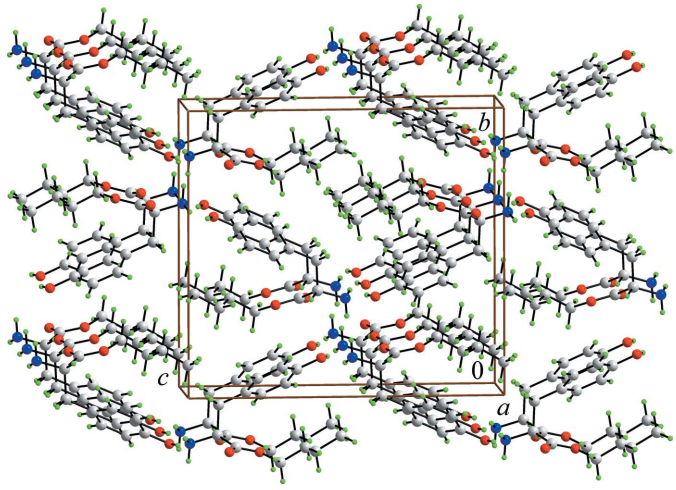


Figure 2
Packing diagram of (I) (Crystal Impact, 2002). Colour code: O (red), N (blue), C (grey) and H (green).

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