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[2-(*tert*-Butylcarbonylmethylamino)-phenyl]acetic acid

Nicolas Raynal, Muriel Amblard, Jean Martinez and Marc Rolland*

Laboratoire des Aminoacides, Peptides et Protéines, Université Montpellier I et II, Faculté de Pharmacie, UMR CNRS 5810, 15 Avenue Charles Flahault, 34060 Montpellier Cedex 2, France

Correspondence e-mail: rolland@colombes.pharma.univ-montp1.fr

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.050
wR factor = 0.128
Data-to-parameter ratio = 15.1

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

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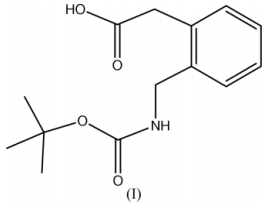
We present here the crystal structure of the title compound, $C_{14}H_{19}NO_4$. It contains O···H—O and N···H—O hydrogen bonds, connecting the molecules into centrosymmetric dimers and into infinite chains.

Comment

Peptides and proteins are important targets for drug discovery, but their metabolic instability and poor oral bio-availability frequently limit their use as therapeutic agents. A major effort has been devoted to the development of peptidomimetics containing non-peptidic scaffolds (Olson *et al.*, 1993; Fairlie *et al.*, 1995). As part of a general programme towards the synthesis of pseudopeptide hormone agonists or antagonists, we selected several compounds in the literature that could be used as dipeptide mimetics and present here the crystal structure of such a compound.



(I)

The title compound, (I), is an aromatic amino-acid derivative with a free carboxylic acid group. In order to use this compound as a dipeptide mimetic in the synthesis of helical peptides, the amine function of (2-aminomethylphenyl)acetic acid was protected with a *tert*-butyloxycarbonyl group. The O1···H2—O2 hydrogen bond between the O atoms of the carboxylic acid group forms a centrosymetric acid dimer. This gives almost equal distances to the C1—O1 and C1—O2 bonds, with similar O1—C1—C2 and O2—C1—C2 angles. This appears to be an average of classical distances and angles for atoms in carboxylic acid groups. The difficulty in differentiating the O1 and O2 atoms in the model led us to refine the COOH group with a half hydrogen on each. An intermolecular N1—H1*A*···O3 hydrogen bond connects the translated molecules into an infinite chain along the *b* axis. Both hydrogen bonds seem essential for the crystal packing; we could not detect any interplanar interactions, the distances between chains being close to the sums of van der Waals radii. The benzene ring is almost perfectly planar [maximum deviation from the least-squares plane is 0.0041 (12) Å].

Experimental

Compound (I) was synthesized according to the literature (Zhilian & Pelletier, 1998). Crystals appropriate for data collection were obtained by slow evaporation from diethyl ether solution at 277 K.

Crystal data

$C_{14}H_{19}NO_4$	D_x = 1.262 Mg m^{-3}
M_r = 265.31	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 8934 reflections
a = 12.6392 (6) Å	θ = 1.0–26.2°
b = 5.1470 (2) Å	μ = 0.09 mm^{-1}
c = 22.5450 (1) Å	T = 293 (2) K
β = 107.744 (3)°	Prism, colourless
V = 1396.85 (9) Å^3	0.40 × 0.30 × 0.20 mm
Z = 4	

Data collection

Nonius KappaCCD area-detector diffractometer	R_{int} = 0.033
	θ_{max} = 26.2°
φ scans	$h = -15 \rightarrow 15$
8934 measured reflections	$k = -6 \rightarrow 6$
2702 independent reflections	$l = -27 \rightarrow 26$
2362 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.4704P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	
$wR(F^2)$ = 0.128	
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
2702 reflections	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
179 parameters	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.253 (2)	C2—C3	1.506 (2)
O2—C1	1.248 (2)	C3—C8	1.398 (2)
N1—C9	1.4549 (19)	C8—C9	1.505 (2)
C1—C2	1.503 (2)		
O2—C1—O1	123.55 (16)	O1—C1—C2	117.21 (15)
O2—C1—C2	119.14 (15)		
O2—C1—C2—C3	35.3 (2)	C2—C3—C8—C9	2.2 (2)
O1—C1—C2—C3	−148.17 (16)	C3—C8—C9—N1	−63.65 (19)
C1—C2—C3—C8	95.20 (18)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.86	2.668 (2)	166
O2—H2···O1^{i}	0.82	1.87	2.668 (2)	162
N1—H1A···O3^{iii}	0.86	2.18	3.004 (2)	160

Symmetry codes: (i) $2-x, 2-y, -z$; (iii) $x, 1+y, z$.

H atoms were placed at calculated positions and treated as riding atoms (O—H 0.82 Å; C—H 0.96, 0.97 and 0.98 Å), with a displacement parameter U_{iso} set equal to 1.2 (OH, CH and CH_2) or 1.5 (CH_3) times U_{eq} of the parent atom. Although the atom C12 displays an elongated ellipsoid axis, due to the short-distance interaction between the methyl and the phenyl ring of a symmetry-related (x, $\frac{3}{2}-y$, $\frac{1}{2}+z$) molecule, no disordered model with chemically sensible disorder model could be defined.

Data collection: *KappaCCD Softwar*e (Nonius, 1997); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and *PLATON* (Spek, 2001); software used to prepare material for publication: *maXus* (Mackay *et al.*, 1999).

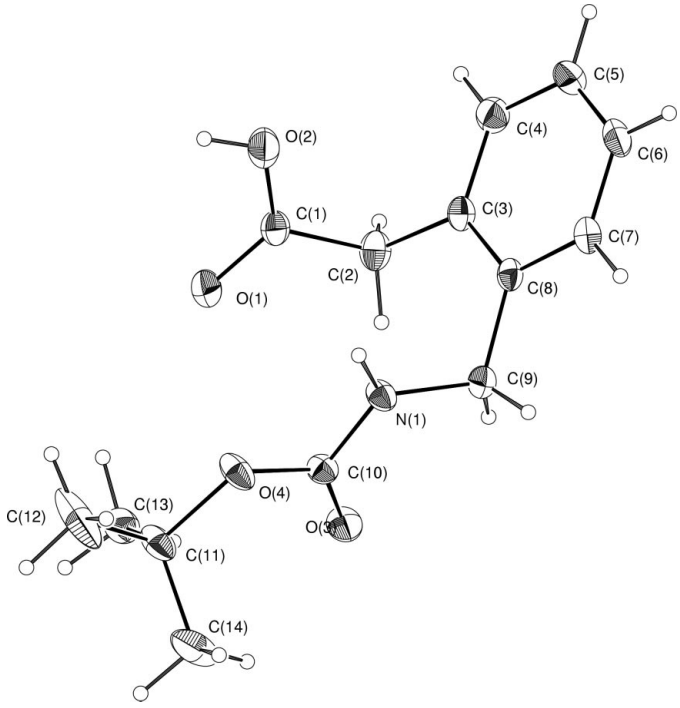


Figure 1
An *ORTEP*III (Burnett & Johnson, 1996) view of the molecular structure of (I), showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as circles of arbitrary radius.

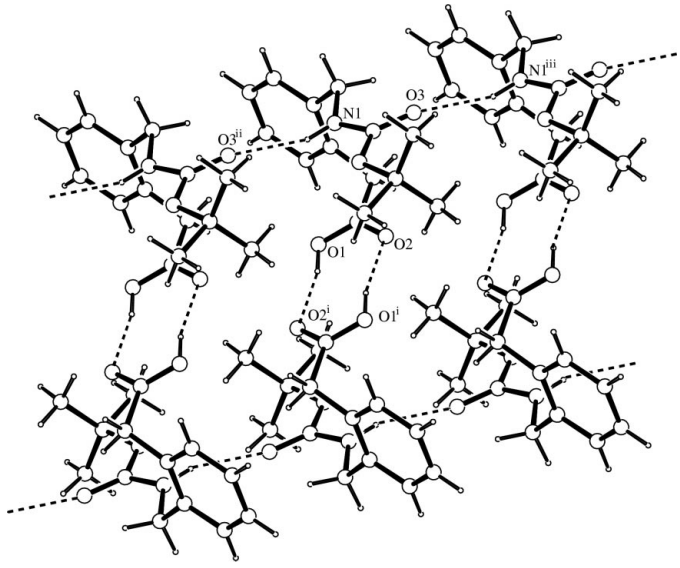


Figure 2
A *PLATON* (Spek, 2001) view of the hydrogen-bonded motif of (I). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $2-x$, $2-y$, $-z$; (ii) x, $y-1$, z; (iii) x, $y+1$, z.]

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