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(2*S*)-Methyl 1-[(2*R*)-3-(benzylsulfanyl)-2-(*tert*-butoxycarbonylamino)propanoyl]pyrrolidine-2-carboxylate

Minna T. Räisänen,* Martti Klinga, Markku Leskelä and Timo Repo

University of Helsinki, Department of Chemistry, Laboratory of Inorganic Chemistry, FIN-00014 Helsinki, Finland

Correspondence e-mail:
minna.t.raisanen@helsinki.fi

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In the title compound, $C_{21}H_{30}N_2O_5S$, all bond lengths and angles are within normal ranges. Several hydrogen bonds stabilize its molecular packing. The secondary structure of the title compound is best described as a β-pleated sheet-type arrangement.

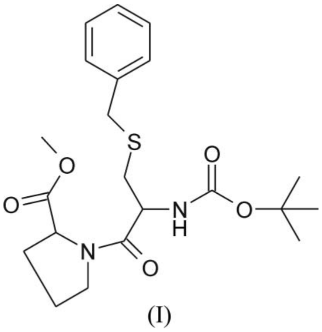
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.048
wR factor = 0.104
Data-to-parameter ratio = 18.9

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

Comment

The title compound, (I) (Fig. 1), was synthesized as an intermediate for L-cysteine-L-proline methyl ester. All bond lengths and angles are within normal ranges (Kashino *et al.*, 1974; Oku *et al.*, 2003). The observed distance of 3.853 (4) Å between the two C_α atoms (C9 and C14) is typical of the *trans* configuration, whereas the ω angle (C14—N1—C10—C9) of −169.4 (2)° is considerably distorted from the expected 180°. Other peptide backbone torsion angles, φ (C17—N2—C9—C10) and ψ (N2—C9—C10—N1), are −147.0 (2) and 173.2 (2)°, respectively. Hydrogen bonds are commonly found in peptide compounds (Kashino *et al.*, 1974; Oku *et al.*, 2003) and the structure of the title compound is stabilized by C—H···O, C—H···S and NC—H···O hydrogen bonds (Table 1). The secondary structure of the title compound can be best described as a β-pleated sheet-type arrangement.



(I)

Experimental

The *N*- and *S*-protected dipeptide *N*-*tert*-butoxycarbonyl-*S*-benzyl-L-cysteine-L-proline methyl ester, (I), was prepared in a solution phase according to literature procedures. *S*-Benzyl-L-cysteine, (1) (Koerber-Ple & Massiot, 1995), and L-proline methyl ester hydrochloride, (2) (Ryono & Weller, 1987), were prepared first, followed by L-proline methyl ester, (3) (Miles *et al.*, 1985). *N*-*tert*-Butoxycarbonyl-*S*-benzyl-L-cysteine, (4), was synthesized from (1) by protecting the amine group (Koerber-Ple & Massiot, 1995). Finally, (I) was obtained as white microcrystals by coupling (3) and (4) with *N*,*N*′-dicyclohexylcarbodiimide (Ondetti & Condon, 1979). Colourless crystals of (I) suitable for X-ray analysis were obtained from CH_2Cl_2.

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Crystal data

$C_{21}H_{30}N_2O_5S$
$M_r = 422.53$
Monoclinic, $P2_1$
$a = 6.013$ (1) Å
$b = 15.597$ (3) Å
$c = 12.347$ (3) Å
$\beta = 103.34$ (3)°
$V = 1126.7$ (4) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.18$ mm^{-1}
$T = 173$ (2) K
0.40 × 0.40 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\rm min} = 0.933$, $T_{\rm max} = 0.989$
15659 measured reflections
5015 independent reflections
3347 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.058$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.104$
$S = 1.03$
5015 reflections
266 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max} = 0.30$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.52$ e Å^{-3}
Absolute structure: Flack (1983), 2345 Friedel pairs
Flack parameter: −0.06 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H22$\cdots$O1	0.860 (17)	2.20 (3)	2.617 (3)	109 (2)
C12—H12$B\cdots$S1$^{\rm i}$	0.99	2.89	3.689 (3)	138
C7—H7$B\cdots$O1$^{\rm ii}$	0.99	2.48	3.319 (4)	142
C8—H8$B\cdots$O1$^{\rm ii}$	0.99	2.53	3.353 (4)	140
C21—H21$B\cdots$O2$^{\rm iii}$	0.98	2.58	3.405 (4)	142

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

The H atom connected to N was located in a difference map and refined isotropically; $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$. Other H atoms were introduced in their calculated positions [C—H = 0.95, 0.98, 0.99 or 1.00 Å, $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms] and refined with fixed geometry with respect to their carrier atoms.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DIRAX* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

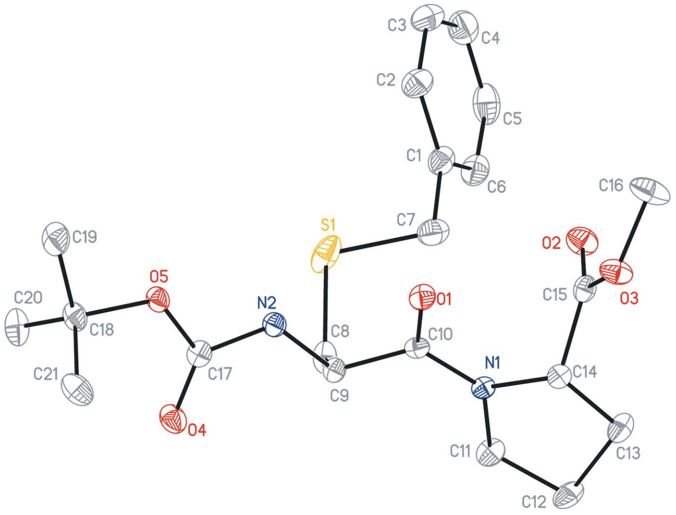

Figure 1
The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

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