

C6'	-0.2950 (2)	0.7931 (3)	0.2687 (2)	0.0392 (13)
C7'	-0.2019 (2)	0.7731 (3)	0.25413 (14)	0.0376 (13)
C8'	-0.1350 (2)	0.7929 (3)	0.29075 (14)	0.0276 (11)
C9'	-0.1506 (2)	0.8839 (3)	0.31147 (13)	0.0279 (11)
C10'	-0.2484 (2)	0.9073 (3)	0.32456 (14)	0.0307 (11)
C11'	-0.0845 (2)	0.9008 (3)	0.34968 (15)	0.0383 (13)
C12'	0.0071 (2)	0.8745 (3)	0.33793 (14)	0.0334 (12)
C13'	0.0283 (2)	0.8263 (3)	0.30278 (14)	0.0276 (11)
C14'	-0.0389 (2)	0.7891 (3)	0.26950 (14)	0.0293 (11)
C15'	-0.0145 (2)	0.6941 (3)	0.25739 (15)	0.0352 (12)
C16'	0.0821 (2)	0.6781 (3)	0.24660 (15)	0.0339 (12)
C17'	0.1408 (2)	0.7080 (3)	0.28619 (15)	0.0332 (12)
C18'	0.1241 (3)	0.8056 (3)	0.29322 (15)	0.0330 (12)
C19'	0.1851 (3)	0.8670 (3)	0.2887 (2)	0.0365 (13)
C20'	0.2810 (3)	0.8451 (3)	0.2826 (2)	0.0457 (14)
C21'	0.3000 (3)	0.7508 (3)	0.2967 (2)	0.0496 (15)
C22'	0.2384 (2)	0.6890 (3)	0.2745 (2)	0.0493 (15)
C23'	-0.4402 (2)	0.9133 (3)	0.23454 (14)	0.0374 (13)
C24'	-0.4632 (2)	0.8684 (3)	0.31232 (14)	0.0378 (13)
C25'	-0.2815 (2)	0.8606 (3)	0.36764 (14)	0.0354 (12)
C26'	-0.1413 (2)	0.7221 (3)	0.32800 (14)	0.0386 (12)
C27'	-0.0308 (2)	0.8457 (3)	0.22630 (14)	0.0384 (13)
C28'	0.1213 (3)	0.6599 (3)	0.3297 (2)	0.0398 (13)
C29'	0.1659 (3)	0.9643 (3)	0.2888 (2)	0.0509 (15)
C30'	0.3118 (3)	0.8619 (4)	0.2338 (2)	0.070 (2)
C31'	0.0755 (3)	0.5298 (3)	0.3624 (2)	0.065 (2)
C32'	-0.4046 (4)	0.9468 (4)	0.1601 (2)	0.077 (2)
C33'	-0.3429 (3)	0.9929 (4)	0.1307 (2)	0.094 (2)
C34'	-0.5292 (3)	1.0985 (3)	0.2709 (2)	0.0473 (14)
C35'	-0.6248 (2)	1.1081 (3)	0.2821 (2)	0.055 (2)
C36'	-0.3691 (3)	1.1595 (3)	0.3853 (2)	0.0371 (13)
C37'	-0.3754 (3)	1.2567 (3)	0.3868 (2)	0.0533 (15)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Molecule 1		Molecule 2	
O1—C36	1.341 (5)	O1'—C36'	1.362 (5)
O1—C2	1.458 (4)	O1'—C2'	1.463 (5)
O2—C36	1.187 (5)	O2'—C36'	1.211 (5)
O3—C34	1.364 (5)	O3'—C34'	1.365 (5)
O3—C3	1.458 (5)	O3'—C3'	1.463 (4)
O4—C34	1.196 (5)	O4'—C34'	1.177 (5)
O5—C32	1.331 (5)	O5'—C32'	1.294 (5)
O5—C23	1.429 (5)	O5'—C23'	1.438 (5)
O6—C32	1.220 (5)	O6'—C32'	1.196 (5)
O7—C28	1.370 (5)	O7'—C28'	1.345 (5)
O7—C31	1.445 (5)	O7'—C31'	1.420 (5)
O8—C28	1.199 (5)	O8'—C28'	1.217 (5)
C12—C13	1.312 (5)	C12'—C13'	1.321 (5)
C18—C19	1.321 (5)	C18'—C19'	1.332 (5)
C13—C12—C11	126.5 (4)	C13'—C12'—C11'	124.4 (4)
C12—C13—C18	120.2 (4)	C12'—C13'—C18'	120.0 (4)
C12—C13—C14	122.3 (4)	C12'—C13'—C14'	123.8 (4)
C18—C13—C14	117.5 (4)	C18'—C13'—C14'	116.3 (4)
C19—C18—C13	124.0 (4)	C19'—C18'—C13'	122.7 (4)
C19—C18—C17	122.4 (4)	C19'—C18'—C17'	124.3 (4)
C13—C18—C17	113.4 (3)	C13'—C18'—C17'	112.9 (4)
C18—C19—C20	123.4 (4)	C18'—C19'—C20'	121.9 (5)
C18—C19—C29	123.2 (4)	C18'—C19'—C29'	124.2 (4)
C20—C19—C29	113.4 (4)	C20'—C19'—C29'	113.8 (4)

Absence of crystal decay in the X-ray beam was confirmed by checking equivalent reflections at the beginning and end of the data collection, which lasted about 8 h. The two largest (Δ/σ) values of 1.429 and 0.382 are associated with the methyl H atoms and indicate that these atoms, attached to C33' and C31, may be in free rotation. All non-H atoms were given anisotropic displacement parameters; the methylene and methyl H atoms were given common isotropic displacement parameters and were allowed to ride on their attached C atoms.

The structure was solved using *SIR92* (Altomare *et al.*, 1994) in the *CRYSTAN* (Edwards, Gilmore, Mackay & Stewart, 1995) system. Refinement was completed with *SHELXL93* (Sheldrick, 1993) and the molecular plot was obtained using *SNOOPI* (Davies, 1983).

We thank the SERC X-ray Crystallographic Service at The University of Wales, Cardiff, for collecting the data, and Dr C. J. Gilmore for assistance in solving the structure.

Lists of structure factors, torsion angles, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1010). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-(Phenylmethylthio)benzaldehyde

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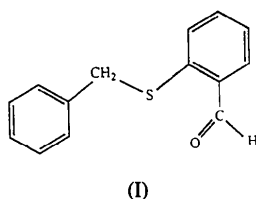
(Received 4 January 1995; accepted 9 May 1995)

Abstract

In the title compound, $\text{C}_{14}\text{H}_{12}\text{OS}$, the two planar benzaldehyde and benzyl groups are inclined at $77.7(1)^\circ$. The torsion angle about the central C—S bond is $174.9(2)^\circ$. The molecules are held together in the crystal by van der Waals interactions.

Comment

As part of our program to design and study chelating ligands containing both O and S donors (Wong, Lee & Cheung, 1995), we were interested in the coordination properties of the title compound, (I).



An ORTEPII plot (Johnson, 1976) of the molecule is shown in Fig. 1. The benzaldehyde moiety is essentially planar (maximum deviation 0.035 Å). It is also coplanar with the central C—S bond (maximum out-of-plane distance 0.237 Å), while the phenyl ring is at 77.7° to this plane. The torsion angle about the central C—S bond is 174.9(2)°. The non-bonding O···S distance is 2.811(2) Å, which indicates that a weak interaction is present. This distance is comparable with those found in 2,2'-thiodibenzoyl chloride [2.728(4) and 2.941(5) Å] (Parkanyi, Kálmán, Kucsman & Kapovits, 1989) and methyl 2-(methylthio)benzoate [2.721(4) Å] (Kucsman, Kapovits, Parkanyi, Argay & Kálmán, 1984) in which weak intramolecular O···S interactions have been observed.

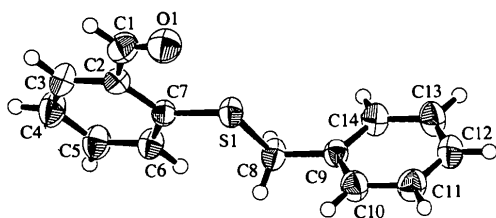


Fig. 1. An ORTEPII (Johnson, 1976) drawing of the title molecule with 50% probability ellipsoids and the atom-numbering scheme.

Experimental

2-(Phenylmethylthio)benzaldehyde was prepared according to the literature method (Nation, Taylor & Wainwright, 1992). Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol/petroleum ether (b.p. 313–333 K) mixture over a period of 2 d.

Crystal data

C₁₄H₁₂OS

M_r = 228.31

Monoclinic

*P*2₁/*n*

a = 5.425(1) Å

b = 17.618(2) Å

c = 12.173(2) Å

β = 97.07(2)°

V = 1154.6 Å³

Z = 4

D_x = 1.313 Mg m⁻³

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 10–15°

μ = 0.243 mm⁻¹

T = 293 K

Needle

0.42 × 0.14 × 0.12 mm

Colourless

Data collection

Nonius CAD-4 diffractometer

ω–2θ scans

Absorption correction:

none

2603 measured reflections

2354 independent reflections

1640 observed reflections

[*I* > 3σ(*I*)]

*R*_{int} = 0.012

θ_{max} = 26°

h = 0 → 6

k = 0 → 21

l = –14 → 14

3 standard reflections

frequency: 120 min

intensity decay: 2%

Refinement

Refinement on *F*

R = 0.038

wR = 0.062

S = 1.912

1640 reflections

149 parameters

H-atom parameters not

refined

w = 4*F*_o²/[σ²(*F*_o²) + 0.04*F*_o²]²

(Δ/σ)_{max} = 0.03

Δρ_{max} = 0.214 e Å⁻³

Δρ_{min} = –0.234 e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables*

for X-ray Crystallography

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
S1	0.0602 (1)	0.14245 (4)	0.93754 (4)	3.35 (1)
O1	–0.3539 (4)	0.2323 (1)	0.8637 (2)	4.96 (4)
C1	–0.3209 (5)	0.2261 (2)	0.7681 (2)	4.40 (6)
C2	–0.1335 (5)	0.1787 (1)	0.7245 (2)	3.38 (5)
C3	–0.1370 (6)	0.1768 (2)	0.6094 (2)	4.60 (6)
C4	0.0293 (6)	0.1347 (2)	0.5605 (2)	4.80 (6)
C5	0.2022 (5)	0.0911 (2)	0.6261 (2)	4.30 (6)
C6	0.2125 (5)	0.0927 (2)	0.7404 (2)	3.61 (5)
C7	0.0472 (4)	0.1370 (1)	0.7923 (2)	2.99 (4)
C8	0.3324 (4)	0.0853 (2)	0.9835 (2)	3.67 (5)
C9	0.3830 (4)	0.0903 (1)	1.1083 (2)	3.06 (4)
C10	0.2395 (5)	0.0503 (2)	1.1746 (2)	3.94 (5)
C11	0.2884 (5)	0.0548 (2)	1.2891 (2)	4.24 (6)
C12	0.4808 (5)	0.0989 (2)	1.3373 (2)	4.11 (5)
C13	0.6238 (5)	0.1393 (2)	1.2721 (2)	4.32 (6)
C14	0.5753 (5)	0.1350 (2)	1.1575 (2)	3.80 (5)

Table 2. Selected geometric parameters (Å, °)

S1—C7	1.764 (2)	C6—C7	1.396 (3)
S1—C8	1.817 (2)	C8—C9	1.513 (3)
O1—C1	1.205 (3)	C9—C10	1.380 (3)
C1—C2	1.465 (4)	C9—C14	1.383 (3)
C2—C3	1.399 (3)	C10—C11	1.389 (3)
C2—C7	1.408 (4)	C11—C12	1.373 (4)
C3—C4	1.361 (4)	C12—C13	1.375 (4)
C4—C5	1.386 (4)	C13—C14	1.390 (3)
C5—C6	1.386 (3)		
C7—S1—C8	102.1 (2)	C2—C7—C6	117.8 (2)
O1—C1—C2	126.4 (2)	S1—C8—C9	108.5 (2)
C1—C2—C3	116.8 (2)	C8—C9—C10	120.8 (2)
C1—C2—C7	123.4 (3)	C8—C9—C14	120.1 (3)
C3—C2—C7	119.8 (2)	C10—C9—C14	119.1 (3)
C2—C3—C4	121.6 (2)	C9—C10—C11	120.4 (2)
C3—C4—C5	119.2 (2)	C10—C11—C12	120.1 (2)
C4—C5—C6	120.5 (3)	C11—C12—C13	119.9 (2)
C5—C6—C7	121.2 (2)	C12—C13—C14	120.1 (2)
S1—C7—C2	119.8 (2)	C9—C14—C13	120.3 (2)
S1—C7—C6	122.4 (2)		

Intensity data were corrected for Lorentz–polarization effects. Assignment of space group *P*2₁/*n* was based on unique systematic absences. The structure was solved by direct

methods (*MULTAN*11/82; Main *et al.*, 1982) and refined by full-matrix least-squares analysis. H atoms were generated in idealized positions and included in the structure-factor calculations, but not refined. All calculations were performed using *SDP* (Enraf–Nonius, 1985) on a MicroVAX II computer.

We thank the Hong Kong Research Grants Council and the University of Hong Kong for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(3*R*,4*S*,5*S*)-5-Benzyl-4-hydroxy-3-[1(*S*)-(1,1-dimethylethoxycarbonylamino)-2-phenylethyl]pyrrolidin-2-one

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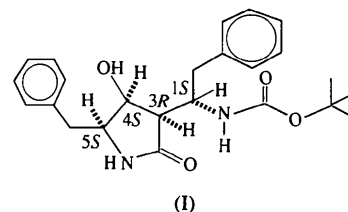
Abstract

The title compound, *tert*-butyl {1(*S*)-[(3*R*,4*S*,5*S*)-5-benzyl-4-hydroxy-2-oxo-3-pyrrolidinyl]-2-phenylethyl}-carbamate, C₂₄H₃₀N₂O₄, is an example of an all-*cis*

trisubstituted pyrrolidin-2-one. The absolute configurations of the C atoms in the lactam ring were assigned to be 3*R*, 4*S* and 5*S* relative to the known chirality, 1*S*, of the C^α atom of the *L*-phenylalanine fragment in the compound. There is one intramolecular hydrogen bond between the pyrrolidin-2-one carbonyl O atom and the N—H group of the *L*-phenylalanine fragment [N—H...O 2.936 (5) Å].

Comment

The aldol addition of dilithiated methyl 3(*S*)-(1,1-dimethylethoxycarbonylamino)-4-phenylbutanoate to *N,N*-dibenzyl-*L*-phenylalaninol resulted in the formation of two diastereomeric aldol products. *N,N*-Dibenzyl deprotection of the minor product gave the title compound, (I), as the sole product (Ettmayer, Hübner & Gstach, 1994).



The purpose of the structure analysis was the determination of the absolute configuration of the chiral centres in the pyrrolidinone ring relative to the known configuration of the C^α atom of the phenylalanine fragment

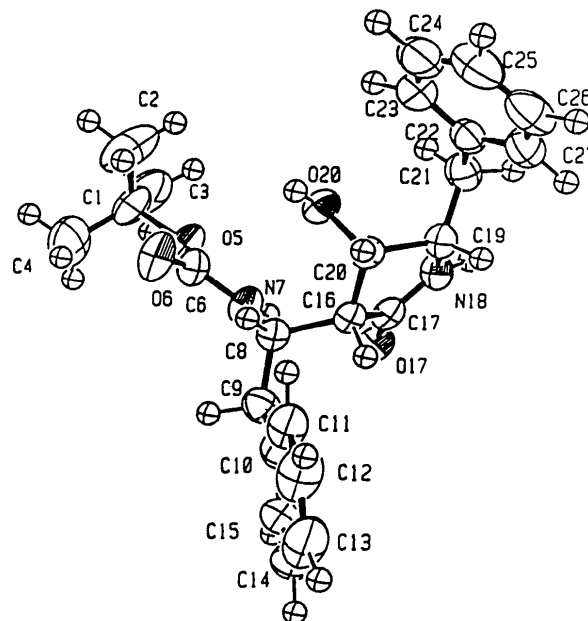


Fig. 1. ORTEP (Johnson, 1965) view of the title compound showing the atom-numbering scheme. Displacement ellipsoids of the non-H atoms are plotted at the 50% probability level; H atoms are represented by spheres of 0.15 Å radii.