

William Clegg,* Richard F. W.
Jackson† and Neil WishartSchool of Natural Sciences (Chemistry),
University of Newcastle upon Tyne,
Newcastle upon Tyne NE1 7RU, England† Present address: Department of Chemistry,
University of Sheffield, Sheffield S3 7HF,
England.

Correspondence e-mail: w.clegg@ncl.ac.uk

Key indicators

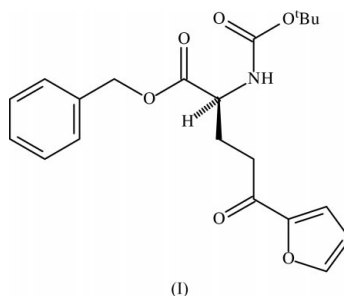
Single-crystal X-ray study
 $T = 160\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.031
 wR factor = 0.081
Data-to-parameter ratio = 6.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Benzyl (2S)-2-tert-butoxycarbonylamino-
5-(2-furyl)-5-oxopentanoate

The title compound, $\text{C}_{21}\text{H}_{25}\text{NO}_6$, prepared using a procedure for the synthesis of enantiomerically pure 5-oxo- α -amino acids, has three fully extended substituents on the central α -C atom of the amino acid. Relatively weak $\text{N}-\text{H}\cdots\text{O}=\text{C}$ hydrogen bonds ($\text{N}\cdots\text{O} > 3\text{ \AA}$) link the molecules into chains.

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Comment

The title compound, (I), is an example of a 5-oxo- α -amino acid prepared by a route that does not involve the formation of one of the four bonds at the α centre and hence retains the chirality already established at this atom (Jackson *et al.*, 1998); the synthetic procedure thus produces enantiomerically pure compounds from appropriate starting materials. The crystal structure of (I) has been determined in order to demonstrate the enantiomeric purity and to investigate whether hydrogen bonding is intermolecular or intramolecular, given the presence of one donor $\text{N}-\text{H}$ group and three potential acceptor carbonyl groups.



The molecular structure of (I) is shown in Fig. 1. All three substituents on the α -C atom, C6, have an essentially fully extended conformation, as shown by the torsion angles in Table 1, each of the two planar rings being rotated only slightly out of the mean plane of the corresponding chain. This extended conformation is apparently more energetically favourable in the solid state than the formation of an intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond, which would form either a five- or a seven-membered ring involving atom O3 or O5, respectively, as acceptor. Instead, relatively weak intermolecular hydrogen bonds are formed ($\text{N}\cdots\text{O} > 3\text{ \AA}$; Table 2), which link the molecules into chains along the a axis. We have reported a similar situation, with an extended molecular conformation and weak intermolecular $\text{N}-\text{H}\cdots\text{O}=\text{C}$ hydrogen bonding, in a 4-oxo- α -amino acid (Clegg *et al.*, 1994).

Finally, we note that the structure, in space group $P1$ with $Z = 1$, shows no overlap of molecules in a projection of the

packing along the short *a* axis (one unit cell deep), and can serve as a useful illustration of pure translation symmetry in crystallography.

Experimental

The title compound was prepared by reaction of the organocopper reagent (*S*)-CuCH₂CH₂CH(NHCO₂^tBu)(CO₂CH₂Ph) with six equivalents of 2-furoyl chloride in tetrahydrofuran solution at 240 K for 3 h, according to a literature procedure (Jackson *et al.*, 1998), and was recrystallized by slow evaporation of a solution in chloroform/dichloromethane. Spectroscopic data (¹H NMR, MS and IR) were in accordance with the expected structure; m.p. 391 K.

Crystal data

C ₂₁ H ₂₅ NO ₆	<i>Z</i> = 1
<i>M_r</i> = 387.42	<i>D_x</i> = 1.266 Mg m ⁻³
Triclinic, <i>P</i> 1	Mo <i>K</i> α radiation
<i>a</i> = 5.200 (2) Å	Cell parameters from 32 reflections
<i>b</i> = 10.132 (4) Å	<i>θ</i> = 10.7–12.5°
<i>c</i> = 10.768 (4) Å	<i>μ</i> = 0.09 mm ⁻¹
<i>α</i> = 65.66 (2)°	<i>T</i> = 160 (2) K
<i>β</i> = 83.87 (2)°	Plate, colourless
<i>γ</i> = 79.63 (3)°	0.56 × 0.52 × 0.20 mm
<i>V</i> = 508.1 (3) Å ³	

Data collection

Stoe–Siemens four-circle diffractometer	<i>R</i> _{int} = 0.038
<i>ω</i> / <i>θ</i> scans with on-line profile fitting (Clegg, 1981)	<i>θ</i> _{max} = 25.0°
Absorption correction: none	<i>h</i> = -6 → 6
1795 measured reflections	<i>k</i> = -10 → 12
1786 independent reflections	<i>l</i> = -5 → 12
1729 reflections with <i>I</i> > 2σ(<i>I</i>)	5 standard reflections
	frequency: 60 min
	intensity decay: 2%

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 0.0741P]$
$R[F^2 > 2\sigma(F^2)] = 0.031$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.081$	(Δ/σ) _{max} < 0.001
<i>S</i> = 1.05	$\Delta\rho_{max} = 0.18 \text{ e } \text{Å}^{-3}$
1786 reflections	$\Delta\rho_{min} = -0.23 \text{ e } \text{Å}^{-3}$
257 parameters	Extinction correction: <i>SHELXTL</i>
H-atom parameters constrained	Extinction coefficient: 0.031 (8)

Table 1

Selected torsion angles (°).

C15–C6–C7–O4	–91.0 (2)	C6–C15–C16–C17	–179.35 (18)
N–C6–C7–O4	146.13 (18)	C15–C16–C17–C18	163.45 (19)
C7–C6–C15–C16	172.11 (18)	C16–C17–C18–C19	–10.9 (4)
N–C6–C15–C16	–65.3 (2)	C16–C17–C18–O6	172.68 (18)
C7–C6–N–C5	–77.3 (3)	O1–C5–N–C6	173.1 (2)
C15–C6–N–C5	161.5 (2)	N–C5–O1–C4	177.16 (19)
C6–C7–O4–C8	178.39 (19)	C1–C4–O1–C5	178.8 (2)
C9–C8–O4–C7	178.54 (19)	C2–C4–O1–C5	–63.7 (3)
O4–C8–C9–C10	15.3 (3)	C3–C4–O1–C5	61.2 (3)
O4–C8–C9–C14	–165.2 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N–H0...O2 ⁱ	0.88	2.20	3.032 (3)	158

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

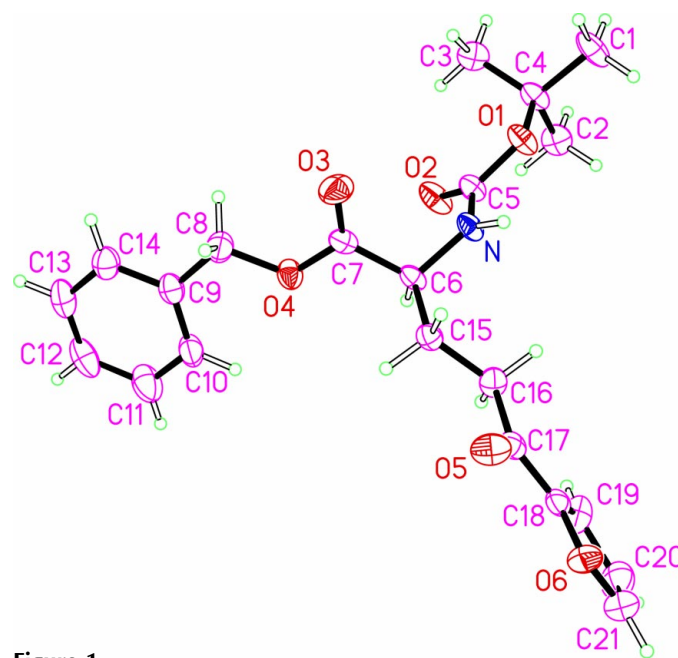


Figure 1

The molecular structure of (I), with atom labels and 50% probability ellipsoids for non-H atoms.

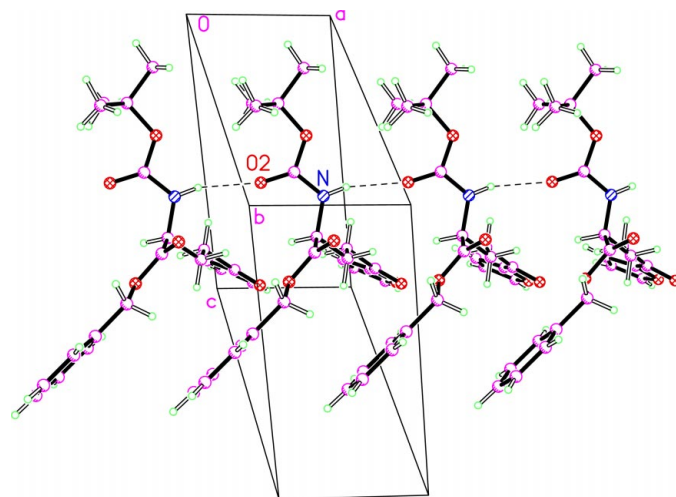


Figure 2

A section of the crystal packing in (I), showing intermolecular hydrogen bonds as dashed lines.

H atoms were placed geometrically and refined with a riding model (including free rotation about C–C bonds), and with *U*_{iso} constrained to be 1.2 (1.5 for methyl groups) times *U*_{eq} of the carrier atom. The absolute stereochemistry could not be established from the diffraction experiment, because of the lack of significant anomalous dispersion effects; Friedel pairs in the data set were averaged. The space group *P*1, with *Z* = 1, indicates enantiomeric purity for the crystal studied, and the absolute configuration was assumed from that known for the starting material in the synthesis.

Data collection: *DIF4* (Stoe & Cie, 1988); cell refinement: *DIF4*; data reduction: local programs; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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