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(2*R*,4*S*)-3-*N*-(Fluoren-9-ylmethoxycarbonyl)-4-methyl-2-nonyloxazolidin-5-one

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Key indicators

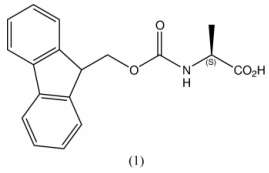
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
R factor = 0.053
wR factor = 0.151
Data-to-parameter ratio = 10.0

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

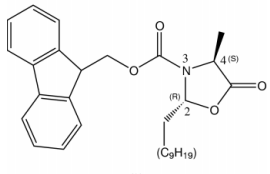
The title compound, $C_{38}H_{35}NO_4$, was isolated as one of two diastereoisomeric products. The molecule was found to have *trans*-configured *n*-nonyl and methyl substituents on the 5-oxazolidinone ring, the *S* configuration being assigned to C4 with reference to L-alanine. The *n*-nonyl group is fully extended and the oxazolidinone ring adopts a half-chair conformation.

Comment

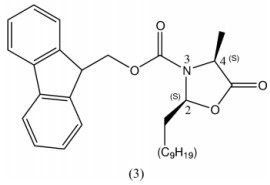
In the course of preparation of an *N*-alkyl-L-alanine derivative, the intermediate (1) was treated with *n*-decanal and camphorsulfonic acid in refluxing toluene. The reaction gave two diastereoisomeric 5-oxazolidinones, (2) and (3), as products in which the molecules have either *trans*- or *cis*-configured *n*-nonyl and methyl substituents about the 5-oxazolidinone ring. Other workers have prepared similar structures in studies concerned with the manipulation of α-amino acids generally for the construction of α,α-dialkyl-α-amino acids (see, for example, Karady *et al.*, 1984). Compounds (2) and (3) were separated by column chromatography. As the ^{1}H NMR spectra of both compounds were similar we were unable to determine the configuration at the C2 asymmetric center, the *S* configuration being assigned to C4 with reference to L-alanine. Following column chromatography, the minor diastereoisomer crystallized and so an X-ray analysis was undertaken to ascertain its structure as either (2) or (3).



(1)



(2)



(3)

As structure (2) was found to have *trans*-configured *n*-nonyl and methyl substituents about the 5-oxazolidinone ring (Fig.

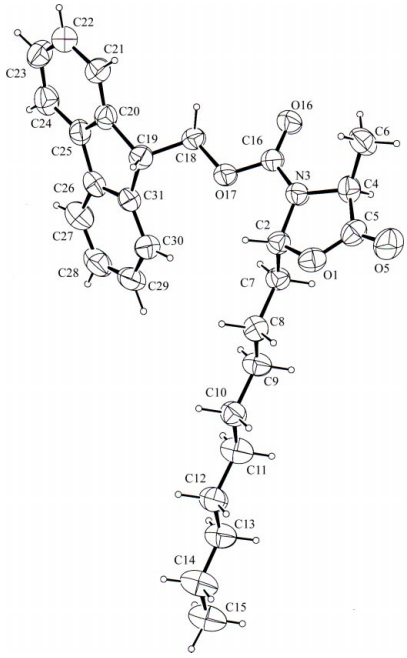


Figure 1
A perspective drawing of the molecule, with displacement ellipsoids drawn at the 40% probability level (Johnson, 1976).

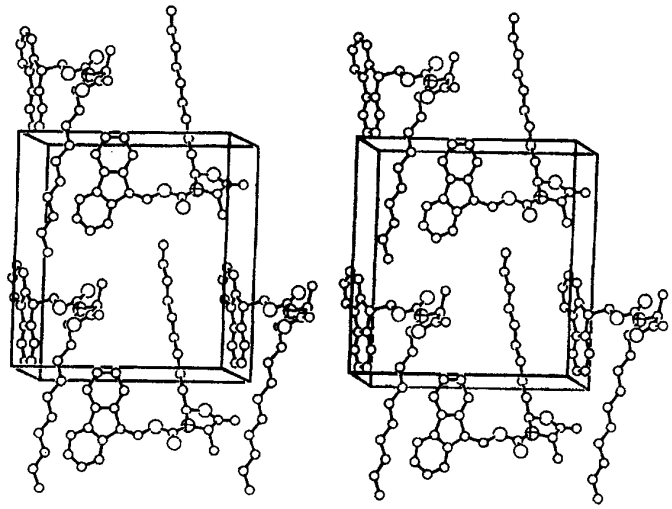


Figure 2
Stereoview of the crystal packing, viewed down the *a* axis with the *c* axis vertical. The larger open and crossed circles represent O and N atoms, respectively.

1), the configuration at C2 is *R*. The nonyl side chain at C2 is fully extended, the pertinent torsion angles ranging from 176.9 (3) to 179.6 (4)°. The oxazolidinone ring adopts a half-chair conformation as indicated by the pseudo-rotation parameters (Altona *et al.*, 1968) Δ = 3.5 and φ_m 21.1°. The fluorenyl substituent at C18 is not strictly planar as is generally observed for other fluorenyl moieties, but rather exhibits a slight bowing of the rings. The mean plane through the thirteen atoms shows that only five atoms (C19, C21, C22, C29 and C30) lie −0.070 (3), 0.037 (3), 0.042 (3), 0.045 (4) and 0.032 (3) Å, respectively, from the plane, while the other eight atoms lie roughly in the plane (r.m.s. δ < 0.02 Å). This characteristic, although more pronounced, has been noted recently in another crystal structure containing a fluorenyl moiety (Meyers *et al.*, 2001). The other dimensions associated with the central five-membered ring are similar to those generally observed for other comparable structures.

The crystal packing, illustrated in Fig. 2, shows that the molecules are orientated in the crystal with the linear *n*-nonyl groups aligned along the *b* axis and adjacent to the fluorenyl moieties of adjacent molecules. Some intermolecular interaction between the two groups could account for the slight bowing of the fluorenyl rings. Intermolecular interactions were also suggested previously as causing this effect.

Experimental

N-Fmoc-L-alanine, (1) (Fmoc = fluoren-9-ylmethoxycarbonyl; 500 mg, 1.6 mmol), camphorsulfonic acid (325 mg, 1.4 mmol) and acetic acid (92 µl, 1.6 mmol) were dissolved in toluene (200 ml). To this solution was added *n*-decanal (602 µl, 3.2 mmol) and the mixture was then heated under reflux (Dean–Stark trap) for 18 h. The solution was concentrated *in vacuo* and the residue was taken up in ethyl acetate. The solution was then washed with 5% sodium bicarbonate solution (3 × 50 ml). The organic phase was dried ($MgSO_4$) and concentrated *in vacuo*. The residue was purified by flash column chromatography, eluting with 10% ethyl acetate–hexane, to give the diastereoisomers (2) and (3) as a mixture (570 mg, 79%). A ^{13}C NMR spectrum of the mixture indicated the ratio of compounds (2):(3) was approximately 1:2.6 (ratio obtained by integration of the methyl signals at δ 17.70 and 13.99 p.p.m., respectively). The diastereoisomers were separated by column chromatography, eluting with 25% diethyl ether–hexane. The first fraction eluted was the minor compound (2), and crystals (m.p. 350 K) were formed by slow evaporation from a solution of diethyl ether/*n*-hexane at room temperature.

Crystal data

$C_{28}H_{35}NO_4$
M_r = 449.59
Monoclinic, $P2_1$
a = 5.1400 (10) Å
b = 15.575 (3) Å
c = 16.050 (3) Å
β = 96.995 (3)°
V = 1275.3 (4) Å^3
Z = 2
D_x = 1.171 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5736 reflections
θ = 5–27.5°
μ = 0.08 mm^{-1}
T = 292 (2) K
Tablet, colourless
0.5 × 0.4 × 0.1 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995; Sheldrick, 1996)
T_{min} = 0.964, T_{max} = 0.992
11309 measured reflections
3001 independent reflections
2268 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 27.5°
$h = -6 \rightarrow 6$
$k = -20 \rightarrow 20$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.151
S = 1.06
3001 reflections
300 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0928P)^2 + 0.0192P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}

The molecule crystallized in the non-centrosymmetric space group $P2_1$; however, owing to the absence of atoms heavier than O, the absolute configuration could not be directly determined but was deduced by reference to L-alanine. Moreover the Friedel pairs were merged and no attempt was made to refine the Flack (1983) parameter.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1986); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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