Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 2(*S*)-(*N*-fluoren-9-ylmethoxycarbonylamino)-3-(2-pyridyl)propionate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.007 Å
R factor = 0.052
wR factor = 0.090
Data-to-parameter ratio = 6.8

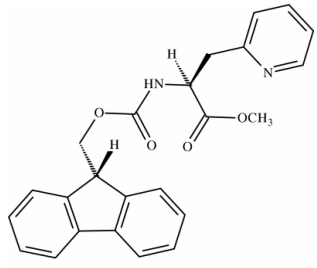
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 August 2003
Accepted 4 August 2003
Online 8 August 2003

The title compound, $C_{24}H_{22}N_2O_4$, is a methyl ester of a 2-pyridylalanine in which the amine group is protected. The molecule has four essentially planar segments. Apart from a relatively weak N—H···O hydrogen bond, molecules are held together in the solid state only by van der Waals interactions.

Comment

The title compound, (I), was prepared in a study of the preparation of enantiomerically pure pyridylamino acids (Tabanella *et al.*, 2003). It was obtained from a palladium-catalysed coupling reaction of 2-bromopyridine with a fluorenylmethyoxycarbonyl-protected (Fmoc-protected) zinc reagent derived from iodoalanine methyl ester. Its structure was determined in order to confirm the identity of the product and to establish the nature of any inter- or intramolecular hydrogen bonding.



(I)

The molecule consists of four planar segments (Fig. 1), *viz*. the fluorene group, the ester and amine linkage from C7 to C11, the methyl ester attached to C7, and the pyridylmethyl group. The main torsion angles defining the conformational arrangement of these segments are given in Table 1. Bond lengths and angles are unexceptional.

Only one, relatively weak, intermolecular hydrogen bond is found, linking the single donor, the N—H group, with atom O2 of an adjacent molecule, with an N···O distance of 3.214 (5) Å and an angle of 142° at the H atom. Neither of the carbonyl O atoms acts as a hydrogen-bond acceptor.

Although complexes with deprotonated β-(2-pyridyl)-α-alanine as a ligand have been crystallographically characterized (Ebner *et al.*, 1979, 1980; Ebner & Angelici, 1981), no crystal structures have been reported for the amino acid itself or for protected derivatives.

Experimental

The synthesis of the title compound is described by Tabanella *et al.* (2003).

Crystal data

$C_{24}H_{22}N_2O_4$
M_r = 402.44
Orthorhombic, $P2_12_12_1$
a = 5.524 (4) Å
b = 14.988 (11) Å
c = 23.557 (18) Å
V = 1950 (3) Å^3
Z = 4
D_x = 1.371 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2523 reflections
θ = 2.2–25.8°
μ = 0.09 mm^{-1}
T = 173 (2) K
Needle, colourless
0.48 × 0.09 × 0.06 mm

Data collection

Bruker SMART 1K CCD diffractometer
Narrow-frame ω scans
Absorption correction: none
6519 measured reflections
1863 independent reflections
1042 reflections with $I > 2\sigma(I)$
R_{int} = 0.158
θ_{max} = 25.0°
$h = -6 \rightarrow 6$
$k = -11 \rightarrow 15$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.090
S = 0.80
1863 reflections
273 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0258P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0073 (12)

Table 1
Selected geometric parameters (°).

N1—C1—C6—C7	30.3 (6)	C6—C7—N2—C10	136.1 (4)
C5—C1—C6—C7	−146.7 (5)	C8—C7—N2—C10	−93.4 (5)
C1—C6—C7—C8	−75.6 (5)	C7—N2—C10—O4	163.1 (4)
C1—C6—C7—N2	52.0 (6)	N2—C10—O4—C11	176.3 (3)
C6—C7—C8—O2	−42.9 (6)	C10—O4—C11—C12	122.7 (4)
N2—C7—C8—O2	−171.9 (4)	O4—C11—C12—C13	−72.2 (5)
C7—C8—O2—C9	−173.3 (4)	O4—C11—C12—C19	173.4 (3)

H atoms were positioned 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. In the absence of significant anomalous scattering effects, the absolute configuration could not be confirmed from the diffraction data; it was assumed from the known configuration of the starting material, and Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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.

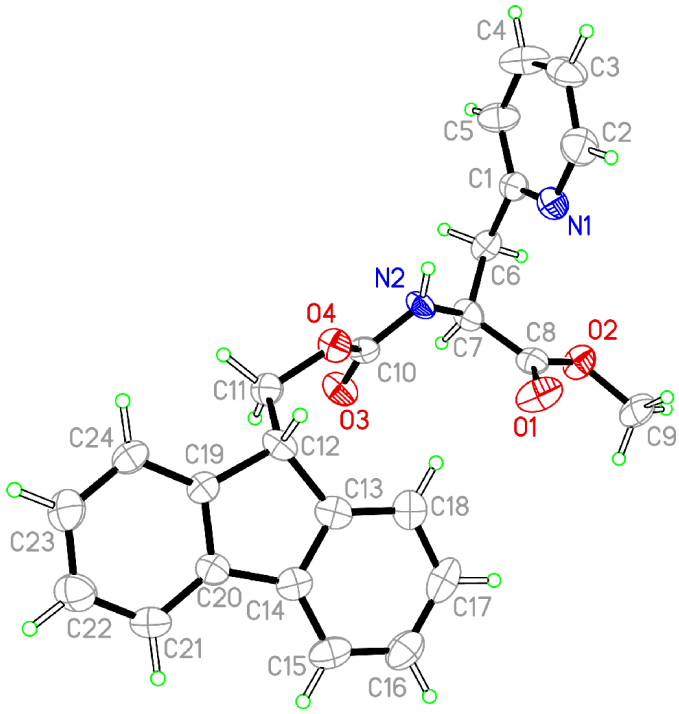


Figure 1
The molecular structure with atom labels and 50% probability ellipsoids for non-H atoms.

We thank the EPSRC for financial support, and Professor Richard Jackson and Ingrid Valancogne for supplying the sample.

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