

José Giralés, Mark L.  
McLaughlin and Frank R.  
Fronczek\*Department of Chemistry, Louisiana State  
University, Baton Rouge, LA 70803-1804, USACorrespondence e-mail:  
fronz@chxray1.chem.lsu.edu

## Key indicators

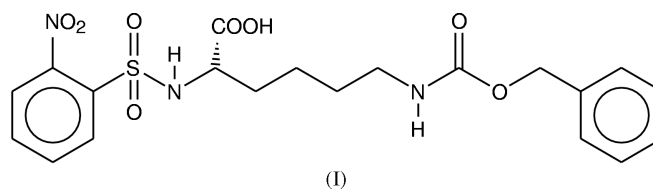
Single-crystal X-ray study  
 $T = 120$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.039  
 $wR$  factor = 0.077  
Data-to-parameter ratio = 14.4For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>. $N^\epsilon$ -Benzyloxycarbonyl- $N^\alpha$ -(2-nitrobenzenesulfonyl)-  
L-lysine

The title compound,  $\text{C}_{20}\text{H}_{23}\text{N}_3\text{O}_8\text{S}$ , is in an extended conformation. The COOH group does not form hydrogen-bonded carboxyl dimers, but forms an intermolecular hydrogen bond with the NCOO carbonyl O atom [ $\text{O} \cdots \text{O}$  2.666 (2) Å]. The N atom bonded to S is pyramidal, and forms a bifurcated intramolecular hydrogen bond with carboxy and nitro O atoms.

Received 22 November 2000  
Accepted 28 November 2000  
Online 14 December 2000

## Comment

The solid-phase peptide synthesis by the use of  $N^\alpha$ -protecting groups such as Fmoc (9-fluorenylmethoxycarbonyl) is widely used as described by Wenschuh *et al.* (1994). In an effort to increase the yield of the amino acid coupling reaction, the title compound, (I), was synthesized. The 2-nitrobenzenesulfonyl protecting group is smaller than the Fmoc group and is expected to increase the amino acid coupling yield due to decreased steric interactions with the nucleophile. The crystal structure determination of the title compound was carried out to confirm its successful synthesis.



The title compound is in an extended conformation. The COOH group does not form hydrogen-bonded carboxy dimers, but forms an intermolecular hydrogen bond with the carbonyl O5 atom. The lysine N1 atom is pyramidal, and forms a bifurcated intramolecular hydrogen bond with the carboxy O3 and nitro O8 atoms.

The cell dimensions of the title compound at 296 K are  $a = 11.8092$  (13),  $b = 34.832$  (4),  $c = 5.4033$  (6) Å and  $V = 2222.6$  (7) Å<sup>3</sup>.

## Experimental

The title compound was prepared by reacting  $N^\epsilon$ -benzyloxycarbonyl-L-lysine with chlorotrimethylsilane (1.85 equivalents), diisopropylethylamine (2.3 equivalents) and *ortho*-nitrobenzenesulfonyl chloride (0.9 equivalents) in anhydrous dichloromethane under argon over 12 h (273 K to reflux), followed by aqueous work-up and extraction. The amino acid derivative was recrystallized by dissolving the crude material in a hot ethanol solution, followed by slow cooling to room temperature.

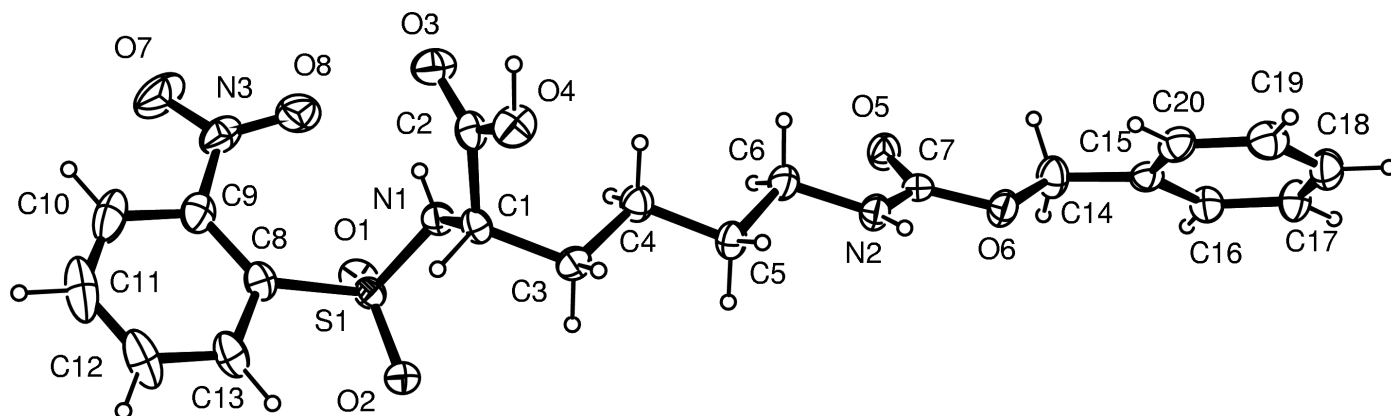


Figure 1

The atom-numbering scheme for (I) with displacement ellipsoids at the 50% probability level.

### Crystal data

$C_{20}H_{23}N_3O_8S$   
 $M_r = 465.47$   
 Orthorhombic,  $P2_12_12_1$   
 $a = 11.7629$  (2) Å  
 $b = 34.5731$  (6) Å  
 $c = 5.3477$  (2) Å  
 $V = 2174.8$  (2) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.422$  Mg m<sup>-3</sup>

### Data collection

KappaCCD diffractometer (with Oxford Cryosystems Cryostream cooler)  
 $\omega$  scans with  $\kappa$  offsets  
 Absorption correction: multi-scan (HKL SCALEPACK; Otwinowski & Minor, 1997)  
 $T_{\min} = 0.937$ ,  $T_{\max} = 0.980$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.077$   
 $S = 1.05$   
 4345 reflections  
 302 parameters  
 H atoms treated by a mixture of independent and constrained refinement

Mo  $K\alpha$  radiation  
 Cell parameters from 13981 reflections  
 $\theta = 2.5$ – $27.5^\circ$   
 $\mu = 0.20$  mm<sup>-1</sup>  
 $T = 120$  K  
 Fragment, colorless  
 $0.23 \times 0.12 \times 0.10$  mm

13 981 measured reflections  
 4345 independent reflections  
 3563 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.050$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -15 \rightarrow 15$   
 $k = -44 \rightarrow 44$   
 $l = -5 \rightarrow 5$

$w = 1/[\sigma^2(F_o^2) + (0.0239P)^2 + 0.7661P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.24$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL97  
 Extinction coefficient: 0.0039 (6)  
 Absolute structure: Flack (1983); 1439 Friedel pairs  
 Flack parameter =  $-0.07$  (7)

Table 1

Selected geometric parameters (Å, °).

S1—O2	1.4301 (15)	O5—C7	1.238 (3)
S1—O1	1.4323 (15)	O6—C7	1.336 (3)
S1—N1	1.6065 (18)	N1—C1	1.473 (3)
S1—C8	1.791 (2)	N2—C7	1.326 (3)
O3—C2	1.207 (3)	N2—C6	1.464 (3)
O4—C2	1.331 (3)		
S1—N1—C1—C3	101.17 (18)	O8—N3—C9—C8	−23.1 (3)
N1—C1—C2—O3	5.7 (3)	O6—C14—C15—C20	−44.1 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O4—H40 <sup>i</sup> ···O5 <sup>i</sup>	0.93 (3)	1.75 (3)	2.666 (2)	172 (3)
N1—H1N···O3	0.80 (3)	2.26 (3)	2.659 (3)	111 (2)
N1—H1N···O8	0.80 (3)	2.19 (3)	2.843 (3)	138 (2)
N2—H2N···O5 <sup>ii</sup>	0.87 (2)	2.41 (3)	3.233 (3)	160 (2)
C1—H1···O1 <sup>iii</sup>	1.00	2.22	3.197 (3)	167
C11—H11···O1 <sup>iii</sup>	0.95	2.42	3.254 (4)	146

Symmetry codes: (i)  $1 - x, 1 - y, z - 1$ ; (ii)  $x, y, z - 1$ ; (iii)  $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$ .

H atoms on C atoms were placed in calculated positions with C—H bond distances in the range 0.95–1.00 Å and  $U_{\text{iso}} = 1.2U_{\text{eq}}$  of the attached atom, and thereafter treated as riding. H atoms on O and N atoms were placed by difference maps and refined individually. The absolute configuration was determined by refinement of the Flack (1983) parameter, and corresponds with that of L-lysine.

Data collection: COLLECT (Nonius, 2000); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK (Otwinowski & Minor, 1997); program(s) used to solve structure: direct methods using SIR (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

The purchase of the diffractometer was made possible by grant No. LEQSF(1999-2000)-ESH-TR-13, administered by the Louisiana Board of Regents.

### References

- Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Nonius (2000). COLLECT. Nonius BV, Delft, The Netherlands.  
 Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.  
 Sheldrick, G. (1997). SHELXL97. University of Göttingen, Germany.  
 Wenschuh, H., Beyermann, M., Krause, E., Brudel, M., Winter, R., Schumann, M., Carpino, L. & Bienert, M. (1994). *J. Org. Chem.* **59**, 3275–3280.