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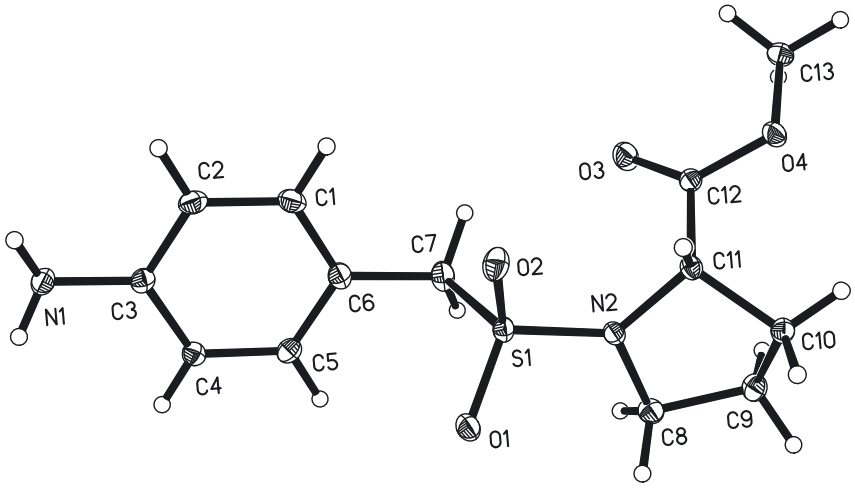
Crystal structure of methyl ((4-aminobenzyl) sulfonyl)-*L*-prolinate, $C_{13}H_{18}N_2O_4S$



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Abstract

$C_{13}H_{18}N_2O_4S$, orthorhombic, $P2_12_12_1$ (no. 19), a = 6.8619(2) Å, b = 12.0735(4) Å, c = 16.6591(5) Å, V = 1380.16(7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0236, $wR_{ref}(F^2)$ = 0.0604, T = 150 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.14 × 0.12 × 0.10 mm
Wavelength:	Cu *K*α radiation (1.54178 Å)
μ:	2.24 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	79.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	20,828, 2946, 0.042
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2872
$N(param)_{refined}$:	183
Programs:	Bruker [1], SHELX [2]

1 Source of material

(4–Nitrophenyl)methanesulfonyl chloride (2.81 g, 12.0 mmol), methyl pyrrolidine-2-carboxylate hydrochloride (2.39 g, 14.4 mmol) and 4-dimethylaminopyridine (3.52 g, 28.8 mmol) were dissolved in 48 mL of dichloromethane and reacted at room temperature for 3.0 h. The response endpoint was detected by thin layer chromatography (TLC). When the reaction was stopped, an appropriate amount of ethyl acetate was added to the reaction system, stirred and allowed to stand for 10 min. The filtrate obtained after filtering off the precipitate was concentrated under vacuum. The crude product was purified by silica-gel column chromatography (petroleum ether:ethyl acetate = 3:1, *v*:*v*). The white solid (1.62 g, 4.94 mmol) obtained after the reaction was dissolved in 20 mL of super-dry methanol with

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5972 (3)	0.10403 (16)	0.13039 (11)	0.0262 (4)
H1	0.517115	0.168137	0.128250	0.031*
C2	0.5164 (3)	0.00354 (16)	0.15321 (10)	0.0250 (4)
H2	0.381954	−0.000393	0.166545	0.030*
C3	0.6311 (3)	−0.09186 (15)	0.15674 (9)	0.0198 (3)
C4	0.8288 (3)	−0.08351 (15)	0.13699 (10)	0.0201 (3)
H4	0.909299	−0.147475	0.138953	0.024*
C5	0.9078 (3)	0.01737 (15)	0.11460 (10)	0.0214 (4)
H5	1.042463	0.021778	0.101744	0.026*
C6	0.7935 (3)	0.11253 (15)	0.11059 (10)	0.0224 (4)
C7	0.8846 (3)	0.22168 (16)	0.08884 (10)	0.0261 (4)
H7A	0.786612	0.269128	0.062011	0.031*
H7B	0.993146	0.209151	0.050759	0.031*
C8	1.2917 (3)	0.40152 (16)	0.12293 (10)	0.0227 (4)
H8A	1.314161	0.356074	0.074345	0.027*
H8B	1.375060	0.373634	0.167020	0.027*
C9	1.3305 (3)	0.52418 (16)	0.10640 (11)	0.0252 (4)
H9A	1.465720	0.544398	0.121083	0.030*
H9B	1.308981	0.541944	0.049063	0.030*
C10	1.1842 (3)	0.58433 (15)	0.15935 (10)	0.0207 (3)
H10A	1.160560	0.660597	0.139818	0.025*
H10B	1.229401	0.587452	0.215737	0.025*
C11	1.0013 (2)	0.51285 (13)	0.15131 (9)	0.0177 (3)
H11	0.919980	0.519648	0.200867	0.021*
C12	0.8778 (2)	0.53899 (14)	0.07726 (10)	0.0185 (3)
C13	0.7337 (3)	0.68444 (15)	0.00261 (10)	0.0237 (4)
H13A	0.611150	0.642766	0.001641	0.036*
H13B	0.805258	0.671372	−0.047409	0.036*
H13C	0.705502	0.763671	0.007956	0.036*
N1	0.5498 (2)	−0.19452 (12)	0.17535 (9)	0.0238 (3)
H1A	0.437345	−0.187300	0.201944	0.025*
H1B	0.631536	−0.240553	0.194209	0.035*
N2	1.0839 (2)	0.40101 (12)	0.14615 (9)	0.0191 (3)
O1	1.1177 (2)	0.21852 (11)	0.21203 (7)	0.0263 (3)
O2	0.8100 (2)	0.32514 (11)	0.22329 (8)	0.0276 (3)
O3	0.8086 (2)	0.47123 (11)	0.03267 (8)	0.0301 (3)
O4	0.8508 (2)	0.64820 (11)	0.07014 (7)	0.0244 (3)
S1	0.97480 (6)	0.29069 (3)	0.17654 (2)	0.01842 (13)

stannous chloride (5.57 g, 24.7 mmol) and refluxed at a controlled temperature of 343 K for 2.0 h. The response endpoint was detected by thin layer chromatography (TLC). When the reaction was stopped, an appropriate amount of saturated aqueous Na_2CO_3 solution was added to the reaction system, the pH was adjusted to 7–8, an appropriate amount of ethyl acetate was added, and stirred for 1.0 h. The filtrate was obtained by filtration of diatomaceous earth. The filtrate was successively washed with deionized water and brine, dried over anhydrous sodium sulfate and condensed under vacuum. The crude product was purified by silica-gel column chromatography (petroleum ether:ethyl acetate = 2:1, *v*:*v*). The title compound was recrystallized from dichloromethane and methanol (1:1, *v*:*v*) system to attain suitable crystals.

2 Experimental details

The H atoms were placed in idealized positions and treated as riding on their parent atoms. The H atoms at N1 were placed in difference maps and treated as riding.

3 Comment

Sulfonamides are important compounds with a wide range of applications [3–5]. The title compound was obtained from the raw material (4-nitrophenyl)methanesulfonyl chloride by substitution and reduction reactions. We further obtained the title compound, methyl ((4-aminobenzyl)sulfonyl)-*L*-prolinate, as a single crystal.

The ORTEP diagram is presented in the Figure. Bond lengths and angles are all in the expected ranges [6]. The whole molecule contains two parts, *p*-aminobenzenesulfonyl and methyl *L*-prolinate, linked by N(2)–S(1). The bond length of N(2)–S(1) is 1.6096(15) Å. The bond angle of C(7)–S(1)–N(2) is 106.80(8)° [7]. For the *p*-aminobenzenesulfonyl side, the amino group at the C(3) position is obtained by reduction of the nitro group, and can act as a proton donor and form hydrogen bonds with other atoms [8, 9]. For the methyl *L*-prolinate, the C(11) is the chiral center and the molecule adopts the *L* stereoselective configuration. The carboxyl methyl ester at the C(11) can improve the molecular lipid solubility.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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