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N-Chlorosulfonyl-L-proline Benzyl Ester

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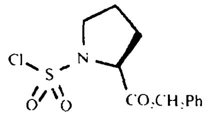
Abstract

The crystal structure reported for the title compound, $C_{12}H_{14}ClNO_4S$, (I), is the first for a sulfamoyl chloride.

Comment

N,*N*-Dialkylsulfamoyl chlorides (otherwise called *N*-chlorosulfonylamines) are a well-studied class, but the simple examples are mostly noxious liquids and no other crystal structure has been reported for any member of the class. The nearest related structures to have been described (Barton, Rogido & Clardy, 1970; Furst, Wachsman, Pieroni, White & Moriconi, 1973; Paquette, Lau & Rogers, 1988) are all complex *N*-chlorosulfonylamides.



(I)

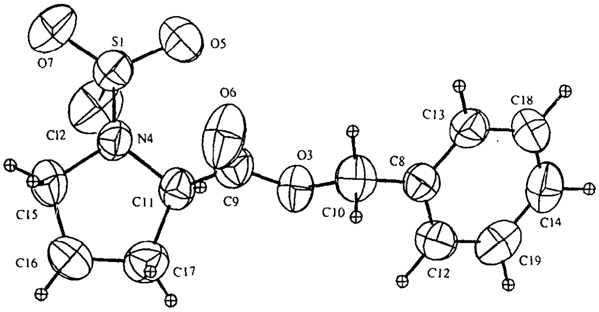


Fig. 1. Structure of chlorosulfonyl-L-proline benzyl ester, showing the atom-labelling scheme.

Experimental

The title compound was prepared by T. J. Cheeseright and J. H. Jones, Dyson Perrins Laboratory, Oxford. The action of sulfuryl chloride on L-proline benzyl ester in toluene at 273 K afforded a 35–40% yield (m.p. 338–339 K) after recrystallization from chloroform–light petroleum.

Crystal data

$C_{12}H_{14}ClNO_4S$
$M_r = 303.8$
Orthorhombic
$P2_12_12_1$
$a = 5.6987\ (2)$ Å
$b = 14.384\ (2)$ Å
$c = 17.218\ (3)$ Å
$V = 1411$ Å^3
$Z = 4$
$D_x = 1.43$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 35.4$–$49.8°$
$\mu = 3.89$ mm^{-1}
$T = 295\ (2)$ K
Needle-like
0.8 × 0.4 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4F diffractometer
ω–2θ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.614$, $T_{\max} = 1.354$
2229 measured reflections
1608 independent reflections
1090 observed reflections [$I > 3\sigma(I)$]

$R_{\text{int}} = 0.058$
$\theta_{\max} = 72°$
$h = -1 \rightarrow 7$
$k = -1 \rightarrow 17$
$l = -1 \rightarrow 21$
3 intensity and 3 orientation standard reflections
frequency: 60 min
intensity variation: −50%

Refinement

Refinement on F
$R = 0.045$
$wR = 0.054$
$S = 1.09$
1090 reflections
173 parameters
H-atom parameters not refined
Chebychev weighting scheme (Tukey–Prince algorithm, 15)
$w = [weight] \times [1 - (\Delta F/6\sigma F)^2]^2$

$(\Delta/\sigma)_{\max} = 0.402 \times 10^{-4}$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 3.02×10^{-4}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S(1)	0.8326 (2)	0.3296 (1)	0.65260 (8)	0.0613
Cl(2)	1.1850 (3)	0.3568 (1)	0.6516 (1)	0.0874
O(3)	0.7488 (7)	0.1276 (3)	0.8359 (2)	0.0612
N(4)	0.8137 (8)	0.2204 (3)	0.6442 (2)	0.0544
O(5)	0.7560 (9)	0.3558 (3)	0.7272 (2)	0.0790
O(6)	0.4726 (8)	0.1448 (4)	0.7445 (3)	0.0863
O(7)	0.7439 (9)	0.3727 (3)	0.5848 (2)	0.0837
C(8)	0.669 (1)	0.1147 (3)	0.9715 (3)	0.0512
C(9)	0.675 (1)	0.1452 (4)	0.7649 (3)	0.0593

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C(10)	0.571 (1)	0.0999 (4)	0.8924 (3)	0.0651
C(11)	0.884 (1)	0.1618 (4)	0.7123 (3)	0.0540
C(12)	0.548 (1)	0.1673 (4)	1.0241 (3)	0.0629
C(13)	0.879 (1)	0.0753 (4)	0.9941 (3)	0.0656
C(14)	0.839 (1)	0.1431 (5)	1.1203 (3)	0.0736
C(15)	0.885 (1)	0.1744 (5)	0.5705 (3)	0.0702
C(16)	1.056 (2)	0.1004 (5)	0.5955 (4)	0.0799
C(17)	0.967 (1)	0.0719 (4)	0.6738 (3)	0.0714
C(18)	0.965 (1)	0.0898 (5)	1.0687 (4)	0.0743
C(19)	0.633 (1)	0.1811 (4)	1.0985 (3)	0.0719

Table 2. *Selected geometric parameters* (Å, °)

S(1)—Cl(2)	2.046 (2)	C(8)—C(12)	1.368 (7)
S(1)—N(4)	1.581 (4)	C(8)—C(13)	1.378 (8)
S(1)—O(5)	1.409 (4)	C(9)—C(11)	1.514 (8)
S(1)—O(7)	1.415 (4)	C(11)—C(17)	1.529 (8)
O(3)—C(9)	1.318 (6)	C(12)—C(19)	1.383 (8)
O(3)—C(10)	1.462 (6)	C(13)—C(18)	1.389 (8)
N(4)—C(11)	1.497 (6)	C(14)—C(18)	1.373 (9)
N(4)—C(15)	1.488 (6)	C(14)—C(19)	1.35 (1)
O(6)—C(9)	1.206 (7)	C(15)—C(16)	1.51 (1)
C(8)—C(10)	1.489 (7)	C(16)—C(17)	1.498 (9)
N(4)—S(1)—Cl(2)	104.9 (2)	C(11)—C(9)—O(3)	109.6 (5)
O(5)—S(1)—Cl(2)	105.1 (2)	C(11)—C(9)—O(6)	125.3 (5)
O(5)—S(1)—N(4)	109.2 (2)	C(8)—C(10)—O(3)	107.9 (5)
O(7)—S(1)—Cl(2)	105.1 (2)	C(9)—C(11)—N(4)	110.4 (4)
O(7)—S(1)—N(4)	109.6 (2)	C(17)—C(11)—N(4)	102.7 (4)
O(7)—S(1)—O(5)	121.6 (3)	C(17)—C(11)—C(9)	111.7 (5)
C(10)—O(3)—C(9)	116.7 (5)	C(19)—C(12)—C(8)	121.0 (6)
C(11)—N(4)—S(1)	118.0 (3)	C(18)—C(13)—C(8)	120.3 (5)
C(15)—N(4)—S(1)	120.1 (4)	C(19)—C(14)—C(18)	119.9 (6)
C(15)—N(4)—C(11)	110.1 (4)	C(16)—C(15)—N(4)	104.3 (4)
C(12)—C(8)—C(10)	119.6 (5)	C(17)—C(16)—C(15)	103.3 (6)
C(13)—C(8)—C(10)	121.9 (5)	C(16)—C(17)—C(11)	105.3 (5)
C(13)—C(8)—C(12)	118.6 (5)	C(14)—C(18)—C(13)	119.9 (6)
O(6)—C(9)—O(3)	125.0 (5)	C(14)—C(19)—C(12)	120.3 (6)

Analysis of the intensity data revealed that the systematic absences were consistent with the space group $P2_12_12_1$. Corrections were applied for Lorentz and polarization effects. The structure was solved using the direct methods program *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989), which revealed the positions of all the non-H atoms. All other computations were performed using the *CRYSTALS* program (Watkin, Carruthers & Betteridge, 1985). The H atoms were subsequently placed geometrically during the refinement process. Full-matrix least-squares refinement was used for the positions and isotropic temperature factors of all the non H-atoms; a parameter to allow for the effects of secondary extinction was included.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HE1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,6-Bis(*p*-nitrophenylthiomethyl)pyridine

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Abstract

In the title compound, $C_{19}H_{15}N_3O_4S_2$, the pyridine ring, one thiomethyl group and the adjoining nitrobenzene group are approximately in the same plane. The C—C—S—C torsion angle is −179.9 (3)°. The C atom of the second thiomethyl group is also in this plane, but the S(2) atom is *anti* to the plane resulting in a C—C—S—C torsion angle of 67.7 (4)°.

Comment

As a part of our study on metal complexes of *S,S'*-alkyl and *S,S'*-aryl substituted 2,6-bis(thiomethyl)pyridine derivatives (Teixidor, Sánchez-Castelló, Lucena, Escriche, Kivekäs, Sundberg & Casabó, 1991), we now report the crystal structure of 2,6-bis(*p*-nitrophenylthiomethyl)pyridine, (I). This type of ligand is a source of an NS_2-coordinating