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1-Chloromethylsulfinyl-2-nitrobenzene

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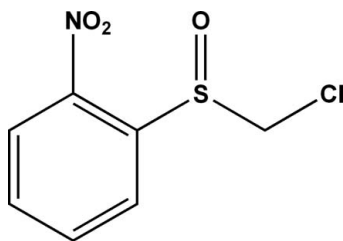
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Key indicators: single-crystal X-ray study; $T = 180$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.026; wR factor = 0.071; data-to-parameter ratio = 18.4.

In the title compound, $\text{C}_7\text{H}_6\text{ClNO}_3\text{S}$, the nitro group forms a dihedral angle of $2.7(4)^\circ$ with the benzene ring. The bond-angle sum at the S atom is 303.7° . In the crystal, molecules are linked by weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming layers lying parallel to $(\bar{1}01)$.

Related literature

For the biological and pharmacological activity of sulfoxides, see, for example: Melzig *et al.* (2009); Huang *et al.* (2010). For related structures, see: Yan (2010); Kobayashi *et al.* (2003).



Experimental

Crystal data

 $\text{C}_7\text{H}_6\text{ClNO}_3\text{S}$
 $M_r = 219.65$

 Monoclinic, $P2_1/c$
 $a = 12.2394(5)$ Å

 $b = 5.5009(2)$ Å

 $c = 14.5537(11)$ Å

 $\beta = 116.631(4)^\circ$
 $V = 875.92(9)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.65$ mm⁻¹
 $T = 180$ K

 $0.23 \times 0.20 \times 0.18$ mm

Data collection

Agilent Xcalibur (Eos, Gemini

ultra) diffractometer

Absorption correction: multi-scan

 (*CrysAlis PRO*; Agilent, 2011)

 $T_{\min} = 0.900$, $T_{\max} = 1.000$

10209 measured reflections

2172 independent reflections

 1799 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.071$
 $S = 1.05$

2172 reflections

118 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C4}-\text{H4}\cdots\text{O12}^i$	0.95	2.44	3.384 (2)	173
$\text{C7}-\text{H7A}\cdots\text{O1}^{ii}$	0.99	2.36	3.2478 (18)	149
$\text{C7}-\text{H7B}\cdots\text{O1}^{iii}$	0.99	2.50	3.332 (2)	142

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

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported by the Unité de Recherche de Chimie de l'Environnement et Moléculaire Structurale (CHEMS), Université Mentouri-Constantine, Algeria. Thanks are due to MESRS (Ministère de l'Enseignement Supérieur et de la Recherche Scientifique and ANDRU (l'Agence Nationale pour le Développement de la Recherche Universitaire) for financial support *via* the PNR programm.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6974).

References

- Agilent (2011). *CrysAlis PRO*. Agilent Technologies Ltd, Yarnton, England.
 Brandenburg, K. & Berndt, M. (2001). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
 Burla, M. C., Caliendo, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Huang, J.-Y., Li, S.-J. & Wang, Y.-G. (2010). *J. Carbohydr. Chem.* **29**, 142–153.
 Kobayashi, K., Sato, A., Sakamoto, S. & Yamaguchi, K. (2003). *J. Am. Chem. Soc.* **125**, 3035–3045.
 Melzig, L., Rauhut, C. B. & Knochel, P. (2009). *Chem. Commun.* pp. 3536–3538.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Yan, Z. (2010). *Acta Cryst.* **E66**, o3311.

supporting information

Acta Cryst. (2012). E68, o3207 [doi:10.1107/S1600536812043553]

1-Chloromethylsulfinyl-2-nitrobenzene

Sabrina Benmebarek, Mhamed Boudraa, Sofiane Bouacida and Jean-Claude Daran

S1. Comment

The use of sulfoxides as pharmaceutical has shown promise in recent years (e.g. Melzig *et al.*, 2009 and Huang *et al.* 2010). As part of our ongoing studies on the synthesis, structures and biological activity of organometallic sulfanilamide complexes we have synthesized and determined the crystal structure of the title compound (I). The molecular geometry and the atom-numbering scheme are shown in Fig 1. In the crystal structure of the title compound, there are two pairs of molecules enantiomers in the unit cell. In each molecule, the nitro group forms a dihedral angle of $2.7(4)^\circ$ with the phenyl ring very different to that found in 2-(methylsulfinyl)benzamide (25.6°) (Yan, 2010) and in benzamide (26.31°) (Kobayashi *et al.*, 2003). The crystal packing is stabilized by weak C—H \cdots O hydrogen bonds (Fig. 2) forming non-interacting layers parallel to (-101) planes.

S2. Experimental

O-chloronitrobenzene (1.60 g, 10 mmol) and thioacetic acid (0.80 g, 10 mmol) were dissolved in 75 ml aqua ethanol solution (25 ml water + 50 ml ethanol) and refluxed for 3 h under continuous stirring. Then the obtained product was evaporated at room temperature to dryness. The residue was diluted in 50 ml pure ethanol. After few days, orange blocks were recovered, as the solvent slowly evaporated.

S3. Refinement

All non-H atoms were refined with anisotropic atomic displacement parameters. Approximate positions for all H atoms were first obtained from the difference electron density map. However, the H atoms were situated into idealized positions and the H-atoms have been refined within the riding atom approximation. The applied constraints were as follow: $C_{\text{aryl}}-H_{\text{aryl}} = 0.95 \text{ \AA}$ and $C_{\text{methylene}}-H_{\text{methylene}} = 0.99 \text{ \AA}$. $U_{\text{iso}}(H_{\text{aryl/methylene}}) = 1.2U_{\text{eq}}(C_{\text{aryl/methylene}})$.

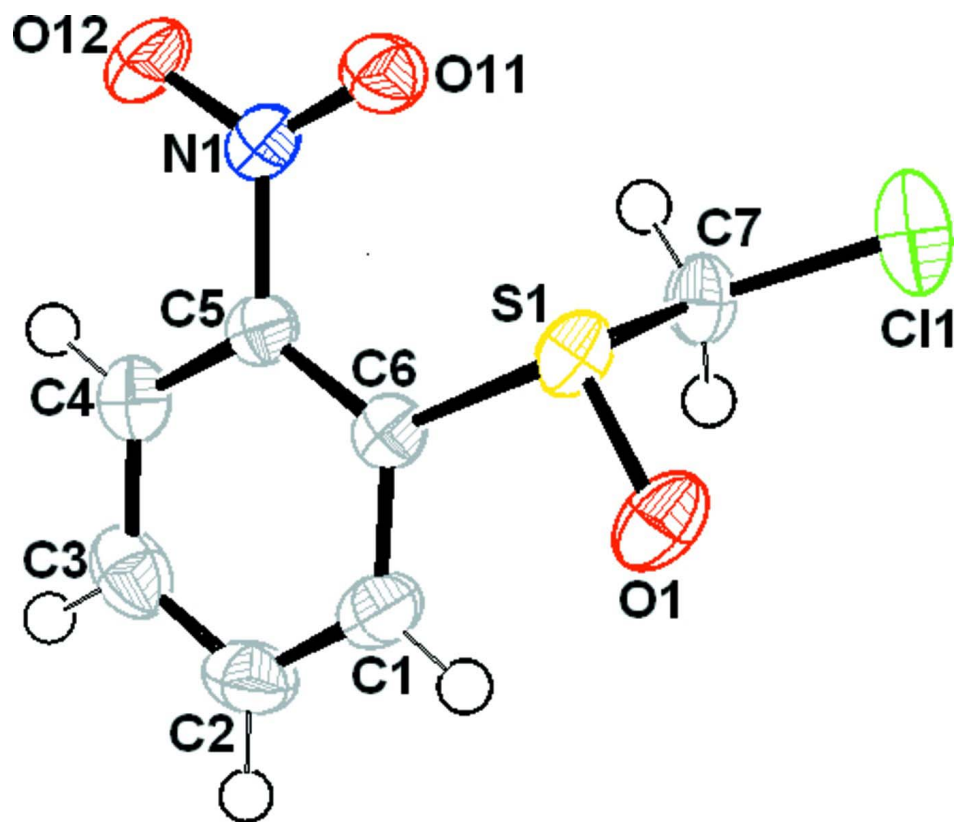


Figure 1

Drawing of asymmetric unit of, (I), with displacement ellipsoids drawn at the 50% probability level.

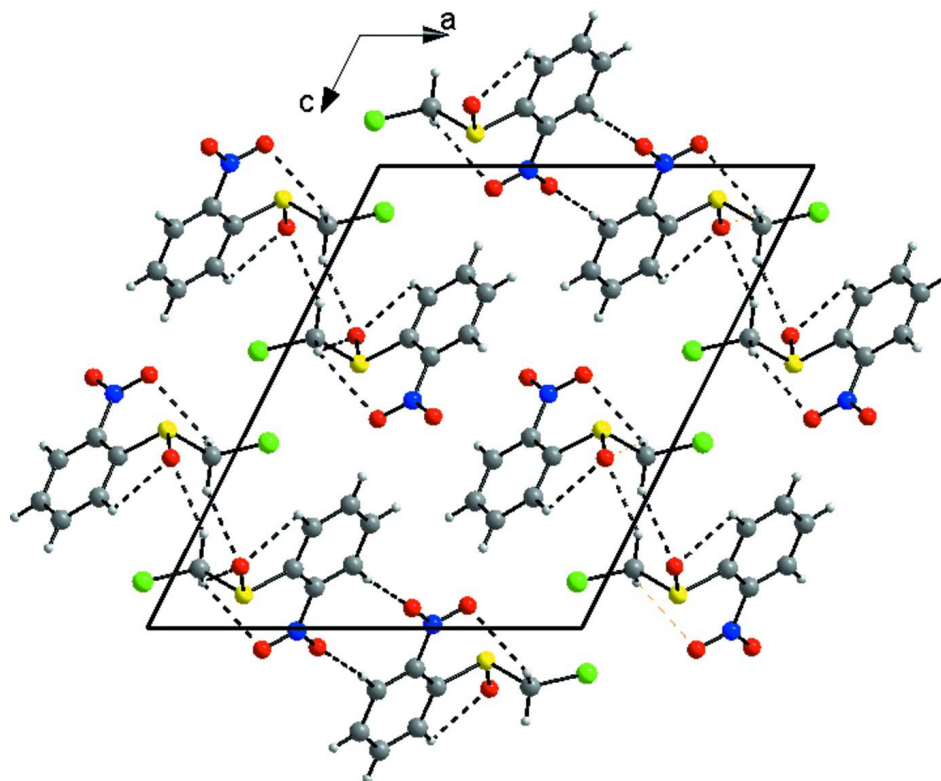
**Figure 2**

Diagram packing of (I) viewed *via* *b* axis showing alternating layers parallel to (-101) planes.

1-Chloromethylsulfinyl-2-nitrobenzene

Crystal data

$C_7H_6ClNO_3S$

$M_r = 219.65$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 12.2394$ (5) Å

$b = 5.5009$ (2) Å

$c = 14.5537$ (11) Å

$\beta = 116.631$ (4)°

$V = 875.92$ (9) Å³

$Z = 4$

$F(000) = 448$

$D_x = 1.666$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 6334 reflections

$\theta = 3.3$ – 29.2 °

$\mu = 0.65$ mm⁻¹

$T = 180$ K

Block, orange

$0.23 \times 0.20 \times 0.18$ mm

Data collection

Agilent Xcalibur (Eos, Gemini ultra)
diffractometer

Graphite monochromator

Detector resolution: 16.1978 pixels mm⁻¹

ω scans

Absorption correction: multi-scan
(*CrysAlis PRO*; Agilent, 2011)

$T_{\min} = 0.900$, $T_{\max} = 1.000$

10209 measured reflections

2172 independent reflections

1799 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 29.2$ °, $\theta_{\min} = 3.7$ °

$h = -16 \rightarrow 15$

$k = -7 \rightarrow 7$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.026$	H-atom parameters constrained
$wR(F^2) = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.1537P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2172 reflections	$(\Delta/\sigma)_{\max} = 0.002$
118 parameters	$\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. Absorption correction: Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm. CrysAlisPro (Agilent, 2011)

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	1.06418 (4)	0.73513 (8)	0.59930 (3)	0.03956 (13)
S1	0.81710 (3)	0.81595 (6)	0.57165 (3)	0.02049 (10)
O1	0.85724 (9)	1.04534 (18)	0.63222 (8)	0.0286 (2)
O11	0.71137 (9)	0.42553 (19)	0.45234 (7)	0.0271 (2)
O12	0.58622 (10)	0.15605 (19)	0.45752 (8)	0.0312 (3)
N1	0.65255 (10)	0.3330 (2)	0.49290 (9)	0.0204 (2)
C1	0.73727 (13)	0.7493 (3)	0.71720 (11)	0.0256 (3)
H1	0.7829	0.8934	0.7453	0.031*
C2	0.67575 (14)	0.6385 (3)	0.76600 (11)	0.0298 (3)
H2	0.6802	0.7065	0.8276	0.036*
C3	0.60801 (13)	0.4300 (3)	0.72587 (11)	0.0290 (3)
H3	0.5672	0.354	0.7604	0.035*
C4	0.59970 (12)	0.3319 (3)	0.63530 (11)	0.0238 (3)
H4	0.5518	0.1908	0.6063	0.029*
C5	0.66235 (12)	0.4428 (2)	0.58775 (10)	0.0186 (3)
C6	0.73266 (11)	0.6514 (2)	0.62775 (10)	0.0188 (3)
C7	0.94884 (12)	0.6136 (3)	0.62770 (11)	0.0231 (3)
H7A	0.9786	0.6027	0.703	0.028*
H7B	0.926	0.4484	0.5982	0.028*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0267 (2)	0.0429 (3)	0.0519 (3)	-0.00401 (16)	0.02018 (18)	0.00852 (19)

S1	0.02044 (17)	0.01600 (17)	0.02053 (17)	-0.00089 (12)	0.00517 (13)	0.00213 (13)
O1	0.0302 (5)	0.0156 (5)	0.0326 (5)	-0.0035 (4)	0.0074 (5)	-0.0018 (4)
O11	0.0317 (5)	0.0286 (6)	0.0247 (5)	-0.0052 (4)	0.0161 (4)	-0.0025 (4)
O12	0.0313 (6)	0.0277 (6)	0.0311 (6)	-0.0120 (4)	0.0109 (5)	-0.0111 (5)
N1	0.0192 (5)	0.0183 (6)	0.0205 (5)	0.0001 (4)	0.0061 (5)	-0.0008 (4)
C1	0.0237 (7)	0.0241 (7)	0.0235 (7)	0.0014 (6)	0.0057 (6)	-0.0049 (6)
C2	0.0300 (8)	0.0382 (9)	0.0207 (7)	0.0071 (6)	0.0110 (6)	-0.0023 (6)
C3	0.0263 (7)	0.0370 (9)	0.0270 (7)	0.0044 (6)	0.0148 (6)	0.0079 (7)
C4	0.0210 (7)	0.0228 (7)	0.0271 (7)	0.0004 (5)	0.0103 (6)	0.0032 (6)
C5	0.0171 (6)	0.0184 (6)	0.0178 (6)	0.0033 (5)	0.0054 (5)	0.0006 (5)
C6	0.0177 (6)	0.0171 (6)	0.0192 (6)	0.0033 (5)	0.0061 (5)	0.0022 (5)
C7	0.0191 (6)	0.0206 (6)	0.0279 (7)	0.0001 (5)	0.0091 (5)	0.0043 (6)

Geometric parameters (Å, °)

C11—C7	1.7703 (14)	C2—C3	1.382 (2)
S1—O1	1.4908 (10)	C2—H2	0.95
S1—C6	1.8196 (14)	C3—C4	1.385 (2)
S1—C7	1.8236 (14)	C3—H3	0.95
O11—N1	1.2276 (15)	C4—C5	1.3837 (19)
O12—N1	1.2234 (15)	C4—H4	0.95
N1—C5	1.4618 (17)	C5—C6	1.3953 (18)
C1—C2	1.386 (2)	C7—H7A	0.99
C1—C6	1.3865 (19)	C7—H7B	0.99
C1—H1	0.95		
O1—S1—C6	104.99 (6)	C5—C4—C3	118.91 (13)
O1—S1—C7	105.15 (6)	C5—C4—H4	120.5
C6—S1—C7	93.53 (6)	C3—C4—H4	120.5
O12—N1—O11	123.31 (12)	C4—C5—C6	122.02 (12)
O12—N1—C5	118.97 (11)	C4—C5—N1	117.44 (12)
O11—N1—C5	117.72 (11)	C6—C5—N1	120.53 (12)
C2—C1—C6	120.49 (14)	C1—C6—C5	117.97 (13)
C2—C1—H1	119.8	C1—C6—S1	115.84 (11)
C6—C1—H1	119.8	C5—C6—S1	126.16 (10)
C3—C2—C1	120.60 (13)	C11—C7—S1	107.62 (7)
C3—C2—H2	119.7	C11—C7—H7A	110.2
C1—C2—H2	119.7	S1—C7—H7A	110.2
C2—C3—C4	119.97 (13)	C11—C7—H7B	110.2
C2—C3—H3	120	S1—C7—H7B	110.2
C4—C3—H3	120	H7A—C7—H7B	108.5
C6—C1—C2—C3	-0.6 (2)	C4—C5—C6—C1	-0.9 (2)
C1—C2—C3—C4	-0.9 (2)	N1—C5—C6—C1	179.16 (12)
C2—C3—C4—C5	1.5 (2)	C4—C5—C6—S1	-179.03 (10)
C3—C4—C5—C6	-0.6 (2)	N1—C5—C6—S1	1.02 (18)
C3—C4—C5—N1	179.38 (12)	O1—S1—C6—C1	-8.09 (12)
O12—N1—C5—C4	3.01 (18)	C7—S1—C6—C1	98.62 (11)

O11—N1—C5—C4	-177.48 (12)	O1—S1—C6—C5	170.09 (11)
O12—N1—C5—C6	-177.04 (12)	C7—S1—C6—C5	-83.20 (12)
O11—N1—C5—C6	2.47 (18)	O1—S1—C7—C11	-67.86 (9)
C2—C1—C6—C5	1.4 (2)	C6—S1—C7—C11	-174.44 (8)
C2—C1—C6—S1	179.78 (11)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C4—H4...O12 ⁱ	0.95	2.44	3.384 (2)	173
C7—H7A...O1 ⁱⁱ	0.99	2.36	3.2478 (18)	149
C7—H7B...O1 ⁱⁱⁱ	0.99	2.50	3.332 (2)	142

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+2, y-1/2, -z+3/2$; (iii) $x, y-1, z$.