Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(4-Chloro-2-nitrophenyl)-*N*-(methylsulfonyl)acetamide

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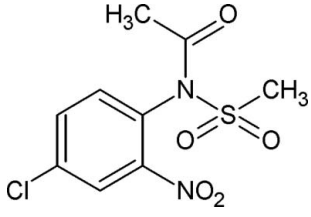
Received 10 September 2008; accepted 6 October 2008

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.104; data-to-parameter ratio = 18.3.

The title compound, $C_9H_9ClN_2O_5S$, is of interest as a precursor to biologically active substituted quinolines and related compounds. The structure displays intermolecular C—H···O interactions. Each molecule is linked to two adjacent neighbours *via* weak centrosymmetric dimer-forming interactions, forming chains in the [101] direction.

Related literature

For synthesis and biological evaluation of sulfur-containing heterocyclic compounds, see: Zia-ur-Rehman *et al.* (2005, 2006, 2007, 2008); Wen *et al.* (2005); Zhang, Xu, Wen *et al.* (2006). For related molecules, see: (Wen *et al.*, 2006; Zhang, Xu, Zou *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_9H_9ClN_2O_5S$
M_r = 292.70
Monoclinic, $P2_1/c$
a = 9.8071 (4) Å
b = 9.4310 (4) Å
c = 13.5679 (7) Å
β = 105.883 (2)°
V = 1207.00 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.50 mm^{-1}
T = 296 (2) K
0.25 × 0.15 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.913, T_{max} = 0.956
13383 measured reflections
2988 independent reflections
2077 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.02
2981 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O5^{i}	0.93	2.55	3.404 (3)	153
C9—H9B···O3^{ii}	0.96	2.58	3.521 (3)	169

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

The authors are grateful to the PCSIR Laboratories Complex, Lahore, Pakistan, for provision of the necessary chemicals, and the Higher Education Commission of Pakistan for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2142).

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supporting information

Acta Cryst. (2008). E**64**, o2092 [doi:10.1107/S1600536808032157]

N-(4-Chloro-2-nitrophenyl)-*N*-(methylsulfonyl)acetamide

Muhammad Zia-ur-Rehman, Nosheen Akbar, Muhammad Nadeem Arshad and Islam Ullah Khan

S1. Comment

N-(Substituted phenyl)acetamides are well known for their importance as intermediates in organic synthesis. They are used as precursors for the synthesis of many heterocyclic compounds, *e.g.* 2,5-piperazinedione (Wen *et al.*, 2006), (quinolin-8-yloxy)acetamide (Zhang, Xu, Wen *et al.*, 2006) and 2,2-(1,3,4-thiadiazolyl-2,5-dithio)diacetamide (Wen *et al.*, 2005). In the present paper, the structure of *N*-(4-chloro-2-nitrophenyl)-*N*-(methylsulfonyl)acetamide (**I**) has been determined as part of a research program involving the synthesis and biological evaluation of sulfur containing heterocyclic compounds (Zia-ur-Rehman *et al.*, 2005, 2006, 2007, 2008).

In the molecule of **I** (Fig. 1), the bond lengths and bond angles are similar to those in related molecules (Wen *et al.*, 2006; Zhang, Xu, Zou *et al.*, 2006) and are within normal ranges (Allen *et al.*, 1987). The nitro group is slightly twisted out of the plane of the benzene ring, as indicated by O1—N1—C3—C2 and O2—N1—C3—C2 torsion angles of -16.7 (3) and 160.9 (2)°, respectively. Each molecule is linked to its neighbour *via* a centrosymmetric head-to-tail interaction between the methyl hydrogen H9B and the carbonyl oxygen [C9—H9B···O3]. Adjacent pairs of these molecules are then linked into chains *via* intermolecular [C2—H5···O5] interactions along the [101] direction (Table 1 and Fig. 2).

S2. Experimental

A mixture of *N*-(4-chloro-2-nitrophenyl)methane sulfonamide (2.507 g; 10.0 mmoles) and acetic anhydride (10.0 ml) was heated to reflux for half an hour and then poured over crushed ice. Resultant solids were then washed with cold water and dried under reduced pressure. Yellow crystals were obtained by slow evaporation of an ethanolic solution over a period of two days.

S3. Refinement

H atoms bound to C were placed in geometric positions (C—H distance = 0.95 Å) using a riding model with U_{iso}(H) = 1.2 U_{eq}(C).

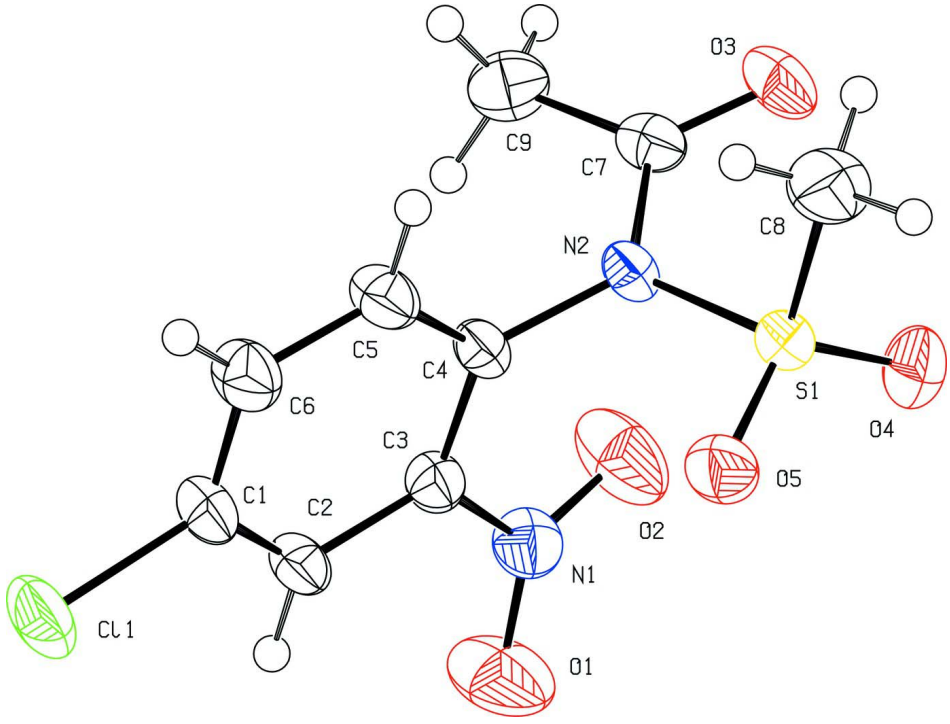

Figure 1

The asymmetric unit of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

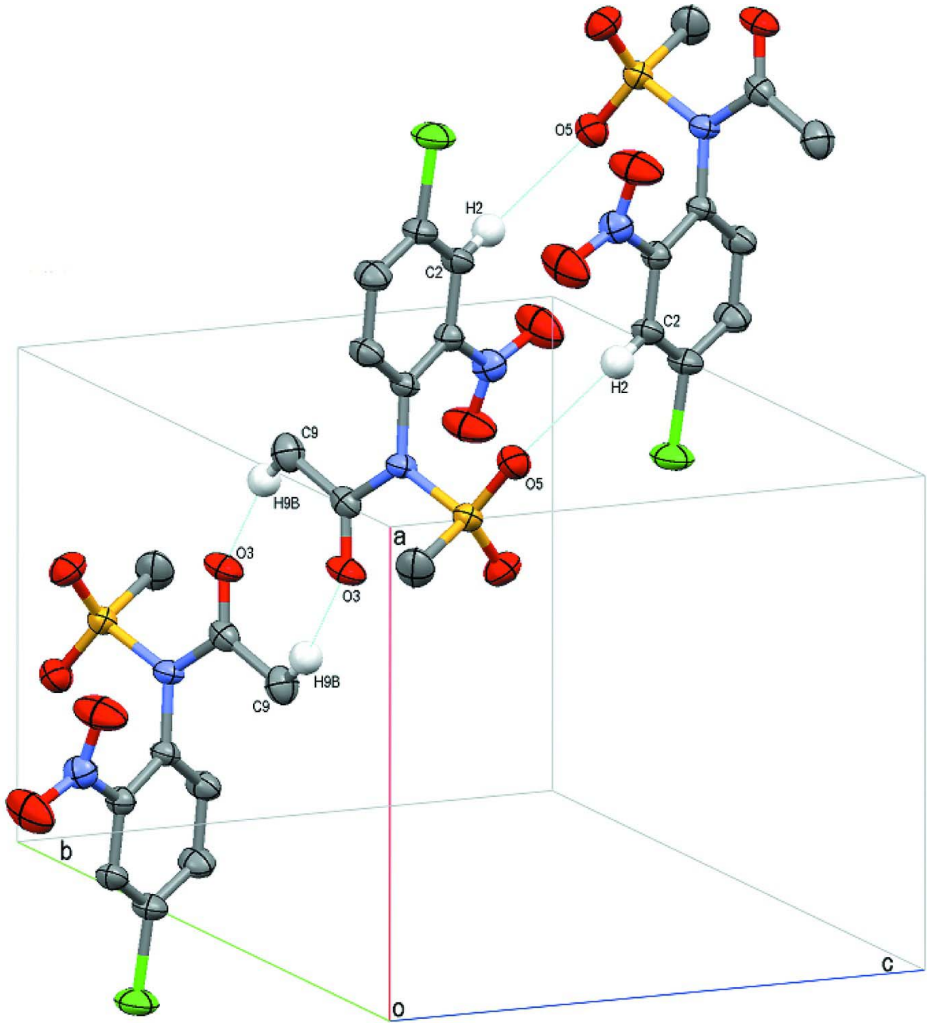

Figure 2

Perspective view of a portion of the crystal packing, viewed approximately down the b-axis, showing hydrogen bond interactions (dashed lines) along the [101] direction. H atoms not involved in hydrogen bonding have been omitted for clarity.

N-(4-Chloro-2-nitrophenyl)-*N*-(methylsulfonyl)acetamide

Crystal data

$C_9H_9ClN_2O_5S$
$M_r = 292.70$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.8071\ (4)$ Å
$b = 9.4310\ (4)$ Å
$c = 13.5679\ (7)$ Å
$\beta = 105.883\ (2)°$
$V = 1207.00\ (9)$ Å^3
$Z = 4$

$F(000) = 600$
$D_x = 1.611$ Mg m^{-3}
Melting point: 401 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3121 reflections
$\theta = 2.7–27.2°$
$\mu = 0.50$ mm^{-1}
$T = 296$ K
Needle, light yellow
0.25 × 0.15 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.5 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.913, T_{max} = 0.956
13383 measured reflections
2988 independent reflections
2077 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.3°, θ_{min} = 2.7°
h = −12→13
k = −12→12
l = −18→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.02
2981 reflections
163 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.5587P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Special details

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.43935 (5)	0.98058 (6)	0.14520 (4)	0.03097 (15)
Cl1	−0.20712 (6)	0.67766 (7)	−0.00125 (6)	0.0511 (2)
N2	0.34896 (17)	0.91810 (19)	0.22582 (14)	0.0276 (4)
O5	0.34507 (18)	0.96150 (18)	0.04574 (13)	0.0439 (4)
C4	0.2107 (2)	0.8605 (2)	0.17969 (16)	0.0269 (5)
O3	0.52800 (17)	0.97095 (19)	0.36537 (13)	0.0454 (4)
N1	0.0872 (2)	1.0934 (2)	0.18329 (16)	0.0361 (5)
C3	0.0874 (2)	0.9425 (2)	0.15362 (17)	0.0283 (5)
O1	−0.0130 (2)	1.1651 (2)	0.13957 (18)	0.0695 (7)
C1	−0.0476 (2)	0.7464 (3)	0.07164 (19)	0.0365 (5)
O4	0.49004 (18)	1.11878 (17)	0.17659 (15)	0.0477 (5)
O2	0.1842 (2)	1.1381 (2)	0.25205 (18)	0.0651 (6)
C5	0.1996 (2)	0.7188 (2)	0.15196 (19)	0.0359 (5)
H5	0.2800	0.6618	0.1689	0.043*
C6	0.0708 (2)	0.6607 (3)	0.09955 (19)	0.0401 (6)
H6	0.0639	0.5647	0.0833	0.048*

C7	0.4104 (2)	0.9221 (2)	0.33238 (17)	0.0315 (5)
C2	−0.0409 (2)	0.8874 (2)	0.09933 (18)	0.0327 (5)
H2	−0.1215	0.9441	0.0816	0.039*
C8	0.5815 (3)	0.8637 (3)	0.1615 (2)	0.0501 (7)
H8A	0.5463	0.7690	0.1460	0.075*
H8B	0.6390	0.8679	0.2312	0.075*
H8C	0.6374	0.8899	0.1163	0.075*
C9	0.3259 (3)	0.8606 (3)	0.39765 (19)	0.0415 (6)
H9A	0.2285	0.8887	0.3716	0.062*
H9B	0.3621	0.8944	0.4666	0.062*
H9C	0.3323	0.7591	0.3969	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0248 (3)	0.0320 (3)	0.0347 (3)	0.0000 (2)	0.0057 (2)	0.0040 (2)
Cl1	0.0311 (3)	0.0557 (4)	0.0567 (4)	−0.0129 (3)	−0.0043 (3)	−0.0082 (3)
N2	0.0209 (8)	0.0327 (9)	0.0269 (10)	−0.0027 (7)	0.0024 (7)	−0.0027 (8)
O5	0.0386 (9)	0.0575 (11)	0.0322 (10)	−0.0048 (8)	0.0039 (8)	0.0063 (8)
C4	0.0208 (9)	0.0328 (11)	0.0253 (11)	−0.0021 (8)	0.0032 (8)	−0.0017 (9)
O3	0.0317 (9)	0.0564 (11)	0.0388 (10)	−0.0073 (8)	−0.0064 (8)	−0.0053 (8)
N1	0.0334 (10)	0.0337 (10)	0.0424 (12)	0.0039 (8)	0.0123 (9)	−0.0019 (9)
C3	0.0268 (10)	0.0283 (11)	0.0292 (12)	−0.0009 (8)	0.0066 (9)	−0.0012 (9)
O1	0.0571 (13)	0.0447 (11)	0.0897 (17)	0.0213 (10)	−0.0088 (12)	−0.0094 (11)
C1	0.0256 (11)	0.0426 (13)	0.0364 (14)	−0.0069 (10)	0.0002 (10)	−0.0021 (10)
O4	0.0457 (10)	0.0354 (9)	0.0607 (13)	−0.0111 (8)	0.0124 (9)	0.0025 (8)
O2	0.0447 (11)	0.0471 (11)	0.0889 (17)	0.0022 (9)	−0.0063 (11)	−0.0287 (11)
C5	0.0277 (11)	0.0326 (11)	0.0422 (15)	0.0037 (9)	0.0010 (10)	−0.0035 (10)
C6	0.0368 (13)	0.0329 (12)	0.0447 (15)	−0.0034 (10)	0.0012 (11)	−0.0067 (11)
C7	0.0308 (11)	0.0307 (11)	0.0291 (13)	0.0039 (9)	0.0016 (10)	−0.0025 (9)
C2	0.0202 (10)	0.0403 (12)	0.0356 (13)	0.0008 (9)	0.0043 (9)	0.0024 (10)
C8	0.0370 (14)	0.0579 (17)	0.0606 (19)	0.0155 (12)	0.0219 (13)	0.0118 (14)
C9	0.0489 (14)	0.0445 (14)	0.0309 (14)	0.0029 (11)	0.0104 (12)	0.0004 (11)

Geometric parameters (Å, º)

S1—O4	1.4182 (17)	C1—C2	1.379 (3)
S1—O5	1.4232 (18)	C1—C6	1.380 (3)
S1—N2	1.6913 (19)	C5—C6	1.382 (3)
S1—C8	1.743 (2)	C5—H5	0.9300
Cl1—C1	1.732 (2)	C6—H6	0.9300
N2—C7	1.406 (3)	C7—C9	1.487 (3)
N2—C4	1.435 (2)	C2—H2	0.9300
C4—C5	1.384 (3)	C8—H8A	0.9600
C4—C3	1.397 (3)	C8—H8B	0.9600
O3—C7	1.208 (3)	C8—H8C	0.9600
N1—O1	1.207 (2)	C9—H9A	0.9600
N1—O2	1.212 (3)	C9—H9B	0.9600

N1—C3	1.479 (3)	C9—H9C	0.9600
C3—C2	1.374 (3)		
O4—S1—O5	118.97 (11)	C4—C5—H5	119.5
O4—S1—N2	109.18 (10)	C1—C6—C5	119.4 (2)
O5—S1—N2	104.46 (9)	C1—C6—H6	120.3
O4—S1—C8	109.91 (13)	C5—C6—H6	120.3
O5—S1—C8	109.27 (13)	O3—C7—N2	119.2 (2)
N2—S1—C8	103.88 (11)	O3—C7—C9	124.0 (2)
C7—N2—C4	123.11 (18)	N2—C7—C9	116.74 (19)
C7—N2—S1	120.17 (14)	C3—C2—C1	118.6 (2)
C4—N2—S1	116.71 (14)	C3—C2—H2	120.7
C5—C4—C3	117.84 (19)	C1—C2—H2	120.7
C5—C4—N2	118.61 (18)	S1—C8—H8A	109.5
C3—C4—N2	123.35 (18)	S1—C8—H8B	109.5
O1—N1—O2	123.1 (2)	H8A—C8—H8B	109.5
O1—N1—C3	117.83 (19)	S1—C8—H8C	109.5
O2—N1—C3	119.05 (19)	H8A—C8—H8C	109.5
C2—C3—C4	121.92 (19)	H8B—C8—H8C	109.5
C2—C3—N1	116.14 (18)	C7—C9—H9A	109.5
C4—C3—N1	121.95 (18)	C7—C9—H9B	109.5
C2—C1—C6	121.1 (2)	H9A—C9—H9B	109.5
C2—C1—Cl1	119.02 (18)	C7—C9—H9C	109.5
C6—C1—Cl1	119.86 (18)	H9A—C9—H9C	109.5
C6—C5—C4	121.1 (2)	H9B—C9—H9C	109.5
C6—C5—H5	119.5		
O4—S1—N2—C7	−50.92 (18)	O1—N1—C3—C4	163.6 (2)
O5—S1—N2—C7	−179.19 (16)	O2—N1—C3—C4	−18.8 (3)
C8—S1—N2—C7	66.30 (19)	C3—C4—C5—C6	0.5 (4)
O4—S1—N2—C4	130.04 (16)	N2—C4—C5—C6	−174.5 (2)
O5—S1—N2—C4	1.77 (17)	C2—C1—C6—C5	−3.1 (4)
C8—S1—N2—C4	−112.74 (18)	Cl1—C1—C6—C5	175.8 (2)
C7—N2—C4—C5	−91.8 (3)	C4—C5—C6—C1	2.1 (4)
S1—N2—C4—C5	87.2 (2)	C4—N2—C7—O3	179.2 (2)
C7—N2—C4—C3	93.5 (3)	S1—N2—C7—O3	0.2 (3)
S1—N2—C4—C3	−87.5 (2)	C4—N2—C7—C9	1.0 (3)
C5—C4—C3—C2	−2.1 (3)	S1—N2—C7—C9	−178.00 (16)
N2—C4—C3—C2	172.6 (2)	C4—C3—C2—C1	1.2 (4)
C5—C4—C3—N1	177.6 (2)	N1—C3—C2—C1	−178.6 (2)
N2—C4—C3—N1	−7.7 (3)	C6—C1—C2—C3	1.5 (4)
O1—N1—C3—C2	−16.7 (3)	Cl1—C1—C2—C3	−177.41 (18)
O2—N1—C3—C2	160.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O5^{i}	0.93	2.55	3.404 (3)	153

C9—H9*B*···O3^{ii}	0.96	2.58	3.521 (3)	169

Symmetry codes: (i) −*x*, −*y*+2, −*z*; (ii) −*x*+1, −*y*+2, −*z*+1.