organic compounds

N-(3,5-Dichlorophenyl)-4-methyl-benzenesulfonamide

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In the crystal structure of the title compound, $C_{13}H_{11}Cl_2NO_2S$, the conformation of the N—C bond in the C—SO_2—NH—C segment is *gauche* with respect to the SO bonds. The two benzene rings are tilted by 79.6 (1)° relative to each other. In the crystal, inversion dimers linked by pairs of N—H···O hydrogen bonds occur.

Related literature

For the preparation of the title compound, see: Shetty & Gowda (2005). For background literature, see: For a study of the effect of substituents on the crystal structures of *N*-(aryl)-arylsulfonamides, see: Gowda *et al.* (2008, 2009*a,b*). For bond parameters in related aryl sulfonamides, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006).
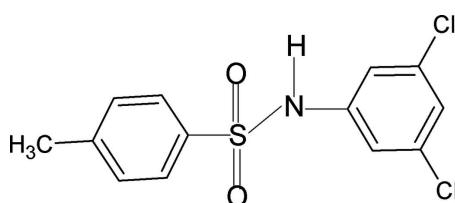
Experimental

Crystal data

$C_{13}H_{11}Cl_2NO_2S$	b = 8.9627 (8) Å
M_r = 316.19	c = 22.944 (2) Å
Monoclinic, $P2_1/n$	β = 91.801 (8)°
a = 6.7388 (8) Å	V = 1385.1 (2) Å^3
Z = 4	T = 299 K
Cu $K\alpha$ radiation	0.42 × 0.35 × 0.13 mm
μ = 5.61 mm^{-1}	

Data collection

Enraf–Nonius CAD-4 diffractometer	2424 independent reflections
Absorption correction: ψ scan (North *et al.*, 1968)	2049 reflections with $I > 2\sigma(I)$
T_{min} = 0.140, T_{max} = 0.482	R_{int} = 0.104
3363 measured reflections	3 standard reflections frequency: 120 min intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.236	
S = 1.11	$\Delta\rho_{max}$ = 0.48 e Å^{-3}
2424 reflections	$\Delta\rho_{min}$ = −0.64 e Å^{-3}
176 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^i	0.87 (5)	2.02 (6)	2.888 (5)	176 (5)

Symmetry code: (i) $-x+2, -y, -z+1$.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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supporting information

N-(3,5-Dichlorophenyl)-4-methylbenzenesulfonamide

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S1. Comment

As part of a study of the substituent effects on the crystal structures of *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2008; 2009*a, b*), in the present work, the crystal structure of 4-methyl-*N*-(3,5-dichlorophenyl)benzenesulfonamide (I) has been determined. The conformation of the N—C bond in the C—SO$_2$—NH—C segment of the structure has "*trans*" and "*gauche*" torsions with respect to the SO bonds (Fig. 1). The molecule is bent at the S atom with the C—SO$_2$—NH—C torsion angle of 69.3 (4)° compared to the values of -51.6 (3)° and 68.3 (2)°, respectively, for 4-methyl-*N*-(phenyl)-benzenesulfonamide (II)(Gowda *et al.*, 2009*b*) and *N*-(3,5-dichlorophenyl)-benzenesulfonamide (III) (Gowda *et al.*, 2008). The two benzene rings in (I) are tilted relative to each other by 79.6 (1)°, compared to the values of 68.4 (1)° for the compound II and 57.0 (1)° for III.

The other bond parameters in (I) are similar to those observed in (II) (Gowda *et al.*, 2009*b*), (III) (Gowda *et al.*, 2008), 4-methyl-*N*-(3,4-dimethylphenyl)benzenesulfonamide (Gowda *et al.*, 2009*a*) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007). The packing of molecules *via* N—H⋯O(S) hydrogen bonds (Table 1) into supramolecular structure is shown in Fig. 2.

S2. Experimental

The solution of toluene (10 cc) in chloroform (40 cc) was treated dropwise with chlorosulfonic acid (25 cc) at 0 ° C. After the initial evolution of hydrogen chloride subsided, the reaction mixture was brought to room temperature and poured into crushed ice in a beaker. The chloroform layer was separated, washed with cold water and allowed to evaporate slowly. The residual 4-methylbenzenesulfonylchloride was treated with 3,5-dichloroaniline in the stoichiometric ratio and boiled for 15 minutes. The reaction mixture was then cooled to room temperature and added to ice cold water (100 cc). The resultant 4-methyl-*N*-(3,5-dichlorophenyl)benzenesulfonamide was filtered under suction and washed thoroughly with cold water. It was then recrystallized to constant melting point from dilute ethanol. The purity of the compound was checked and characterized by recording its infrared and NMR spectra (Shetty & Gowda, 2005). The prism like colourless single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and its position refined [N—H = 0.87 (5) Å]. The other H atoms were positioned with idealized geometry using a riding model [C—H = 0.93–0.96 Å]. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

Two reflections (-5 1 9 and -3 2 11) were omitted from the refinement as a statistical analysis showed that they were anomalous.
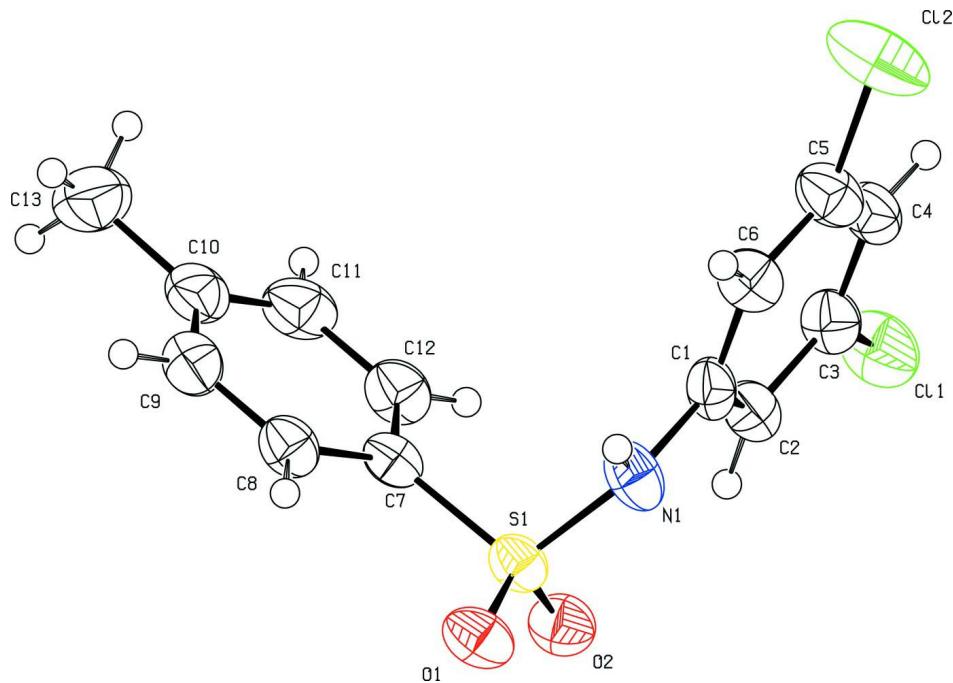
Figure 1

Molecular structure of (I), showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level.
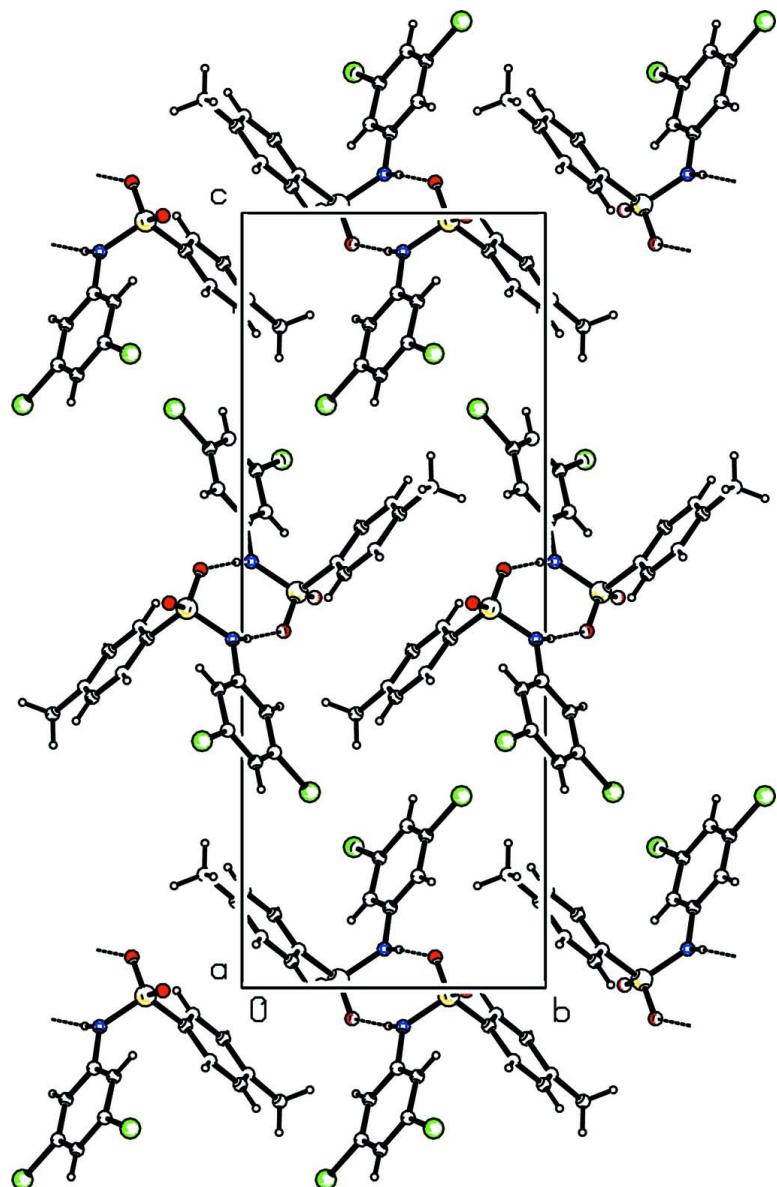
Figure 2

Molecular packing of (I) with hydrogen bonding shown as dashed lines.

N-(3,5-Dichlorophenyl)-4-methylbenzenesulfonamide

Crystal data

$C_{13}H_{11}Cl_2NO_2S$	$F(000) = 648$
$M_r = 316.19$	$D_x = 1.516$ Mg m^{-3}
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation, $\lambda = 1.54180$ Å
Hall symbol: -P 2yn	Cell parameters from 25 reflections
$a = 6.7388$ (8) Å	$\theta = 3.9$–18.2°
$b = 8.9627$ (8) Å	$\mu = 5.61$ mm^{-1}
$c = 22.944$ (2) Å	$T = 299$ K
$\beta = 91.801$ (8)°	Prism, colourless
$V = 1385.1$ (2) Å^3	$0.42 \times 0.35 \times 0.13$ mm
$Z = 4$	

Data collection

Enraf–Nonius CAD-4 diffractometer	2424 independent reflections
Radiation source: fine-focus sealed tube	2049 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.104$
$\omega/2\theta$ scans	$\theta_{max} = 66.9°$, $\theta_{min} = 3.9°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -7 \rightarrow 2$
	$k = -10 \rightarrow 0$
$T_{min} = 0.140$, $T_{max} = 0.482$	$l = -27 \rightarrow 27$
3363 measured reflections	3 standard reflections every 120 min
	intensity decay: 1.0%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.056$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.236$	
$S = 1.11$	H atoms treated by a mixture of independent and constrained refinement
2424 reflections	
176 parameters	$w = 1/[\sigma^2(F_o^2) + (0.1586P)^2 + 1.125P]$ where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} = 0.011$
	$\Delta\rho_{max} = 0.48$ e Å^{-3}
	$\Delta\rho_{min} = -0.64$ 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	$U_{iso}*/U_{eq}$
C1	0.6639 (6)	0.0125 (4)	0.60350 (17)	0.0403 (9)
C2	0.4841 (6)	0.0772 (5)	0.61451 (17)	0.0442 (9)
H2	0.4215	0.1394	0.5872	0.053*
C3	0.3986 (6)	0.0473 (5)	0.66742 (18)	0.0441 (9)
C4	0.4832 (7)	−0.0443 (5)	0.70890 (18)	0.0470 (10)
H4	0.4223	−0.0636	0.7439	0.056*
C5	0.6627 (7)	−0.1064 (5)	0.69595 (19)	0.0496 (10)
C6	0.7556 (7)	−0.0807 (5)	0.64425 (18)	0.0454 (9)
H6	0.8771	−0.1249	0.6369	0.054*
C7	0.9133 (6)	0.3137 (4)	0.54905 (17)	0.0393 (9)
C8	1.1092 (6)	0.3369 (5)	0.53436 (18)	0.0430 (9)
H8	1.1630	0.2845	0.5036	0.052*
C9	1.2229 (7)	0.4380 (5)	0.56558 (19)	0.0495 (10)
H9	1.3544	0.4532	0.5560	0.059*
C10	1.1436 (8)	0.5185 (5)	0.6117 (2)	0.0522 (11)

C11	0.9505 (8)	0.4940 (6)	0.62479 (19)	0.0547 (11)
H11	0.8965	0.5466	0.6554	0.066*
C12	0.8329 (7)	0.3936 (5)	0.59400 (18)	0.0476 (10)
H12	0.7010	0.3799	0.6034	0.057*
C13	1.2739 (10)	0.6246 (7)	0.6464 (2)	0.0714 (15)
H13A	1.2902	0.7152	0.6248	0.086*
H13B	1.4014	0.5795	0.6540	0.086*
H13C	1.2133	0.6468	0.6827	0.086*
N1	0.7620 (6)	0.0309 (4)	0.55009 (16)	0.0467 (9)
H1N	0.872 (8)	−0.020 (6)	0.549 (2)	0.056*
O1	0.8754 (5)	0.1373 (3)	0.46032 (12)	0.0478 (8)
O2	0.5716 (4)	0.2390 (4)	0.50360 (13)	0.0490 (7)
Cl1	0.17240 (18)	0.13146 (17)	0.68157 (5)	0.0653 (5)
Cl2	0.7786 (3)	−0.22328 (19)	0.74746 (6)	0.0826 (6)
S1	0.76828 (14)	0.18214 (11)	0.51047 (4)	0.0392 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.049 (2)	0.0334 (18)	0.039 (2)	−0.0094 (16)	0.0126 (16)	−0.0055 (15)
C2	0.051 (2)	0.042 (2)	0.040 (2)	−0.0025 (17)	0.0062 (17)	0.0030 (17)
C3	0.044 (2)	0.045 (2)	0.044 (2)	0.0012 (16)	0.0111 (17)	−0.0025 (18)
C4	0.061 (3)	0.042 (2)	0.038 (2)	−0.0064 (18)	0.0116 (18)	−0.0019 (17)
C5	0.067 (3)	0.039 (2)	0.044 (2)	0.0051 (19)	0.0065 (19)	0.0027 (18)
C6	0.053 (2)	0.041 (2)	0.043 (2)	0.0065 (17)	0.0083 (17)	−0.0036 (17)
C7	0.046 (2)	0.039 (2)	0.0330 (18)	0.0042 (16)	0.0065 (15)	0.0040 (15)
C8	0.048 (2)	0.041 (2)	0.040 (2)	0.0033 (17)	0.0104 (17)	0.0008 (17)
C9	0.052 (3)	0.047 (2)	0.049 (2)	−0.0016 (18)	0.0036 (19)	0.0065 (19)
C10	0.072 (3)	0.040 (2)	0.045 (2)	−0.001 (2)	−0.003 (2)	0.0070 (19)
C11	0.078 (3)	0.050 (2)	0.037 (2)	0.006 (2)	0.008 (2)	−0.0056 (18)
C12	0.055 (3)	0.046 (2)	0.042 (2)	0.0047 (18)	0.0138 (18)	−0.0040 (18)
C13	0.089 (4)	0.068 (3)	0.056 (3)	−0.008 (3)	−0.014 (3)	−0.004 (3)
N1	0.055 (2)	0.0387 (18)	0.047 (2)	0.0038 (15)	0.0219 (17)	0.0029 (15)
O1	0.0598 (18)	0.0507 (17)	0.0337 (14)	0.0098 (13)	0.0148 (12)	−0.0022 (12)
O2	0.0481 (17)	0.0543 (18)	0.0446 (16)	0.0038 (13)	0.0051 (12)	0.0002 (14)
Cl1	0.0525 (7)	0.0842 (9)	0.0604 (7)	0.0132 (6)	0.0205 (5)	0.0067 (6)
Cl2	0.1076 (12)	0.0872 (10)	0.0542 (8)	0.0448 (9)	0.0193 (7)	0.0235 (7)
S1	0.0456 (6)	0.0388 (6)	0.0338 (6)	0.0031 (4)	0.0110 (4)	−0.0010 (4)

Geometric parameters (Å, º)

C1—C2	1.374 (6)	C8—H8	0.9300
C1—C6	1.384 (6)	C9—C10	1.399 (7)
C1—N1	1.420 (5)	C9—H9	0.9300
C2—C3	1.387 (5)	C10—C11	1.362 (7)
C2—H2	0.9300	C10—C13	1.506 (7)
C3—C4	1.368 (6)	C11—C12	1.379 (7)
C3—Cl1	1.740 (4)	C11—H11	0.9300

C4—C5	1.373 (6)	C12—H12	0.9300
C4—H4	0.9300	C13—H13A	0.9600
C5—C6	1.378 (6)	C13—H13B	0.9600
C5—Cl2	1.746 (5)	C13—H13C	0.9600
C6—H6	0.9300	N1—S1	1.633 (4)
C7—C12	1.380 (6)	N1—H1N	0.87 (5)
C7—C8	1.389 (6)	O1—S1	1.435 (3)
C7—S1	1.753 (4)	O2—S1	1.424 (3)
C8—C9	1.374 (6)		
C2—C1—C6	120.5 (4)	C10—C9—H9	119.5
C2—C1—N1	123.1 (4)	C11—C10—C9	118.3 (4)
C6—C1—N1	116.3 (4)	C11—C10—C13	122.0 (5)
C1—C2—C3	118.2 (4)	C9—C10—C13	119.7 (5)
C1—C2—H2	120.9	C10—C11—C12	121.9 (4)
C3—C2—H2	120.9	C10—C11—H11	119.0
C4—C3—C2	123.3 (4)	C12—C11—H11	119.0
C4—C3—Cl1	118.6 (3)	C11—C12—C7	119.3 (4)
C2—C3—Cl1	118.1 (3)	C11—C12—H12	120.4
C3—C4—C5	116.3 (4)	C7—C12—H12	120.4
C3—C4—H4	121.8	C10—C13—H13A	109.5
C5—C4—H4	121.8	C10—C13—H13B	109.5
C4—C5—C6	123.1 (4)	H13A—C13—H13B	109.5
C4—C5—Cl2	118.4 (3)	C10—C13—H13C	109.5
C6—C5—Cl2	118.5 (3)	H13A—C13—H13C	109.5
C5—C6—C1	118.5 (4)	H13B—C13—H13C	109.5
C5—C6—H6	120.7	C1—N1—S1	126.7 (3)
C1—C6—H6	120.7	C1—N1—H1N	113 (4)
C12—C7—C8	120.1 (4)	S1—N1—H1N	112 (4)
C12—C7—S1	120.0 (3)	O2—S1—O1	120.10 (18)
C8—C7—S1	119.8 (3)	O2—S1—N1	108.55 (19)
C9—C8—C7	119.4 (4)	O1—S1—N1	103.68 (17)
C9—C8—H8	120.3	O2—S1—C7	108.50 (19)
C7—C8—H8	120.3	O1—S1—C7	107.86 (19)
C8—C9—C10	120.9 (4)	N1—S1—C7	107.5 (2)
C8—C9—H9	119.5		
C6—C1—C2—C3	0.3 (6)	C9—C10—C11—C12	−0.2 (7)
N1—C1—C2—C3	177.7 (4)	C13—C10—C11—C12	−178.1 (5)
C1—C2—C3—C4	−0.6 (7)	C10—C11—C12—C7	0.9 (7)
C1—C2—C3—Cl1	179.2 (3)	C8—C7—C12—C11	−1.4 (6)
C2—C3—C4—C5	0.6 (7)	S1—C7—C12—C11	178.5 (3)
Cl1—C3—C4—C5	−179.1 (3)	C2—C1—N1—S1	37.0 (6)
C3—C4—C5—C6	−0.4 (7)	C6—C1—N1—S1	−145.5 (4)
C3—C4—C5—Cl2	179.6 (3)	C1—N1—S1—O2	−47.9 (4)
C4—C5—C6—C1	0.2 (7)	C1—N1—S1—O1	−176.6 (4)
Cl2—C5—C6—C1	−179.8 (3)	C1—N1—S1—C7	69.3 (4)
C2—C1—C6—C5	−0.2 (6)	C12—C7—S1—O2	37.9 (4)

N1—C1—C6—C5	−177.7 (4)	C8—C7—S1—O2	−142.2 (3)
C12—C7—C8—C9	1.1 (6)	C12—C7—S1—O1	169.5 (3)
S1—C7—C8—C9	−178.7 (3)	C8—C7—S1—O1	−10.7 (4)
C7—C8—C9—C10	−0.4 (6)	C12—C7—S1—N1	−79.3 (4)
C8—C9—C10—C11	0.0 (7)	C8—C7—S1—N1	100.6 (3)
C8—C9—C10—C13	177.9 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1[i]	0.87 (5)	2.02 (6)	2.888 (5)	176 (5)

Symmetry code: (i) −*x*+2, −*y*, −*z*+1.